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THE UNIVERSITY OF ALBERTA

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by



Erastus Lamanya Keya

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## ABSTRACT

The physico-chemical properties of tropical root starches from arrowroot, cassava, sweet potato, taro, and yam were studied in order to clarify their possible incorporation and role in a bread making formulation. These properties were then compared with those of starches from a Canadian western red spring wheat (CWRSW) (cv. Neepawa) of good baking, and a soft white spring wheat (SWSW) (cv. Fielder) of inferior baking quality. A flour composite formula of 85% starch and 15% vital gluten was adopted for all starches. Bread from pure wheat flours obtained from Neepawa and Fielder were used as the standard and internal references. Based on the influences in bread making of starch granule size distribution and morphology, percent amylose and mineral contents, water binding capacities, swelling power and solubilities, gelatinization properties and enthalpies of fusion, gel viscosity, retrogradation in gels, affinity for gluten, interaction with monoglycerides, dough rheological properties, bread qualities in the presence and absence of monoglyceride, and sensory evaluation, it was concluded that wheat starches made better composite breads than root starches with the same grade of vital gluten. Of the root starches, cassava produced the best composite bread next in quality to those of the wheat starches, followed respectively by yam, sweet potato, taro and lastly by arrowroot starch. Subtle differences between starch composite breads and the standard wheat flour

bread indicated the need for the establishment of proper formulations and baking conditions for the composite breads. The study, however, demonstrated that root starches possess varying potentials of being used in composite bread making with gluten or strong wheat flours. The study, in addition, has provided fundamental results which could be useful in further baking investigations involving root tuber flours, since such flours are predominantly composed of starch.

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## 1. INTRODUCTION

Wheat bread products are staple foods in many countries (Pomeranz and Shellenberger, 1971). Consumption patterns vary widely from one region to another, but they are generally higher and on the increase in developing countries. In recent years high population increase rates and urban migration of people in search of better occupations and standards of living are more responsible for this occurrence than any other factors.

In contrast, wheat cultivation on a large scale is limited to only a few areas in the United States, Canada, Europe, the USSR, Argentina, Australia, and New Zealand. These countries account for about 80% of world wheat production, yet have a total of only about one-third of the population (Kim and Ruiter, 1969). Wheat production in the developing countries is generally on a low scale and is often hampered by poor soils, bad climate, disease and pests. The yields are therefore low in most of cases and have to be supplemented by importation, which is expensive and uses up valuable foreign currency reserves, thus worsening the balance of payments.

Developing countries can reduce this problem by adopting composite flour technology, which provides the possibility of extending the utilization of the limited wheat supplies for bread and other wheat-based foods through mixed flours, including commonly available non-wheat sources (Kim and Ruiter, 1969; Blaise and Okezie, 1980). Protein

flours from oil seeds, such as sunflower, cottonseed, peanut, soya and several types of beans and peas can be used to substitute for part of the wheat flour in bread formulations to make good and acceptable bread. Similarly, starch flours from tropical tubers and cereals can be used.

Composite flours can be formulated to improve the diet (MacConell *et al.*, 1974 ; Hoover, 1975; Fleming and Sosulski, 1977). Officially recommended levels of minerals and vitamins (Gage, 1978) may also be added. When wheat flour has strong gluten, compositing may be necessary to dilute the gluten in order to obtain a flour of desired baking end use. Starchy flours can be very useful in this particular requirement.

Pure wheat bread has attractive organoleptic properties which have made it a popular food worldwide. Composite bread, being less expensive than wheat bread, would be economically more desirable in poorer nations. But in order for composite bread to be acceptable to the consumers, it should have organoleptic properties close to those of pure wheat bread.



## 2. OBJECTIVES OF THE INVESTIGATIONS

The main objective of the investigations was to determine the possibility of using starches from some tropical root crops in breadmaking. Such bread would need to have qualities acceptable to consumers.

### 2.1 Determination and Comparison of the Physico-chemical Properties of Starches from some Tropical Root Crops and Canadian Wheats

Tropical root crops, unlike wheat, are not customarily used for breadmaking. It was therefore necessary to determine and understand the physical and chemical properties of the root starches in order to relate them to those of wheat.

Arrowroot, cassava, sweet potato, taro and yam starches were selected for the study. Flours and starches of Neepawa (superior bread making) and Fielder (inferior bread making) Canadian spring wheat, were included.

The properties investigated for every starch were: grain size distribution and morphology; amylose and mineral contents; grain swelling power and solubility; gelatinization behaviour; retrogradation with storage time; complexing with monoglycerides; affinity for gluten; and role in breadmaking.

## 2.2 Breadmaking with Root Starches and Gluten Composite Flours and its Comparison with Bread made from Spring Wheat Flours

The dry matter content of flour for baking is mainly starch (75-80%) and gluten (11-15%). In order to investigate the compatibility and role of root starches in bread, composite flours consisting of 85% starch and 15% gluten were made and used throughout the study. Interference from the other ingredients usually present in bread formulations was avoided by making bread intended for instrumental analysis from a lean formula of flour, sugar and salt, while bread for panel tasting and overall quality assessment had shortening included in its formulation in order to improve its organoleptic properties. Of particular interest were the handling properties of the composite doughs and the shelf life of their corresponding breads.

### 2.2.1 Determination and Comparison of the Dough Rheological Characteristics of Wheat and Composite Flours

Prior to using the composite flours in breadmaking, it was necessary to determine the rheological properties of their doughs during mixing. Knowledge of the handling properties of the doughs during mixing was required in order to make doughs of equivalent consistencies by subjecting each to its optimal mixing requirements. Hence, the optimal flour water absorption; dough arrival, development, stability and departure times; and mixing tolerance under

standard farinographic conditions were determined for each composite flour and compared with those of wheat flours

### **2.2.2 Determination and Comparison of the Shelf Life**

#### **Properties of Bread from Wheat and Composite Flours in Absence and Presence of Monoglycerides**

Starting soon after baking, chemical and physical changes occur in bread, making it stale. Stale bread is firm and has very poor organoleptic properties. As a result, it has low consumer acceptability, thereby inflicting economic losses on processors and retailers of bread.

Since monoglycerides are known to reduce the tendency of bread to firm during storage, they are widely used in the bread industry. Breads containing monoglycerides or none at all was made from composite flours and compared with similar bread from wheat flour. Experiments to investigate their shelf life stability were designed to cover crumb compressibility, penetration resistance, and crystallinity of the starch in the crumb as a function of time.

### **2.3 Quality Evaluation of the Bread by Taste Panel**

Knowledge of the quality of the composite bread, as judged by consumers, was needed. To provide this information, a taste panel was organized to evaluate and give organoleptic appraisal to the composite types of bread, and compare them with wheat bread according to the bread quality scoring and evaluation chart recommended by the

American Institute of Baking (Matz, 1960).

### 3. LITERATURE REVIEW

#### 3.1 Tropical Root Starches

Several tropical root crops are known to be excellent sources of starch. The main examples are arrowroot, cassava sweet potato, cocoyams (taro and tannia), and yams. Examination of literature on tropical root crops (Shipman, 1967; Edmond and Ammerman, 1971; Onwueme, 1978; Tu *et al.*: 1979; Wang, 1983) shows that cassava is by far the most industrially exploited root crop in terms of starch production. Sweet potato starch, according to Phillips (1974), is of more significant industrial importance in Japan than elsewhere due to political protection. Arrowroot provides a minor source for industrial starch production, while taro, tannia and yams are important staple foods in the tropical areas where they are grown, but have not been given serious examination for industrial starch production.

##### 3.1.1 Occurrence

Synthesis of starch occurs first in the leaves of plants. Starch cannot however be directly translocated within the plant. It must therefore first be broken into sugars, usually sucrose and glucose, to facilitate translocation to the amyloplasts (plastids) for resynthesis and storage (Badenhuizen and Dutton, 1956; Porter, 1962). Such starch acts as a stored energy resource capable of being remobilized by enzyme action whenever needed by the

plant.

For tropical root crops, starch storage occurs in the plastids of the tuberous roots of cassava, yams and sweet potatoes as well as in the arrowroot rhizomes and in the tannia and taro corms.

Starch may be deposited as a single granule or several granules per amyloplast. Starch is said to be simple in the former case and compounded in the latter (Buttrose, 1962; Badenhuizen, 1965). Cassava and sweet potato are examples in which compounded starches occur. In most cases, compounded starches are disintegrated during the starch Isolation process.

The various root crops contain different amounts of starch, depending on type of crop, cultivars, soils, climate and age of the crop. Fresh, mature arrowroots contain 22-28% starch (Shipman, 1967). Mature cassava contains about 32% when tubers are peeled (Knightly, 1969), and about 20-25% in unpeeled fresh roots (Purseglove, 1968). Wijeratne (1974) reported that Sri Lankan cassava varieties contain 22-23% starch. Sweet potatoes contain up to 30% starch (Knightly, 1969). Keitt (1909) reported the lowest starch content of 14.43-16.46% and the highest content of 16.46-19.07% for American sweet potatoes. Taro corms contain 13-29% carbohydrates, of which 77.7% is starch (Coursey, 1968; Oyenuga, 1968; Onwueme, 1978). Carbohydrates form about 1/4 the mass of fresh yam tubers; 28% having been reported for *Dioscorea alata* (Onwueme, 1978).

### 3.1.2 Starch Isolation from Tubers

Production of starch from cassava tubers and arrowroot rhizomes follows the same process (Shipman, 1967). Fresh tubers or rhizomes are washed with high pressure water jets. The cleaned roots are rasped into a pulp, which is suspended in plenty of clean water and sent to a series of shaker or rotary type screens for separation of the fibrous debris. Sulfur dioxide (0.2% -- Shipman, 1967; 0.05% -- Onwueme, 1978) is added to the final screening and washing steps in the process to improve the color of the starch, aid in settling and hinder the actions of molds and bacteria on the starch. In addition,  $\text{SO}_2$  keeps the screens free of gummy substances which would otherwise block them (Shipman, 1967).

The starch milk is passed through a cyclone to remove sand and other debris. The starch is then recovered from the milky suspension by centrifugation or settling, and is then dried to a moisture content of 10-14% (Shipman, 1967; Knightly, 1969; Onwueme, 1978). The starch is usually pulverized before bagging.

Sweet potato starch is isolated in a process in which an alkaline pH (9) is maintained with calcium hydroxide solution (Knightly, 1969). After washing and maceration, the pulp is forced through screens.  $\text{SO}_2$  is added to prevent formation of melanin color from tyrosine. The starch is washed and separated from the pulp in centrifuges lined with screens. Further cleaning is achieved in centrifuges before sending the starch milk through hydrocyclones for

purification. Water is removed and the starch dried (Knightly, 1969).

Literature on utilization of taro and yams for starch production is scanty. Starch from yam tubers may, however, be produced using the same process as for potatoes, with appropriate pH adjustment. Starch from the taro corms can be obtained using the same process as for arrowroot and cassava.

### 3.2 Wheat Starch Properties

From 75 to 85% of the wheat kernel is made up of the endosperm. The endosperm consists of starch grains embedded in a matrix of gluten-forming proteins. During wheat milling, the endosperm is scraped out, being separated from the bran and the germ before grinding to flour (Britton, 1969; Ziegler and Green, 1971; Alf, 1975; Jenkins, 1975).

The starch content of wheat kernels and flour varies inversely with the protein content (Hopkins and Graham, 1935), the total amount being affected by soils and climatic conditions (Pomeranz, 1980). Hard wheats contain less starch than soft wheats. Starch content in flour is affected by the degree of extraction and refinement (D'Appolonia *et al.* 1971). For instance, 80% extraction flour with 14% moisture has 65-71% starch (Herd and Kent-Jones, 1931; Hopkins and Graham, 1935, Dimler *et al.* 1944; Fraser and Holmer, 1956; Pomeranz, 1980). On a dry basis, bread making wheat flours have 75-80% starch (Tipples, 1969).



### 3.2.1 Isolation of Starch from Wheat Kernels

Earlier methods of starch isolation from wheat, for instance the Halle process, aimed at destroying the wheat protein. Whole wheat kernels were softened by steeping in water followed by one to four weeks of fermentation in which the gluten was broken down by chemical and biochemical reactions (Radley, 1953; Anderson, 1967). Dissolved protein was eliminated as soluble waste by decantation. The starch was separated in revolving screens, and purified by repeated agitation in water, sedimentation and decantation. The clean starch was then dried (D'Appolonia *et al.* 1971).

The Halle process was superceded by the Alsation process by which starch and gluten could both be isolated from the wheat kernels. Wheat was squeezed in net bags through a series of rollers, the starch being washed out at the same time with water, then recovered from the suspension and dried. Gluten was recovered from the residue in the bags. The process was quite laborious (Radley, 1953; Anderson, 1967).

Modern starch isolation from wheat uses the Martin or Batter processes, both of which start with wheat flour. The cleaned wheat kernels are tempered (AACC, 1982, method 26-95) before milling. Tempering is necessary to toughen the bran on the kernels and condition the endosperm moisture content to facilitate clean and more complete isolation and separation of the endosperm from the bran and the germ (Britton, 1969; Ziegler, 1971; Alf, 1975; Jenkins, 1975).

The endosperm is then reduced to flour by rollers.

In the Martin Process, a dough is made and starch is washed out from it with plenty of water. The gluten left behind is carefully dried to save its vitality. The starch is centrifuged from the slurry and flash dried to about 10-12% moisture content (Knightly, 1969; Anderson, 1967; D'Appolonia *et al.*, 1971).

The Batter process differs from the Martin process in that a slack dough is made and disintegrated in a large amount of water. Gluten forms into small curds and the starch is suspended in the water. The gluten and starch are separated by screening. The gluten is then washed and dried, and the starch is recovered as in the Martin process (Knightly, 1965, 1969; Anderson, 1967; D'Appolonia, 1971).

Chemical methods using 0.1% NaOH (Knightly, 1969), 0.3%N NaOH (Dimler *et al.*, 1944) or 0.2N  $\text{NH}_4\text{OH}$  (Phillips, 1966) for the solubilisation of protein from wheat flour, leaving starch behind, have been described. The starch produced by the Martin and Batter processes is, however, purer than that obtained by the chemical processes.

### 3.3 Starch Properties

For almost five decades starch researchers have been interested in the properties of native starches and their functionality in foods (Alsberg, 1935; Pulkki, 1938; Sandstedt *et al.*, 1939; Harris and Sibbit, 1941, 1942; Burham and Clapp, 1942; Whistler *et al.*, 1955; Schoch and Maywald,

1956; Schock, 1965; Medcalf and Gilles, 1965; D'Appolonia and Gilles, 1971; Hosney *et al.* 1971; Dahle, 1971; Kulp, 1972; Rasper *et al.* 1974; Sterling, 1978; Pomeranz, 1980; Hoover and Hadziyev, 1981; Christianson *et al.* 1982; and many others).

Several properties of starch have received intensive investigation. These include: grain size distribution and morphology; composition and structure of the grain; starch swelling power and solubility; viscosity of the starch pastes; gelatinization characteristics; retrogradation; affinity for gluten; and complexing with monoglycerides to counteract the effect of retrogradation. These properties affect the texture and quality of foods containing starch. They are reviewed further below in relation to root crops and wheat starches.

### 3.3.1 Starch Grain Size Distribution and Morphology

Starches from different sources are so characteristic in size and shape that they can be identified this way (Schock and Maywald, 1956; D'Appolonia *et al.* 1971). In addition to size and shape, other properties of interest are the presence or absence of hilum, its position, and whether it is centric or acentric. The presence or absence of striations is also an important feature in relation to whether or not they surround the hilum. The appearance of the granule when viewed in polarized light, normally called birefringence, is also important.

### 3.3.1.1 Arrowroot Starch

Arrowroot starch grains vary in size from 5-50  $\mu\text{m}$  (Seidemann, 1964). This size range is larger compared to 3.9-15.6  $\mu\text{m}$  reported by Ciacco and D'Appolonia (1977). These researchers reported arrowroot starch to have a centric polarization cross. The grains had a circular appearance under polarized light.

### 3.3.1.2 Cassava Starch

Cassava starch grain size distribution has been presented as 0-5  $\mu\text{m}$  (7.3%); 6-10  $\mu\text{m}$  (21.3%); 11-15  $\mu\text{m}$  (19.63%); 16-20  $\mu\text{m}$  (36.0%); 21-25  $\mu\text{m}$  (8.52%); 26-30  $\mu\text{m}$  (4.24%); 31-35  $\mu\text{m}$  (2.68%); and 36-40  $\mu\text{m}$  (0.2%) [Seidemann, 1963]. Wivinis and Maywald (1967) described cassava starch grains as round, truncated-egg, and cap-shaped, with moderate polarization crosses. Ciacco and D'Appolonia (1977) found them to vary from 7.8-19.5  $\mu\text{m}$  in size and they were round in polarized light. Onwueme (1978) reported cassava starch grains to be 3-35  $\mu\text{m}$  in size, while Pyler (1979) reports an average size of 20  $\mu\text{m}$  and describes them as round or oval in shape, characterized by an indentation on one side and the presence of a fissured centric hilum.

### 3.3.1.3 Sweet Potato Starch

According to Keitt (1912) and Thurber (1933), the grains of sweet potato starch are similar to those of cassava. Matwejew (1958), cited by Seidemann (1966), found that sweet potato starch grains varied in size from 0-10  $\mu\text{m}$ .

(64-89%); 11-40  $\mu\text{m}$  (10-36%); and 41-70  $\mu\text{m}$  (0.07%). This agreed with the work of Reichert (1913) and of Seidemann (1963). The granules of sweet potato starch have been described as round and polyhedral, some them having rounded facets. The hilum is centric. Polarization crosses vary from strong in the rounded ones to weak in the polyhedral ones (Wivinis and Maywald, 1967).

#### 3.3.1.4 Taro Starch

Payne et al. (1941) reported the size of taro starch grains to vary from 2.5-9.3  $\mu\text{m}$ . This was corroborated by Amin (1955), whereas Seidemann (1966) reported a size distribution of 3-15  $\mu\text{m}$ , with a few large ones going up to 21  $\mu\text{m}$ . Wivinis and Maywald (1967) found that all taro starch grains were less than 1  $\mu\text{m}$  in size. Radley (1940)

more recently, Higashihara et al. (1975) and Griffin (1979) concluded that taro starch grains vary in size from 1-6.5  $\mu\text{m}$ . According to Seidemann (1966), the grains are similar in shape regardless of species. They are compounded in groups of 2-6 or more grains. Individual grains result from the disintegration of the compound granules. Individual grains have weak polarization crosses and acentric fissures.

#### 3.3.1.5 Yam Starch

Yam starches have been reported to range in size from 1-70  $\mu\text{m}$  Seidemann (1964). Some species of yam vary greatly, while others are quite similar in size and morphology of

their starch grains. According to Onwueme (1978) the largest starch grains are located in the pith of the tuber, decreasing in size towards the tuber ends and surface.

The starch grains of *Dioscorea alata* vary in size in the ranges 5-20, 21-40, 41-50, and 51-70  $\mu\text{m}$  (Planchion and Juillet, 1909). Rao and Beri (1955) found them to be 36  $\mu\text{m}$  long and 21  $\mu\text{m}$  wide on average. Ciacco and D'Appolonia (1977) reported them to be 25.4-31.2  $\mu\text{m}$  in width and 27-50  $\mu\text{m}$  in length, homogeneous in shape, being mainly triagonally rounded and possessing an acentric polarization cross. Seidemann (1964) described the grains as elongated, oval, partly rounded or slightly curved, triangularly rounded or ellipsoidal. Onwueme (1978) reported an average of 55  $\mu\text{m}$  for the granule size.

*Dioscorea rotunda* starch grains vary in size from 5-20, 21-55, and 56-60  $\mu\text{m}$  (Ching-Shen, 1955). They are elongated, oval, egg- or ellipsoidal-shaped (Seidemann, 1964). The starch grains of *D. batatas* are similar to those of *D. rotunda*.

*D. dumentorum*, *D. hispida* and *D. esculenta* have very small grains of starch, all 1-5  $\mu\text{m}$  (Seidemann, 1964). Onwueme (1978) reported an even smaller size range of 1-2  $\mu\text{m}$  for *D. esculenta*.

*D. bulbifera* has starch grains of 5-45  $\mu\text{m}$ , the majority being 20-40  $\mu\text{m}$ . The size ranges for *D. cayenensis* are 3-10, 10-20 and 20-25  $\mu\text{m}$ . The two species are very similar morphologically. The starch grains are rounded,

triangular or trapezoidal, with a pronounced hilum (Seidemann, 1964; Onwueme, 1978).

### 3.3.1.6 Wheat Starch

Hoyer (1911) found the size of wheat starch grains to be from 30-55  $\mu\text{m}$ , while Remenovskiy (1921) found the limit to be 57  $\mu\text{m}$ . Brehmer (1928) examined several wheat varieties and reported starch grain size ranges of 14-39  $\mu\text{m}$  (*T. turgidum*); 15.4-39.6  $\mu\text{m}$  (*T. Spelta*); 11.1-30.1  $\mu\text{m}$  (*T. dicoccum*); and 12-27  $\mu\text{m}$  (*T. monococcum*).

The presence of equatorial grooves on some wheat starch grains was reported by Burham and Clapp (1942), Sandstedt (1955), and many others afterwards. Kerr (1950) differentiated wheat starch grains in two categories: small donut-like ones of size 2-10  $\mu\text{m}$  and large lenticular ones of size 20-35  $\mu\text{m}$ . According to Lentner (1956), small starch grains up to 10  $\mu\text{m}$  in size account for about 88.4% of all starch in wheat, while about 6.2, 5.2 and 0.8% of the grains are found in the size ranges of 11-20; 21-30; and 31-45  $\mu\text{m}$ , respectively. MacMasters and Waggle (1963) asserted that there is no discrete size division of wheat starch grains into large and small, but that there is a gradual continuous change in the size distribution. Many researchers, including Seidemann (1966) and Pyler (1979), tend to generally support Kerr's findings.

Some of the larger lenticular starch grains have a faint centric hilum, the surface being smooth with no striations (Whistler, 1965; D'Appolonia *et al.* 1971). Pyler

(1979), however, reported that the hilum is acentric. Although large granules form only about 12.5% of all the starch in wheat, they account for most of the weight and surface area of free wheat starch (Grewe and Bailey, 1927; Stamberg, 1939; Hanssen *et al.* 1953). Stamberg (1939) reported that the specific surface area of 1.0 g of wheat starch is 2,004 cm<sup>2</sup>.

### 3.3.2 Starch Granule Composition and Structure

The starch granule is made up of basically two polysaccharide polymers. Amylose has a straight chain configuration, containing D-glucose units linked through  $\alpha$ -1,4-glucosidic bonds. Amylopectin is a branched chain polymer of D-glucose units, connected as in amylose but through the  $\alpha$ -1,6-glucosidic bonds at the junctions (Hough and Jones, 1953; Greenwood, 1956; Wolf from and ElKhadem, 1965; Schoch, 1961, 1962).

— Amylose contains 500-2000 units of D-glucose and has an average molecular weight range of 80,000-320,000 (Schoch, 1961; Pyler, 1979). Amylopectin has about 20-30 glucose units per link and contains several hundreds of such links. For wheat starch, Potter and Hassid (1948) found that branching in amylopectin occurred after every 23 glucose units. According to D'Appolonia *et al.* (1971), these values only indicate the overall degree of branching. They do not show the inner or outer chain length of amylopectin. Lee *et al.* (1968) asserted that there was in fact no symmetrical



branching in amylopectin. The total molecular weight of amylopectin has been estimated at one million or over (Schoch, 1961; Pyler, 1979).

Minor branchings have been noticed and reported at some of the carbon 2 and 3 positions of the glucose units in the chains (Fruton and Simmonds, 1958). Leach and Schoch (1962) also reported the existence in corn of an intermediate, slightly branched polymer in addition to amylose and amylopectin. D'Appolonia *et al.* (1971), however, state that these observations are minor to the established presence of only amylose and amylopectin in the starch granule.

The starch granule as a whole is a spherocrystal. Inside the granule, amylose and amylopectin chains are organized in concentric radial shells (Badenhuizen, 1959). Adjacent amylose and amylopectin chains become bundled tightly into crystalline, randomly distributed regions called micelles, which are held together by hydrogen bonding. A single amylose or any of the free amylopectin chains may be involved in more than one micelle. Hence, micelles hold the starch granule firmly together and are responsible for birefringence observed as a polarization cross when starch is viewed in a light polarizing microscope. They are also responsible for the X-ray diffraction patterns observed on starch. Evidence provided by Montgomery and Senti (1958) tends to suggest that it is amylopectin which is the major participant in micelle formation.

Amorphous regions occur radially within and tangentially between shells where the amylose and amylopectin chains are less closely packed. These areas are not birefringent and are easily penetrated by water. Further submicrostructure of the starch grain has been detailed by Whistler and Turner (1955) and Nikuni (1957, 1977).

The amylose content of starch may be determined by potentiometric iodine titration (Bates *et al.* 1943; Schoch, 1964) or by a colorimetric method based on blue color formation of an amylose-iodine complex (McCready and Hassid, 1943). Using these methods, especially the former one, the amylose contents of several types of starches have been determined. Bates *et al.* (1943) reported an amylose content of 24% in wheat starch. Schoch (1945) found 26% using pentasol precipitation of amylose. Deatherage *et al.* (1955) found in several American and foreign wheats a range of 17-29% amylose. More recently, Medcalf and Gilles (1965) found wheat starch to have 23.4-27.5% amylose, which agrees with the range of 23.4-26.9 reported by Ciacco and D'Appolonia (1977).

Greenwood *et al.* (1955) found 20.5, 16.7 and 17.8% amylose in arrowroot, cassava and sweet potato starches, respectively. Onwueme (1978) reported 17% amylose in cassava starch. According to Ciacco and D'Appolonia (1977), arrowroot, cassava, and yam starches contain 13.8; 14.5 and 23.3% amylose, respectively. Onwueme (1978) reported taro starch to contain 17-28% amylose.

### 3.3.3 Starch Grains Swelling, Gelatinization, Solubility and Paste Viscosity

Swelling, gelatinization, solubility and paste viscosity of starch in an aqueous system are interrelated starch characteristics which have been reviewed extensively by Leach (1965) and D'Appolonia *et al.* (1971).

Native starch grains are insoluble in cold water despite of their molecules being highly hydroxylated. This is due to the presence of an internal micellar network holding the starch granule firmly together through hydrogen bonds. The starch granules, however, are able to absorb water (and other solvents), swelling in the process. In a cold aqueous system, the swelling is limited and reversible. If the starch grains are heated with sufficient water, they remain unchanged in appearance until a critical temperature is reached at which point some of the grains begin to swell tangentially and irreversibly. They are said to be gelatinizing (Leach, 1965).

Schoch (1964) devised a method for determining the swelling power (SP) and solubility of starch. It involves heating and stirring at 200 rpm 0.5 g of starch in a total of 180 g of distilled water in a 250 ml centrifuge bottle at a controlled temperature for 30 minutes. Stirring is stopped and the stainless steel paddle rinsed clean into the bottle with a little distilled water followed by addition of water to a total of 200 g water. The bottle is then corked and the contents mixed by inversion before centrifugation at 3000

rpm for 15 min. Dissolved starch is determined as a percentage from 50 ml aliquots of the centrifugate by evaporation. The swelling power of the starch is determined from the paste residue in the bottle after correction for dissolved starch. It is expressed as grams of starch paste per gram of starch on dry basis.

$$SP = \frac{(g. \text{ starch paste} \times 100)}{(Wt \text{ of sample, g}) \times (100 - \% \text{ solubles, db})}$$

Swelling power, gelatinization, starch solubility and viscosity are functions of temperature, and vary with different starches.

Gelatinization does not occur at one temperature, but proceeds over a temperature range which is characteristic of starch species, and even varies between granules of the same starch. This is due to differences in the internal three dimensional molecular associations in the starch grains. Also larger grains of starch swell and gelatinize more easily than the smaller ones (Leach, 1965; Sterling, 1974, 1976; Chung and Hudziyev, 1980).

Gelatinization entails rupture of the hydrogen bonds inside the starch granules. This action results in increased diffusion of water into the granules, where it hydrates the starch molecules on the hydroxyl groups set free by the cleavage of the hydrogen bonds. The starch grains swell, but still retain their outlines (Schoch, 1965).

Gelatinization and swelling begin in the amorphous areas of the starch granule where bonding is weaker than in the crystalline micellar areas. In fully gelatinized

granules, the crystalline structure is completely disarrayed, as shown by x-ray diffraction analysis and the absence of birefringence (Leach, 1965).

The fully hydrated linear starch fraction diffuses from the swollen grains into the aqueous medium. Shorter chains leach out preferentially (Leach, 1965). At pasting temperature, the swollen starch grains begin to form a cohesive paste, the viscosity of which starts to rise exponentially, mainly due to the swelling of the starch granules as they absorb water. If the paste is stirred, then some of the swollen granules are broken, thus the final viscosity of the paste is a balance in consistency due to the unbroken swollen grains, broken fragments and dissolved starch (Anker and Geddes, 1944; D'Appolonia *et al.* 1971). Maximum viscosity is recorded when the maximum number of swollen grains exist in the paste, there after, the viscosity drops as the number of broken swollen grains increases (D'Appolonia *et al.* 1971). The viscosity of a paste is influenced by concentration, temperature, the type of starch and the environmental conditions during formation of the starch (Alsberg and Rask, 1924; Mangels and Bailey, 1933, 1934; Anker and Geddes, 1944). Popular instruments for measuring starch paste viscosity are the Brabender amylograph, Corn Industries and the Haake viscometer.

Several methods exist for studying gelatinization of starch. Amylographic methods depend on viscosity (Bean and Osman, 1959). Optical methods utilize photopastography

(Seidemann, 1967), and disappearance of birefringence in the polarized light (Watson, 1964; Miller 1973). Differential scanning calorimetry (DSC) involves thermal analysis of a sample over a selected temperature range including temperatures above 100°C, where other methods are not applicable (Stevens and Elton, 1971; Donovan, 1979; Wootton and Bamunuaracchchi, 1979; Donovan and Mape, 1980; Biliaderis *et al.* 1980; Hoover and Hadziyev, 1982). DSC enables one to work with different water/starch ratios without the problems of moisture loss control. It facilitates better determinations of transition temperatures and enthalpies.

Donovan (1979) showed that gelatinization was a function of the water present in starch. In excess water the amorphous areas and the crystallites gelatinized completely, giving one endotherm peak. At lower water levels, the crystallites do not become completely hydrated to cleave the hydrogen bonds. As a consequence, they melt at higher temperatures, giving a shoulder endotherm to the main gelatinization endotherm. At low water volume fractions of less than 0.5, the gelatinization endotherm becomes progressively smaller as the crystallites' melting endotherm increases. The presence of clathrates in cereal starches (e.g wheat) results in a melting endotherm near 100°C. This is absent in root and tuber starches.

The gelatinization enthalpy of starch in the presence of excess water represents the total of enthalpies required for swelling, hydration of molecules and melting of crystallites. When the water content is not sufficient, the enthalpy changes are mainly due to melting of the crystallites (Donovan, 1979). According to Stevens and Elton (1971), the decrease in the enthalpy of gelatinization observed as the water fraction in starch/water systems becomes smaller is due to a reduction in the degree of disorder inside the starch granule.

Several factors affect the swelling power, gelatinization, solubility and paste viscosity of starches. Starches high in amylose are more resistant to swelling and gelatinization due to a high level of internal association. Their paste viscosities are also low (Leach *et al.* 1959; Leach, 1965). Waxy starches have higher swelling powers than normal starches. Evidence provided by Leach and Schoch (1961) indicates that amylopectin is the one responsible for swelling power since starches from which amylose had been digested enzymatically still had normal swelling power.

Cereal starches swell less than root starches, while root starches swell less than tuber starches (Leach, 1965). The presence of chemical adjuncts, ionized esterified phosphate groups, natural and added surfactants and monoglycerides, fatty acids, compounds that compete for water and derivatization influence the starch swelling power and gelatinization. Solubility and viscosity are

consequently affected (Leach, 1965; D'Appolonia *et al.* 1971).

Sodium nitrate, urea, sodium hydroxide, ammonium hydroxide, potassium thiocyanate, potassium iodide and similar chemicals which cleave hydrogen bonds reduce the internal molecular association of starch granules. The result is reduced gelatinization temperatures and higher swelling powers. Depending on the severity of the treatment, such starches may swell and gelatinize at room temperature. Since the granules are internally weakened, they break easily under shear stirring force and hence have low paste viscosity. Similarly, treatment of granular starches with warm dilute acid and hypochlorite below their gelatinization temperatures results in starch of low paste viscosity and high solubility. Desolvating agents such as sodium sulphate will inhibit swelling of starch grains. In fact they are used in derivatizations where swelling and gelatinization of starch granules are not required (Leach, 1965; Roberts *et al.* 1965).

Excessive swelling and solubility in white potato starch has been attributed to the presence of ionized estersified phosphate groups which repel each other as a result of like charge (Leach, 1965).

Naturally occurring fatty acids and added surfactants and monoglycerides interact with starch to form clathrates, resulting in reduced swelling power and solubility. Starch so affected is very resistant to gelatinization and has low



water binding ability (Leach and Schoch, 1961; Gray and Schoch, 1962; D'Appolonia *et al.* 1971; Longley and Miller, 1971; Lonkhuysen and Blankestijn, 1974; Ohashi *et al.* 1979; Pomeranz, 1980; Chiasi *et al.* 1982; Hoover and Hadziyev, 1982).

The size of starch grains also affects their gelatinization temperature. Sterling (1974, 1976) and Chung and Hadziyev (1980) observed that larger starch granules gelatinize at lower temperature than smaller ones.

Esterification and etherification of starches results in reduced internal attraction forces of the starch granule. Consequently, these starches have high swelling ability and reduced gelatinization temperatures. They also have higher solubility and viscosity. The higher the degree of substitution, the greater the effect (Leach, 1965; Wurzburg and Szymanski, 1970)

Reactions of sodium trimetaphosphate, epichlorhydrin, phosphorus oxychloride and several other polyfunctional compounds, including anhydrides of adipic and acetic acids, react with starch granules to produce cross-bonded starches which resist gelatinization. Cross-bonding can be controlled to produce starches of different properties (Leach, 1965; Roberts, 1965; Knightly, 1965, 1967; Osman and Elizabeth, 1967; Wurzburg and Szymanski, 1970).

Organic substances, such as sugar, competing for water reduce the hydration capacity and swelling power of starch. They also delay gelatinization (Bean and Osman, 1959).

### 3.3.4 Starch Retrogradation

Glucose molecules in amylose chains have three free hydroxyl groups through which association by hydrogen bonding between neighboring chains can occur (Schoch, 1961; Pyler, 1979). If a hot dilute aqueous solution of amylose is cooled slowly, the amylose chains realign and form a precipitate. If the solution is cooled rapidly, the ability of the (amylose chains) to realign completely is hindered. A gel is formed which consists of randomly distributed sacs of fluid in a network of amylose chains held together through small micellar regions in which only parts of the amylose chains participate, the rest remaining as free loops (Meyer, 1950; Schoch, 1961). This physical instability of hydrated amylose molecules, resulting in precipitate and gel formation, is known as retrogradation (Katz, 1928, 1930; Schoch 1942, 1961, 1965; Hellman *et al.* 1954; Collison, 1968; Hellendoorn, 1971; Pyler, 1979).

A cooked paste of starch contains the swollen starch granules, amylose leached out of the granules and fragments of the broken granules. Retrogradation in this case involves intermolecular bonding within and between the swollen starch granules, their fragments and the free amylose (Meyer, 1942, 1950).

Retrograded starch is crystalline, and has the 'B' type X-ray diffraction patterns (Katz and Itallie, 1930; Foster, 1965). It has reduced swelling power, solubility, water binding capacity, iodine affinity and hydrolysis by acids

and enzymes (Volz and Ranstad, 1952; Sterling, 1957).

Retrograded cereal starches can be completely resolubilized

at high temperatures of about 125°C (Bechtel *et al.*

1965). The waxy starches can be resolubilized at lower

temperatures of 50-100°C (Osman and Cummisford, 1959;

Sastry, 1965; Pyler, 1977).

According to Katz (1928) and Sterling (1960), maximum retrogradation occurs at -2°C, while the minimum rate is at

below -20°C; and at or above 60°C. Odd cases have been

reported where the retrogradation rates at 37 and 62°C were

the same (Lampitt *et al.* 1948), while the rate at 70°C was

greater than at room temperature (Sterling, 1960). Kalb and

Sterling (1961) observed that the retrogradation rate was

reduced if the starch was originally gelatinized at a higher temperature.

Retrogradation of starch is strongly accelerated by higher concentration of amylose and low molecular weight.

The shorter the length of the amylose chains, the higher the

starch retrogradation rate (Lampitt *et al.* 1948; Whistler

and Johnson, 1948; Lansky *et al.* 1949; Radley, 1953; Loewus

and Briggs, 1957; Schoch, 1965; Whistler, 1965).

Furthermore, Lampitt *et al.* (1948) and Pyler (1979) reported

that chain uniformity played an important role in

accelerating the rate of retrogradation. Several researchers

have reported on the influence of pH. Retrogradation is

favorable by adjusting pH to 5.0 before gelatinization (Hollo,

1960; Kalb and Sterling, 1961) and to 1.3-2.2 after

gelatinization (Schoch, 1941; Kalb and Sterling, 1961). Pyler (1979) reported a pH of 7 to favor retrogradation most, while Foster (1965) reported that the retrogradation rate was faster at pH 6.5 than at pH 4, where it was almost non-existent.

According to Hellendoorn (1971), the rate of retrogradation in mashed potato products increased as the moisture content decreased, to a maximum at 30% moisture. Duckworth and Smith (1963) found that the absolute minimum rate for retrogradation existed at monomolecular layer moisture content.

The presence of monovalent anions and cations, especially iodide and potassium, retard the rate of retrogradation, while polyvalent anions and cations accelerate it (Loewus and Briggs, 1957). Ciasco and Fernandes (1979) have reported that the rate of retrogradation is progressively increased by anions  $I^-$ ,  $Br^-$ ,  $Cl^-$ , and  $F^-$ , while similar increases for cations are in the order of  $K^+$ ,  $Li^+$  and  $Na^+$ .

Formation of clathrates due to interaction of starch with fatty acids and surfactants reduces starch hydrophilicity and hence hinders retrogradation (Zobel, 1963). Greenwood and Hourston (1967) reported that starch derivatization through esterification and etherification introduced substitutes in the starch granule, making it difficult for the amylose and the free end chains of amylopectin to realign and retrograde. McIver *et al.* (1968)

reported that retrogradation rate decreased as a function of time. Their findings were confirmed by Brennan and Sodah-Ayenor (1973) and Knightly (1977). It has been observed that starch gels contract and exude water as the amylose molecules align and associate during retrogradation. The gels become opaque as they age.

### 3.3.5 Starch Complexing with Monoglycerides

Knowledge of the nature of monoglycerides is necessary in understanding their reactions with starch.

A monoglyceride is an ester of one fatty acid with glycerol. Esterification to any of the end carbons produces 1(3)- $\alpha$  monoglycerides while esterification on the central carbon always produces  $\beta$ -monoglycerides. (Doerfert, 1968; Pyler 1979).

The fatty acid chain is lipophilic and is easily dispersed in oils and fats, while the glycerol group is hydrophilic, hence is easily dispersed in water.

Consequently, monoglycerides possess a hydrophilic - lipophilic character, which is however, not evenly balanced because of the nature of the fatty acid and glycerol (Griffin, 1949; Krog, 1981).

The hydrophile - lipophile balance (HLB) represents the ratio of size and strength of the polar (hydrophilic) to nonpolar (lipophilic) groups. It is expressed on a numerical scale on which the monoglyceride becomes more lipophilic the smaller below 9 its HLB becomes and more hydrophilic as its

HLB grows bigger than 11.0 (Griffin, 1949). Commercial monoglycerides have an HLB of 2.8-3.5 (MacDonald, 1964). This low HLB renders them excellent in softening bread and hence retarding the staling process (Knightly, 1968). They are however not good dough strengtheners because of the low HLB (Del Vecchio, 1975). Special monoglycerides with increased HLB have been made by introduction of organic acids such as lactic, tartaric, succinic and fumaric into their structure through ethoxylation and hydroxylation (Pitt, 1971).

Monoglyceride molecules arrange themselves into crystals with the terminal methyl group of the fatty acids on the surface and the glycerol groups at the center. They are said to be in their  $\beta$ -crystallinity form (Birnbaum, 1971). If a monoglyceride in its  $\beta$ -crystallinity form is heated near its melting point in aqueous medium with appropriate pH and then allowed to cool down to room temperature, the hydrated monoglyceride molecules reverse their orientation: the polar glycerol groups are on the surface and fatty acids chains in the centre of the crystal. The monoglycerides are in their  $\alpha$ -crystallinity form when they assume this orientation (Wren, 1968; Krog and Jensen, 1970; Langendijk and Penning, 1970; Lankhuysen and Blankestijn, 1974, 1976; Hoover and Hadžiyev, 1982). The use of  $\alpha$  and  $\beta$  in the structure of monoglyceride molecules is therefore different from their meaning when used in describing monoglyceride crystals.

Commercial monoglycerides are manufactured by direct esterification of fatty acids, interesterification, or glycerolysis of fats at 200°C in an excess of glycerol and in the presence of an alkaline catalyst (Pyler, 1974). Glycerolysis of fats is more commonly used for manufacture of food grade monoglycerides (MacDonald, 1974; Lauridsen, 1976). The alkali is neutralized, while the excess glycerol is removed by vacuum (Pitt, 1977). The process gives about 50% monoglycerides, which are concentrated to 90-95% by molecular distillation under high vacuum (Birnbaum, 1965). At least 90% of the concentrate consists of  $\alpha$ -monoglycerides (Brokaw *et al.*, 1955).


Several factors affect the reaction between starch and monoglycerides. They include type of monoglyceride and its physical state, type of starch and its amylose content, and temperature.

According to Krog (1981) and Hoover and Hadziyev (1982), the length and unsaturation of the fatty acid chain significantly affects the ability of a monoglyceride to combine with starch. Previously, it had been found that glyceryl-monopalmitate was more reactive with starch than -monomyristate, -monolaurate, -monostearate, -monoarachidate, -monooleate, and -monolinoleate (Lagendijk and Pennings, 1970). It was also found that the amount of amylose-monomonoglyceride complex formed decreased with the degree of unsaturation. This was explained by the fact that a saturated monoglyceride has a straight chain of about 4 Å

outer diameter and fits well into the amylose helix, which has an inner diameter of about 6 Å (Rundle and French, 1943). In comparison, unsaturated monoglycerides have bent chains due to double or more bonds and, as a result, are not readily fully accommodated by the amylose helix. In their work, Lagendijk and Penning (1970) also reported that the quantity of clathrates formed by a monoglyceride was favored by long fatty acid chains.

From previous work, Krog and Jensen (1970) had found that  $\alpha$ -crystallinity form monoglycerides were more reactive than the same monoglyceride in  $\beta$ -crystallinity form. Their finding was confirmed by Lagendijk and Penning (1970); Lonkhuysen and Blankestijn (1974, 1976) and Hoover and Hadziyev (1982).

Osman *et al.* (1961) reported that wheat starch amylose bound more monoglyceride per gram than cassava starch amylose. This was confirmed by Lonkhuysen and Blankestijn (1974, 1976), who also reported that, while wheat starch bound more monoglycerides at 30°C, gelatinized cassava starch bound more monoglyceride than gelatinized wheat starch. According to their work, surface area of starch did not appear to be a significant factor influencing ability of starch to bind monoglycerides. They were of the opinion that other structural factors were important. Gelatinized cassava starch was found to disintegrate to a higher degree on gelatinization than wheat starch. Exposure of more reactive sites accounted for the ability of gelatinized cassava





starch to bind more monoglycerides than gelatinized wheat starch (Lonkhuysen and Blankestijn, 1974, 1976).

Jongh (1961) observed that a suspension of starch was flocculated by added monoglycerides. This was confirmed by Lonkhuysen and Blankestijn (1976). Observations made by these three researchers as well as those of Krog (1970) and Lagendijk and Penning (1970), that starch treated with monoglycerides was firmer and resisted gelatinization more than starch without monoglycerides, support Schoch (1965) who indicated that monoglycerides are able to penetrate into the starch granules and clathrate with amylose. This immobilizes the amylose inside the granules. This interaction reduces the hydration capacity of amylose and the linear end chains of amylopectin, resulting in reduced swelling power of the starch. Hoover and Hadziyev (1982), working with potato starch, reported that monoglycerides absorbed by potato starch grains reduced their swelling power, solubility, rehydration rates, water holding capacity and iodine affinity.

### 3.3.6 Starch Affinity for Gluten

Starch affinity for gluten refers to the ability of starch to associate with gluten in the dough and in the crumb. The association is pH and heat sensitive.

Yoshima and Matsumoto (1966) showed that wheat proteins were positively charged and behaved as colloids at pH 5 and 6. Dahle (1971) reported that wheat starch and protein

associated better in acid to neutral pH and that the affinity between the two decreased with the increase in pH. He observed that both starch and gluten had negative charges in alkaline pH and this reduced affinity between them due to repulsion.

Bennet and Ewart (1962) showed that the rheological properties of dough were sensitive to pH. They reported that dough extensibility decreased as pH increased, indicating increased affinity within the system. Hlynka and Chanin (1957) showed that loaf volume was affected by pH of the dough. Minimum loaf volume was obtained at pH 7 and maximum at pH 5.7. Loaf volume decreased at pH 5.3 and 4.7. Takuechi (1969) and Dahle (1971) reached a similar conclusion that the association between starch and gluten in the dough was due to attraction between oppositely charged colloids within the system.

Dennett and Sterling (1979) found that there was no affinity between raw gluten and ungelatinized starch. Gelatinized starch had, however, the highest affinity for raw gluten in a system simulating the early baking stage in bread making. The affinity of gelatinized starch for gluten dropped by 30-50% when the gluten was denatured in a system simulating fully baked bread. This seemed to corroborate observations made by Dahle (1971) that heated flour had poor bread making properties and decreased protein solubility.

Dennett and Sterling (1979) also found that the amylose content of starch had a direct influence on the affinity of starch for gluten. Since affinity between starch granules and gluten is a surface phenomenon, amylose must therefore be able to modify the surface of the granules to enhance the affinity. Lowry *et al.* (1951) found that amylose had greater affinity for gluten than amylopectin.

Since there is no affinity between ungelatinized starch and native gluten (Dennett and Sterling, 1979), it is implied that gelatinization and swelling of starch granules is necessary for the development of affinity for gluten. Swelling of the starch grains therefore seems to increase their porosity, while at the same time exposing a maximum number of hydrophilic sites for bonding with gluten.

The starch granules swell during gelatinization and absorb water from the gluten matrix. Hence, the gluten is dehydrated and denatured during baking. Gluten in this state has reduced hydrophilicity and therefore reduced affinity for the gelatinized starch. This has the advantage of resulting in softer and more flexible crumb. In fact a negative correlation between affinity of starch for gluten and both the fractional volume changes and firmness has been observed (Dennett and Sterling, 1979). High starch affinity for gluten in the early baking stage plays an important role of ensuring optimal association between starch and protein hence maximum development of the dough before the protein is denatured (Dahle, 1971).

### 3.3.7 Role of Starch in Bread Making

Starch constitutes about 65-70% of flour (Pomeranz, 1980) and plays a significant role in bread making. Its functions in this process have been outlined by Sandstedt (1961). It dilutes the wheat protein such that the wheat flour is of good consistency for baking. Some of the starch granules get damaged during milling. A low level of starch damage is acceptable (Tipples, 1969) as it provides an easily available substrate for enzymatic activity to produce sugars used by the yeast during dough mixing and fermentation. Too much starch damage destroys both the baking quality of the starch and its products (Alsberg and Griffing, 1925). Damaged starch granules absorb water and swell excessively during dough mixing and fermentation (Sandstedt, 1955), being completely hydrolysed by the combined action of  $\alpha$  and  $\beta$  amylases. Intact starch granules are resistant to  $\beta$  amylase activity (Sandstedt *et al.* 1960) and are only slowly attacked by  $\alpha$  amylase (Sandstedt and Gates, 1954).

Starch provides an active surface for gluten to adhere to during bread making operations. Dough rheological and crumb properties are significantly influenced by this association (Takuechi, 1969; Dahle, 1971; Dennett and Sterling, 1979).

Starch granules orientate themselves in the protein matrix lining the gas-cell walls (MacMasters, 1961). As the starch granules gelatinize during baking, they swell, but do

not disintegrate due to limited water availability (Farrand, 1972). Limited water absorption renders them flexible, which in turn extends the elasticity of the gas-cell walls in the crumb. They absorb water from the gluten matrix in which they are embedded and cause it to set, hence providing support for the whole crumb structure.

The functionality of starch in baking thus depends on its gelatinization in a protein matrix to form porous and elastic crumb. Factors affecting starch gelatinization will therefore affect its function in bread making. In addition to those already mentioned above, a few more merit to be mentioned.

Small size starch grains have higher gelatinization temperature and hydration capacities (Kulp, 1973; Dennett and Sterling, 1979). An inverse relationship between loaf volume and size of starch granules has been reported (Ponte *et al.* 1963). Starches high in amylose content are more resistant to gelatinization (Medcalf, 1968; Kulp, 1973). Yasanuga *et al.* (1968) found that time-temperature relationship were important in influencing starch gelatinization. Furthermore different starches respond somewhat differently (Sandstedt, 1961). Harris and Sibbit (1941, 1942) found that starches from different wheats differed in their baking trials with gluten. This observation was confirmed by Hoseney *et al.* (1971). Additives such as sugar (Leach, 1965) and monoglycerides (Yasanuga, 1968) reduce starch swelling capacity by

competing for water or affecting the ability of starch to absorb water.

Firming of bread is due to starch retrogradation. This is an undesirable behavior since it results in staling of the bread.

### 3.3.8 Gluten

Gluten is the main wheat protein deposited in the kernel endosperm. It forms the protein matrix in which the starch granules are embedded (Bradbury *et al.* 1956; MacMasters *et al.* 1971). On milling, the endosperm is separated from the bran, followed by reduction to flour. The flour contains from 11-15% protein (Tipples, 1969), of which gluten is 85% and nongluten proteins 15% (Pomeranz, 1980). Gluten may then be isolated from the flour using either the Martin or Batter processes (Knightly, 1965, 1967). The extract consists of 75-80% gluten, 5-15% residue starch, 5-10% lipids and a small amount of minerals (Pyler, 1979).

### 3.3.9 Composition and Behavior of Gluten

Based on solubility in different solvents, gluten has been separated into its components by Osborne (1907). They consist of gliadin and glutenin, which account for the properties of gluten. These two constitute 85% of all the protein in wheat flour. The nongluten-forming protein fraction consists of albumins, globulins, peptides, amino acids and flour enzymes. These proteins are soluble in water

or dilute salt solutions, and foam and coagulate relatively easily (Holme, 1966). The presence of a large number of aliphatic and aromatic groups in the nonpolar side chains results in their observed hydrophobic bonding in aqueous medium.

The polypeptide chains in gliadin and glutenin are bound together by disulfide bonds. In gliadin, they are mainly intramolecular, connecting parts of the same polypeptide chain into folds; while in glutenin, most occur between polypeptide chains, resulting in large molecular aggregates. This difference is due to the amino acid sequences in gliadin and glutenin (Pence and Nimmo, 1964).

About one third of protein exists in aggregates which cannot be extracted by dilute acetic acid (Mecham *et al.* 1962; Seckinger and Wolf, 1970). Dough mixing or addition to it of reducing agents, such as glutathione and cysteine, cleaves the disulfide bonds and disaggregates the protein globules into smaller units which are more easily extracted (Tsen, 1970). In addition, mixing straightens them out into chains. Disaggregation of globular proteins in flour is, therefore, a prerequisite to the formation of gluten films and matrix in the dough. Disaggregation involves mainly the scission of inter-chain disulfide groups and is related to the sulfhydryl-disulfide interchange reactions during dough mixing.

Sulfhydryl (-SH) groups were first reported by Sullivan *et al.* (1936). Sulfhydryl groups contained in the amino acid cysteine play a role in dough development by formation of disulfide bonds (-SS-) through the oxidising activity of flour oxidants such as iodate and bromate. Crosslinks are made if the -SH groups from different polypeptides are oxidised. The dough becomes more rigid as the network of cross-bonded polypeptides forms (Sokol and Mecham, 1960). Compounds that react with sulfhydryl groups for example iodoacetamide suppress disulfide bonds formation resulting in reduced dough resistance to mixing and accelerated breakdown (Binger, 1965). Such treatment inactivates oxidising agents in the dough. As they are mixed in air, stable doughs lose their sulfhydryl groups faster compared to weaker ones (Sokol *et al.* 1960).

Gliadin is soluble in 70% alcohol and has a molecular weight of 25,000-100,000. It is extensible with low elasticity when hydrated and forms a viscous fluid mass. It is soluble in acids and bases.

Glutenin is dispersible in dilute alkaline solutions. It has a high molecular weight of over 100,000. It is elastic, but has low extensibility and hence forms a tough rubbery mass when hydrated. It complexes readily with lipids.

MacDonald and Gilles (1967) reported that gliadin and glutenin contain high levels of glutamic acid and appreciably high amounts of proline. Helical formations in

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gluten protein molecules were found to be much limited as compared to other proteins due to the presence of proline which introduces a bend in the amino acid chains. The cohesiveness and elastic characteristics of the gluten were ascribed to the presence of a high level of glutamic acid (Dimler, 1963). Low solubility for gliadin and glutenin is accounted for by their deficiency in basic and acidic groups responsible for solubilization of most proteins.

The concentration of sulfhydryl and disulfide groups in flour is very low. Bloksma (1964) reported only one micromole of sulfhydryl and 10 of disulfide bonds per gram of flour. In spite of their low concentration, only a fraction of sulfhydryl groups is available for oxidation during dough mixing (Bushuk, 1961). A similar condition exists for the disulfide bonds, for which intra- and intermolecular bonds are important. Intermolecular -SS- bonds have been found to be the more reactive and hence play a significant role in influencing dough rheological properties. As the dough is mixed, some of the disulfide bonds are broken, but get reformed by interacting with the sulfhydryl groups within or between protein molecules. Strain is thus reduced between protein fragments and as a result, dough breakdown is delayed as mixing proceeds (Goldstein, 1957). Furthermore, sulfhydryl-disulfide interchanges during mixing of dough generate sulfhydryl groups which may be oxidised or participate in further exchange reactions. As such, sulfhydryl groups are never

used up, but keep on being regenerated. Hence very few sulfhydryl groups can produce extensive changes in dough consistency.

### 3.3.10 Role Played by Gluten in Bread Making

When water is added to wheat flour, the proteins gliadin and glutenin become hydrated and form gluten on mixing (Jenkins, 1975; Pyler, 1979; Pomeranz, 1980). Hydrated, well-mixed gluten is a tenacious colloidal system. It is responsible for retaining carbon dioxide in the dough during fermentation. That accounts for the superiority of wheat flour over flour from other cereals or root crops in processing of leavened products.

Gluten forms the background matrix in which starch is embedded in the dough and bread crumb. It is denatured during baking to form a resilient internal grid that supports the bread structure. The amount of water absorbed by flour during dough preparation is a function of gluten quality and quantity (Bushuk, 1975; Jenkins, 1975). It is high for flours containing a high percentage and good quality gluten. Volume and texture of bread are similarly functions of gluten quality and quantity (Bushuk et al.: 1968). Flours high in quality gluten content are better able to withstand the deleterious effects of damaged starch in the dough and bread (Tipples, 1969). Dough properties are at their best when the dough has sufficient gluten of good quality. The resulting bread has better shelf

life and consumer acceptability.

### 3.3.10.1 Flour Baking Performance

The baking performance of wheat flour is dependent on several quality factors. Investigations have shown that the factors are all interdependent. They include maturity of the flour since milling, quantity and quality of the gluten, starch content and degree of its damage during milling, levels of  $\alpha$  and  $\beta$  amylases, flour lipids and the presence of flour additives.

Freshly milled flour has been found unsuitable for baking because it imparts a yellow pigment to the bread. The bread is also shrunken in volume (Jenkins, 1975). Color is due to the presence of pigments in the fresh flour. These get bleached over time by atmospheric oxidation, but current practice utilizes bleaching agents such as chlorine, chlorine dioxide, ammonium chloride, acetone peroxide and benzoyl peroxide. Shrunken volume of bread from fresh flour is due to inelasticity of proteins in freshly milled flour. Storage up to about six weeks removes the inelasticity. Storage is avoided by using flour maturing agents. Except for benzoyl peroxide, the above named bleaching agents are also flour maturing agents or dough improvers. Additional dough improvers cited in the literature are ascorbic acid, potassium bromate, ammonium persulfate, azodicarbonamide, L-cysteine and monocalcium sulfate (Pratt, 1971; Jenkins, 1975).

A good flour for baking absorbs a large optimal quantity of water and forms a dough that develops in a reasonable time during which it is stable to mixing (Wehrli and Pomeranz, 1970; Bushuk, 1975; Jenkins, 1975; Pyler, 1979; Pomeranz, 1980). The percent water absorption of flour is affected by the quality of protein present in it. An inverse relationship exists between them. The starch present also affects the water absorption. Sandstedt (1955) reported that dry undamaged starch absorption is increased if the starch is damaged, the increase being proportional to the degree of starch damage (Tipples, 1969). Excessive water absorption due to damaged starch results in sticky doughs that are difficult to handle. Flours with low protein content and quality are affected most. The excess water has been found to decrease later during fermentation (Halton, 1961) and in the oven during baking (Atkinson and Fueher, 1960).

The amylase content of flour plays an important role in dough behavior and bread quality. Normal wheat flours have less  $\alpha$ - than  $\beta$ - amylase contents. If flour is made from sprouted wheats, it will contain more  $\alpha$ - and  $\beta$ - amylases. This is an abnormal situation because it results in excessive gasing and dextrinization of starch. Since cereal  $\alpha$ -amylase is still active when starch gelatinizes, dextrinization of starch during baking has been found to be proportional to  $\alpha$ -amylase present, but not to starch damage (Tipples, 1969). The crumb becomes sticky and the loaf

tends to curve in and collapse. In addition, dextrinization reduces the water binding capacity of starch. Only a limited degree of dextrinization is beneficial.

Lipid-gluten interaction in dough is essential to good quality in bread. In the dough, the interaction helps to seal in gas during mixing and fermentation periods. During baking, the lipids interact with starch and promote freshness in bread (Hoseney *et al.* 1970). It has been reported that polar lipids improve the crumb softness better than nonpolar lipids (Pomeranz *et al.* 1966; Pomeranz *et al.* 1969; Wehrli and Pomeranz, 1969). Flours from which lipids have been extracted have insoluble proteins and perform badly in baking (Chung *et al.* 1977). Some limited proteolysis to mellow gluten during fermentation is desirable.

### *3.3.10.2 Flour Additives in Bread making*

The essential components of a bread formula are flour, water, yeast and salt. In order to improve the quality of the bread, several other additives have been found necessary (Pomeranz and Shellenberger, 1971; Tipples, 1975; Jenkins, 1975; Pyler, 1979).

Fats for baking may be in the form of lard, regular shortening and emulsified shortenings. According to Pyler (1979), fats have a beneficial influence on bread quality, although their contribution to the general characteristics of bread is of limited significance. Fat is totally omitted in sponge cakes, while the level at which it is used in

yellow, white and pound cakes, some cookies and pastries is considerable as it plays an important role in their structural development.

Usage of fat in baking improves the aeration and handling properties of doughs. Fat gives tenderness to the product and increases its eating and keeping qualities. Bread with fat has a fine grain and tender texture. Stability in cake batters is significantly improved by the presence of fat. But the level of fat in the dough must be controlled up to about 3% since an excess leads to weaker doughs, reduced fermentation rate, as a result of fat coating on yeast and yeast food, and creamy crumbs.

Emulsified shortenings increase the water absorption capacity of the dough. Most of the water is retained during baking (up to 35%), and results in softer crumbs. Incorporated monoglycerides interact with starch to retard bread staling. Dough improvers such as succinylated monoglyceride (Meisner, 1969), sodium stearyl fumarate (Geminder *et al.* 1965; Brachfeld *et al.* 1966), tartaric acid esters of mono and diglycerides (Birnbaum, 1955) and acyl lactilates, for example calcium stearoyl-2-lactylate (Marnett and Tenney, 1961), are used to strengthen the dough against mechanical breakdown during mixing and handling. Dough improvers also called dough conditioners, have a lipophilic chain that binds amylose and a negatively-charged moiety that reacts with active positively-charged sites in gluten. The gluten and the

starch are bound more closely, bringing about strength in the dough as a whole. The grain and texture of the crumb are improved. Volume of the bread and its resistance to staling are increased.

Salt is added in order to improve the ability of gluten to absorb water. Doughs made without salt are slack and sticky. These conditions disappear when salt is added and mixed in well (Jenkins, 1975). Commercial dough improvers already mentioned have a similar effect. Salt can be added up to 3% in order to impart a characteristic flavor to bread.

Sugar is added in order to augment the sugars already in the flour for yeast nutrition and for color development during baking. In North America, excess sugar is added wherever sweet bread is preferred. Yeast needs sugar to produce carbon dioxide required for leavening the bread. Yeast food, providing required salts and nitrogen for yeast cellular development, is added in the form of ammonium salts, which are provided with starch as a filler. The yeast itself is added at a level of about 3% on flour.

Sometimes a flour is deficient in  $\alpha$  amylase activity. In this case bacterial  $\alpha$  amylase is added to officially recommended levels to ensure uniform gassing required for optimal grain and loaf volume.

Non-fat dry milk addition to flour has the best benefit in weaker doughs, where it lightens the dough in a similar manner as does salt. If used with stronger flours,

water absorption has to be increased to remove excess dough tightening and to obtain better consistency for handling and fermentation. Proteins from the milk additive increase the nutritional value of the bread. Lactose in the milk additive is not yeast-fermentable, but contributes to good crust color development. Addition of milk solids to flour is particularly beneficial where a long fermentation period is used and where the flour lacks amylolytic activity.

Eggs are used as additives in special baked products that fetch a higher price. Egg proteins reinforce flour proteins and as with the addition of milk solids, will tighten dough consistency. This is corrected by adjusting the water absorption accordingly. It has been reported (Jenkins, 1975) that egg whites used instead of additional gluten produce thin and crisp crust in baked products without excessive volume changes observed when gluten is added, which agrees with the fact that egg proteins do not entrap as much gas as gluten.

Enrichment in terms of officially recommended levels of vitamins and minerals may be included in bread formulation (Gage, 1978). Mold inhibitors are added so as to delay spoilage of the baked goods.

### *3.3.10.3 The use of Composite Flours in Bread Making*

There is an increasing need for composite flours because the demand for bread and other baked products based on wheat flour has risen rather sharply in many areas of the world, especially in the developing nations, without a



corresponding increase in wheat production in those areas, (Kim and Ruiter, 1969; Seyam and Kidman, 1975; Olatunji and Akinrele, 1978; Blaise and Okezie, 1980). To reduce expenditure on imported wheat grain, utilization of composite flours that involve local sources of starchy flours and oil seed flours high in protein is a reasonable approach to extend the use of the available wheat.

In compositing the flour, it is necessary to balance out the nutritional requirements as set by FAO standards (Seyam and Kidman, 1975). Too much starch and too little protein is a situation to be avoided (Milner, 1974). Fortification using locally available protein flours from beans, cotton seed, sunflower and others should be encouraged (MacConnell and Bushuk, 1974). Addition of vitamins and minerals (Gage, 1978) and limiting amino acids should be considered to give consumers a more nutritionally balanced product. But in order for high protein bread to have a significant contribution in diets, it must be visually and organoleptically acceptable, otherwise ~~few will~~ buy or consume it (Fleming and Sosulski, 1977).

Research in composite flour technology has concentrated mainly on the influences of partial substitution of wheat flour with pure starch, plant protein concentrates, or flours high in starch or plant protein content on dough rheological properties and bread quality. Composites with soya bean flour have been investigated by

Pomeranz *et al.* (1969), Tsen and Tang (1971), and Tsen and Hoover, 1973. Composites with chick pea flour have been studied by Shehata and Fryer (1970), while Hussein *et al.* (1974) investigated composites of wheat flour with broad bean flour. Jeffers *et al.* (1978) studied composite flours of field pea and wheat flours. Blends of fababean and wheat flour have been studied by Lowerenz *et al.* (1979), and navy bean and wheat flour blends by D'Appolonia (1978). Fleming and Sosulski (1977) studies involved the use of composite flours of defatted soya flour, dehulled and defatted sunflower flour, fababean and field pea protein concentrates. They reported that acceptable bread could be made using 12% sunflower flour or 15% soy, fababean and field pea concentrates. However, they found it necessary to include in their formulations 2% vital gluten and 1.0-1.5% dough conditioner to restore the quality of the composite bread.

Cassava and yam have been considered potentially useful in composite flours (Kim and Ruiter, 1969; Pringle *et al.* 1969; Dendy *et al.* 1970). Rasper *et al.* (1974) found cassava starch superior to yam in composite flours for baking. They attributed this to the closeness in pasting temperature of cassava and wheat starches. The performance of cassava flour was, however, found inferior to yam flour in composites with wheat flour for baking which led them to conclude that other components in the non-wheat flours had functional properties of significant influence in baking.

qualities of the flours.

D'Appolonia (1977) found that the baking quality of tuber starches was a function of their physico-chemical properties. Hahn and Rasper (1974) reported that nonstarch water soluble polysaccharides from tuber flours increased bread volume, while the insoluble fractions had a deleterious effect. The presence of fiber in cassava flour was reported as responsible for poor quality of bread with cassava flour composites as compared to composites with pure cassava starch (Hudson and Ogunsua, 1976).

Ciaccio and D'Appolonia (1977) showed that, although yam and cassava flours had only 7.8 and 1.2% protein compared to 11-15% in wheat, they were richer in some amino acids, especially lysine, histidine, arginine, aspartic acid, threonine, and alanine. Yam flour alone was better than wheat flour in its contents of serine, glycine, valine, isoleucine, leucine and phenylalanine. But wheat flour was found to contain much higher levels of glutamic acid, proline, cystine and tyrosine. The same investigators reported in 1978 that acceptable bread could be made from blends of 15% cassava starch or yam flour. Good bread could be obtained from 10% cassava flour in the blend, but dough handling problems became limiting. Good French-type bread was made from blends containing 10% yam flour, while blends with 5 and 10% cassava starch produced good white pan bread. The internal properties of bread containing cassava starch were improved by addition of 0.5% sodium

stearoyl-2-lactylate. They found that blends with cassava starch were more amenable to continuous processing methods than blends with yam. They observed that cassava starch always produced better bread than cassava flour irrespective of the processing method.

Ciaccio and D'Appolonia (1977) found that cassava and arrowroot starches gelatinized during baking to greater extents than wheat or yam starches. They concluded that pasting temperature is an important factor for tuber starches when used in baking, which corroborated findings by Anker and Geddes (1944) and Seyam and Kidman (1975).

Olatunji and Akinrele (1978) studied yam, cocoyams (taro and tannia), cassava and breadfruit flours in a composite bread making with wheat flour. They concluded that 10% substitution level of wheat flour by any of the other flours gave bread acceptably close in quality to pure wheat bread.

Addition of starch or non-wheat flours to wheat depresses loaf volume, weakens the dough and results in harsh crumb texture. Deleterious effects in the crumb can be corrected by proper handling of the dough and use of additives such as gluten, dough conditioners of high HLB, and emulsifiers that react with starch and retard retrogradation. Rheological changes accompanying partial substitution of wheat flour with non-wheat flours, starch or plant protein concentrates are discussed below.

### 3.3.11 Dough Rheological Properties

The rheological properties of a dough refer to its physico-mechanical characteristics. As a whole, a dough is a composite system consisting of distinct phases that differ in their rheological properties (Bloksma, 1971).

The swollen gluten continuous phase is, however, considered responsible for nearly all the physico-mechanical properties of a dough during its mixing and development. On the other hand, starch accounts for about 60% of the volume fraction of a dough, hence Hanssen *et al.* (1952) and Sandstedt (1954) have stated that, due to the high concentration of starch in the dough, rheological properties are more likely influenced by the interaction between starch and gluten, but not gluten alone. In fact the presence of starch makes the dough system more rigid.

Dough rheological properties are important in the baking industry as they significantly influence the quality of bread and other baked products (Bloksma, 1971). As dough is mixed, air is trapped in its protein continuous phase (Baker and Mize, 1946). The dough becomes coherent and begins to withdraw from the mixing equipment as it develops. Development is complete when the dough is fully elastic and is smooth and dry to the touch (Bushuk *et al.* 1968). Further mixing beyond this stage results in dough breakdown, which is shown by gradual loss in elasticity accompanied by an increase in dough extensibility, stickiness and fluidity.

Doughs of flours from different sources or of different blends respond differently to mixing. Their mixing characteristic are affected by additives such as salt, oxidising and reducing agents (Bushuk and Hlynka, 1961), monoglycerides and dough conditioners (Pyler 1979).

In conventional dough mixing, additional development of the protein structure is afforded by fermentation, punching and molding. Gluten lamellae are formed between gas-cells by the stretching and folding operations. The starch granules are entrapped in the gluten lamellae around the gas-cells (Burhans and Clapp, 1942). These operations are less important in mechanical dough development where the gluten is already highly developed by mechanical action at the end of the mixing operation.

Dough rheological properties can be followed and measured by the Brabender farinograph (Locken *et al.* 1960), the Swanson and Working mixograph, and other mixer-recorders (Swanson and Working, 1933; Lamour and Working, 1939; Johnson *et al.* 1946; Voisey *et al.* 1963, 1966; Shuey and Gilles, 1966).

Dough extensibility, strength and resistance to extension can be measured by the Brabender extensigraph (Halton, 1949); the Chopin extensigraph [alveograph] (Chopin, 1957; Bennett *et al.* 1956; Maes and Pirotte, 1956); or the Halton extensigraph [research extensometer] (Halton, 1949).

The research extensometer is similar to the Brabender extensigraph. One advantage the Brabender extensigraph has over the Chopin extensigraph is that it can show the effects of added dough improvers, which the Chopin extensigraph and the Brandender farinograph can hardly do.

The rheograph graphically records the rheological changes occurring in dough as it is subjected to mechanical development. Doughs of 700 g are mixed until the fatigue point is reached. The time required to reach the fatigue point is characteristic of a flour and is influenced by such factors as gluten content and strength, milling, and dough ingredients (Pyler 1979). The Patterson mixatron can be used to electronically measure the instantaneous consistency of the dough during mixing and the mixing time (Selman, 1949; Pyler, 1979).

The Brabender farinograph is the most commonly used instrument in the determination of the rheological properties of dough. It involves rapid addition of water to and mixing of flour of 14% moisture basis at a constant temperature and speed with a pair of z-shaped mixers in the mixing chamber of the instrument. The consistency of the dough in Brabender units (BU) is recorded against time.

The curve (farinogram) just straddling the 500 BU line represents dough with maximum consistency. The water absorbed to effect this consistency is read off as percent water absorption, which is characteristic of a particular flour. Other rheological properties provided by the

farinogram are dough arrival and development times, dough stability, mixing tolerance, and degree of breakdown after 20 min. of mixing (Pyler, 1979).

The Brabender extensigraph is used in measuring dough extensibility, resistance to extension and strength (Bloksma, 1971; Pyler, 1979). A sample of 150 g of dough prepared in the Brabender farinograph is scaled-off, molded, and fermented for 45 min. The dough is clamped and a hook stretched downwards through it until it snaps. The force exerted is transmitted through an arm to the stylus and is recorded on a chart. The dough is reshaped and the process repeated twice after 45 and 90 min. fermentation time to simulate the fermentation period and dough punching.

The vertical axis of the chart represents force in Brabender units, while the horizontal axis represents the extensibility in mm. The height of the curve at 50 mm from the start of stretching represents the resistance to extension.

Maximum resistance is taken at the peak of the curve. The area under the curve represents both the total force used in stretching the dough and the strength of the dough. Good doughs show good resistance to extension. They also have good extensibility (Harris, 1943; Pyler, 1979). For maximum gas retention of a particular dough, some extensibility/resistance ratio exists which is represented by a particular extensigram. Oxidation decreases extensibility, hence increases the ratio (Bloksma, 1971).



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Rheological studies on composite flours have shown that water absorption, mixing tolerance index in BU, and resistance to extension of a dough increase as the level of substitution of the wheat flour by the non-wheat flour, starch or protein concentrate increases (Seyam and Kidman, 1975; Olatunji and Akinrele, 1978; Sathe *et al.* 1981).

Ciacco and D'Appolonia (1977), however, found that, whereas substitution of wheat flour with arrowroot and cassava starches raised the mixing tolerance index, substitution with yam reduced it. Lorenz (1978), using unshelled sunflower meal flour, observed a drop in water absorption in the blends with wheat flour, yet Flemming and Sosulski (1977) observed an increase in water absorption by using dehulled and defatted sunflower flour.

Partial substitution of wheat flour has been reported to reduce dough development times and stability (Seyam and Kidman, 1975; Ciacco and D'Appolonia, 1977, 1978; Olatunji and Akinrele, 1978; Lorenz, 1978; Lorenz *et al.* 1979; D'Appolonia and MacArthur, 1979; Sathe *et al.* 1981). Lorenz *et al.* (1979), however, noted that substitution with fababean flour to a level beyond 5% resulted in increased arrival times. A similar observation was made with substitution with fababean protein concentrate.

Ciacco and D'Appolonia (1977) observed that blending wheat flour with yam starch raised peak time and stability of the dough. Arrowroot and cassava starches had the opposite effect. Ciacco and D'Appolonia (1978) also observed

that arrival and dough development times rose if cassava flour was used for substitution, but not in the case of the starch.

Using yam flour for the substitution at 10% and above reduced arrival times, while dough development time did not start to fall until 15% or more substitution level. Stability of the dough varied with level of substitution. It rose above the control at 5%, was equal to the control at 15%, but dropped at 20% substitution levels with yam flour.

According to the same investigators, addition of 0.5% sodium stearyl-2-lactylate decreased the water absorption at all levels, including the control. Arrival and dough development times increased in the control, but dropped in the case of cassava flour and starch. For yam flour, a decrease in the same properties was observed at and above a 15% level of substitution.

### 3.4 Dough and Crumb Hydration Capacities

Hydration capacity of a dough or bread crumb is defined as the water uptake per gram of dry matter (Yasanuga *et al.* 1968; Dennett and Sterling, 1979). The difference between dough and crumb hydration capacities represents hydration due to gelatinization (Dennett and Sterling, 1979).

Yasanuga *et al.* (1968) found that the hydration capacity of bread crumb is influenced by several factors. Dough moisture content, time, and temperature of baking were found to have direct influence on the degree of

gelatinization and, consequently, the crumb hydration capacity. The staling of bread decreased the crumb hydration capacity. The presence of monoglycerides and shortening reduced the rate of decrease in the hydration capacity of the crumb during storage time. Physical damage to starch granules increased crumb hydration capacity. A similar observation was made when malt was added to the baking formula.

Dennett and Sterling (1979) examined the hydration capacities of dough and crumbs made from composites of 85% starch and 15% vital gluten in a formula containing 4.0% sugar, 1.5% salt, and 3.0% yeast. They found that dough from amylo maize, potato, wheat, maize, tapioca, rice, and amioca starch/gluten composites had hydration capacities corresponding to 1.2, 1.1, 0.9, 0.8, 0.8, 0.8, and 1.1 g water/g dry matter. Similarly, the respective bread crumbs had hydration capacities of 2.2, 3.3, 2.9, 3.1, 5.4, 4.8, and 4.3 g water/g dry matter.

Further studies indicated that there was a statistically significant negative correlation between amylose content and crumb hydration capacity. A significant positive correlation was found between soluble amylose content and crumb hydration capacity.

### 3.5 Dough and Crumb Microstructure

The structure of the dough developed during mixing, fermentation, punching and proofing is maintained in the crumb after baking.

The dough is not homogeneous in its microstructure. Unleavened dough consists of protein, starch, and gas-cells, while leavened dough has yeast-cells in addition.

The development of the dough depends on optimal hydration, optimal mixing and stretching of gluten, and the presence or absence of oxidising and reducing agents (Evans *et al.* 1981) Hydrated gluten forms a continuous phase consisting of a network of thin protein films between gas-cells. The starch granules are embedded in the gluten films and line the gas-cell walls (Burhans and Clapp, 1942; Baker and Mize 1946; Sandstedt *et al.* 1954; Hanssen, 1957).

The protein network of developed gluten is interconnected with hydrogen and occasional disulfide bonds which confer to it viscoelastic properties. The resilience of the structure as a whole depends on the number and strength of the bonds. Electrostatic bonds, Van der Waalls forces, and protein chain entanglements are also thought to contribute to the strength of the whole system (Bloksma, 1971). If the gluten is overmixed, the protein films rupture and become filamented (Grosskreutz, 1961).

Flour lipids and added shortenings do not occur as distinct phases within the dough, but as films on the starch granule surfaces (Barhans and Clapp, 1942). Pomeranz, (1973)

indicated that lipids interact mainly with gluten in the dough and mainly with starch during baking as the crumb is forming thus accounting for the freshness of bread. (

Studies by Grosskreutz (1961) of hydrated gluten structure led him to postulate that developed gluten consist of 2-5% lipoprotein and that the proteins in the hydrated gluten consists of folded polypeptide chains in the  $\alpha$ -helix conformation arranged in plateletes of about 70 Å thickness. These orientate themselves in bimolecular leaflets bound to the outer surfaces of bimolecular phospholipid films through salt linkages between the acidic groups in the phospholipids and basic sites on the proteins.

Horseney *et al.* (1970) reached the conclusion that gliadin and glutenin, the main components of gluten, are bound to each other through glycolipid membranes, with the free polar lipids connected to the gliadin through hydrophilic bonds.

Wehrli and Pomeranz (1970) supported the conclusion of Horseney *et al.* (1970) by reporting that infrared spectroscopy had demonstrated the existence of hydrogen bonds between glycolipids and gelatinized starch or glycolipids and gluten components. Similarly, Van der Waal's forces were reported between glycolipids and gluten components. Nuclear magnetic resonance spectra were reported by the same investigators to have indicated hydrophobic bonding between glutenin and the glycolipids. A conclusion was made that binding of polar lipids to both gliadin and

glutenin played a role in perfecting the ability of gluten to retain gas in the dough.

Flemming and Sosulski (1979), in their studies on the effect of concentrated plant proteins on bread crumb microstructure, observed that foreign proteins disrupted the well-defined protein-starch complex as seen in the pure wheat flour bread. The supplemented bread crumbs had thick cell walls with small pores scattered in them. These pores could have accounted for loss of gas from the supplemented breads, which had reduced loaf volumes, compact or coarse crumb grains and firm textures.

### 3.6 Bread Staling

Bread staling refers to all the physico-chemical changes that occur in bread after baking. It has been the subject of many reviews (Herz, 1965; Waldt, 1968; Elton, 1969; Zobel, 1973; Willhoft, 1971, 1973; Maga, 1975; Kim and D'Appolonia, 1977; Knightlyly, 1977; D'Appolonia and Morad, 1981; Roewe *et al.* 1982).

Changes in bread caused by staling are undesirable. The bread crumb becomes firm during storage. The fresh bread crust, which is normally dry, crispy, and brittle, becomes soft and leathery. The bread loses its fresh pleasant aroma and develops a faintly bitter taste, especially in the crust (Pyler, 1979).

Staling of bread was first associated with starch retrogradation by Lindet (1902). Katz (1928) reported that bread with sufficient moisture staled as a function of temperature. He observed that bread remained fresh at 60°C or higher temperatures, as was observed earlier by Boussingault (1852) Bechtel *et al.* (1953) and von Bibra (1961), who observed that at least 30% moisture in the bread was necessary for this to occur. The bread became progressively more stale with decrease in temperature to the freezing temperature of bread (about 6.7°C) (Pence *et al.* 1956). Bread stored at -10°C or lower remained fresh. X-ray diffraction analysis showed fresh bread to have the 'V' pattern typical of amorphous starch, while stale bread had the 'B' pattern typical of crystalline starch. These observations were confirmed by other researchers (Prentice *et al.* 1954; Bechtel, 1959; Zobel and Senti, 1959).

Schoch and French (1945) established that staling of bread was due to gradual and spontaneous aggregation of the free linear chains of amylopectin. This formed crystalline structures throughout the crumb, but they could be dissociated by heating at 50-60°C, as opposed to higher temperatures of about 125°C required for breaking similar, but stronger association involving amylose. In a follow up, Schoch (1965) explained that during baking amylose diffuses out of the starch granules into the intergranular areas between starch granules within the crumb. amylose chains associate so fast that they are already retrograded

by the time the bread is cooled. Further retrogradation would therefore be due to amylopectin within the starch granules. Pomeranz (1980) confirmed Schoch's work by reporting that staling was due to both amylose and amylopectin in the first day after baking, thereafter being influenced only by amylopectin. McIver *et al.* (1968), using differential scanning calorimetry, also confirmed that amylopectin was more responsible for bread staling than amylose.

Cornford *et al.* (1964) used the Avrami equation to study and relate bread crumb firming to crumb elastic modulus, time, and various temperatures above the freezing point of bread. It was observed that the extent of crystallization could be measured by the increase 'E' in the crumb elastic modulus relative to its limiting or the final value 'E<sub>1</sub>', during storage time. For a linear relationship, the uncrystallized fraction  $\theta$  after a time of storage 't' could be quantified by  $\theta = (E_1 - E) / (E_1 - E_0)$  where 'E<sub>0</sub>' is the initial modulus. This function was found to follow the equation  $\theta = \exp(-kt^n)$  in which 'k' is a constant characteristic of crystal growth, while 'n' is an integer (1-4) designating the mode of nucleation. They showed that above the freezing temperature of bread, the relative rate of modulus increase become greater with decreasing temperatures. Concomitant decreases in the time constant (time required for a given fraction of the crumb to become stale) were also observed. It was concluded that firming in



bread crumb as it stales is a physical process involving ordered arrangement of atoms or molecules as occurs in crystallization. The process was attributed to crystallization of starch as it retrogrades. Similar observations were made in later studies by Cornford *et al.* (1964); Axford *et al.* (1968); McIver *et al.* (1968); Colwell *et al.* (1969), and Kim and D'Appolonia (1977). Their work gives firm support to observations reported above after Schoch and French (1945) and Schoch (1965).

According to Willhoft (1971, 1973) denaturation of gluten during baking causes structural changes, reducing the ability of gluten to bind water. There is a transfer of water from the denatured gluten to the gelatinized starch during staling. The softening of starch resulting from such water transfer is compensated for by firming due to retrogradation. Taylor *et al.* (1959), however, reported that the starch gel lost from 58-51% of its moisture-sorption capacity over seven days storage, while gluten maintained its capacity. Cornford *et al.* (1964) also found a negative temperature coefficient for bread staling. They concluded that diffusion of moisture from gluten to starch during staling was not a primary occurrence. The softening of the bread crust during staling seems to indicate a decisive transfer of moisture from the crumb to the crust.

Several factors affect the staling of bread. Factors that reduce loaf specific volume enhance staling (Elton *et al.* 1969; Axford *et al.* 1968). Flours high in good

quality protein produce bread with high specific volume. Such bread had a lower tendency to stale as compared to bread made from flour low in quantity and quality of protein. Chorleywood Process bread was found to stale less than conventional process bread. Properly fermented bread was found to stale less than over or under fermented bread. Optimum hydration of the dough slowed down the rate of staling in bread (Swortfiguer, 1971).

Water soluble and water insoluble pentosans slow the rate of retrogradation by affecting the amylopectin and amylose fractions of starch, hence reducing the total amount of starch available for retrogradation (Pomeranz, 1980; D'Appolonia and Morad, 1981).

The presence of non polar lipids has been found to reduce crumb firming only slightly (Pomeranz and Chung, 1979,) while free polar lipids were observed to reduce it significantly (Pomeranz, 1969). Surfactants combine with starch and reduce its tendency to stale. Monoglycerides reduce staling by penetrating the starch granule and immobilising in it the the unleached amylose fraction (Schoch, 1965). Several investigators (Hutchinson, 1936; Platt and Powers, 1940; Carlin, 1947) have found that the presence of shortening in bread reduces staling.

Freezing of bread is the most effective way of prolonging bread storage for any length of time. Development of bad aroma is the only limiting factor (Bailey, 1932; Cathcard, 1939; Cathcart, 1941; Pence *et al.* 1956). Staling

of bread has been investigated using changes in crumb compressibility (Bailey, 1930; Stramb and Hirsch, 1935; Platt and Powers, 1940; Noznick and Geddes, 1943; Pence *et al.* 1955; Waldt, 1968; Marston and Short, 1969; Maleki *et al.* 1981; Roewe and Kulp; 1982). Kim and D'Appolonia (1977) followed retrogradation by measuring the solubility of starch in the crumb to estimate the degree of staling. Crumb x-ray diffraction analysis has also been employed in the analysis of retrogradation (Dragsdorf and Marston, 1979). Other investigators have used starch susceptibility to amylase attack. Bechtel and Meisner (1952) used changes in crumbliness of the crumb to measure its level of retrogradation.

### 3.7 Bread Quality Evaluation

The reasons for bread quality evaluation are to access consumer acceptability, routine quality checks on the suitability of raw materials used in the formulations, and regulation of the production process to achieve specified quality standards in the product.

The bread is examined for external and internal properties. The external properties of interest are loaf volume, crust color, symmetry of form, character of the crust, and break and shred. The internal characteristics are crumb grain, crumb color, aroma, taste, mastication (chewability), and texture (Matz, 1960; Pyler, 1979).

The characteristics of the bread can be analysed by any of the sensory tests (difference analysis, ranking difference, and preference evaluation) coupled with statistical analysis where necessary (Fleming and Sosulski, 1977; Sosulski and Fleming, 1979; Blaise and Okeizie, 1980).

In the triangle difference analysis test, panelists are asked to identify the odd sample from a set of three, two of which are identical. The degree of difference is then judged as extreme, much, moderate or slight.

In ranking difference test, the characteristic of interest, for example, crumb color, crust color, crumb grain or crumb texture, is examined in different loaves of bread. The loaves are then ranked according to the panelists' preference based on the degree of the differences observed.

As regards the preference test, bread characteristics are evaluated according to a hedonic scale of numerals. The level of preference for a characteristic is indicated by numerals selected from the scale of, for example, 1-10, where 10 is the perfect score and 1 represents a poor score or unacceptable quality (Ciaccio and D'Appolonia, 1977, 1978; D'Appolonia and MacArthur, 1979). Investigators have selected scales they find suitable. Olatunji and Akinrele (1978) used a scale of 1-100, with 81-100 scores representing acceptable and 56 or below being unacceptable. Sosulski and Fleming (1979) used a 5-point scale to evaluate flavor, texture and overall acceptability.

Numerical scores from the various panelists are then subjected to statistical analysis of variance and Duncan's multiple range test (Sokal and Rohlf, 1969; Lamond, 1970).

According to the American Institute of Baking the external properties of a loaf account for 30% of the total score, of which volume is allocated a maximum score of 10 and color of the crust a score of 8. The other external properties are each assigned a maximum score of 3 for the best quality. Of the 70% scores allocated to the internal properties, taste and texture account for 15 scores each for the best quality. The rest of the internal properties are assessed individually on a maximum score of 10 for the highest quality. Bread loaves lose scores if specified faults are detected in any of the properties.

## 4. EXPERIMENTAL

### 4.1 Materials

#### *Arrowroot Starch*

Native arrowroot (*Maranta arundinacea*) starch was isolated from the flour of a Kenyan cultivar.

#### *Cassava Starch*

Native cassava (*Manihot utilissima*) starch was isolated from flour of a Kenyan cultivar and tubers of a Fijian cultivar. Commercially available (A.E. Staley Manufacturing Co., Decatur, Illinois) tapioca starch was also used.

#### *Sweet Potato Starch*

Native sweet potato (*Ipomoea batatas*) starches were isolated from the tubers of the cultivars Centennial, Porto Rico and Georgia Red.

#### *Taro Starch*

Native taro (*Colocasia esculenta*) starch was extracted from tubers of a Jamaican cultivar.

#### *Yam Starch*

Native yam (*Dioscorea cayenensis*) starch was extracted from tubers of a Jamaican cultivar.

#### *Wheat Flour and Starch*

Wheat flours were obtained by milling hard red spring (cv. Neepawa) and soft white spring (cv. Fielder) Canadian wheats. Wheat starch was then isolated from the flours.

### *Vital Wheat Gluten*

Vital wheat gluten, Whetpro-80 (Industrial Grain Products Ltd., Montreal), consisting of 80.0% protein, 11.5% carbohydrates, 6.5% moisture, 1.0% fat and 1.0% ash, was used in composite flours with all starches.

### *Yeast*

Active dry yeast (Standard Brands Canada Ltd.) was locally obtained.

### *Sugar and Salt.*

Commercial food grade sugar and salt were used.

### *Shortening*

Crisco (Procter and Gamble Inc., Toronto), a hydrogenated vegetable oil shortening consisting of mono- and diglycerides, was used.

### *Monoglycerides*

Types C<sub>16</sub> and C<sub>18</sub> distilled monoglycerides, at least 90%, prepared from hydrogenated palm oil with palmitic acid enrichment, and edible saturated cottonseed oil were obtained from Vauxhall Foods Ltd., Alberta and Eastman Kodak Co., NY.

### *Photographic Paper and Dry Mounting Tissue*

Photographic papers and dry mounting tissue were all Kodak brand.

### *Chemicals*

Kodak type Dektol developer and fixer were used

Tri (dimethyl amino methyl) phenol-DMP-30 and Afaldite (502) resin were obtained from Ladd Research Industries, Inc. (Burlington, CT).

Dodecenyl succinic anhydride (DDSA) was obtained from Ernest Fullum, Inc. (Schenectady, NY).

Propylene oxide was obtained from Eastman Kodak Co. (Rochester, NY).

Other chemicals used were reagent grade obtained from Fisher Scientific Co.

#### 4.2 Equipment

Precision balances made by Mettler, Zurich, CH, were used in taking accurate weights of chemicals, samples and materials.

Ovens: Vacuum oven (National Appliances Co., Portland, OR) was used in all moisture content determinations.

Iso-temp draft oven (Fisher) was used for drying purposes.

White Westinghouse domestic oven was used for baking bread at 210°C. Still air oven was used for storing bread at 24°C.

Waring blender Model 702 BAW (Waring Products Corp., Winsted, CT) with variable autotransformer (Superior Electric Co., CT) was used in the isolation of the starches.

Centrifuges: International centrifuge size 2 (International Co., Boston, MA) and Beckman Model J21B with rotors JA-14 and JA-20 (Beckman Instrument Inc., Palo Alto, CA) were used in starch and starch-monoglyceride clathrate isolation steps.



Cyclone sample mill (UD Corporation, Boulder, CO) was used in preparation of whole wheat flour.

Buehler laboratory flour mill - type MLU-202 (Buehler Brothers Ltd. Engineering Works, Uzwil, Switzerland) was used in milling tempered wheat grain to white straight grade flour.

Camfro type RZR1-64 stirrer (Camfro Ltd., Warton, Ontario) and Lo-temptrol water bath (Precision Scientific Co., Chicago., IL) were used in the determination of starch swelling power and solubility.

Control temperature water bath with Thermix 1441 stirrer (Braun Melsungen Ag, W. Germany) was used in providing constant temperature when required.

Potentiometric titrimeter (Fisher Scientific Co) was used in the determination of amylose content of the starches.

Technicon autoanalyser II, (Technicon Industrial Systems, Tarrytown, NY) was used in the determination of the phosphorus content of the starches.

A Haake rotovisco model RV3 with NV sensor system (Haake, Karlsruhe, Germany) was used in all starch viscosity determinations.

Spectrophotometers: A Beckman DU-8, UV-visible spectrophotometer (Beckman Instruments Inc., Irvine, CA) was used in the determination of amylose complexing indices of monoglycerides. A Perkin-Elmer 297-1R spectrometer (Perkin Elmer Ltd, Beaconsfield, Buckinghamshire, UK.) was used in

the examination of the structure of the  $\alpha$ - and  $\beta$ -monoglycerides. A Perkin-Elmer 505 atomic absorption spectrophotometer (Perkin-Elmer Ltd, Toronto,) was used in determining the mineral contents except phosphorus of the starches.

Controlled environment incubator shaker (New Brunswick Scientific Inc., Edison, NJ) was used in maintaining constant temperature and shaking during determination of the starch monoglyceride interactions.

Microscopes: Light microscope (Ernst Leitz Wetzlar, W. Germany) with a calibrated dark field was used in the determination of the starch granule size distribution. Scanning electron microscope (SEM), Stereoscan 150, Cambridge Scientific Instruments Ltd. (Cambridge, England) was used in studying the morphology of the starches, dough and crumb.

A transmission electron microscope ultramicrotome (Reichert "OM U2", Reichert Optische Werke AG, Vienna, Austria) was used in sectioning samples for the transmission electron microscope, Philips EM type PW 6000 (Philips Scientific Instruments, Netherlands)

Differential scanning calorimeter, Du Pont model 900 with the 910 cell base (Du Pont Co., Wilmington, DE), was used in the determination of the gelatinization properties of all the starches and the retrogradation of concentrated aged starch gels.

Farinograph type FA/R-Z (C.W. Brabender Instruments Inc., Hackensack, NJ) was used in the determination of percent water absorption of flour and the rheological properties of the doughs.

Hobart mixer type KM32 CDN (Braun AG Frankfurt/M, W.t Germany) was used in mixing doughs for breadmaking.

Temperature-humidity chamber model 417530 (Holpack Canada Ltd, Waterloo) was used during dough fermentation and pan proofing at 35°C and 80% humidity.

Freeze drier (Virtis Co. Inc., Gardiner, NY) was used in freeze drying samples for X-ray diffraction analysis.

X-ray diffractometer: Philips model PW-1730 X-ray generator, with PW-1710 diffractometer control unit and PM-8110 and PM-8203 one line recorders, was used in examining crystallinity in aged starch gels, starch-monoglyceride clathrates and starch isolated from aged bread crumb.

Textrometer model SL14 Minarik speed control unit (Minarik Electric Co., Los Angeles, CA) with speed reducer Type NSH-12RG (Bodine Electric Co. Chicago, IL), Honeywell Electronic 19 chart recorder and type 93 strain gage transducer input module, model 3001D (Daytronic), was used in crumb compressibility and crumb penetration resistance determinations.

### 4.3 Methods

#### 4.3.1 Isolation of Starch from Fresh Tubers

The tubers were washed in tap water and peeled in deionized water containing about 100-150 ppm of  $\text{NaHSO}_3$ . They were then diced and homogenized at medium speed in a Waring blender in deionized water containing as above. The resulting slurry was filtered through 100 mesh polyester filter cloth. The filtrate was collected, while the residue was recycled through the waring blender until all starch had been extracted from it. The starch in the filtrate was allowed to sediment for 1 to 2 hr, after which the supernatant was removed by syphoning.

The starch was cleaned by being resuspended in a little water and centrifuged at 3000 rpm for 15 min in 250 ml centrifuge bottles. The supernatant and the upper brown layer of impurities were eliminated. Any debris at the bottom of the starch layer was also eliminated. The process was repeated (2-3 times) until a clean sample of starch was obtained.

The starch was finally washed with 95% ethanol, followed by another washing with acetone in a Buchner funnel lined with Whatman No. 1 filter paper and connected to an aspirator. The starch was dried at  $40^\circ\text{C}$  for 3 hr in a forced draft Iso-temp oven. Its moisture content was determined by drying weighed samples in a vacuum oven at  $60^\circ\text{C}$  overnight, followed by cooling for 1 hr over  $\text{P}_2\text{O}_5$  in a dessicator,

reweighing and expressing the weight loss as percent moisture content.

For moisture determination, all weights were taken on a precision balance.

#### 4.3.2 Isolation of Starch from Arrowroot and Cassava Flours

A thin slurry of flour was made in water and screened through 100 mesh polyester cloth. The residue was resuspended in water a second time and rescreened through the polyester cloth. This was repeated until practically all starch had been washed out. Starch recovery from the filtrate and subsequent cleaning were performed as given above. Moisture content was determined.

#### 4.3.3 Isolation of Starch from Wheat Kernels

##### 4.3.3.1 Wheat Milling

Wheat kernels were tempered as given in AACC method 26-95 on experimental milling. The grain was then milled using the Buehler laboratory mill. Whole wheat flour was obtained using the cyclone sample mill.

#### 4.3.4 Isolation of Starch from Wheat Flour

Dough from wheat flour was made with cold tap water and given a rest of about 20 min. Starch was then washed out of the dough with water. The gluten was discarded. The starch in the water suspension was recovered and cleaned as given in section 1.3.1. The moisture content of the starch was

then determined.

#### 4.3.5 Determination of Starch Grain Size Distribution

The field of a Leitz Wetzlar light microscope was calibrated with a scale at selected low or high magnifications. An eye piece of magnification 10x carrying a blank scale was mounted and matched with the microscope field calibration.

The value in microns of each division of the blank eye piece scale was determined. The microscope field calibrating scale (mounted on a slide) was removed and replaced with a slide carrying starch a sample. The sizes of 100 random starch grains were measured. Percent size distribution of the starch grains was then determined.

#### 4.3.6 Determination of the Starch Grain Morphology by Scanning Electron Microscopy (SEM)

Native starch was defatted in the Soxhlet apparatus overnight with 95% ethanol, dried in an Iso-temp draft oven for 2 hr at 40°C, and then mounted on aluminium stubs with conductive silver paste. The starch grains were sputter coated twice with 20 nm of gold at 900 V and 40 mA in a vacuum. The morphological properties of the starches were then viewed in a Cambridge stereoscan 150 differential scanning electron microscope at an electrical acceleration potential of 15 kv. Photomicrographs of the starch grains were made.

#### 4.3.7 Determination of the Starch Amylose Content

Amylose content of each starch was determined by potentiometric titration of an aliquot of solubilized starch solution with standard iodine solution, as described by Schoch (1964).

#### 4.3.8 Determination of the Starch Mineral Content

Technicon Autoanalyser II Industrial Method No. 369-75A/A (November, 1975) was used.

To 1.0 g of starch in the Kjeldhal digestion test tube, were added 15.0 g  $K_2SO_4$  and 0.50 g red  $HgO$  (catalyst) and the tube was shaken gently to mix well. Concentrated  $H_2SO_4$  (20 ml) was added. The digestion tubes were again shaken gently to disperse the contents. Hydrogen peroxide (to break down organic matter and minimise foaming) was added in small amounts of about 2.0 ml to avoid excessive reaction, until the contents in the digestion tubes became light in color. Three plain henger boiling chips were added to each tube. Digestion was first done at 250-260°C for 2-3 hr, followed by final digestion at 380°C for 6-8 hr until all digests became clear. The tubes were cooled and the contents of each diluted to 75 ml.

From the solutions,  $Na^+$  and  $K^+$  were determined by flame emission,  $Ca^{++}$  and  $Mg^{++}$  by atomic absorption, and P by Technicon autoanalyser. The results were converted to percent on dry basis of starch. Atomic absorption conditions for  $Ca^{++}$  and  $Mg^{++}$  determination were as follows:

| Condition                     | Calcium   | Magnesium                  |
|-------------------------------|---|----------------------------|
| Wavelength                    | 422.7 nm  | 285.2 nm                   |
| Slit                          | 0.7 nm  | 0.7 nm                     |
| Sensitivity                   | 0.092 mg/L  | 0.0078 mg/L                |
| Flame                         | Air-Acetylene<br>(oxidizing, lean<br>and blue)                        | same as for<br>calcium     |
| Stock<br>Standard<br>solution | 500 mg/L in<br>deionized<br>water acidified<br>with 10 ml of<br>HCl/L | 1000 mg/L in<br>dilute HCl |

Flame emission conditions for the determination of sodium and potassium .

| Condition                  | Na <sup>+</sup>                 | K <sup>+</sup>                  |
|----------------------------|---------------------------------|---------------------------------|
| Wavelength                 | 589.0 nm                        | 776.5 nm                        |
| Slit                       | 0.2 nm                          | 0.4 nm                          |
| Flame                      | Air-acetylene                   | Air-acetylene                   |
| Stock standard<br>solution | 1000 mg/L in<br>deionized water | 1000 mg/L in<br>deionized water |

Phosphorus determination by Technicon autoanalyser technique is presented in Technicon Autoanalyser Industrial methods No. 329-74W/B and No. 369-75A/A. The determination is based on the formation of the blue phosphomolybdenum complex from the reaction between orthophosphate, molybdenum ion and antimony ion followed by reduction with ascorbic acid in acidic pH. The complex absorbance was determined at 660 nm.

#### 4.3.9 Determination of Starch Swelling Power and Solubility

The method followed in determining the starch swelling power and solubility is described by Schoch (1964).



#### 4.3.10 Starch Viscosity Determination

Suspensions of starch (1% w/v) in distilled water were gelatinized and held at 85°C for 30 min. The viscosities of the resulting slurries were determined at 25, 30, 35, 40, 50, 60 and 70°C for 5 and 10 min runs of the Haake Rotovisco RV3 and NV sensing system at 1280 rpm. All results were reported in centipoises (cP).

#### 4.3.11 Differential Scanning Calorimetry of Gelatinization Properties of Starch

The gelatinization properties of the starches were determined at water volume fractions of 3.0, 4.0, 5.0, 6.0, 7.0, and 8.0. All water volume fractions were calculated using 1.55 g/cm<sup>3</sup>, as the average density of starch.

The appropriate volume of distilled water was added to a weighed quantity of starch in a small mortar. The starch was gently, but thoroughly dispersed and mixed with the water using a small glass pestle. Quantities of about 6 mg of the resulting slurry or paste were weighed accurately into the differential scanning calorimeter (DSC) pans; and sealed quickly and firmly in a DSC press. The pans were left at room temperature overnight before being heated in a DSC from 10°C to 150°C, over which temperature range the gelatinization properties of the starches were recorded. An empty DSC pan was used as reference in all determinations. DSC sensitivity was 10X (on the cell) and 5 mV/cm on the chart, with heating rate of 5C°/min and time base setting of

1 min/cm. The onset, peak and gelatinization temperatures, as well as the gelatinization temperature ranges in °C, for each starch were determined from the charts. The charts were also used in the calculation of the enthalpies of gelatinization in cal/g of each starch.

#### 4.3.11.1 Calculation of Enthalpies of Fusion Gelatinization

The enthalpies of gelatinization per gram of starch were calculated from the formula given below

$$\Delta H_{\text{mcal/mg}} = A/M(60BE)\Delta q_s$$

Where:

$\Delta H_{\text{mcal/mg}}$  = Enthalpy of gelatinization in mcal/mg of starch

A = Peak area in in<sup>2</sup>

M = Mass of the sample

B = Time base setting on the differential scanning calorimeter (1 min/0.4 in)

E = Cell calibration constant = 1.01.

$\Delta q_s$  = Y-axis setting on the DSC (0.5 mcal.S  
mcal/mg/1000 = cal/g

Applying the settings used on the DSC,  $\Delta H_{\text{cal/g}} = 4.5441 A/M \text{ cal/g starch.}$

#### 4.3.12 Determination of Retrogradation in Gelatinized, Aged, Concentrated Starch Gels

Taking into account the moisture content of the starch, a total of 15 ml of distilled water was added to 10.0 g of starch, and the resulting slurries gelatinized at 95°C for 1

hr. The gelatinized starches were allowed to cool down and form gels, and the water contents were determined. The gels were stored at 24°C and their retrogradation followed by DSC and x-ray diffraction analysis of fresh, 2, 4, 6, and 40 day old gels.

#### 4.3.12.1 Differential Scanning Calorimeter Determination

For DSC analysis, 10 mg of the starch gel were sealed in DSC pans and heated from 10-150°C using an empty sealed DSC pan for reference. DSC sensitivity was 10x (on the DSC cell) and 5 mV/cm on the chart. The heating rate was 5°C/min at a DSC time base setting of 1 min/cm.

#### 4.3.12.2 X-ray Diffraction Analysis

For X-ray analysis, the starch gel samples were first dried at 45°C in a draft oven. The dry samples were then ground separately in a mortar with a pestle to a powder. The powder was densely compressed in the X-ray diffractometer aluminum sample holder and analysed with copper K  $\alpha$ -radiation (1.5418 Å) at a scanning angular velocity of 1°(2 $\theta$ ) from 3° to 35° (2 $\theta$ ) with a time constant of 4 sec. Chart speed was 1 cm/min.

#### 4.3.13 Determination of Starch Complexing with Monoglycerides

Types C<sub>1</sub> and C<sub>2</sub> Monoglycerides in their  $\alpha$ - and  $\beta$ -crystallinity forms were used. All starches were lintnerized and then used in the investigations as

85  
ungelatinized, gelatinized or solubilized starch.

#### 4.3.13.1 Conversion of Monoglycerides from $\beta$ - to $\alpha$ -Crystallinity forms

The monoglycerides were purchased from the commercial supplier in their  $\beta$ -crystallinity form. They were converted to their more reactive  $\alpha$ -crystallinity form by heating for 20 min in a hot water bath at 65°C, a suspension of the monoglyceride in a measured volume of distilled water adjusted with dilute HCl to pH 2.3 for type C<sub>12</sub> and pH 3.5 for type C<sub>18</sub>.

#### 4.3.13.2 Infrared Analyses of Monoglycerides

The structural characteristic of the  $\beta$ - and  $\alpha$ -monoglycerides were checked by infrared analysis.

The  $\beta$ -monoglycerides were first converted to  $\alpha$ -monoglycerides by heating 15 parts of monoglyceride in 85 parts distilled water adjusted for pH at 65°C for 20 min then cooling down to room temperature.

Samples for infrared analysis were then made in Nujol and scanned from 600-2000 cm<sup>-1</sup>.

Samples for x-ray diffraction analysis were compressed in aluminum sample holders and analysed as in 4.3.12.2.

#### 4.3.13.3 Starch Lintnerization

Slurries of 1:1.5 (w/v) native starch in 2.5% (v/v) HCl in water were hydrolysed at 40°C for 72 hrs in a controlled environment incubator-shaker set at 200 rpm. The starch was then washed thoroughly with distilled water to

remove acid in a Buchner funnel lined with Whatman filter paper No. 1 and connected to an aspirator. The clean starch was dried for 3 hr at 40°C in an Iso-temp forced draft oven. Moisture content was then determined.

#### 4.3.13.4 Solubilization of Lintnerized starch

Five ml. of N KOH were added to 1.0 g (db) samples of lintnerized starches in 35 ml test tubes. Each test tube was stirred by a vortex mixer until the starch was thoroughly dispersed in the KOH. Clear solutions of the starches in the KOH hydroxide were obtained after cooling in a refrigerator at 4°C for 30 min. The solutions were then neutralized with an equal volume of N HCl before interaction with monoglycerides.

#### 4.3.13.5 Gelatinization of Lintnerized Starch

Weighed quantities (1.0 g db) of lintnerized starch were placed in 35 ml test tubes. Five ml of distilled water, or distilled water with pH adjusted to 2.3, or distilled water with pH adjusted to 6.5, were added, depending on whether the final gelatinized starch was going to be reacted with  $\beta$ -C<sub>18</sub> and C<sub>18</sub>; or  $\alpha$ -C<sub>18</sub> and C<sub>18</sub>. The test tubes were corked, stirred on a vortex mixer and placed in a hot water bath at 95°C for 30 min to gelatinize the starch, uncorking 2-3 times to release pressure. The test tubes were then removed from the hot water bath and allowed to cool down to room temperature.



#### 4.3.13.6 Starch-Monoglyceride Interaction

Lintnerized starch samples were interacted with the  $\alpha$ - and  $\beta$ -crystallinity forms of  $C_{18}$  and  $C_{12}$  monoglycerides at levels of 0.0, 0.10, 0.20, 0.30, 0.40, 0.50, 0.80, and 1.0% (db) of starch. Three cases were considered for each starch. The first involved interaction of ungelatinized lintnerized starches with the monoglycerides. The second involved gelatinized starch while the third case covered the interaction of solubilized starch with the monoglycerides.

#### 4.3.13.7 Starch and $\alpha$ -Monoglyceride Interaction

##### Determination

Weights of 0.0, 0.10, 0.20, 0.30, 0.40, 0.50, 0.80, and 1.0 mg of either  $\beta$ - $C_{18}$  or  $C_{12}$  monoglycerides were weighed separately into 250 ml conical flasks. Five ml of distilled water, with pH appropriately adjusted were added to each flask. The monoglycerides were allowed to convert to the  $\alpha$ -crystallinity form as given above.

Ungelatinized starch 1 g (db) was weighed into each flask and the volume made up to a total of 20 ml in each case with pH adjusted distilled water. The flasks were corked and kept for 12 hr at 45°C in a controlled environment shaker incubator at 200 rpm. The samples were centrifuged at 12,000 rpm after the interaction period. The supernatant was used for amylose complexing index determination, while the residue was dried at 40°C in an Iso-temp draft oven and used for X-ray diffraction

crystallinity analysis.

Gelatinized or solubilized starches were similarly analysed in a similar manner to the above. After separate gelatinization or solubilization steps, the samples were transferred quantitatively into 250 ml conical flasks, making the total volume 20 ml with distilled water pH, 2.3 for C<sub>18</sub> and pH 6.5 for C<sub>18</sub>. The samples were then allowed to interact followed by centrifugation. The supernatant was collected for amylose complexing index determination and the residue dried for X-ray diffraction analysis.

#### *4.3.13.8 Starch and $\beta$ -Monoglyceride Interaction*

##### *Determination*

1.0 g (db) of ungelatinized starch was added to each of the 250 ml conical flasks containing 0.0, 0.10, 0.20, 0.30, 0.40, 0.50, 0.80 and 1.0 mg of either  $\beta$ -C<sub>18</sub> or  $\beta$ -C<sub>18</sub>. Twenty ml of distilled water were added and the reaction allowed to proceed as described above. After centrifugation, the supernatants and the residues were saved and used for amylose complexing index determination and X-ray diffraction analysis.

When gelatinized or solubilized starches were used, the  $\beta$ -monoglyceride C<sub>18</sub> or C<sub>18</sub> was added directly into the conical flasks containing the starch samples bring the total volume to 20 ml in each case with distilled water. Determination of the amylose complexing indices and X-ray diffraction analysis were performed as given above.

**Determination of the Amylose Percent Complexing Indices of**

## Monoglycerides

The method of Gilbert and Spragg (1964) was followed, but was modified to give "percent complexing index" instead of the "blue value".

The steps followed were: 1.0 ml of a supernatant was diluted 1:1 (v/v) with distilled water in a 50 ml volumetric flask. 1N, 0.5 ml NaOH was added and the mixture heated for 3 min in a boiling water bath. After cooling, 0.5 ml N HCl was added to neutralize the sodium hydroxide. Potassium hydrogen tartrate buffer 0.09 g was added. Distilled water was added to the flask up to about 45 ml of the total volume. Iodine standard solution 0.05 ml containing 2 mg  $I_2$ /ml and 20 mg KI/ml, was added. Distilled water was added up to the 50 ml mark. The flask was corked and contents mixed by inverting the flask a few times. The solution was kept at 20°C for 20 min. The absorbance of the solution was taken at 680 nm in 1 cm cuvettes using the DU-8 Beckman Spectrophotometer with water as the reference. The amylose percent complexing index was calculated as given below.

### (1) Soluble Starch samples

$$\% \text{Complexing Index} = 100 \times (A_{680\text{TA}} - A_{680\text{SS}}) / A_{680\text{TA}}$$

Where:

$A_{680\text{TA}}$  = The total absorbance at 680 nm of the complex between iodine and amylose from 1.0 g (db) of solubilized starch sample.



$A_{880_{SS}}$  = The absorbance at 680 nm of the iodine-amylose complex obtained after interacting the 1.0 g (db) of soluble starch sample with monoglycerides.

*(II) Ungelatinized and gelatinized starch samples*

$$\% \text{Complexing Index} = 100 \times (A_{880_{GS}} - A_{880_{GMS}}) / A_{880_{TA}}$$

Where:

$A_{880_{GS}}$  = The absorbance at 680 nm of the complex between iodine and amylose from 1.0 g (db) ungelatinized or gelatinized starch before interaction with monoglycerides.

$A_{880_{GMS}}$  = The absorbance at 680 nm of the iodine amylose complex after the starch sample (1.0g db) has reacted with monoglycerides.

A plot of complexing index against percent monoglycerides indicated the optimum amount of monoglycerides for complexing amylose in each starch.

#### *4.3.13.9 X-Ray Diffraction Analysis of the Starch-Monoglyceride Clathrates*

The residues obtained after the starch-monoglyceride interactions were ground to powder in a mortar after drying and analysed for crystallinity by X-ray diffraction analysis as previously described.

#### *4.3.14 Determination of Starch Affinity for Gluten in the Dough, Early and Fully Baked Stages of Bread making*

The method of Dahle (1971) to examine the affinity between different starches and gluten was followed. Starch suspensions 0.5% in distilled water, and a gluten extract

containing 102.60 mg protein/ml, determined by the Lowry method (Lowry *et al.*, 1951) on the centrifugates of 40 g gluten suspension in 200 ml of 0.1 N acetic acid, were reacted in a ratio of 2 ml to 2 ml to simulate protein-starch interaction in the dough. In order to simulate early baking starch-gluten interactions, the starch suspension was first gelatinized for 10 min in a boiling of water bath. Fully baked condition was simulated by denaturing the gluten extract and gelatinizing the starch suspension in a boiling water bath for 10 min and cooling before interaction.

Each sample was mixed and shaken for 2 min, followed by centrifugation for 10 min at 3000 rpm. The centrifugates were retained for further analysis. In the case simulating the dough condition, the unreacted protein in the centrifugate was determined by the Lowry *et al.* (1951) method.

$$\% \text{ Affinity} = \frac{100(1 - \text{Residue soluble protein})}{(\text{original soluble protein})}$$

In the other two cases, the supernatants were appropriately diluted and reacted with 0.05 ml of standard iodine solution and the absorbance taken at 625 nm. Percent bound starch as affinity of starch for gluten was calculated from the formula given below.

$$\% \text{ Affinity} = \frac{100(1 - \text{Absorbance of residue soluble starch})}{(\text{Absorbance of original starch sample})}$$

In both calculations, the dilution factors employed in sample preparation for absorbance reading were taken into account.

#### 4.3.15 Flour Water Absorption Determination

The flour water absorption was determined with the Brabender farinograph using 50 g of flour adjusted to 14.0% moisture basis as per AACC method 82-23.

The farinograph mixing chamber was maintained at a constant temperature of 35°C with circulating water from Lo-Temprol 154 constant temperature water bath. The appropriate quantity of the flour was placed in the mixing chamber and mixed at speed 2 for 1 min for the flour temperature to equilibrate with that of the chamber. Composite flours consisted of 85% starch and 15% vital gluten.

The farinograph chart was then started at zero time. At the same time water at 35°C from the farinograph burette was added to the flour. First, 25 ml of water was added continuously into the flour from the burette. The rest of the water was added in small amounts of about 2 ml and later dropwise until the farinogram just straddled the 500 BU line. The total amount of water used was read from the burette and recorded. A final determination was made in which all the required volume of water was added all at once from the burette to the flour. The determination was complete if the farinogram came to rest proportionally on

the 500 BU line. The flour absorption was then read out from the burette as percent or in ml water/100g flour.

Sometimes the farinogram would not come to rest on the 500 BU line in the final determination. In such cases, more water at the rate of 0.5 ml per 20 BU was added, if the dough was too dry, until the farinogram came to the 500 BU line. If too much water had been added, the determination was repeated with 0.5 ml per 20 BU less water.

#### 4.3.16 Determination of the Rheological Properties of the Doughs

The doughs were made from wheat as well as composite (85% starch + 15% vital gluten) flours containing 4%, 1.5% and 3% yeast.

Flour absorption, including the additives, was determined. Yeast 3% was weighed separately in a 50 ml beaker, and activated for 5 min with 10.0 ml of distilled water at 35°C, the water being part of the percent absorption of a particular flour. The flour equivalent of 50 g on 14.0% moisture basis containing 4% sugar and 1.5% salt was put in the farinograph mixing chamber, mixed for 1 min and stopped. The activated yeast suspension was added to the flour in the mixing chamber, the beaker being rinsed once into the chamber with 10 ml of distilled water. The chart was set at zero time and started. The mixing was started at the same time. The water balance (% absorption - 20 ml) for each flour was added all at once from the farinograph

burette.

Due to the presence of additives in the flour, the farinogram peaks were beyond the required 500 BU line level, requiring a correctional addition of 0.5 ml water per 500 BU beyond the 500 level. A fresh farinogram straddling the 50 BU line was run for 20 min after taking into account the correction. Dough rheological properties, arrival and development times, stability, mixing tolerance and breakdown in consistency after 20 min were determined from the farinograms.

#### 4.3.17 Determination of Dough and Crumb Hydration Capacities

The method of Yaşunaga *et al.* (1968), as modified by Dennett and Sterling (1979), was used in determining the hydration capacities of the wheat and composite flour doughs and bread crumbs. Samples, 20 g each of dough or bread crumb were homogenized for 1 min each in 100 ml of water in a Waring blender and transferred into a 250 ml beaker. The blender was rinsed with 100 ml more water which was again transferred into the 250 ml beaker. The slurry was stirred for 1 hr on a magnetic stirrer before being transferred into 250 ml weighed centrifuge bottles. The beaker was rinsed with 50 ml more water, which was also added into the centrifuge bottles, followed by centrifugation at  $3000 \times g$  for 10 min. The supernatant was carefully discarded and the bottles inverted on paper towels to drain for 5 min. The weights of the sediments were recorded. Dry matter in the

doughs and the crumbs was determined by drying 1-2 g at 70°C in a vacuum to constant weight.

Hydration capacity was expressed as grams of water per gram of dry solids in the dough or the crumb.

#### 4.3.18 Determination of the Dough and Crumb Morphology by Scanning Electron Microscopy

Small cubes of dough, about 2-3 mm in size, were removed from a dough which had been knocked back after fermentation for 1.5 hr. The dough pieces were mounted on aluminium stubs with conductive silver paste and proofed for 0.5 hr in a temperature - humidity chamber at 35°C and 80% humidity, followed by drying in a vacuum at 40°C overnight. About half of each dry sample on the stub was cracked open with a sharp scalpel to reveal fractured surfaces. The samples were then sputter coated with at least 20 nm of gold at 900 V and 40 mA in a vacuum. The morphological characteristics of the unfractured and fractured surfaces of the dough were examined by SEM at 15 kV. Sample photomicrographs were made for each surface.

#### 4.3.19 Determination of Dough and Crumb Internal Structure by Transmission Electron Microscopy

Pieces of dough and crumb about 2 mm<sup>3</sup> each were fixed in 3% glutaraldehyde in phosphate buffer pH 7.4 at 4°C overnight. The samples were washed with buffer and post fixed in 2% osmium tetroxide in the same buffer at pH 7.4

for 4 hr followed by one washing with the buffer. The samples were dehydrated in 70% methanol overnight, then dehydrated further in 80, 90, 95.5 98.5 and 100% methanol series for 15 min at each concentration. The final washing was repeated twice before treatment for 15 min with 1:1 methanol/propylene oxide mixture, followed by two treatments for 15 min each with 100% propylene oxide.

The samples were next treated in a mixture of 50% propylene oxide and 50% of a mixture consisting of 27 ml araldite 502, 23 ml of dodecenyl succinic anhydride (DDSA) and 2 ml of tri (dimethyl amino methyl) phenol (DMP-30). The samples were left to fix in this solution overnight in a slowly rotating shaker. Samples were then imbedded in a resin mixture of 27 ml Araldite 502, 23 ml of DDSA and 2 ml of DMP-30 in rubber molds and the resin allowed to polymerize at 60°C for 2 days. The samples were sectioned into thin slices of 6-8  $\mu$ m thick with an ultramicrotome. The sections were picked up onto 200 mesh copper grids coated with a plastic film of 0.25% formvar (polyvinyl formal) in ethylene dichloride, and dried on filter paper. The samples were stained in 1% uranyl magnesium acetate for 2 min, rinsed in distilled water and finally stained for 2 min in a solution of about 3% lead acetate, 6% sodium hydroxide and 0.3% potassium sodium tartrate (Kay, 1965). The grids were washed with distilled water, dried on filter paper and viewed through the Philips EM-200 type PW-6000 TEM at low magnification. Photomicrographs were made.



#### 4.3.20 Bread Making

Bread was made for either instrumental analysis or for panel tasting using the straight dough method.

##### 4.3.20.1 Formulations

| Ingredient                        | Bread for<br>Instrumental<br>Tasting | Bread for<br>Panel<br>Tasting |
|-----------------------------------|--------------------------------------|-------------------------------|
| Wheat flour or<br>composite flour | 100.0(%)                             | 100.0(%)                      |
| Sugar                             | 7.0                                  | 7.0                           |
| Salt                              | 1.5                                  | 1.5                           |
| Shortening                        | -                                    | 3.0                           |
| Monoglycerides                    | -                                    | 0.5                           |
| Yeast                             | 3.0                                  | 3.0                           |

The amount of water added varied with the type of flour as given below.

|                             | Water    |
|-----------------------------|----------|
| Neepawa flour               | 68-70(%) |
| Neepawa starch/gluten flour | 61-64    |
| Fielder flour               | 57-58    |
| Fielder starch/gluten flour | 55-56    |
| Root Starch/gluten flours:  |          |
| Arrowroot/gluten flour      | 83-85    |
| Cassava/gluten flour        | 63-64    |
| Sweet Potato/gluten flour   | 68-70    |
| Taro/gluten flour           | 68-69    |
| Yam/gluten flour            | 68-69    |

##### 4.3.20.2 Dough Preparation and Baking

Yeast (3% flour basis) was weighed into a beaker, 10 ml of distilled water at 35°C added and left standing for 5 min to disperse and activate the yeast.

The flour and the rest of the dry ingredients were mixed for 1 min in a Hobart mixer at speed 2. The yeast suspension was then added and the beaker rinsed once into the Hobart mixer with 20 ml of water. The rest of the water required was added and the mixer run until the dough had



achieved the desired consistency. The dough was fermented at 35°C and 80% relative humidity for 1.5 hr, punched, rolled and rested for 15 min before scaling known weights into pans greased with a film of shortening. The doughs were pan proofed for 30 min and baked at 210°C for 30 min in an oven. The bread was cooled down for 1.5 hr and wrapped in moisture-proof plastic bags.

#### 4.3.20.3 Determination of Fractional Volume Increases from the Dough to the Bread

The volume ( $V_0$ ) of the dough was taken immediately after panning, but before proofing, using the rape seed displacement technique. The volumes ( $V$ ) of each of the resulting loaves of bread were determined after cooling for 1.5 hrs. using the same technique.

Fractional volume increases were calculated from:

$$\frac{V - V_0}{V_0} \text{ cm}^3$$

#### 4.3.21 Bread Staling Investigations

Bread staling was investigated through experiments on changes in the crumb compressibility, penetration resistance to a standard probe and crystallinity.

##### 4.3.21.1 Crumb Compressibility Determination

Crumb sections 8 mm thick were cut from the center of bread slices and the force required to compress each crumb from 8 to 6 mm recorded on the calibrated chart of the compressimeter. The force was calculated from the chart

peak recordings for each crumb type against the chart range calibration and recorded as kg force/mm of the crumb compressed.

The compressimeter settings used were as follows:

|                           |              |
|---------------------------|--------------|
| Compression head speed    | 4 cm/min     |
| Compression clearance     | 6 mm         |
| Chart sensitivity (range) | 0-4000.0 g   |
| Chart speed               | 1 cm/16 sec. |

Compressibility of bread crumb in the absence and presence of monoglycerides was examined for fresh bread and bread aged for 3.5, 6, and 9 days. Crumb compressibility was then plotted on a graph as a function of bread storage time.

#### 4.3.21.2 Determination of the Crumb Penetration Resistance

Slices of bread 2 cm thick were cut from the centre of the bread, and the force to penetrate 0.5 cm with a standard probe into the crumb recorded. Compressimeter settings given above were also used, except that the compression head was replaced with the penetration probe.

Penetration resistances for fresh bread crumb in presence and absence of monoglycerides and for bread aged 3.5, 6 and 9 days were determined. Crumb penetration resistance in kg force/mm was plotted on a graph as a function of bread storage time.

#### 4.3.21.3 X-Ray Diffraction Analysis of Starch Isolated from Fresh and Aged Crumb

Starch from fresh or aged crumb was isolated by homogenizing about 20 g of crumb in 100 ml water in a Waring blender for 1 min at medium speed. The slurry was transferred into a 250 ml beaker and stirred for 1 hr with a magnetic stirrer, followed by screening through 100 mesh polyester sieve cloth. The starch isolated was separated from the filtrate by centrifugation at 3000 rpm for 10 min. The recovered starch was freeze dried and ground to a powder which was compactly compressed in the aluminum X-ray diffraction sample holder for analysis. The diffractograms obtained were used in describing crystallinity development in the crumb as a function of storage time.

#### 4.4 Bread Quality Evaluation by Panel Tasting

Bread for panel tasting was baked and cooled to room temperature. The volumes of the bread loaves were first determined by rapeseed displacement in a container of known volume. The loaves were then given external and internal quality evaluation by a panel consisting of trained students and support staff members using the American Institute of Baking method for bread quality evaluation and scoring (Matz, 1960).

## 5. RESULTS

### 5.1 A. Starch

#### 5.1.1 The Size Distribution and Morphology of Starch Granules

##### 5.1.1.1 Starch Granules Size Distribution

The particle size distribution of starch granules from wheat (cvs. Neepawa and Fielder), arrowroot, cassava, sweet potato, taro and yam are presented in Table 5.1.

Arrowroot and taro had the smallest starch granules, all below  $4\mu\text{m}$  in size. Yam had the largest starch granules; the smallest being at least  $10\mu\text{m}$  in size and ranging up to  $40\mu\text{m}$ . The majority of the yam starch granules ranged between  $16\text{--}30\mu\text{m}$  in size, with the peak between  $21\text{--}25\mu\text{m}$ .

Most of the sweet potato starch granules (83%) were less than  $10\mu\text{m}$  in size. The largest ones were  $20\mu\text{m}$ . Almost half (49%) the cassava starch granules were  $5\text{--}10\mu\text{m}$  with only 10% less than  $5\mu\text{m}$ , 31%,  $11\text{--}15\mu\text{m}$  and 9%,  $16\text{--}20\mu\text{m}$ . No cassava starch granules larger than  $25\mu\text{m}$  were observed.

Soft white spring wheat (cv. Fielder), had more numerous small granules (47%) of size less than  $5\mu\text{m}$  than Canadian west red spring wheat, cv. Neepawa, with 36% of its starch granules  $<5\mu\text{m}$ . The granule distribution was reversed significantly in the  $5\text{--}10\mu\text{m}$  size range, where Neepawa had more starch granules (40%) than Fielder (30%). Starch granules of size greater than  $25\mu\text{m}$  were not found in

Table 5.1 Starch Granule Size Distribution (%)

| Type of Starch | Granule Size in $\mu\text{m}$ |      |       |       |       |       |       |       |
|----------------|-------------------------------|------|-------|-------|-------|-------|-------|-------|
|                | <4                            | 5-10 | 11-15 | 16-20 | 21-25 | 26-30 | 31-35 | 36-40 |
| Root           |                               |      |       |       |       |       |       |       |
| Arrowroot      | 100                           |      |       |       |       |       |       |       |
| Taro           | 100                           |      |       |       |       |       |       |       |
| Sweet Potato   | 30                            | 53   | 8     | 9     |       |       |       |       |
| Cassava        | 10                            | 49   | 31    | 9     | 1     |       |       |       |
| Yam            | -                             | -    | 3     | 25    | 39    | 19    | 12    | 2     |
| Wheat          |                               |      |       |       |       |       |       |       |
| cv. Fielder*   | 47                            | 30   | 5     | 7     | 8     | 1     | 1     | 1     |
| cv. Neepawa**  | 36                            | 40   | 6     | 12    | 6     |       |       |       |

\*SWSW - Soft White Spring Wheat

\*\*CWRSW - Canadian Western Red Spring Wheat

Neepawa wheat starch, while Fielder had larger starch granules distributed up to  $40\mu\text{m}$ . The effect of starch granule size distribution and starch damage on bread are discussed under dough rheology, microstructure and bread volume.

#### *5.1.1.2 The Morphology of Starch Granules by Scanning Electron Microscopy*

Scanning electron photomicrographs portraying the morphologies of the different starches are given in Plates 5.1 to 5.6, representing arrowroot, taro, sweet potato, yam, cassava (2 Plates), and wheat starches.

Arrowroot (Plate 5.1) and taro (Plate 5.2) starch granules were observed to be polygonal. Most displayed sharp edges between the facets, which in turn possessed curved depressions, made probably by pressure from other starch granules during development. Similar observations were evident with sweet potato starch granules, which also had several rounded facets (Plate 3, a and b). Yam starch granules displayed in Plate 3 (c and d) were ellipsoidal and smooth on the surface. A few had their ellipsoidal outlines expanded and rounded at one end.

Cassava starch granules (Plates 4 and 5) were round or truncated in appearance. The truncated surface was concave, culminating in a sharp sunken central point. Compounded starches were also observed.

Wheat starches (Plate 5.6) were small Platelete, donut-like types or larger ones, lenticular in shape. The

Plate 5.1. Scanning Electron Micrographs of Arrowroot Starches From  
Kenya - a, x 6,900; b, x 4,500; c, x 4,200 and d, x 7,000.

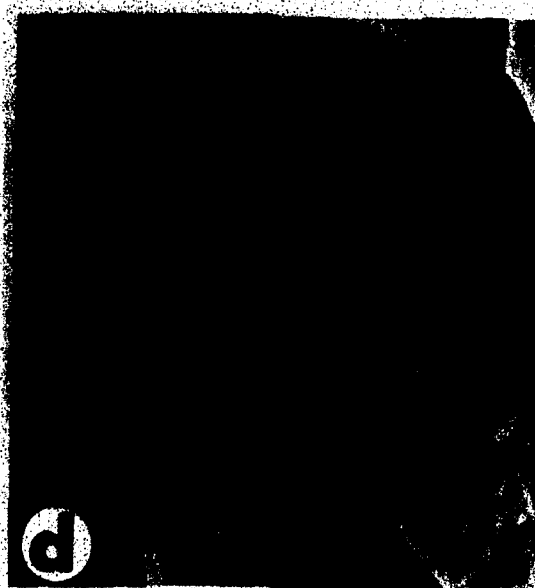




Plate 5.2, Scanning Electron Micrographs of Taro Starches - a, x  
3,300; b, x 6,400; c, x 6,400 and d, x 6,500.

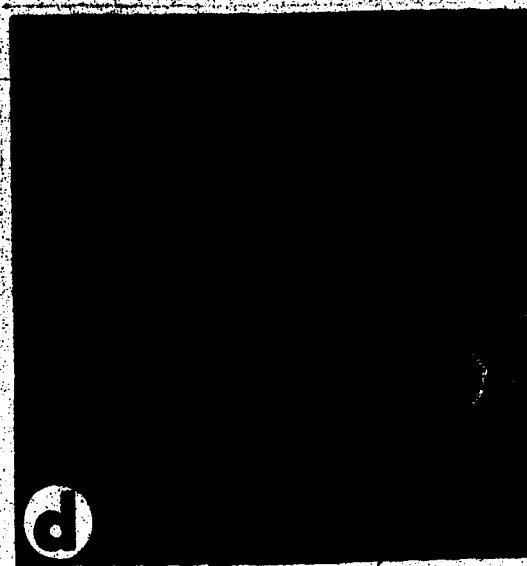
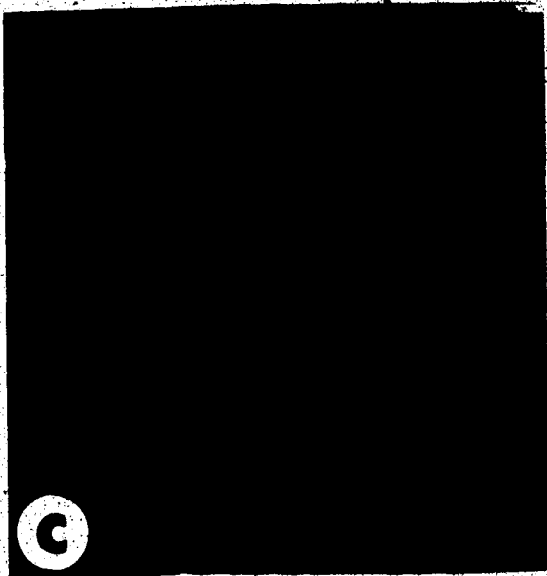
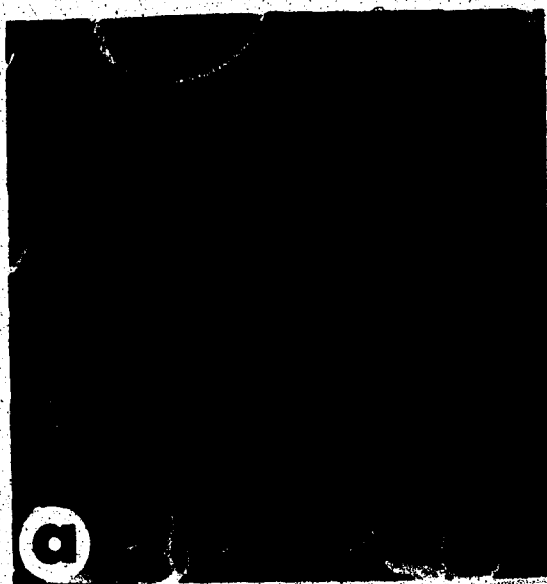


Plate 5.3, Scanning Electron Micrographs of Sweet Potato (a, x 2,600;  
b, x 3,300) and Yam (c, x 620; d, x 1,280) Starches.

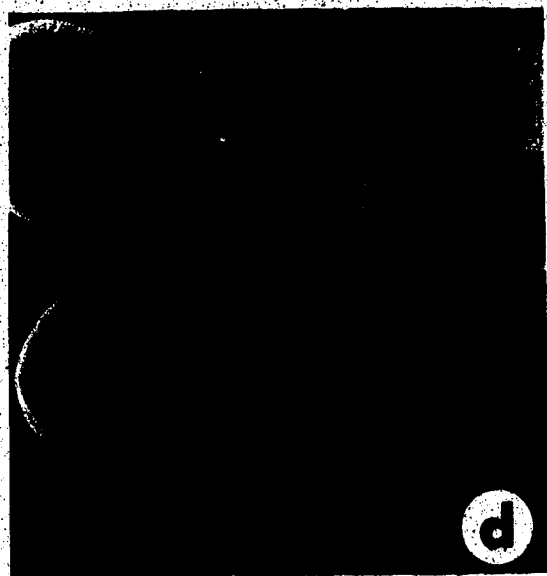
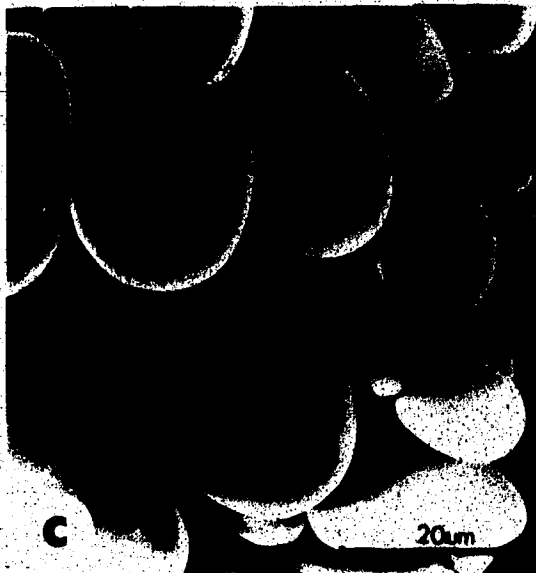
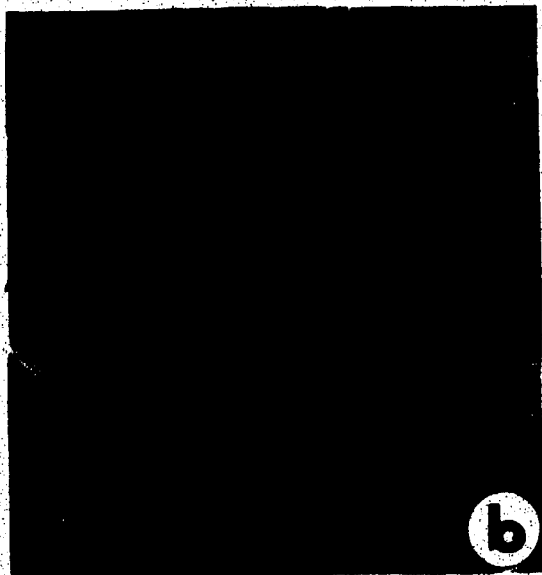


Plate 5.4. Scanning Electron Micrographs of Cassava Starches of  
Commercial (a, x 2,800; b, x 3,700), A.E. Staley Manuf.  
Co., Decatur, IL., and Kenyan (c, x 5,200; d, x 4,500)  
Origins.

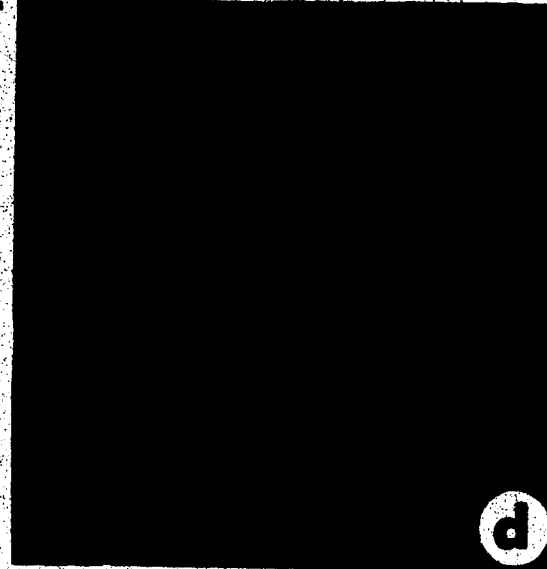


Plate 5.5. Scanning Electron Micrographs of Cassava Starches of (a)  
Commercial (A.E. Staley Manuf. Co., Decatur, IL.), (b)  
Fijian and (c) Kenyan Origins with Magnification Range of  
x 2,000 - x 2,500.

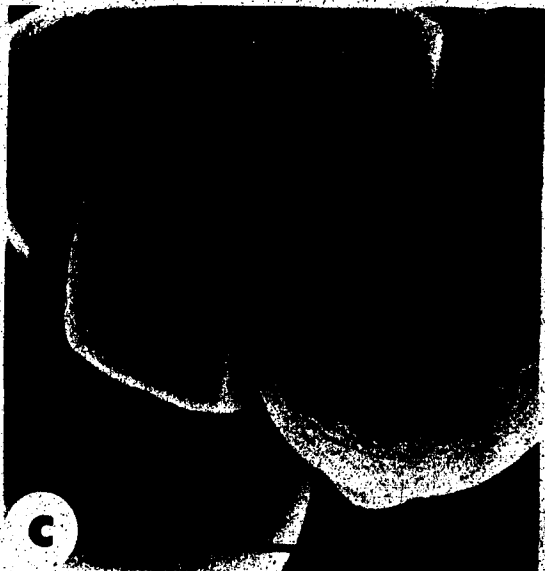
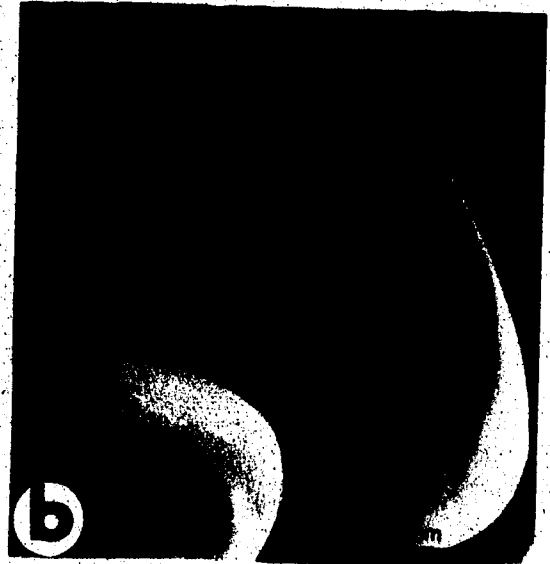




Plate 5.6. Scanning Electron Micrographs of Wheat Starches, cv.

Fielder (a, x 1,120; b, x 2,600); and cv. Neepawa (c, x 1,300; d, x 2,630).



majority of the larger ones possessed pronounced equatorial grooves (Plate 5.6, b and d).

#### 5.1.2 Determination of Amylose Content in Starch

The standard curve for amylose determination in starch by potentiometric titration is given in Figure 5.1.

Figure 5.2 shows typical potentiometric titration curves in presence of arrowroot, cassava and wheat (cv. Neepawa) starches.

Figures 5.3 and 5.4 represent plots for bound iodine versus free iodine in potentiometric titrations involving cassava and wheat (cv. Neepawa) starches. Other starches had similar plots.

Table 5.2 shows the amylose contents of the starches. Also given are the amylose/amylopectin ratios of the various starches.

Wheat (cv. Neepawa) starch had the highest amylose content (27.29%), followed by yam (25.00%) and wheat (cv. Fielder, 23.04%) starches. All the other root starches had less amylose content than the wheat starches. Taro starch had the lowest amylose content (14.93%), next to arrowroot starch (16.73%). Sweet potato and cassava starches had intermediate amylose contents ranging from 18.98-21.0%. The amylose/amylopectin ratios corresponded in magnitude to their respective amylose contents.

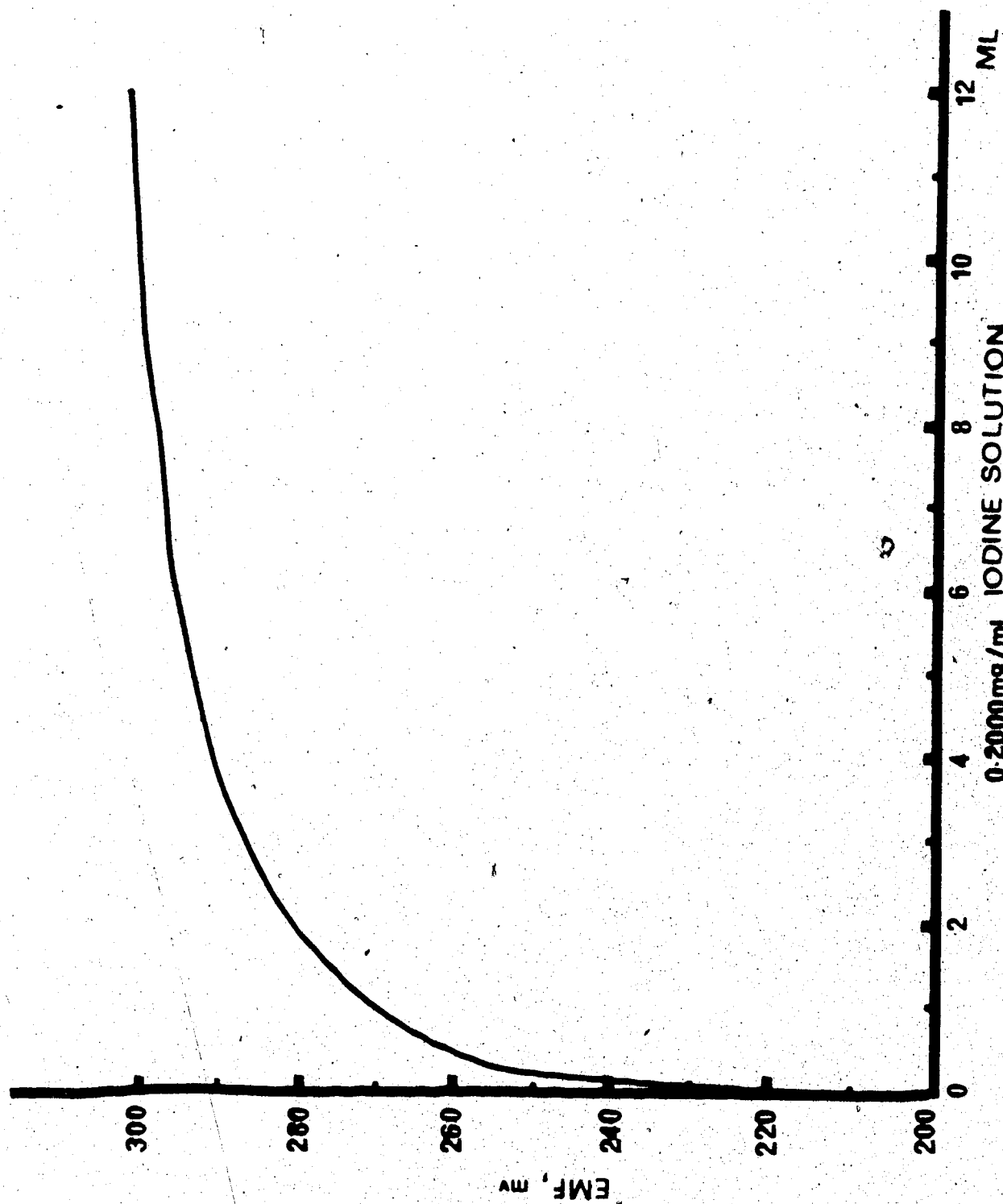


Figure 5.1. Standard Curve of EMF as a Function of Free Iodine Content at 30°C.

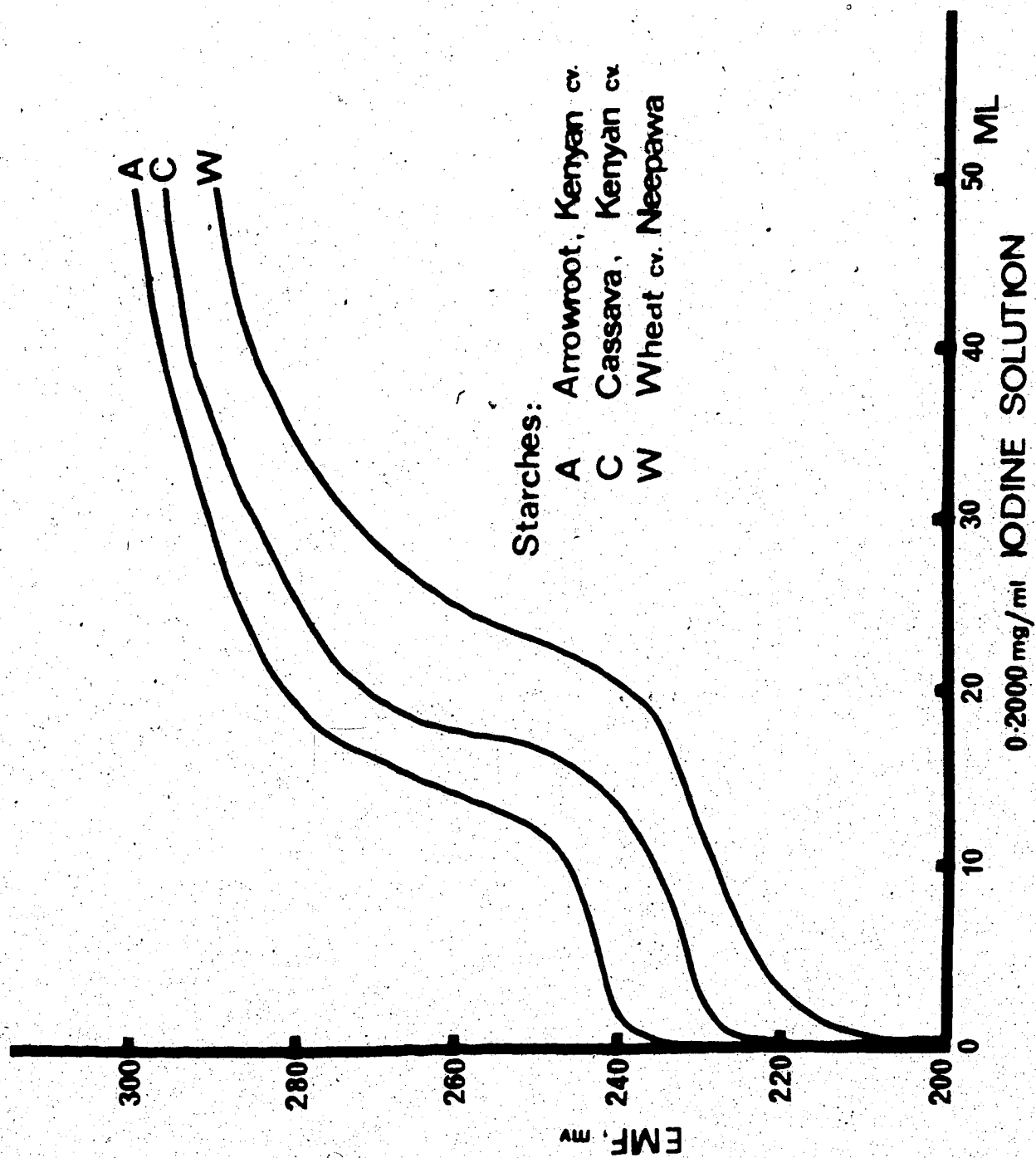


Figure 5.2. Potentiometric Titration of Arrowroot, Cassava and Wheat, cv. Neepawa, Starches with Iodine at 30°C.

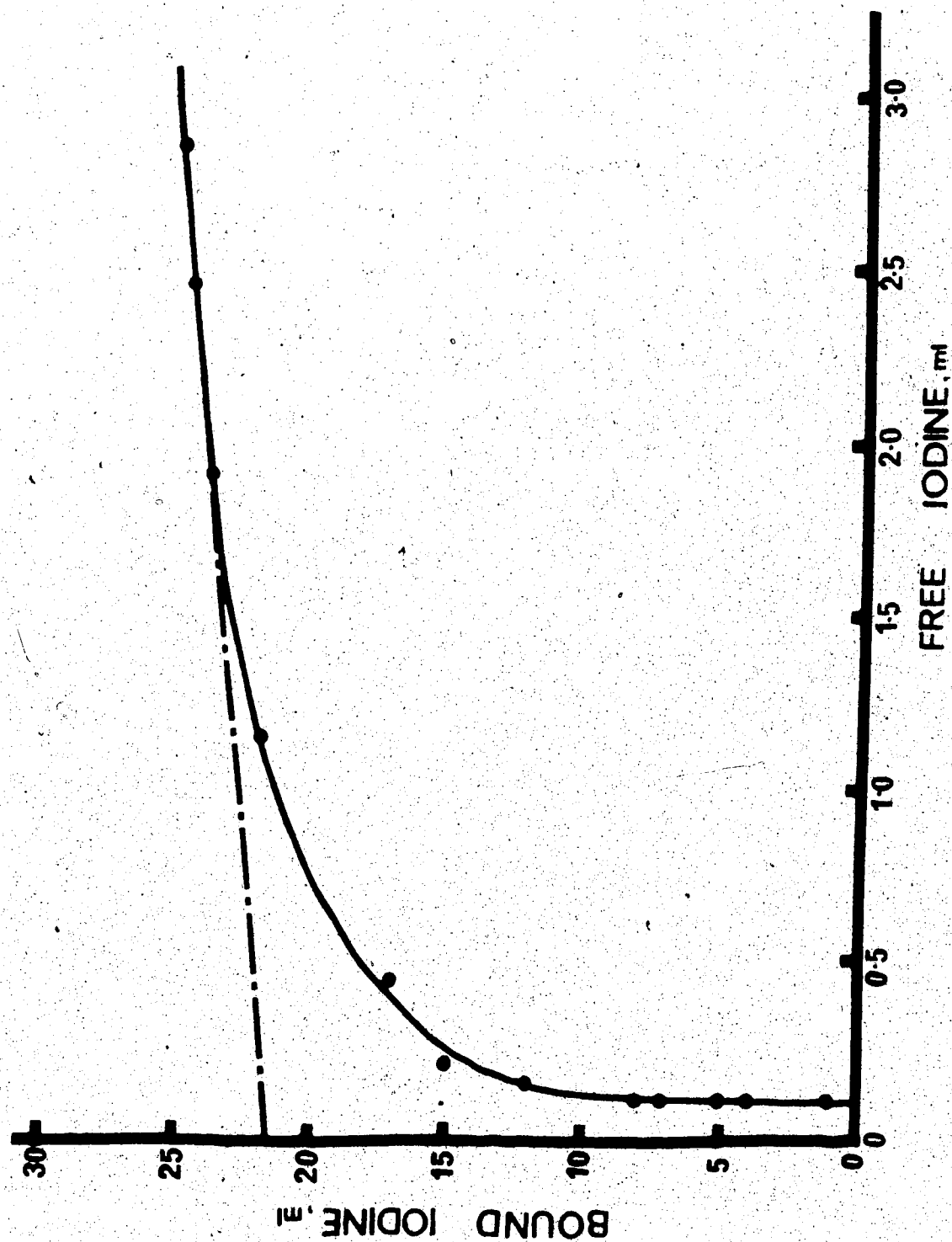


Figure 5.3. Plot of Cassava Starch, Bound versus Free Iodine at 30°C.

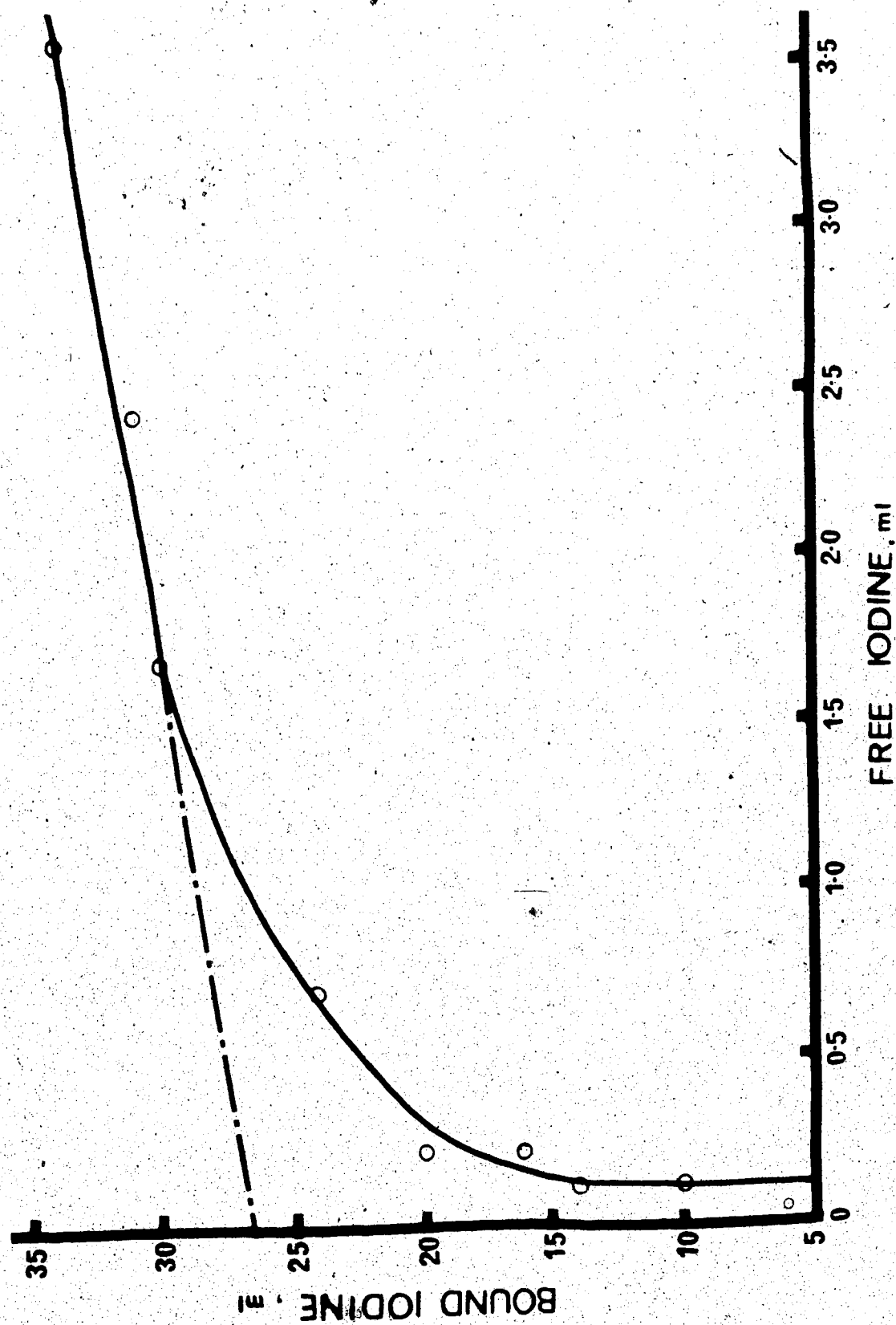


Figure 5.4. Plot of Wheat, cv. Neepawa Starch, Bound versus Free Iodine at 30°C.

Table 5.2 Amylose Contents and Amylose-Amylopectin Ratios of Some Wheat and Tropical Root Starches

| Type of Growth  | Amylose          | Amylose-Amylopectin Ratio |
|-----------------|------------------|---------------------------|
| Root:           |                  |                           |
| Taro            |                  |                           |
| cv. Jamaican    | 14.93 $\pm$ 0.63 | 0.176                     |
| Arrowroot       |                  |                           |
| cv. Kenyan      | 16.73 $\pm$ 0.34 | 0.200                     |
| Cassava         |                  |                           |
| cv. Fijian      | 18.98 $\pm$ 1.51 | 0.234                     |
| cv. Kenyan      | 21.00 $\pm$ 0.67 | 0.266                     |
| Sweet Potato    |                  |                           |
| cv. Georgia Red | 19.66 $\pm$ 0.48 | 0.245                     |
| cv. Porto Rico  | 19.71 $\pm$ 0.61 | 0.246                     |
| cv. Centennial  | 20.00 $\pm$ 0.21 | 0.250                     |
| Yam             |                  |                           |
| cv. Jamaican    | 25.00 $\pm$ 0.35 | 0.333                     |
| Wheat:          |                  |                           |
| cv. Fielder     | 23.04 $\pm$ 0.62 | 0.299                     |
| cv. Neepawa     | 27.29 $\pm$ 1.53 | 0.375                     |



### 5.1.2.1 Starch Mineral Composition

In Table 5.3. are presented the mineral contents of arrowroot, cassava, sweet potato, taro, yam and wheat (cvs. Fielder and Neepawa) starches.

Amongst the root starches, taro and arrowroot, with the smallest starch granule size, had the highest phosphorus contents - approximately twice the amount of phosphorus found in cassava, sweet potato, and wheat (cv. Neepawa) starches. Wheat (cv. Fielder) starch was intermediate in its content of phosphorus. Yam, with the largest granule size had the lowest phosphorus content (0.0064%). Extraction of the wheat starches with water saturated butanol to remove lipid-phosphate complexes reduced their phosphate content from about 0.05% to 0.016 and 0.013% in cvs. Fielder and Neepawa.

All the starches had very low  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$  contents. The wheat starches contained no calcium. In contrast, all the starches had higher contents of  $\text{K}^+$  and  $\text{Na}^+$  compared to their contents of  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$ .

### 5.2 Swelling Power of the Starches

The swelling powers (SP) of arrowroot, cassava, sweet potato, taro, yam, and wheat (cvs. Fielder and Neepawa) starches as a function of temperature are shown in Figure 5.5. and Tables 5.4-5.7.

Table 5.3 Mineral Contents of Some Wheat and Tropical Root Starches

| Type of Starch                 | Moisture, %     | Mineral Content in Percent |                        |                        |                        |                        |
|--------------------------------|-----------------|----------------------------|------------------------|------------------------|------------------------|------------------------|
|                                |                 | P                          | Ca                     | Mg                     | K                      | Na                     |
| Taro<br>cv. Jamaican           | 8.53 $\pm$ 0.15 | 0.0256<br>$\pm$ 0.0008     | 0.0021<br>$\pm$ 0.0011 | 0.0042<br>$\pm$ 0.0004 | 0.0925<br>$\pm$ 0.0071 | 0.0255<br>$\pm$ 0.0006 |
| Arrowroot<br>cv. Kenyan        | 8.24 $\pm$ 0.09 | 0.0227<br>$\pm$ 0.0019     | 0.0081<br>$\pm$ 0.0002 | 0.0072<br>$\pm$ 0.0008 | 0.0936<br>$\pm$ 0.0001 | 0.0321<br>$\pm$ 0.0007 |
| Sweet Potato<br>cv. Centennial | 8.28 $\pm$ 0.31 | 0.0126<br>$\pm$ 0.0017     | 0.0094<br>$\pm$ 0.0017 | 0.0046<br>$\pm$ 0.0008 | 0.0336<br>$\pm$ 0.0008 | 0.0259<br>$\pm$ 0.0036 |
| Cassava<br>cv. Kenyan          | 8.98 $\pm$ 0.22 | 0.0106<br>$\pm$ 0.0008     | 0.0067<br>$\pm$ 0.0005 | 0.0034<br>$\pm$ 0.0004 | 0.0366<br>$\pm$ 0.0007 | 0.0254<br>$\pm$ 0.0006 |
| Yam<br>cv. Jamaican            | 9.10 $\pm$ 0.08 | 0.0064<br>$\pm$ 0.0012     | 0.0003<br>$\pm$ 0.0001 | 0.0024<br>$\pm$ 0.0001 | 0.0156<br>$\pm$ 0.0004 | 0.0194<br>$\pm$ 0.0004 |
| Wheat*                         |                 |                            |                        |                        |                        |                        |
| cv. Fielder                    | 8.45 $\pm$ 0.16 | 0.016<br>$\pm$ 0.006       | ---                    | 0.0050<br>$\pm$ 0.0002 | 0.0220<br>$\pm$ 0.0007 | 0.0249<br>$\pm$ 0.0007 |
| cv. Neepawa                    | 8.25 $\pm$ 0.45 | 0.013<br>$\pm$ 0.002       | ---                    | 0.0048<br>$\pm$ 0.0006 | 0.0221<br>$\pm$ 0.0006 | 0.0187<br>$\pm$ 0.0008 |

\*Refluxed 2 times for 3 hrs with water saturated butanol. If the extraction step was omitted the wheat starch P amounted to 0.05 $\pm$ 0.007.

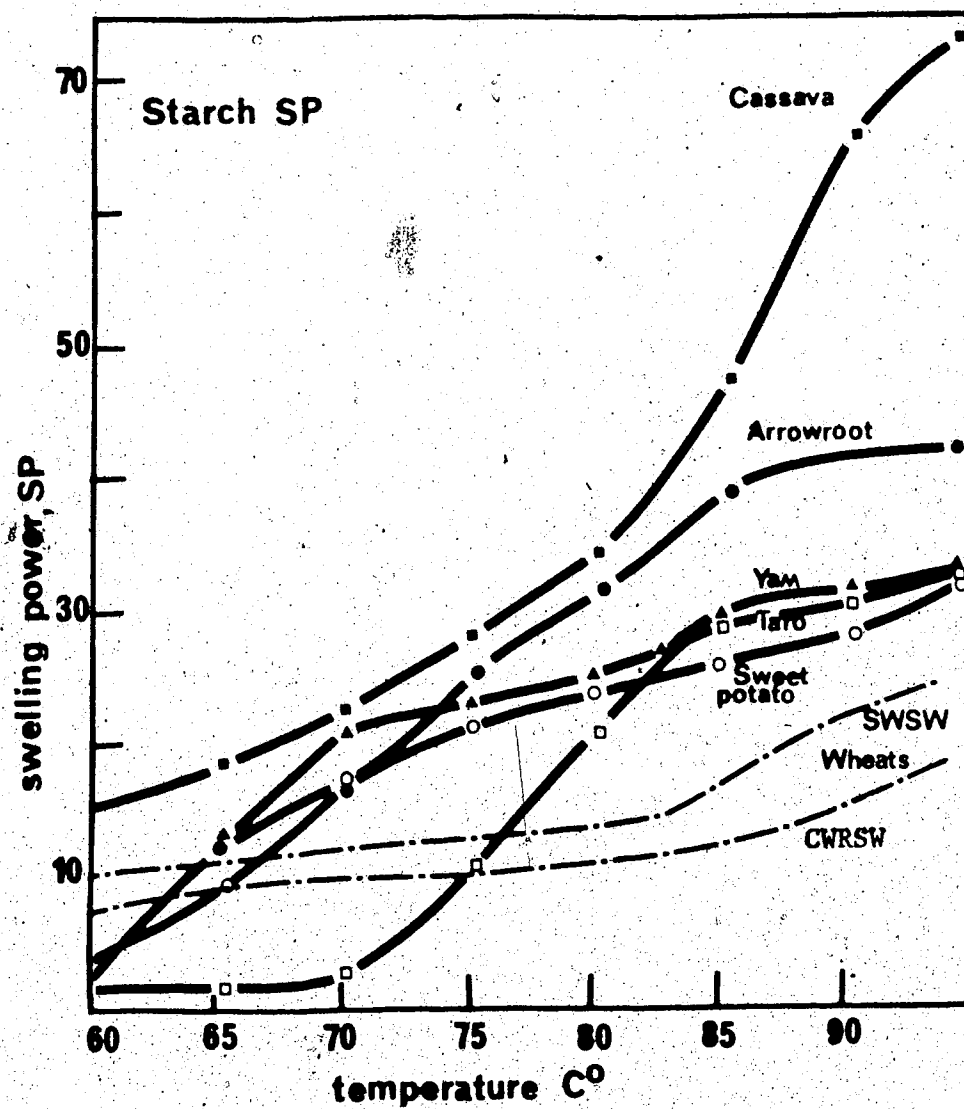


Figure 5.5. Swelling Power as a Function of Temperature and Tropical Root starches for Some Wheat

Table 5.4. Swelling Potentials of Some Taro Root

| Type of Starch           | Temperature °C |            |            |            |            |            |
|--------------------------|----------------|------------|------------|------------|------------|------------|
|                          | 60             | 65         | 70         | 75         | 80         | 85         |
| Arrowroot<br>(Kenyan cv) | 3.57±0.42      | 12.50±0.74 | 17.34±1.17 | 26.11±1.55 | 31.04±2.42 | 39.31±1.55 |
| Taro<br>(Jamaican cv)    | 2.26±0.07      | 2.25±0.17  | 2.56±0.11  | 11.87±1.63 | 21.52±1.34 | 30.09±1.69 |
| Yam<br>(Jamaican cv)     | 2.19±0.19      | 11.99±0.17 | 21.15±0.81 | 22.46±1.28 | 24.68±1.08 | 29.05±1.32 |

95

40.02±0.46

33.00±2.02

33.82±0.17

Table 5.5. Swelling Power of Some Cassava Starches

| Types of Starch   | Temperature °C |            |            |            |            |            |            |            |
|-------------------|----------------|------------|------------|------------|------------|------------|------------|------------|
|                   | 60             | 65         | 70         | 75         | 80         | 85         | 90         | 95         |
| Cassava:          |                |            |            |            |            |            |            |            |
| Commercial Sample | 2.75±0.27      | 13.53±0.73 | 25.47±0.79 | 29.45±2.02 | 35.59±2.30 | 40.34±0.91 | 36.85±1.91 | 39.06±0.64 |
| Fijian cv.        | 9.88±0.30      | 24.60±0.14 | 29.29±0.72 | 35.42±1.92 | 48.29±0.36 | 50.71±1.25 | 50.97±2.14 | 66.99±4.95 |
| Kassan cv.        | 15.57±1.76     | 19.66±1.06 | 22.45±1.06 | 29.24±0.80 | 34.50±2.00 | 47.45±3.62 | 66.77±2.80 | 73.43±4.17 |

Table 5.6. Swelling Power of Some Sweet Potato Starches

| Type of Starch  | Temperature °C |               |                |                |                |                |                |                |  |
|-----------------|----------------|---------------|----------------|----------------|----------------|----------------|----------------|----------------|--|
|                 | 60             | 65            | 70             | 75             | 80             | 85             | 90             | 95             |  |
| Sweet Potatoes  |                |               |                |                |                |                |                |                |  |
| Centennial cv.  | 4.17<br>±0.26  | 9.49<br>±0.43 | 17.68<br>±0.65 | 21.68<br>±0.58 | 23.40<br>±0.98 | 25.84<br>±1.05 | 28.34<br>±0.38 | 32.83<br>±0.70 |  |
| Georgia Red cv. | 2.04<br>±0.07  | 3.34<br>±0.21 | 16.25<br>±1.08 | 22.28<br>±0.48 | 24.44<br>±1.11 | 27.47<br>±0.92 | 30.94<br>±0.69 | 36.68<br>±0.47 |  |
| Porto Rico cv.  | 4.64<br>±0.36  | 9.00<br>±0.49 | 16.57<br>±0.54 | 20.31<br>±1.09 | 24.26<br>±1.14 | 25.46<br>±0.96 | 27.75<br>±1.12 | 31.40<br>±1.06 |  |

Table 5.7. Swelling Power of some Wheat Starches

| Type of Starch | Temperature °C |            |            |            |            |            |            |            |
|----------------|----------------|------------|------------|------------|------------|------------|------------|------------|
|                | 60             | 65         | 70         | 75         | 80         | 85         | 90         | 95         |
| Neepawa        | 7.12±0.45      | 10.00±0.99 | 10.50±1.15 | 9.71±0.98  | 11.44±2.00 | 12.43±1.40 | 15.04±2.0  | 18.83±1.89 |
| Fielder        | 10.04±0.65     | 11.01±1.40 | 12.73±0.98 | 13.17±0.12 | 13.04±1.10 | 16.04±0.67 | 21.94±2.09 | 24.52±1.84 |

Cassava starch had the highest SP at all temperatures. Hard red, followed by soft white, spring wheat starches had the lowest swelling power at temperatures beyond 75°C.

Swelling powers of arrowroot, yam, taro and sweet potato starches were intermediate between cassava and wheat starch swelling powers, but decreased in magnitude from arrowroot to yam, taro, and sweet potato starches at temperatures greater than 85°C. The swelling power of the wheat starches remained fairly constant from 60-85°C, rising only from about 7-10 to 12-16 g of paste/g starch DWB. It then rose fairly slowly from about 15-20 to 18-24 g paste/g at 95°C.

Taro starch did not show any signs of swelling between 60-70°C, after which its swelling power rose steadily almost to a peak at 85°C. It exhibited a sigmoid behavior of its swelling power in relation to temperature.

Yam starch swelled quickly from 2 to 21 g paste/g of starch at 60-70°C and then increased gradually after that to a peak value of about 34 g paste/g of starch. A similar trend was observed for sweet potato starch.

Arrowroot starch rose gradually in its swelling power as a function of temperature up to 85°C then nearly leveled off to a peak value, while cassava continued to rise sharply in its swelling power with further increase of temperature.



### 5.2.1 Starch Solubility

Starch solubility curves as a function of temperature for arrowroot, cassava, sweet potato, taro, yam, and wheat (cvs. Neepawa and Fielder) starches are presented in Figure 5.6, and in Tables 5.8-5.11.

Like with swelling power, cassava starch had the highest solubility at all temperatures. At low temperatures, root starches had higher solubilities than the wheat starches. At temperatures higher than 83°C the solubility of wheat starches increased at a greater rate than those observed for the root starches. At about 87°C, the solubilities of the wheat starches exceeded the solubilities of the root starches, except that of cassava which they equalled only at 95°C.

At all temperatures, hard wheat had lower solubilities than soft wheat. Taro starch solubility as a function of temperature rose in a sigmoid manner as observed for swelling power. Yam and sweet potato starches were very close in their solubility characteristics. Their solubilities rose steadily over the whole temperature range; 60-95°C. Cassava and arrowroot solubilities rose with the highest, but decreasing rates as a function of temperature as indicated by the curves in Figure 5.6.

The solubility of a starch depended on its swelling power. Thus the highest solubility for wheat starches was observed over the temperature range 85-95°C. Solubility rates for taro starch were highest between 70-85°C

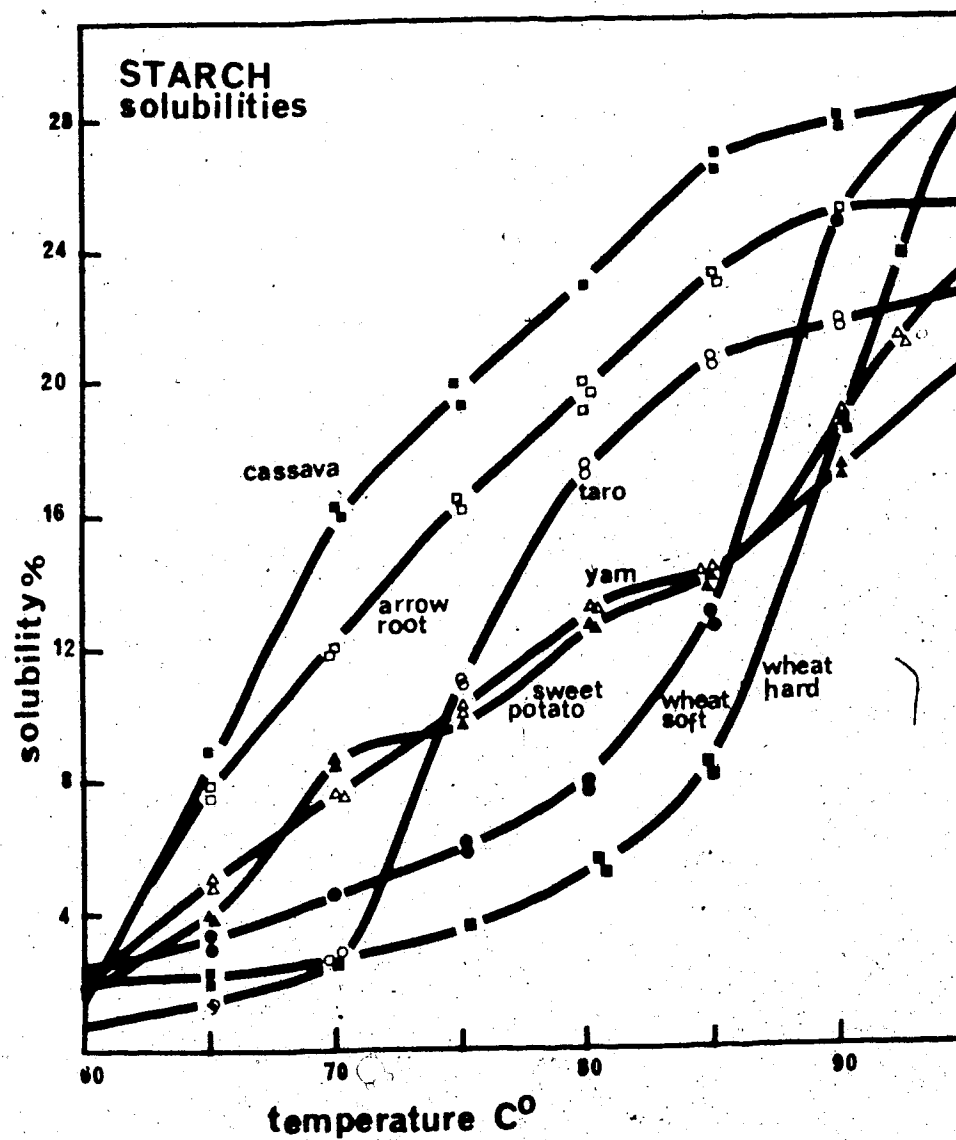


Figure 5.6. Starch Solubility as a Function of Temperature for Some Wheat and Tropical Root Starches.

Table 5.8 Solubility of Arrowroot, Taro and Yam Starches as a Function of Temperature

| Types of Starches        | Temperature °C |           |            |            |            |            |            |            |
|--------------------------|----------------|-----------|------------|------------|------------|------------|------------|------------|
|                          | 60             | 65        | 70         | 75         | 80         | 85         | 90         | 95         |
| Arrowroot<br>(Kenyan cv) | 1.47±0.30      | 7.93±0.99 | 12.04±0.38 | 16.38±0.29 | 19.01±0.83 | 23.28±0.31 | 25.28±0.29 | 24.72±1.0  |
| Taro<br>(Jamaican cv)    | 0.67±0.11      | 1.33±0.17 | 2.29±0.27  | 11.06±1.35 | 17.51±1.39 | 20.87±1.13 | 17.79±1.32 | 22.4±2.14  |
| Yam<br>(Jamaican cv)     | 2.19±0.18      | 5.26±6.88 | 7.47±0.63  | 9.68±1.06  | 13.37±0.21 | 12.89±0.68 | 18.94±0.69 | 23.31±0.39 |

Table 5.9. Solubilities of Some Cassava Starches as a  
Function of Temperature

| Types<br>of Starches | Temperature °C |            |            |            |            |            |            |            |
|----------------------|----------------|------------|------------|------------|------------|------------|------------|------------|
|                      | 60             | 65         | 70         | 75         | 80         | 85         | 90         | 95         |
| Cassava:             |                |            |            |            |            |            |            |            |
| Commercial<br>Sample | 1.49±0.24      | 8.92±0.79  | 16.4±0.36  | 19.14±0.85 | 22.89±0.61 | 27.04±0.27 | 27.51±0.26 | 28.60±0.20 |
| Fijian cv.           | 2.83±0.16      | 10.98±0.14 | 14.18±0.48 | 17.23±0.97 | 23.10±1.27 | 28.25±2.16 | 33.72±2.50 | 35.26±1.97 |
| Kenyan cv.           | 3.31±0.29      | 7.16±0.52  | 6.92±0.29  | 10.05±0.46 | 14.96±1.76 | 20.34±1.33 | 31.14±0.85 | 30.36±0.66 |

Table 5.10. Solubilities of Some Sweet Potato Starches as a  
Function of Temperature

| Types of<br>Starch | Temperature °C |           |           |            |            |            |            |            |
|--------------------|----------------|-----------|-----------|------------|------------|------------|------------|------------|
|                    | 60             | 65        | 70        | 75         | 80         | 85         | 90         | 95         |
| Sweet Potatoes:    |                |           |           |            |            |            |            |            |
| Centennial cv.     | 2.02±0.58      | 3.39±0.29 | 8.76±0.33 | 8.97±0.99  | 12.69±0.18 | 13.93±2.29 | 17.44±0.63 | 20.46±0.81 |
| Georgier Red cv.   | 0.61±0.09      | 1.07±0.12 | 4.24±1.37 | 10.22±0.98 | 11.87±0.84 | 12.85±0.49 | 17.07±0.38 | 17.14±0.52 |
| Porto Rico cv.     | 1.67±0.31      | 2.79±1.33 | 6.67±0.82 | 10.39±0.44 | 15.84±0.47 | 16.64±0.33 | 17.48±0.72 | 18.64±0.32 |

Table 5.11. Solubilities of Wheat, cvs. Neepawa and Fielder  
starches as a Function of Temperature

| Types<br>of Wheat | Temperature °C |           |           |           |           |            |            |            |
|-------------------|----------------|-----------|-----------|-----------|-----------|------------|------------|------------|
|                   | 60             | 65        | 70        | 75        | 80        | 85         | 90         | 95         |
| Neepawa           | 2.07±0.30      | 1.98±0.09 | 3.20±0.20 | 3.55±0.68 | 5.59±0.34 | 9.88±0.17  | 18.91±0.76 | 28.56±0.51 |
| Fielder           | 2.37±0.59      | 3.21±0.51 | 4.65±1.27 | 6.17±1.05 | 7.78±1.08 | 12.85±0.51 | 25.12±0.76 | 28.75±1.10 |

### 5.2.2 Starch Viscosity

Viscosity curves as a function of temperature for arrowroot, cassava, sweet potato, taro, yam and wheat (cvs. Neepawa and Fielder) starches are provided in Figures 5.7-5.10, and in Tables 5.12-5.15.

For all the starches, there was practically no difference between the viscosities after 5 and 10 minute runs of the Haake rotoviscoimeter.

The cassava starches, especially the Fijian cultivar, had the highest viscosities, followed by taro, yam, arrowroot, sweet potato and wheat starches. Of the sweet potato starches, the Porto rico cultivar had the lowest viscosity. Soft wheat (cv. Fielder) was observed to have higher viscosities at all temperatures than hard wheat (cv. Neepawa).

### 5.2.3 Starch Gelatinization Properties

Gelatinization thermograms and data as a function of temperature and water volume fraction,  $v_1=0.3-0.8$ , for some tropical root and wheat starches are given in Figures 5.11-5.13; Tables 5.16-5.20 and Appendices 1-4.

The thermograms obtained showed that the root starches had a gelatinization onset temperature range of 52-63°C, a peak temperature range of 58-68°C, and an end of gelatinization range of 65-73°C. Taro starch was the exception, with a high onset of 74.5°C, a peak of 79°C, and an end of gelatinization temperature of 83.7°C. CWRSW and

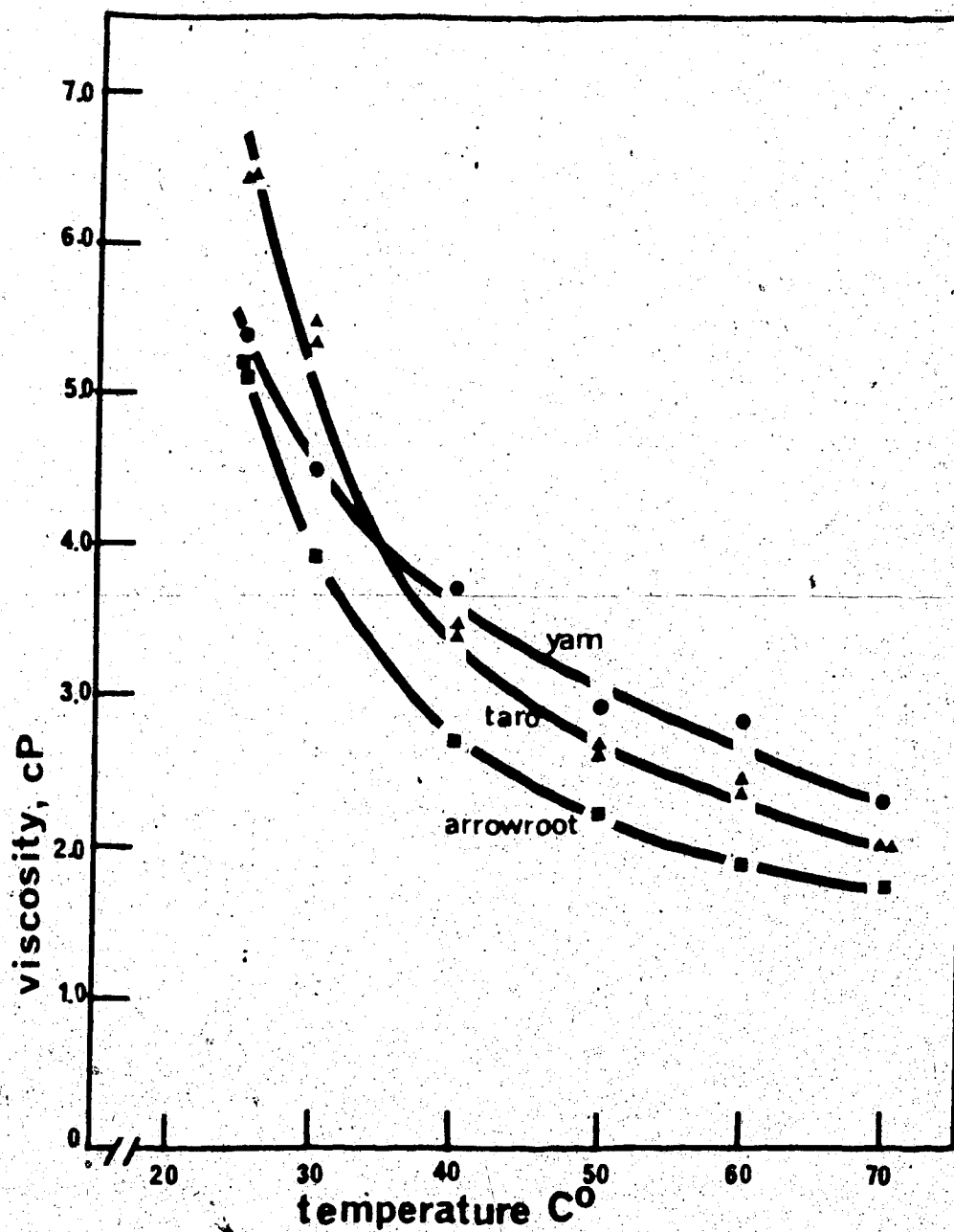


Figure 5.7. Viscosity as a Function of Temperature for Arrowroot, Taro, Yam Starches



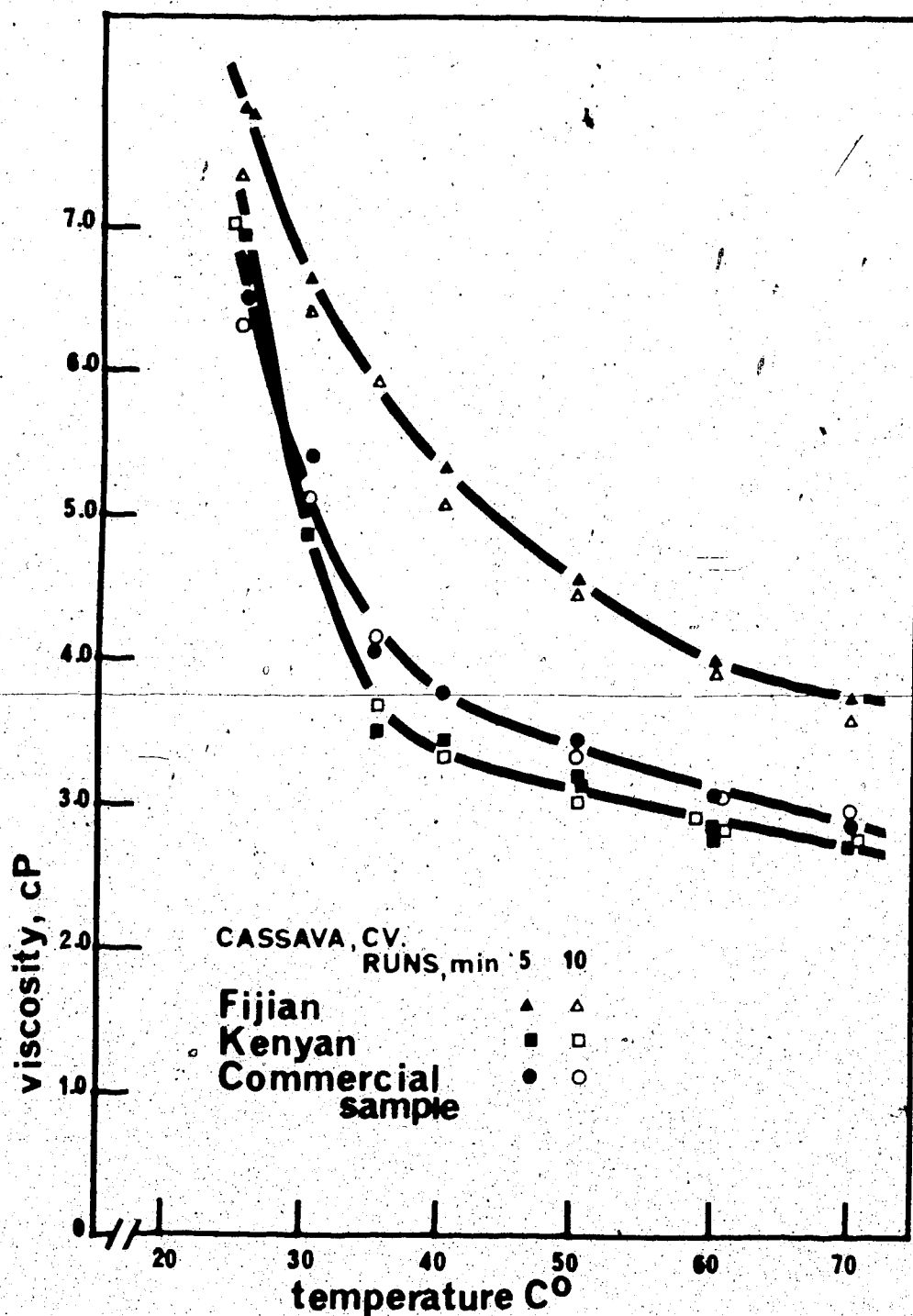


Figure 5.8. Viscosity as a Function of Temperature for Some Cassava Starches

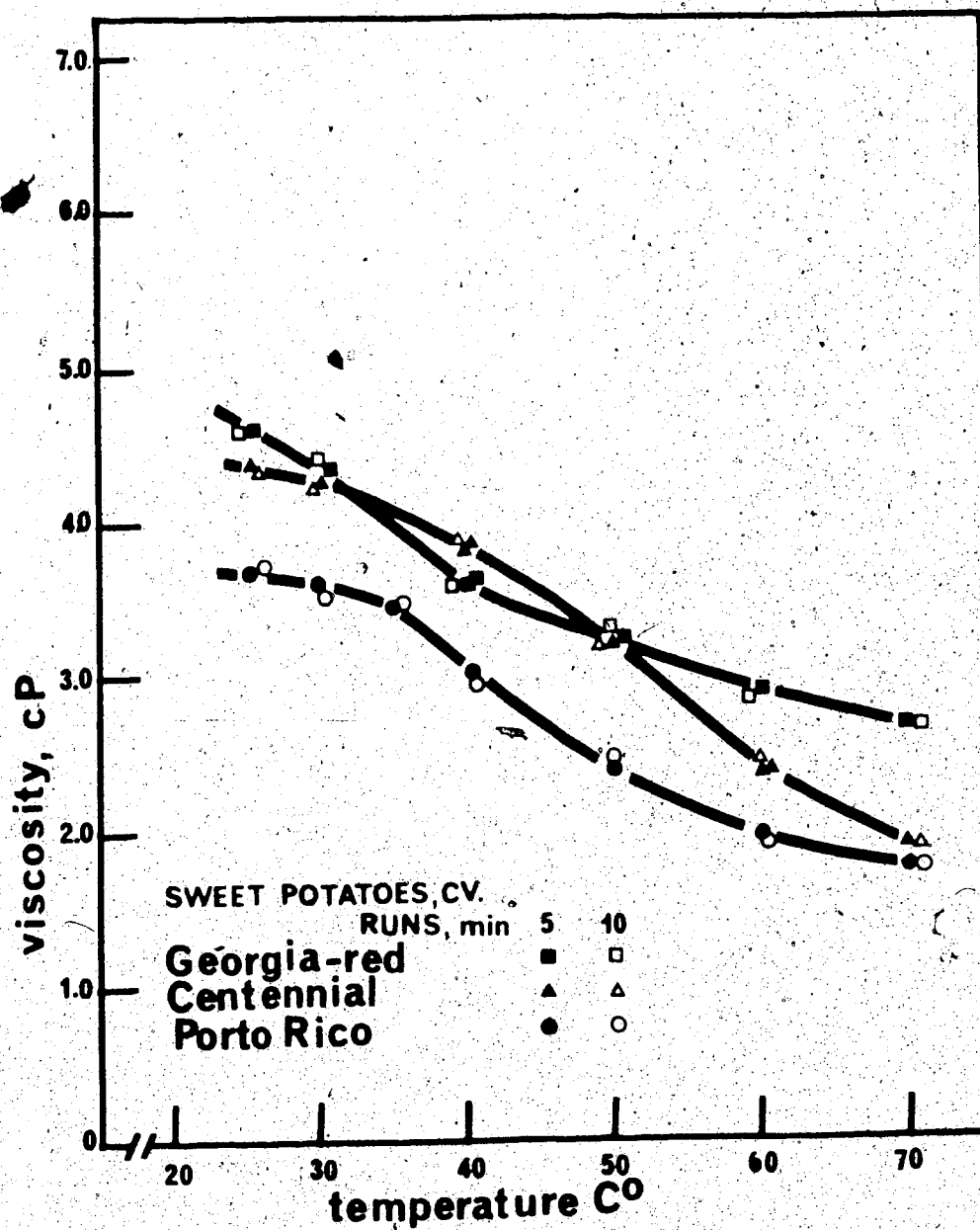


Figure 5.9. Viscosity as a Function of Temperature  
for Some Sweet Potato Starches

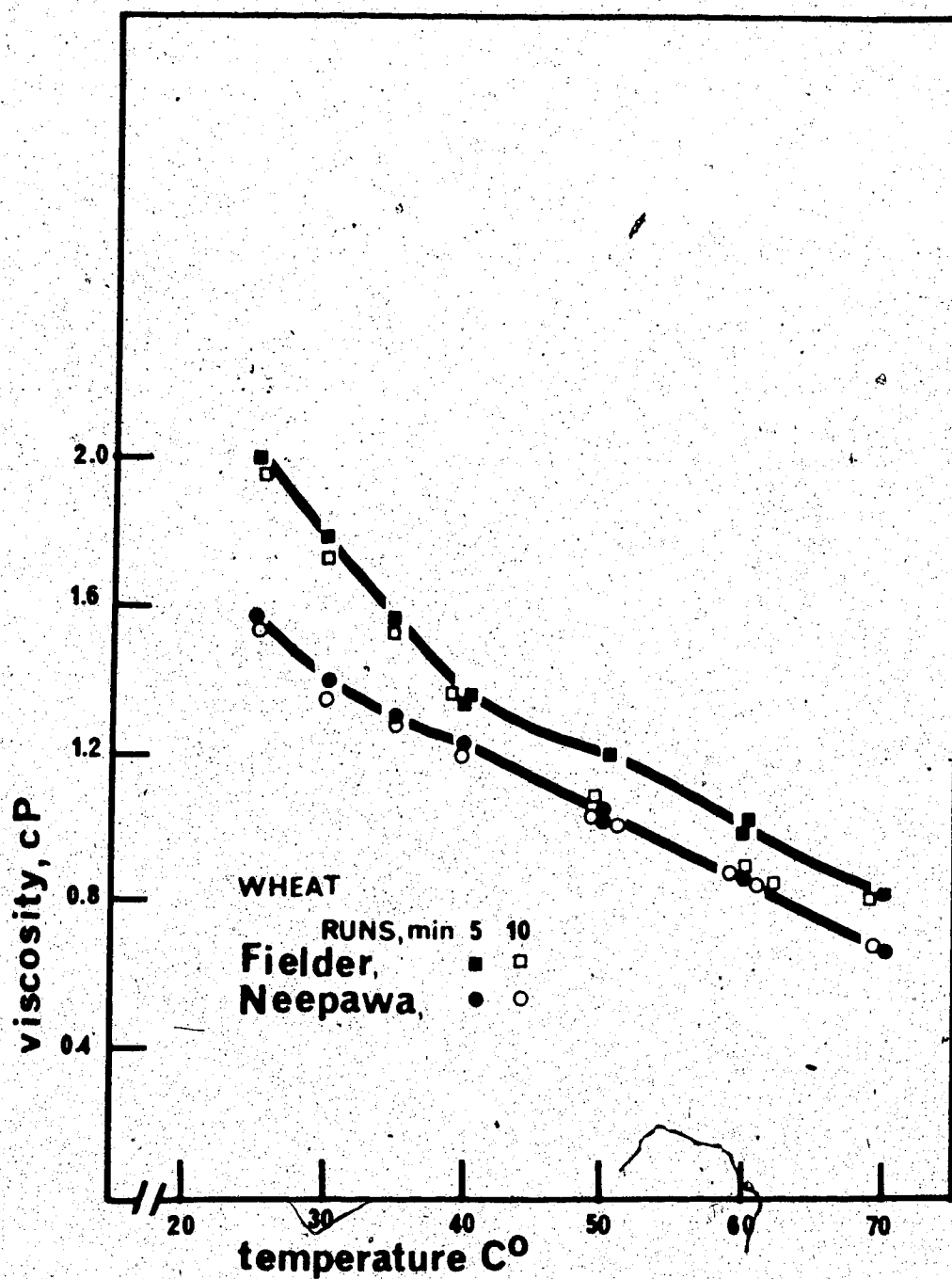


Figure 5.10 Viscosity as a Function of Temperature for  
Some Wheat, cvs. Fielder and Neepawa.

Table 5.12. (Viscosity as a Function of Temperature for Arrowroot, Taro and Yam Starches

| Starch*                 | Duration of<br>Viscometer<br>Run, Min. | Temperature, °C |           |           |           |           |           |           |
|-------------------------|--|-----------------|-----------|-----------|-----------|-----------|-----------|-----------|
|                         |  | 25              | 30        | 35        | 40        | 50        | 60        | 70        |
| Arrowroot<br>cv. Kenyan | 5                                      | 5.25±0.25       | 3.90±0.18 | 3.28±0.04 | 2.69±0.05 | 2.22±0.16 | 1.89±0.09 | 1.70±0.22 |
|                         | 10                                     | 5.08±0.25       | 3.83±0.18 | 3.26±0.06 | 2.63±0.07 | 2.17±0.16 | 1.84±0.16 | 2.39±0.22 |
| Taro<br>cv. Jamaican    | 5                                      | 6.44±0.16       | 5.43±0.10 | 3.88±0.07 | 3.60±0.25 | 2.59±0.12 | 2.46±0.13 | 2.05±0.05 |
|                         | 10                                     | 5.98±0.27       | 5.22±0.15 | 3.76±0.06 | 3.38±0.17 | 2.43±0.15 | 2.28±0.11 | 3.01±0.04 |
| Yam<br>cv. Jamaican     | 5                                      | 5.45±0.13       | 4.47±0.09 | 3.9±0.23  | 3.71±0.15 | 2.90±0.43 | 2.86±0.21 | 2.30±0.14 |
|                         | 10                                     | 5.39±0.13       | 4.40±0.11 | 3.83±0.09 | 3.66±0.16 | 2.85±0.35 | 2.85±0.10 | 2.98±0.14 |

\*In this and following tables, 1% w/v starch suspension was gelatinized at 85°C for 15 min and then cooled prior to viscosity determination at the given temperature.

Table 5.13. Viscosity as a Function of Temperature for Some  
Cassava Starches

| Starch*           | Duration of<br>Viscometer<br>Run, Min. | Temperature, °C |           |           |           |           |           |           |
|-------------------|--|-----------------|-----------|-----------|-----------|-----------|-----------|-----------|
|                   |  | 25              | 30        | 35        | 40        | 50        | 60        | 70        |
| <u>Cassava</u>    |  |                 |           |           |           |           |           |           |
| cv. Kenyan        | 5                                      | 6.95±0.22       | 4.86±0.16 | 3.38±0.06 | 3.45±0.11 | 3.23±0.04 | 2.75±0.02 | 2.71±0.13 |
|                   | 10                                     | 6.72±0.24       | 4.74±0.10 | 3.31±0.12 | 3.40±0.10 | 3.17±0.05 | 2.71±0.02 | 2.75±0.10 |
| cv. Fijian        | 5                                      | 7.86±0.24       | 6.65±0.27 | 6.34±0.09 | 5.31±0.13 | 4.59±0.18 | 4.01±0.19 | 3.78±0.27 |
|                   | 10                                     | 7.28±0.28       | 6.40±0.12 | 5.96±0.04 | 5.05±0.21 | 4.47±0.12 | 3.95±0.22 | 3.58±0.28 |
| Commercial Sample | 5                                      | 6.80±0.07       | 5.46±0.02 | 4.02±0.14 | 3.76±0.13 | 3.44±0.15 | 3.08±0.07 | 2.86±0.26 |
|                   | 10                                     | 6.30±0.14       | 5.24±0.03 | 3.99±0.16 | 3.72±0.08 | 3.36±0.15 | 3.04±0.01 | 3.01±0.22 |

\*In this and following tables, 1% w/v starch suspension was gelatinized at 85°C for 15 min and then cooled prior to viscosity determination at the given temperature.

Table 5.14. Viscosity as a Function of Temperature for Some Sweet Potato Starches

| Starch*             | Duration of<br>Viscometer<br>Run, Min. | Temperature, °C |           |           |           |           |           |           |
|---------------------|--|-----------------|-----------|-----------|-----------|-----------|-----------|-----------|
|                     |  | 25              | 30        | 35        | 40        | 50        | 60        | 70        |
| <u>Sweet Potato</u> |  |                 |           |           |           |           |           |           |
| cv. Centennial      | 5                                      | 4.35±0.13       | 4.25±0.06 | 4.10±0.07 | 3.81±0.04 | 3.18±0.19 | 2.34±0.09 | 1.85±0.07 |
|                     | 10                                     | 4.47±0.15       | 3.97±0.07 | 3.86±0.08 | 3.77±0.04 | 3.17±0.21 | 2.33±0.09 | 1.80±0.10 |
| cv. Porto Rico      | 5                                      | 3.66±0.09       | 3.60±0.01 | 3.44±0.11 | 3.01±0.05 | 2.39±0.14 | 2.00±0.05 | 1.75±0.19 |
|                     | 10                                     | 3.63±0.06       | 3.63±0.06 | 3.39±0.15 | 2.99±0.04 | 2.34±0.14 | 1.97±0.02 | 1.75±0.11 |
| cv. Georgia Red     | 5                                      | 4.63±0.02       | 4.35±0.05 | 4.00±0.08 | 3.55±0.08 | 3.21±0.11 | 2.89±0.16 | 2.65±0.13 |
|                     | 10                                     | 4.54±0.09       | 4.34±0.16 | 4.00±0.10 | 3.50±0.07 | 3.19±0.15 | 2.87±0.13 | 2.65±0.15 |

\*In this and following tables, 1% w/v starch suspension was gelatinized at 85°C for 15 min and then cooled prior to viscosity determination at the given temperature.

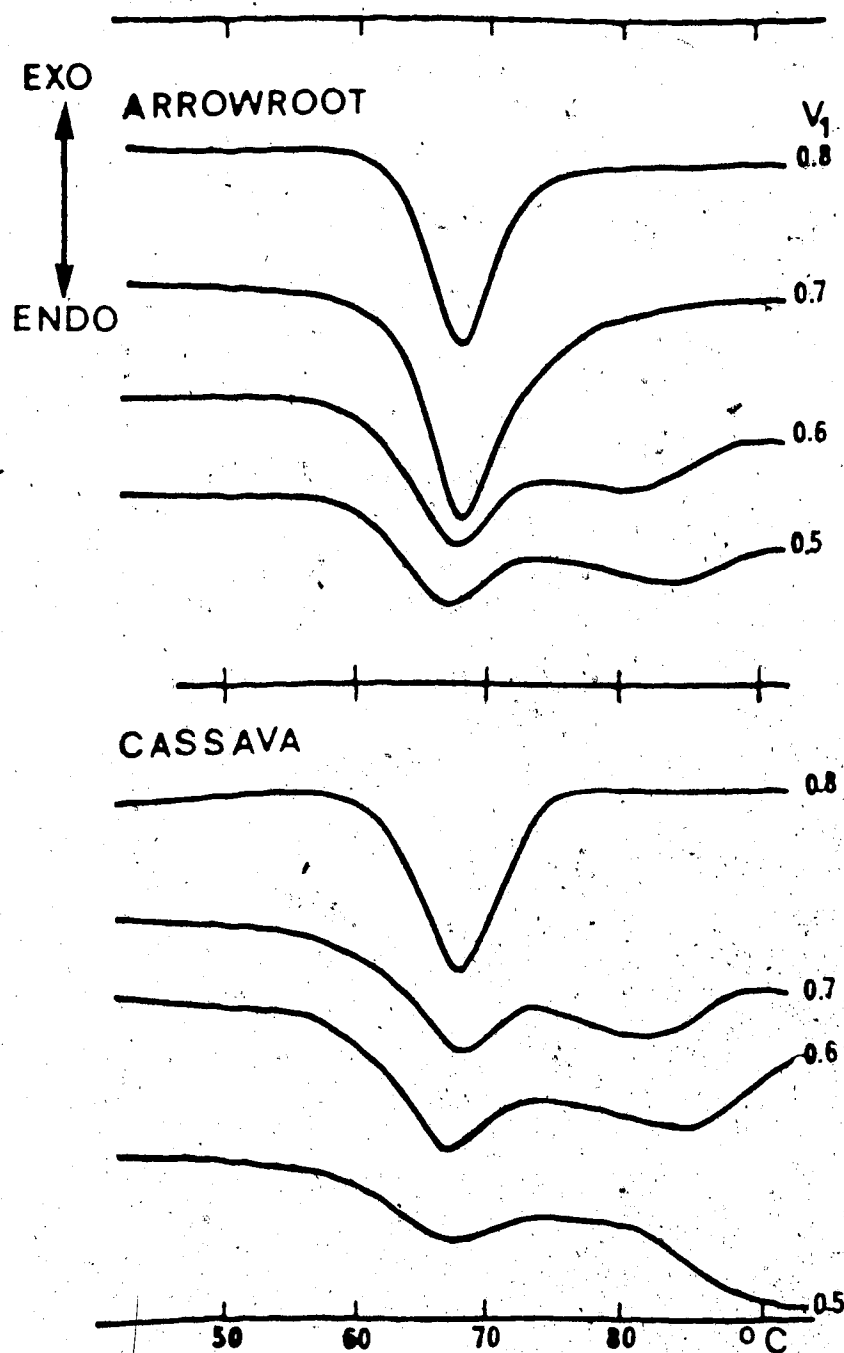


Figure 5.11. DSC-Thermograms for Arrowroot and Cassava Starches as a Function of Temperature and the Water Volume Fraction.

Table 5.16. DSC- Gelatinization Characteristics of Arrowroot and Cassava Starches as a Function of Temperature and Water Volume Fraction

| STARCH TYPE          | V <sub>WATER</sub> VOLUME FRACTION | (G) GELATINIZATION ENDOTHERM         |                                     |                                    |                   |           | (M <sub>1</sub> ) CRYSTALLITES ENDOTHERMS |                                     |                                    |                   |           |
|----------------------|------------------------------------|--------------------------------------|-------------------------------------|------------------------------------|-------------------|-----------|---|-------------------------------------|------------------------------------|-------------------|-----------|
|                      |                                    | <sup>o</sup> C<br>T <sub>ONSET</sub> | <sup>o</sup> C<br>T <sub>PEAK</sub> | <sup>o</sup> C<br>T <sub>END</sub> | CM <sup>2</sup> * | ΔH**      | <sup>o</sup> C<br>T <sub>ONSET</sub>      | <sup>o</sup> C<br>T <sub>PEAK</sub> | <sup>o</sup> C<br>T <sub>END</sub> | CM <sup>2</sup> * | ΔH**      |
| ARROWROOT            | 0.80                               | 62.25±0.29                           | 67.30±0.57                          | 72.25±0.65                         | 3.37±0.18         | 4.08±0.12 | -   | -                                   | -                                  | -                 | -         |
|                      | 0.70                               | 61.10±0.85                           | 67.98±0.39                          | 73.5±0.71                          | 4.08±0.68         | 4.93±0.22 | 71.0±0.35                                 | 76.75±0.35                          | 80.5±2.88                          | 0.55±0.10         | 0.67±0.05 |
|                      | 0.60                               | 60.25±0.35                           | 67.75±0.35                          | 71.75±0.35                         | 3.04±0.10         | 3.68±0.15 | 72.0±0                                    | 79.75±1.77                          | 87.75±3.10                         | 0.63±0.10         | 0.77±0.05 |
|                      | 0.50                               | 60.0±0                               | 67.13±0.13                          | 73.0±0.71                          | 2.17±0.24         | 2.63±0.27 | 73.0±0.0                                  | 83.75±1.10                          | 95.25±3.9                          | 1.53±0.22         | 1.85±0.05 |
|                      | 0.40                               | 60.0±0                               | 67.25±0.21                          | 73.0±0                             | 1.77±0.10         | 2.14±0.11 | 74.0±0                                    | 87.5±2.8                            | 97.5±0.0                           | 1.41±0.30         | 1.71±0.05 |
|                      | 0.30                               | -                                    | -                                   | -                                  | -                 | -         | 91.5±2.50                                 | 119.0±3.9                           | 140±5.0                            | 7.0±2.0           | 8.47±0.10 |
| CASSAVA (KENYAN CV.) | 0.80                               | 52.89±0.88                           | 60.75±0.35                          | 71.25±0.53                         | 3.79±0.31         | 4.59±0.03 | -   | -                                   | -                                  | -                 | -         |
|                      | 0.70                               | 53.38±0.18                           | 58.25±0.35                          | 65.25±1.06                         | 4.05±1.25         | 4.90±0.05 | 67±0.71                                   | 75.75±0.35                          | 80.75±0.35                         | 0.65±0.03         | 0.79±0.05 |
|                      | 0.60                               | 53.25±1.06                           | 58.34±0.53                          | 64.0±0.35                          | 3.10±0.31         | 3.75±0.08 | 70.75±.35                                 | 82.25±6.72                          | 89.25±5.3                          | 3.63±0.16         | 4.20±0.09 |
|                      | 0.50                               | 52.0±0.71                            | 58.0±0.35                           | 65.0±0.71                          | 1.97±0.42         | 2.38±0.10 | 74.0±1.41                                 | 88.5±4.24                           | 96.63±4.10                         | 1.56±0.06         | 1.89±0.03 |
|                      | 0.40                               | 54.5±0                               | 66.75±3.18                          | 77.0±3.89                          | 1.73±0.66         | 2.09±0.05 | 80.25±3.89                                | 106.25±6.0                          | 124.75±0.35                        | 6.23±0.22         | 7.54±0.07 |
|                      | 0.30                               | 60.2±1.5                             | 79.0±2.0                            | 88.0±2.5                           | 1.38±0.50         | 1.67±0.17 | 89.5±0                                    | 131.75±5.3                          | 139.03±3.57                        | 8.48±0.45         | 9.87±0.11 |

\*ENDOTHERM AREA.

\*\*ENTHALPY OF FUSION (cal/g STARCH).



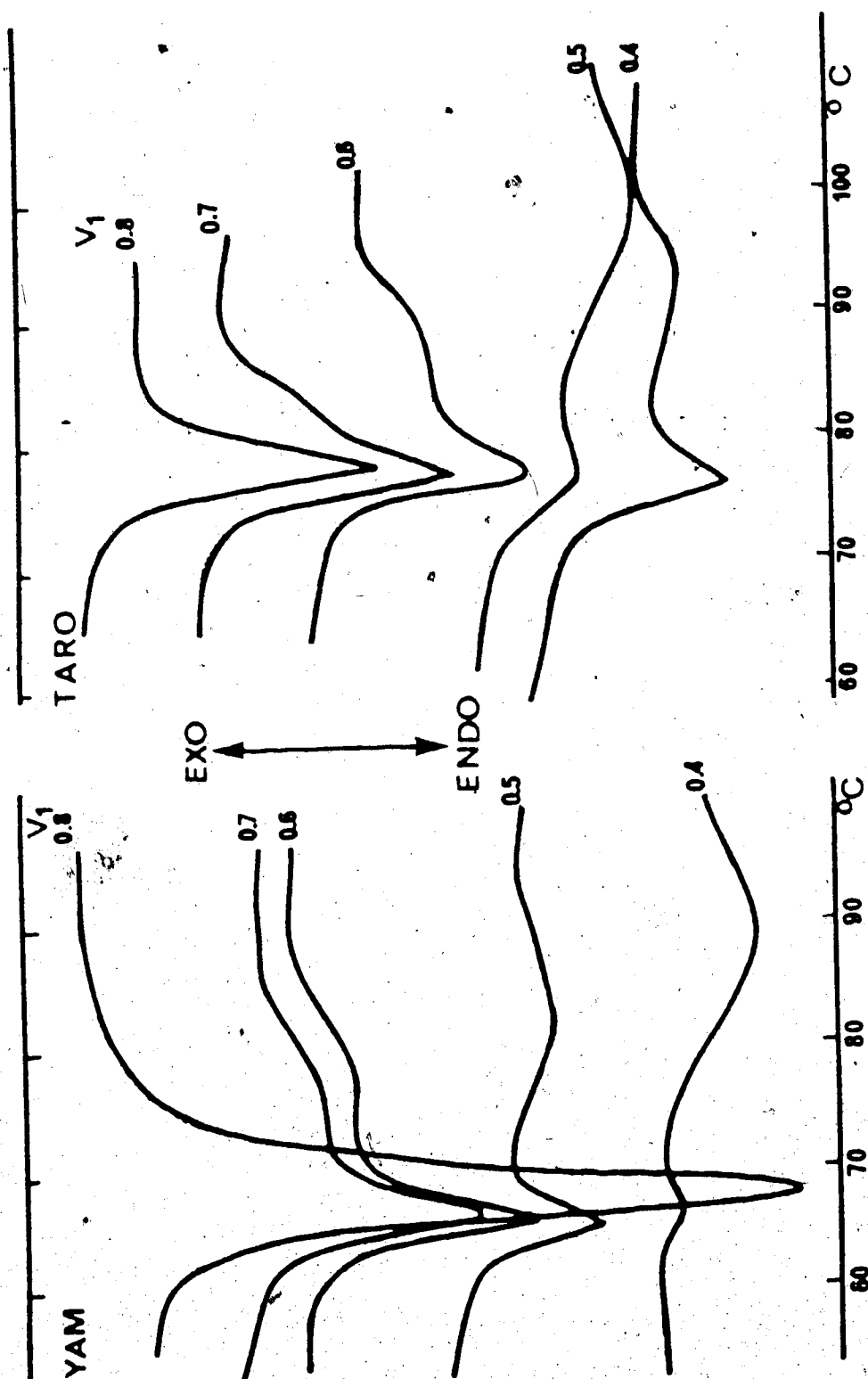


Figure 5.12. DSC - Thermograms of Yam and Taro Starches as a Function of Temperature and the Water Volume Fraction

Table 5.17. DSC- Gelatinization Characteristics of Yam and Taro Starches as a Function of Temperature and Water Volume Fraction.

| STARCH TYPE | V <sub>WATER</sub> VOLUME FRACTION | (G) GELATINIZATION ENDOTHERM |                         |                        |                   |           | (M <sub>1</sub> ) CRYSTALLITES ENDOTHERMS |                         |                        |                   |           |
|-------------|------------------------------------|------------------------------|-------------------------|------------------------|-------------------|-----------|---|-------------------------|------------------------|-------------------|-----------|
|             |                                    | °C<br>T <sub>ONSET</sub>     | °C<br>T <sub>PEAK</sub> | °C<br>T <sub>END</sub> | CM <sup>2</sup> * | ΔH**      | °C<br>T <sub>ONSET</sub>                  | °C<br>T <sub>PEAK</sub> | °C<br>T <sub>END</sub> | CM <sup>2</sup> * | ΔH**      |
| YAM         | 0.80                               | 62.75±0.35                   | 67.42±0.63              | 71.88±1.59             | 4.88±0.93         | 5.91±0.50 | -   | -                       | -                      | -                 | -         |
|             | 0.70                               | 62.5±0                       | 66.5±0                  | 70.0±0                 | 3.77±0.01         | 4.56±0.19 | 70.0±0                                    | 76.25±0                 | 85.5±0                 | 3.12±0.13         | 3.78±0.40 |
|             | 0.60                               | 62.5±0                       | 65.75±0                 | 69.5±0                 | 3.20±1.0          | 3.87±0.25 | 69.75±0                                   | 78.0±0                  | 87.5±0.35              | 3.47±0.35         | 4.20±0.08 |
|             | 0.50                               | 61.17±0.58                   | 65.0±0.5                | 68.5±0                 | 1.81±1.0          | 2.19±0.14 | 70.75±0.35                                | 77.17±3.33              | 91.0±0                 | 1.44±.43          | 1.75±0.35 |
|             | 0.40                               | 60.5±0                       | 64.0±0                  | 67.5±0                 | 0.31±0            | 0.37±0    | 73.25±0.25                                | 87.0±0                  | 99.0±0                 | 3.41±0.04         | 4.12±0.11 |
|             | 0.30                               | -                            | -                       | -                      | -                 | -         | 89.5±0                                    | 103.7±3.01              | 112.5±2.30             | 4.65±0.52         | 5.62±0.25 |
| TARO        | 0.80                               | 75.25±0.25                   | 78.5±0.25               | 83.25±0.25             | 4.37±0.21         | 5.29±0.25 | -   | -                       | -                      | -                 | -         |
|             | 0.70                               | 74.50±0.71                   | 78.0±0.50               | 83.75±0.25             | 5.28±0.35         | 6.38±0.35 | 81.5±0                                    | 86.5±0                  | 96.5±0                 | 1.25±0.05         | 1.51±0.08 |
|             | 0.60                               | 74.50±0                      | 78.5±0                  | 84.0±0.5               | 4.72±0.40         | 5.71±0.52 | 82.0±0                                    | 90.0±0                  | 95.0±0                 | 2.71±0.15         | 3.71±0.20 |
|             | 0.50                               | 74.5±0.25                    | 79.0±0                  | 83.25±1.1              | 2.00±0            | 3.97±0    | 83.5±1.0                                  | 101.0±5.57              | 109.0±0                | 3.11±0.27         | 3.88±0.05 |
|             | 0.40                               | -                            | 79±0                    | 87.5±0                 | 1.76±0.07         | 2.12±0.09 | 81.5±0                                    | 117.25±3.18             | 123.25±3.9             | 3.59±0.25         | 4.34±0.15 |
|             | 0.30                               | -                            | -                       | -                      | -                 | -         | 95.5±0                                    | 136.0±0                 | 146.0±0                | 5.19±1.01         | 6.28±0.19 |

\*ENDOTHERM AREA.

\*\*ENTHALPY OF FUSION (cal/g STARCH).

Figure 5.13. DSC-Thermograms of Wheat, cv. Neepawa Starch as a Function of the Temperature and the Water Volume Fraction.

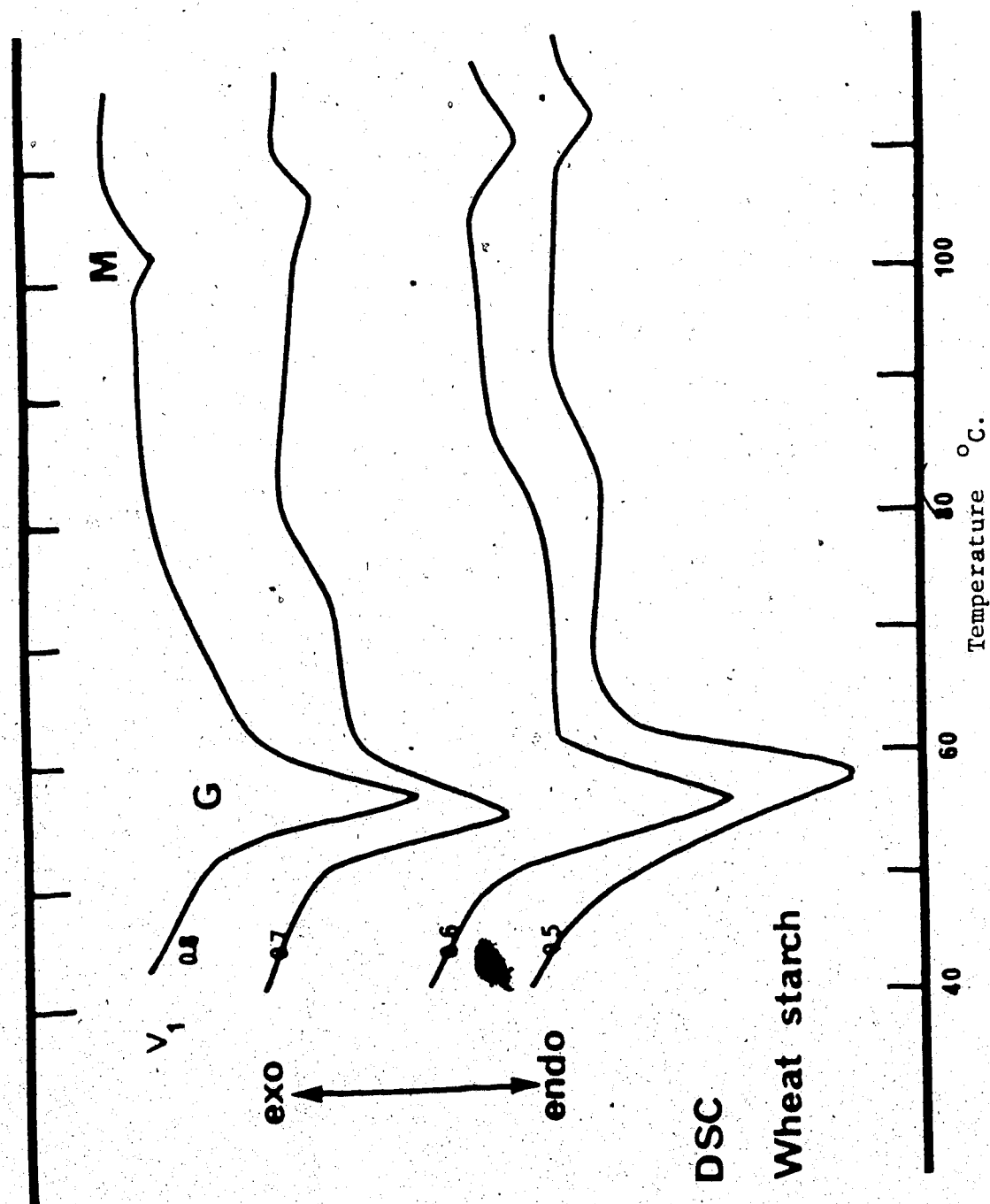


Table 5.18. DSC - Gelatinization Characteristics of Wheat,  
cv. Neepawa Starch as a Function of the Temperature and the  
Water Volume Fraction

| Endotherms         |                 |                        |                       |                      |                               |                        |                       |                      |                 |
|--------------------|-----------------|------------------------|-----------------------|----------------------|-------------------------------|------------------------|-----------------------|----------------------|-----------------|
| Gelatinization (g) |                 |                        |                       |                      | Crystallite (m <sub>l</sub> ) |                        |                       |                      |                 |
| Type of Starch     | Volume fraction | t <sup>o</sup> C Onset | t <sup>o</sup> C Peak | t <sup>o</sup> C End | ΔH/Cal/g*                     | t <sup>o</sup> C Onset | t <sup>o</sup> C Peak | t <sup>o</sup> C End | ΔH/Cal/g*       |
| Wheat:             | 0.8             | 51.04<br>+0.78         | 56.08<br>+0.74        | 62.04<br>+1.15       | 0.410<br>+0.005               | -                      | -                     | -                    | -               |
| cv. Neepawa        | 0.7             | 50.12<br>+1.21         | 55.36<br>+1.18        | 61.22<br>+1.79       | 0.200<br>+0.003               | 62.75<br>+0.35         | 75.50<br>+4.95        | 83.00<br>+0.59       | 0.110<br>+0.050 |
|                    | 0.6             | 49.63<br>+0.50         | 56.40<br>+0.50        | 62.17<br>+2.02       | 0.590<br>+0.004               | 65.25<br>+0.35         | 77.75<br>+0.35        | 91.75<br>+1.77       | 0.180<br>+0.040 |
|                    | 0.5             | 50.50<br>+0.01         | 56.00<br>+0.02        | 58.75<br>+0.35       | 0.220<br>+0.008               | 73.5<br>+1.4           | 84.101<br>+2.60       | 101.00<br>+3.30      | 0.210<br>+0.020 |
|                    | 0.4             | 47.75<br>+1.41         | 52.25<br>+0.00        | 58.75<br>+0.09       | 0.05<br>+0.003                | 84.0<br>+7.8           | 102.30<br>+1.06       | 112.25<br>+2.50      | 0.260<br>+0.030 |
|                    | 0.3             | --                     | --                    | --                   | --                            | --                     | --                    | --                   | --              |

\*Enthalpy of Fusion.

Table 5.19. DSC- Wheat Starch-Lipid Clathrates' Melting Characteristics as a Function of the Temperature and the Water Volume Fraction.

| Wheat: Water<br>Volume<br>Fraction<br>(V <sub>1</sub> ) |     | Clathrate Endotherms (m <sub>2</sub> ) |                          |                         |                 |
|---|-----|--|--------------------------|-------------------------|-----------------|
| cv.<br>Fielder  |     | t <sup>o</sup> C<br>Onset              | t <sup>o</sup> C<br>Peak | t <sup>o</sup> C<br>End | ΔH<br>Cal/g.*   |
|   | 0.8 | 93.25<br>± 3.89                        | 98.75<br>± 3.89          | 103.50<br>± 4.95        | 0.023<br>±0.001 |
|   | 0.7 | 97.75<br>± 3.18                        | 103.25<br>± 3.89         | 107.75<br>± 3.18        | 0.041<br>±0.003 |
|   | 0.6 | 105.75<br>± 1.06                       | 111.75<br>± 0.45         | 115.75<br>± 1.06        | 0.058<br>±0.005 |
|   | 0.5 | --                                     | --                       | --                      | --              |
|   | 0.4 | 122.25<br>± 1.78                       | 128.75<br>± 1.06         | 132.00<br>± 0.10        | 0.060<br>±0.004 |
|   | 0.3 | --                                     | --                       | --                      | --              |
| <hr/>   |     |  |                          |                         |                 |
| cv.<br>Neepawa.   |     |  |                          |                         |                 |
|   | 0.8 | 97.50<br>± 1.00                        | 100.50<br>± 1.00         | 105.00<br>± 2.00        | 0.160<br>±0.010 |
|   | 0.7 | 97.50<br>± 1.00                        | 100.50<br>± 1.00         | 106.50<br>± 1.00        | 0.220<br>±0.010 |
|   | 0.6 | 107.00<br>± 1.00                       | 114.50<br>± 1.50         | 120.50<br>± 2.50        | --              |
|   | 0.5 | --                                     | --                       | --                      | --              |
|   | 0.4 | --                                     | --                       | --                      | --              |
|   | 0.3 | --                                     | --                       | --                      | --              |

\* Enthalpy of Fusion

Table 5.20 Comparison of Gelatinization Characteristics of Some Wheat and Tropical Root Starches

| Type of Starch | Onset*           | Peak*            | End              | Enthalpy* of Fusion<br>cal/g starch |
|----------------|------------------|------------------|------------------|-------------------------------------|
| Wheat          |                  |                  |                  |                                     |
| cv. Neepawa    | 50.08 $\pm$ 0.91 | 55.18 $\pm$ 0.82 | 60.90 $\pm$ 2.02 | 0.66 $\pm$ 0.10                     |
| cv. Fielder    | 50.12 $\pm$ 1.21 | 55.36 $\pm$ 1.18 | 61.22 $\pm$ 1.79 | 0.20 $\pm$ 0.31                     |
| Cassava        |                  |                  |                  |                                     |
| Kenyan cv.     | 53.20 $\pm$ 0.90 | 58.84 $\pm$ 1.28 | 66.38 $\pm$ 2.90 | 4.90 $\pm$ 0.05                     |
| Fijian cv.     | 59.16 $\pm$ 2.61 | 65.42 $\pm$ 1.92 | 74.54 $\pm$ 2.80 | 5.09 $\pm$ 0.81                     |
| Sweet Potato   |                  |                  |                  |                                     |
| Porto Rico     | 52.72 $\pm$ 1.89 | 57.92 $\pm$ 0.59 | 65.50 $\pm$ 0.71 | 3.49 $\pm$ 0.08                     |
| Centennial cv. | 53.63 $\pm$ 1.01 | 61.58 $\pm$ 1.69 | 70.16 $\pm$ 2.59 | 2.59 $\pm$ 0.20                     |
| Georgia Red    | 62.02 $\pm$ 0.81 | 66.47 $\pm$ 1.14 | 72.20 $\pm$ 3.08 | 3.32 $\pm$ 0.86                     |
| Arrowroot      |                  |                  |                  |                                     |
| Kenyan cv.     | 60.72 $\pm$ 0.97 | 67.48 $\pm$ 0.36 | 72.70 $\pm$ 1.79 | 4.93 $\pm$ 0.22                     |
| Yam            | 61.88 $\pm$ 0.99 | 65.73 $\pm$ 1.32 | 69.48 $\pm$ 1.65 | 4.56 $\pm$ 0.19                     |
| Taro           | 74.69 $\pm$ 0.38 | 78.60 $\pm$ 0.42 | 84.35 $\pm$ 1.79 | 6.38 $\pm$ 0.35                     |

\*At water fraction = 0.7.

SWSW had similar endotherms, with an onset temperature of 50°C, a peak of 55.3°C, and an end of 61°C.

Root starches had higher enthalpies of fusion than root starches ranging from 0.3 cal/g starch DWB for  $v_1=0.4$  in yam to 6.38 cal/g starch in taro at  $v_1=0.7$  for the "G" endotherm. The enthalpy of fusion was highest in arrowroot and taro starches, indicating stronger forces within their granules than in other starches. Low enthalpy was found for the wheat starches; 0.66 and 0.20 cal/g of starch, DWB, for cvs. Neepawa and Fielder respectively.

The gelatinization endotherm "G" was shouldered by crystallite melting endotherm  $M_1$  between  $v_1=0.3-0.7$ . The  $M_1$  endotherm became greater at the expense of the "G" endotherm as  $v_1$  decreased.

The  $M_1$  onset, peak and end of gelatinization temperatures were irregular and not as well defined as for the "G" endotherm. Wheat starches, but not root starches, had an additional endotherm  $M_2$  near 100°C, revealing the presence of starch-lipid clathrates (Figure 5.13, and Table 5.19). Onset, peak and end of gelatinization temperatures for the  $M_2$  were also irregular (Table 5.19). Its enthalpy of fusion was small, ranging from 0.023 to 0.06 cal/g of starch, DWB for cv. Fielder and 0.16 to 0.22 for cv. Neepawa for  $v_1=0.7$ .

Data in Table 5.20 compare the gelatinization endotherm characteristics of wheat and root starches. Cassava starch of Kenyan origin and sweet potato starch, cv. Porto rico,

had gelatinization properties close to wheat starch.

#### 5.2.4 Retrogradation of Gels

##### 5.2.4.1 DSC Thermograms

Retrogradation of starch gels, as revealed by DSC thermograms for arrowroot, cassava, sweet potato, taro and wheat (cvs. Fielder and Neepawa) is presented in Figures 5.14-5.16.

It was observed that 60% fresh gels did not have any DSC endotherms. The root starches after two days of storage showed the presence of an endotherm between 50-60°C. The intensity of the endotherm increased with aging time (Figures 5.14-5.15). The wheat starches did not show any endotherm development until after the 6th day in storage at 24°C (Figure 5.16).

Arrowroot, cassava and sweet potato gels exhibited a second set of endotherms between 90-100°C for arrowroot, 78-83°C for cassava and 75-80°C for sweet potato. These were absent in gels of taro, yam and wheat starches.

##### 5.2.4.2 X-Ray Diffraction Patterns

X-ray diffractograms for cassava and wheat (cv. Neepawa) starch gels in Figures 5.17 and 5.18 show that the X-ray diffraction patterns became sharper and more defined with storage time.



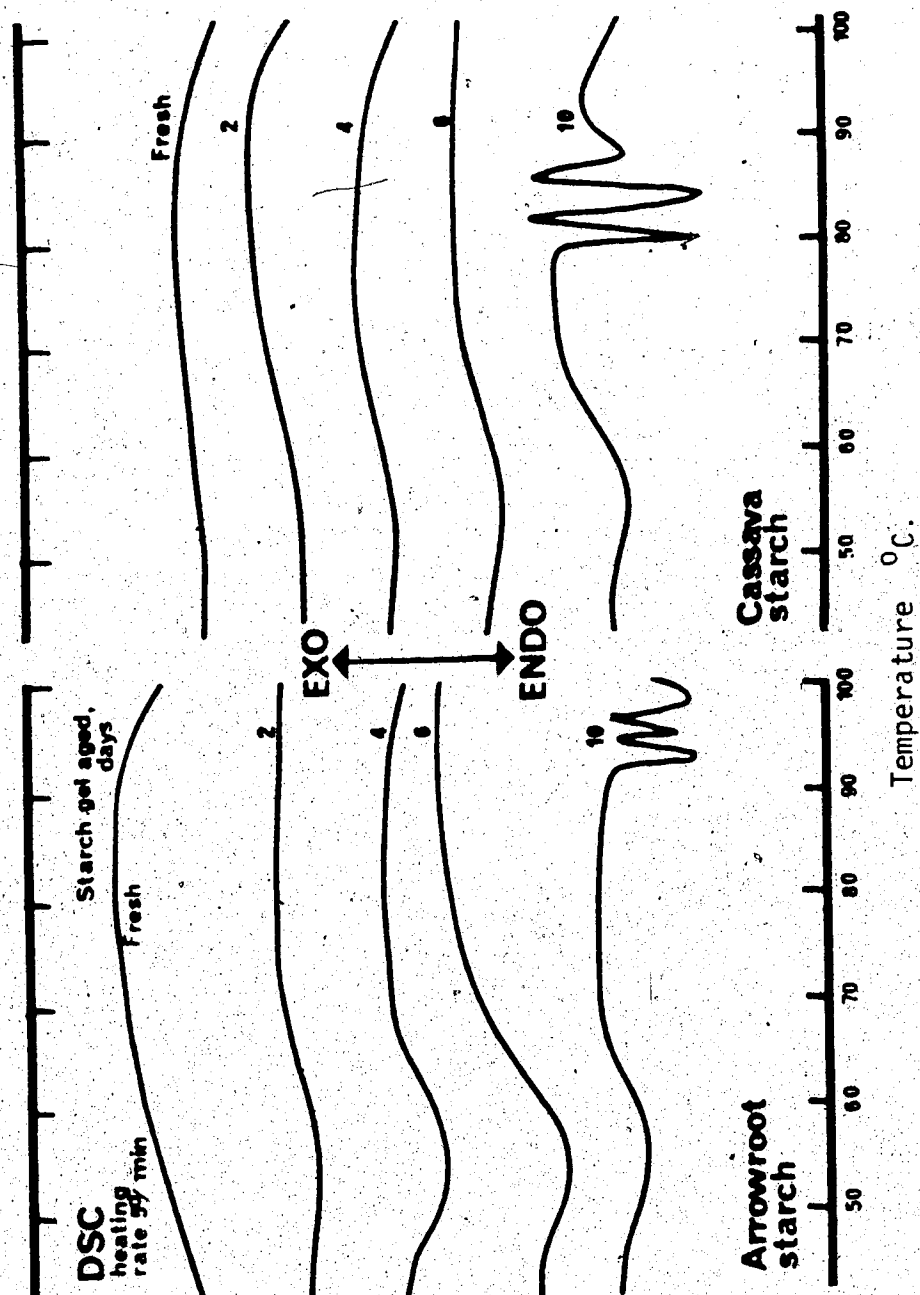


Figure 5.14. DSC- Thermograms of Arrowroot and Cassava Fresh and Aged Starch Gels.

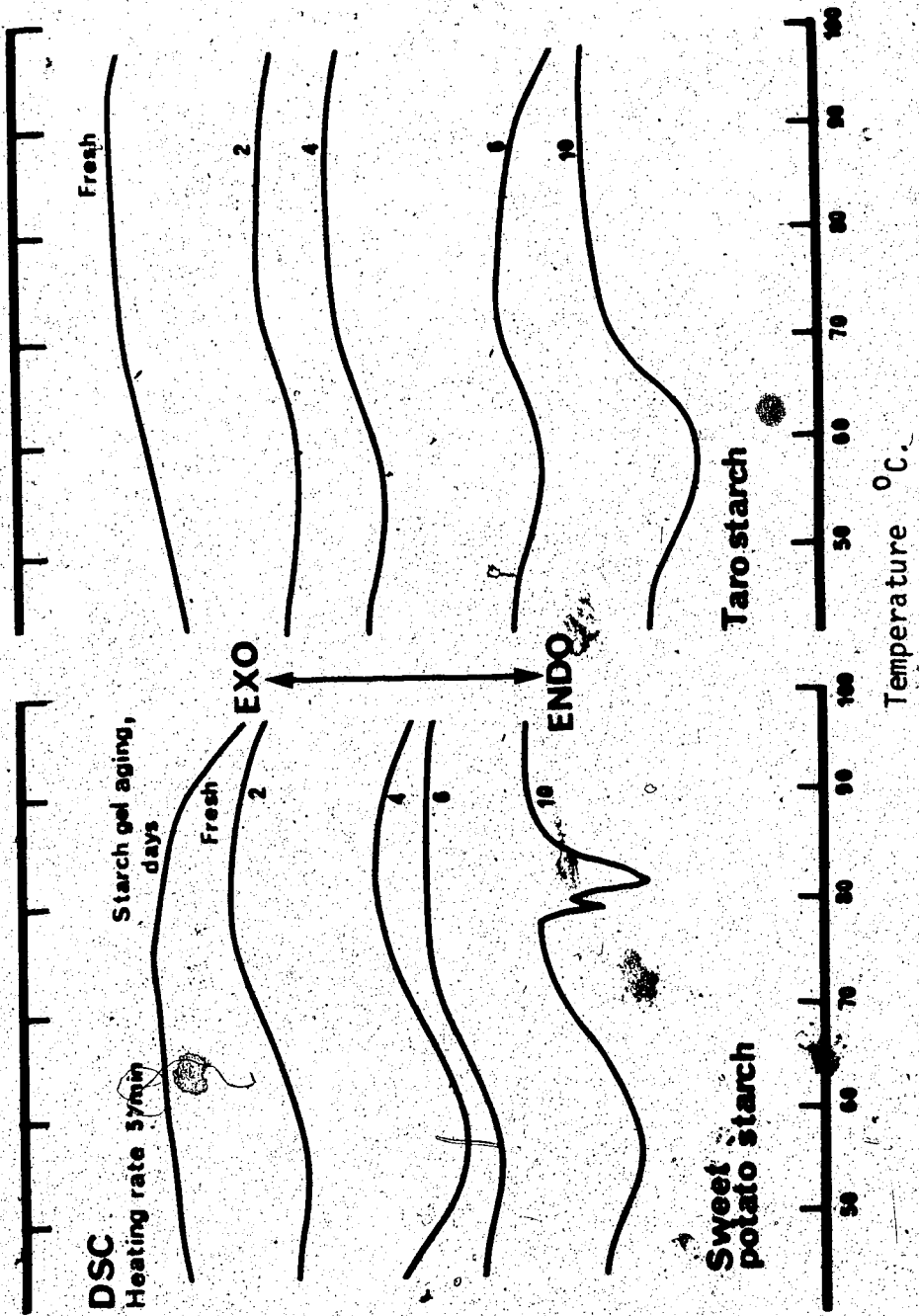


Figure 5.15. DSC- Thermograms of Sweet Potato and Taro Fresh and Aged Starch Gels.

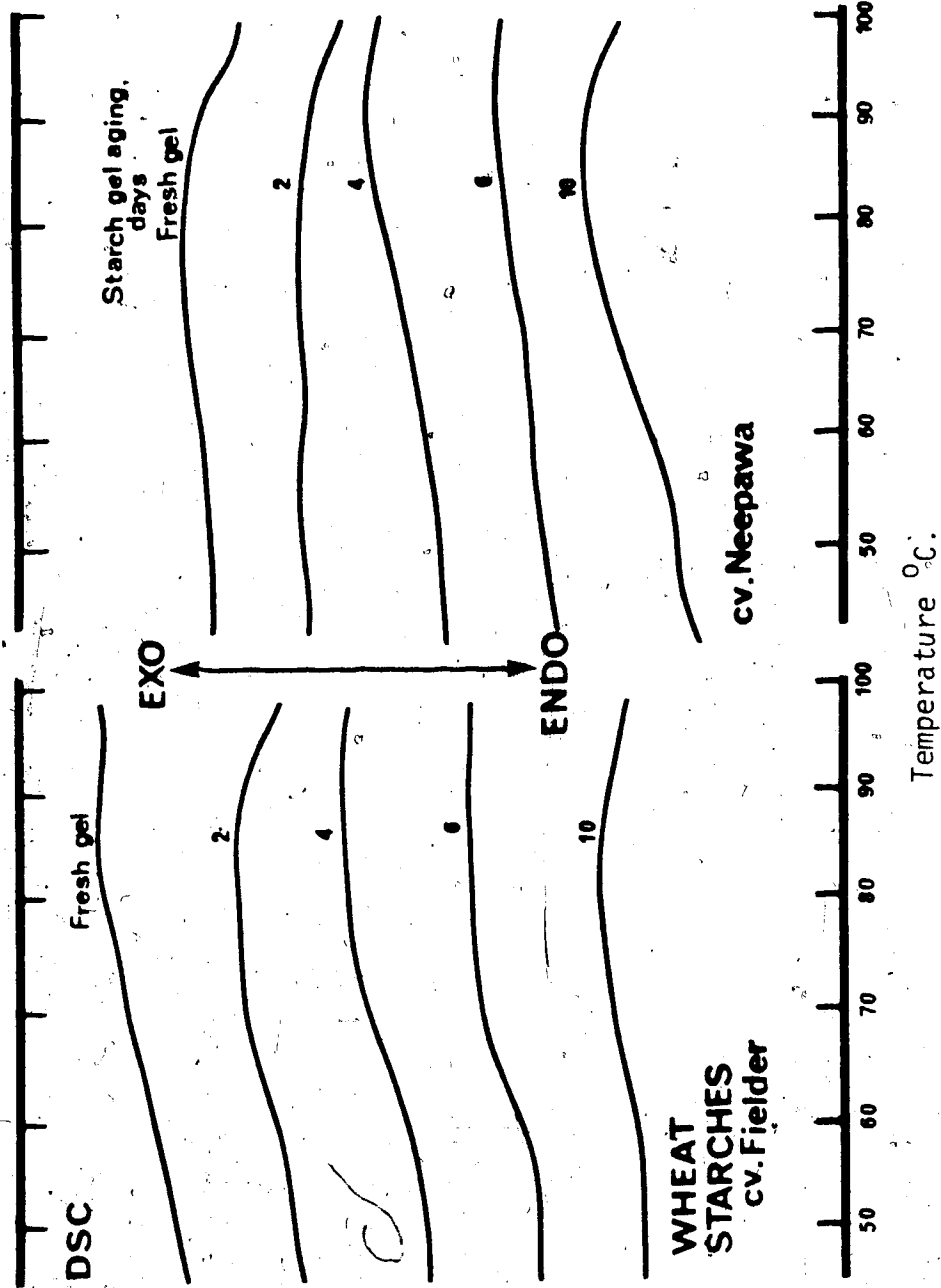


Figure 5.16. Dsc-Thermograms of Wheat, cvs. Fielder and Neepawa Fresh and Aged Starch Gels.

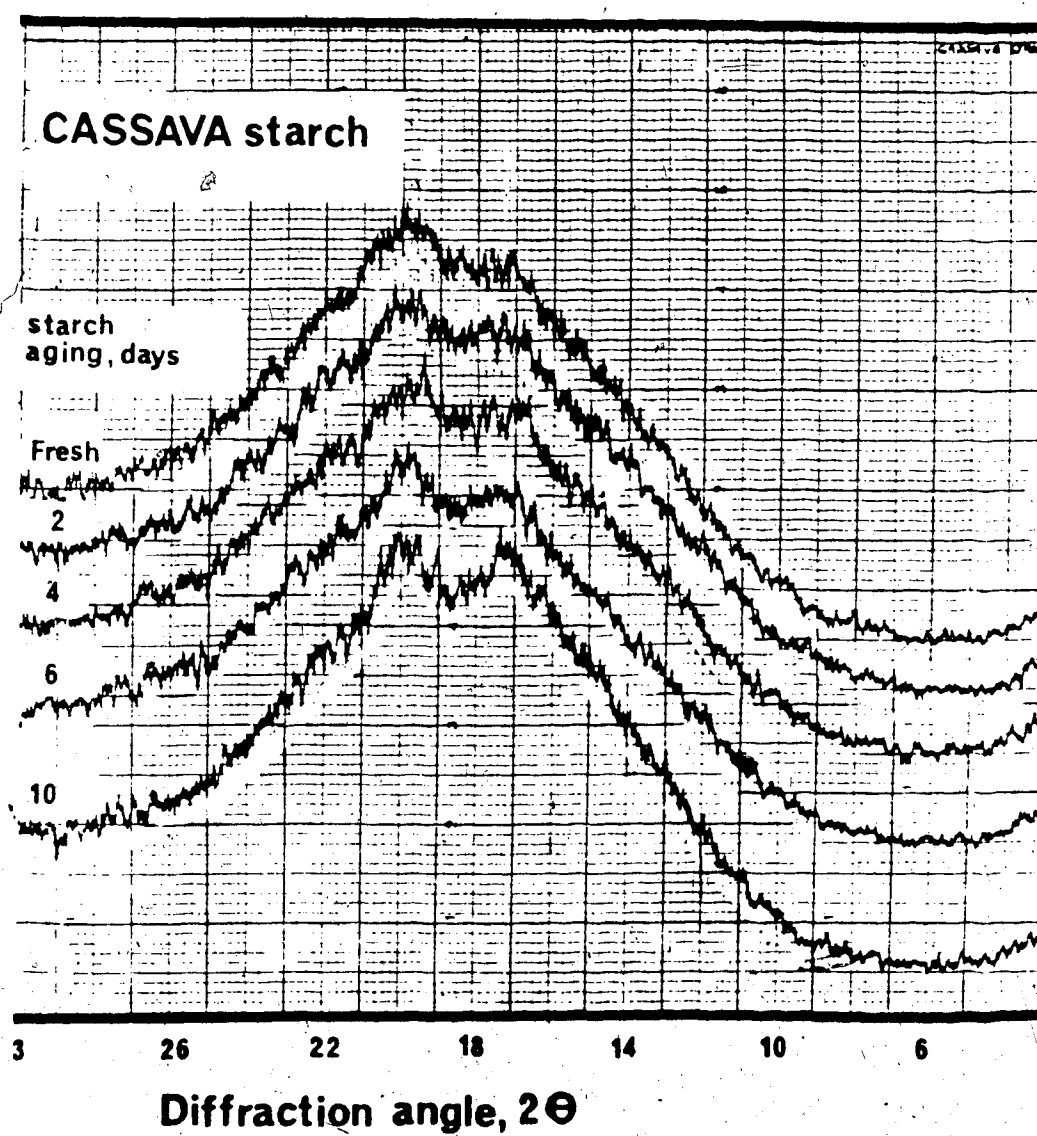


Figure 5.17 X-Ray Diffractograms of Gelatinized Starch Gels as a Function of Storage Time

Table 5.21. X-Ray Diffraction Patterns of Gelatinization and  
Aged Kenyan Cassava Starch

| Fresh        |        | Starch Gel Aged, Days |            |        |                  |            |        |                  |            |        |                  |
|--------------|--------|-----------------------|------------|--------|------------------|------------|--------|------------------|------------|--------|------------------|
|              |        | 2                     |            | 4      |                  | 6          |        | 10               |            |        |                  |
| 2 $\theta$ * | d, Å** | Intensity<br>cps***   | 2 $\theta$ | d, Å   | Intensity<br>cps | 2 $\theta$ | d, Å   | Intensity<br>cps | 2 $\theta$ | d, Å   | Intensity<br>cps |
| 9.917        | 8.9188 | 123                   | 12.265     | 7.2160 | 202              | 12.460     | 7.1037 | 201              | 17.170     | 5.1644 | 398              |
| 13.244       | 6.6848 | 256                   | 14.868     | 5.9581 | 320              | 16.801     | 5.2767 | 401              | 19.918     | 4.4575 | 436              |
| 17.075       | 5.1926 | 419                   | 16.783     | 5.2824 | 397              | 17.634     | 5.0293 | 407              | 21.480     | 4.1369 | 373              |
| 19.361       | 4.5845 | 462                   | 17.781     | 4.9882 | 418              | 19.392     | 4.5772 | 418              | 22.664     | 3.9232 | 338              |
| 19.792       | 4.4857 | 427                   | 19.698     | 4.5068 | 435              | 20.207     | 4.3944 | 402              |            |        |                  |
| 23.302       | 3.8173 | 325                   | 21.985     | 4.0429 | 365              | 21.761     | 4.0840 | 358              |            |        |                  |
| 28.540       | 3.1275 | 221                   | 23.985     | 3.7101 | 287              | 23.445     | 3.7943 | 292              |            |        |                  |
|              |        |                       | 27.356     | 3.2601 | 217              | 25.523     | 3.4899 | 231              |            |        |                  |
|              |        |                       |            |        |                  | 26.561     | 3.3559 | 224              |            |        |                  |

\*2 $\theta$ , Two theta degrees of the angle of diffraction.

\*\*d, Interplanar spacings in Angstrom units.

\*\*\*Diffraction line intensity in cycles per second.

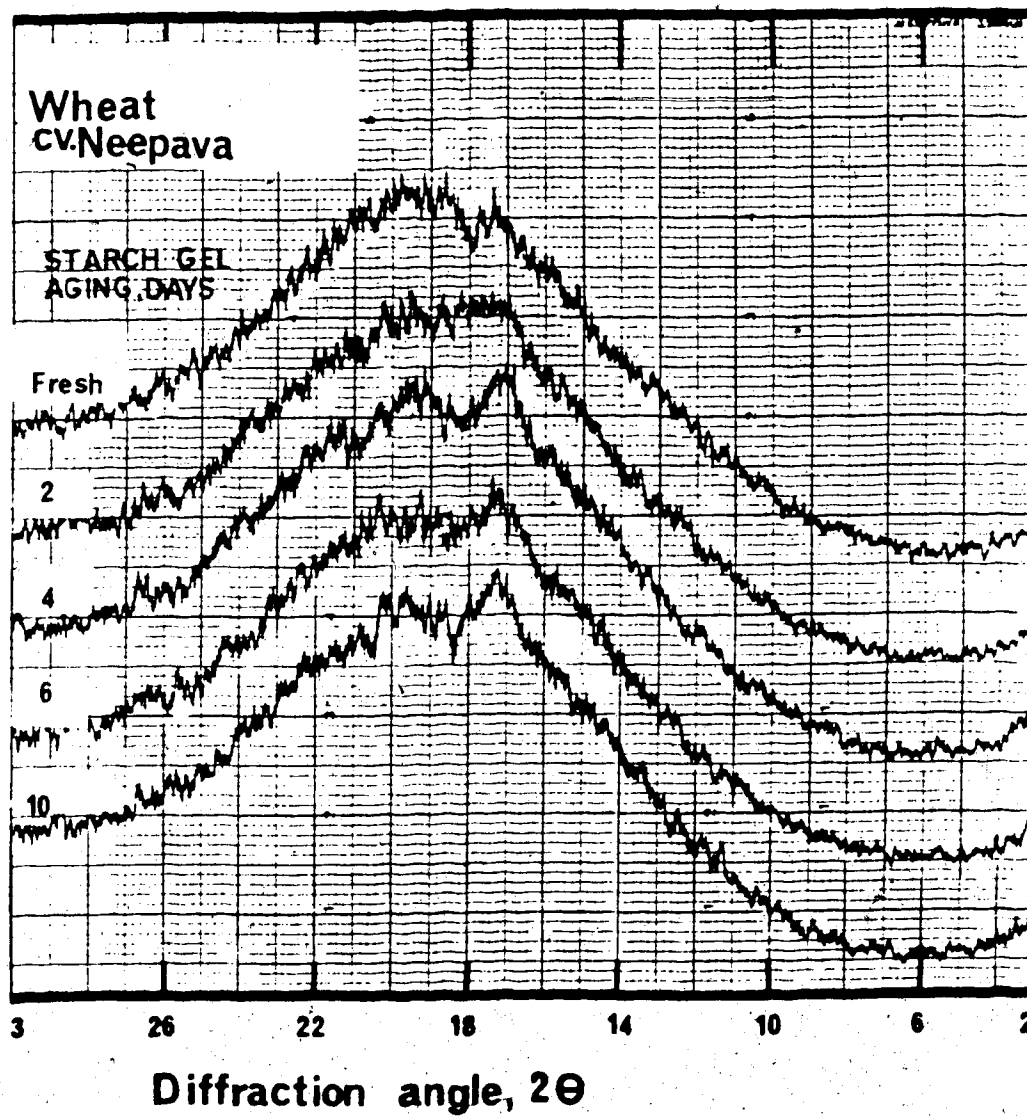


Figure 5.18. X-Ray Diffractograms of Gelatinized Wheat, cv. Neepawa Starch Gels as a Function of Storage Time

Table 5.22. X-Ray Diffraction Patterns of Gelatinized and  
Aged Wheat, cv. Neepawa, Starch

| Starch Gel Aged, Days |        |                  |            |        |               |            |         |               |            |        |               |            |        |               |
|-----------------------|--------|------------------|------------|--------|---------------|------------|---------|---------------|------------|--------|---------------|------------|--------|---------------|
| Fresh                 |        |                  | 2          |        |               | 4          |         |               | 6          |        |               | 10         |        |               |
| 2 $\theta$ *          | d, Å** | Intensity cps*** | 2 $\theta$ | d, Å   | Intensity cps | 2 $\theta$ | d, Å    | Intensity cps | 2 $\theta$ | d, Å   | Intensity cps | 2 $\theta$ | d, Å   | Intensity cps |
| 10.266                | 8.6165 | 143              | 12.416     | 7.1290 | 203           | 5.471      | 16.1519 | 82            | 12.715     | 6.9620 | 207           | 14.377     | 6.1604 | 332           |
| 15.904                | 5.5724 | 353              | 15.443     | 7.376  | 330           | 14.232     | 6.2229  | 290           | 16.942     | 5.2331 | 387           | 15.132     | 5.8550 | 380           |
| 17.395                | 5.0981 | 408              | 17.008     | 5.2131 | 408           | 17.114     | 5.1809  | 442           | 17.445     | 5.0835 | 385           | 17.040     | 5.2033 | 451           |
| 18.843                | 4.7094 | 416              | 19.293     | 4.6004 | 407           | 19.019     | 4.6661  | 426           | 20.263     | 4.3825 | 418           | 19.972     | 4.4457 | 444           |
| 19.579                | 4.5340 | 426              | 20.175     | 4.4013 | 409           | 20.265     | 4.3820  | 413           | 24.243     | 3.6712 | 278           | 22.086     | 4.0246 | 371           |
| 29.146                | 3.0638 | 214              | 21.739     | 4.0880 | 359           | 21.385     | 4.1549  | 381           | 28.235     | 3.1605 | 201           | 27.517     | 3.2414 | 234           |
|                       |        |                  | 23.638     | 3.7638 | 301           | 22.032     | 4.0343  | 365           |            |        |               |            |        |               |
|                       |        |                  |            |        |               | 26.464     | 3.3679  | 243           |            |        |               |            |        |               |

The data in Tables 5.21 and 5.22 show the diffraction angle in " $^{\circ}$  ( $2\theta$ )", the interplanar spacings " $d$ " in " $\text{\AA}$ " and the intensities in cycles per second "cps" of the diffracted lines. Of characteristic interest were the peaks occurring in the neighborhood of diffraction angles  $17-19^{\circ}$  ( $2\theta$ ) and  $20-21^{\circ}$  ( $2\theta$ ).

### 5.2.5 Complexing with Monoglycerides

#### 5.2.5.1 Analysis of Monoglycerides

##### *Infrared Spectra of $C_{16}$ and $C_{18}$ $\alpha$ - and $\beta$ -Crystallinity Monoglycerides.*

Infrared spectra absorption bands of the  $\alpha$ - and  $\beta$ -Crystallinity form of  $C_{16}$  and  $C_{18}$  monoglycerides are presented in Figure 5.19 and Table 5.23.

The absorption bands specific to only  $\alpha$ - or  $\beta$ -crystallinity forms of monoglycerides are presented in Table 5.24.

According to Chapman (1965), different polymorphic types of 1-monoglycerides provide different infrared spectra. On the contrary, the 2<sup>nd</sup>-isomer monoglycerides have only one polymorphic form, hence there is no change in their infrared spectra.

Figure 5.19 shows that  $\alpha$ - and  $\beta$ -crystallinity forms of  $C_{16}$  and  $C_{18}$  monoglycerides have similar infrared spectra. The bands at  $1730-1740\text{ cm}^{-1}$  represent the carbonyl stretching of the ester groups. The bands at  $1460-1480\text{ cm}^{-1}$  represent  $-C-H$  bending of methylene and methyl groups.



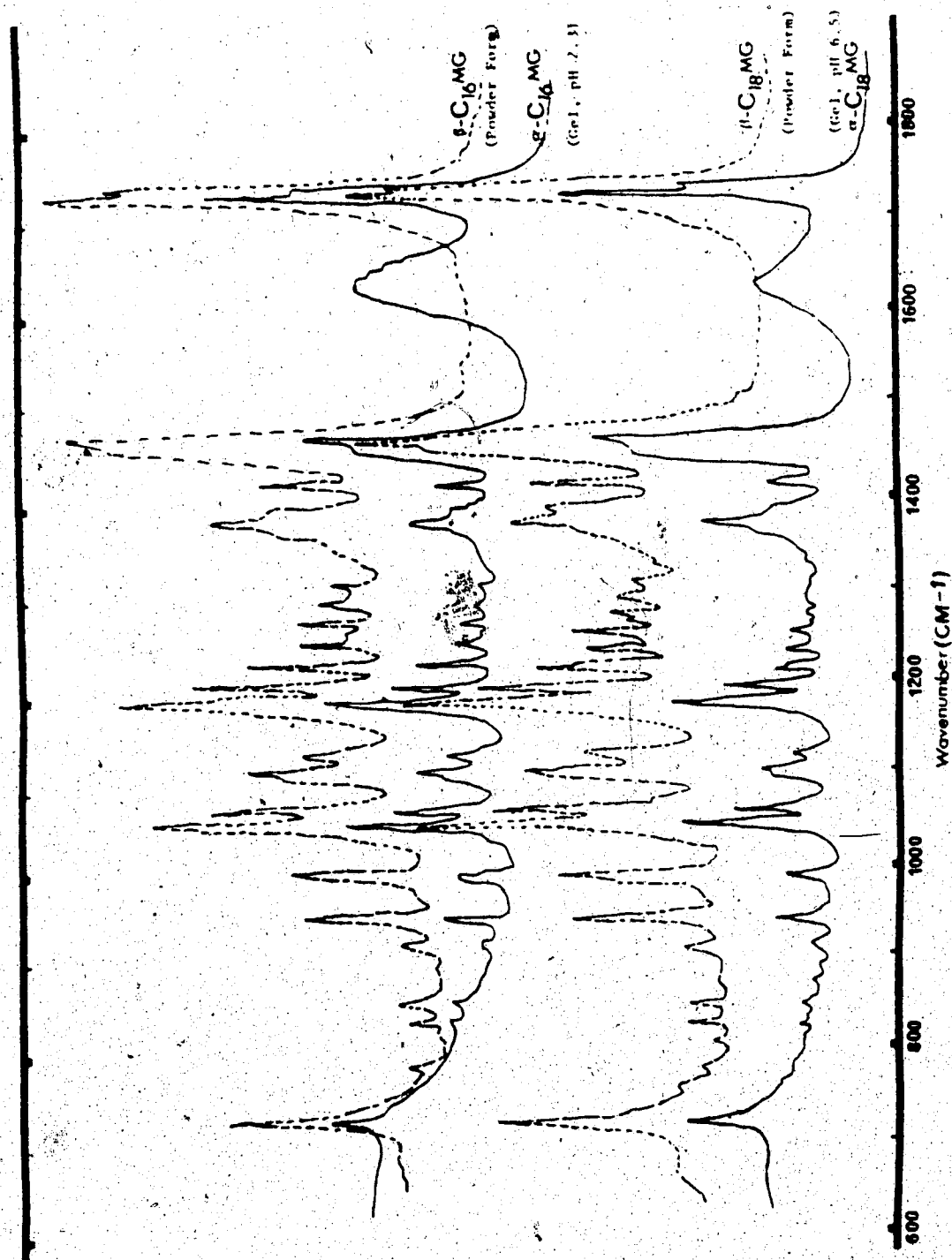


Figure 5.19. Infrared Spectra of The  $\alpha$ - and  $\beta$ -Crystallinity

Forms of the  $\text{C}_{16}$  and  $\text{C}_{18}$  Monoglycerides

| Crystallinity                          | Bands, $\text{cm}^{-1}$ |      |      |      |      |      |      |     |     |     |  |  |
|--|-------------------------|------|------|------|------|------|------|-----|-----|-----|--|--|
| $\alpha$ -C <sub>16</sub> , Gel pH 2.3 | 1737                    | 1630 | 1468 | 1375 | 1288 | 1181 | 1062 | 993 | 851 | 722 |  |  |
|  | 1728                    |      | 1415 | 1309 | 1277 | 1125 | 1048 | 945 | 831 |     |  |  |
|  |                         |      |      |      | 1244 | 1105 |      | 915 |     |     |  |  |
|  |                         |      |      |      | 1221 |      |      |     |     |     |  |  |
|  |                         |      |      |      | 1200 |      |      |     |     |     |  |  |
| $\alpha$ -C <sub>18</sub> , Gel pH 6.5 | 1739                    | 1630 | 1468 | 1375 | 1255 | 1197 | 1062 | 991 | 851 | 720 |  |  |
|  | 1729                    |      | 1415 |      | 1235 | 1180 | 1048 | 945 | 841 |     |  |  |
|  |                         |      |      |      | 1215 | 1125 |      | 915 |     |     |  |  |
|  |                         |      |      |      |      | 1105 |      |     |     |     |  |  |
| $\beta$ -C <sub>16</sub> , Microbeads  | 1736                    |      | 1468 | 1375 | 1288 | 1198 | 1062 | 992 | 851 | 782 |  |  |
|  | 1728                    |      | 1415 | 1308 | 1265 | 1180 | 1048 | 945 | 830 | 722 |  |  |
|  |                         |      |      |      | 1244 | 1122 |      | 915 |     |     |  |  |
|  |                         |      |      |      | 1220 | 1105 |      |     |     |     |  |  |
| $\beta$ -C <sub>18</sub> , Microbeads  | 1737                    |      | 1468 | 1390 | 1292 | 1195 | 1061 | 991 | 851 | 782 |  |  |
|  | 1728                    |      | 1415 | 1375 | 1272 | 1180 | 1048 | 944 | 830 | 721 |  |  |
|  |                         |      |      | 1329 | 1268 | 1122 |      | 912 | 810 |     |  |  |
|  |                         |      |      | 1310 | 1255 | 1105 |      |     |     |     |  |  |
|  |                         |      |      |      | 1242 |      |      |     |     |     |  |  |
|  |                         |      |      |      | 1235 |      |      |     |     |     |  |  |
|  |                         |      |      |      | 1215 |      |      |     |     |     |  |  |

Table 5.23. The Major Infrared Spectra Absorption Frequency

Bands of the  $\alpha$  and  $\beta$ -Crystallinity Forms of C<sub>16</sub> and C<sub>18</sub>

Monoglycerides.

Table 5.24. Monoglyceride Infrared Spectra Absorption Bands  
Specific to the  $\alpha$  or  $\beta$ -Crystallinity Forms

| Type C-16  |         |        |           | Type C-18  |         |        |           |
|------------|---------|--------|-----------|------------|---------|--------|-----------|
| $\alpha$ - | Bands   |        | $\beta$ - | $\alpha$ - | Bands   |        | $\beta$ - |
| Absent     | Present | Absent | Present   | Absent     | Present | Absent | Present   |
| 782        |         |        | 782       |            | 782     | 782    |           |
|            |         |        |           | 830        |         |        | 830       |
|            |         |        |           | 810        |         |        | 810       |
|            | 915     | 915    |           |            |         |        |           |
| 1198       |         |        | 1198      |            |         |        | 1292      |
|            |         |        |           | 1272       |         |        | 1272      |
|            |         |        |           | 1268       |         |        | 1268      |
|            | 1265    | 1265   |           |            |         |        |           |
|            | 1200    | 1200   |           |            |         |        |           |
|            |         |        |           | 1242       |         |        | 1242      |
|            |         |        |           | 1390       |         |        | 1390      |
|            |         |        |           | 1329       |         |        | 1329      |
|            |         |        |           | 1310       |         |        | 1310      |
|            | 1630    | 1630   |           |            | 1630    | 1630   |           |

Symmetrical C-H band of methyl groups occurs at  $1380\text{ cm}^{-1}$ . C-O stretching of primary and secondary -OH groups occur at  $1050$ ,  $1064$ , and  $1180\text{ cm}^{-1}$ . Bands ranging from  $940$ - $995\text{ cm}^{-1}$  are due to C-C single bond stretch. Rocking vibrations of the methylene group occur at  $720\text{ cm}^{-1}$ .

In spite of similarities, the  $\beta$ -crystallinity form is characterized by the C-O stretching of the primary -OH groups at  $1062\text{ cm}^{-1}$ , which is absent in  $\alpha$ -crystallinity form. The  $\alpha$ - and  $\beta$ - forms are also differentiated at  $1705\text{ cm}^{-1}$  due to carbonyl-water hydrogen bonding which is present only in the  $\alpha$ -form. Hence, the presence or absence of these bands can be used to distinguish between the two crystallinity forms of the monoglycerides.

#### *X-Ray Diffraction Patterns of $\alpha$ - and $\beta$ -Crystallinity Forms of $C_{12}$ and $C_{18}$ Monoglycerides*

The X-ray diffraction patterns of the monoglycerides are presented in Figure 5.20. Corresponding data are presented in Tables 5.25 and 5.26.

Medium peaks at  $6.048$  or  $6.141^\circ$  ( $2\theta$ ) and very strong ones at  $21.316$  or  $21.357^\circ$  ( $2\theta$ ) were characteristic of  $\alpha$ -crystallinity forms of  $C_{12}$  and  $C_{18}$  monoglycerides. The latter had an extra weak peak at  $5.312^\circ$  ( $2\theta$ ).

$\beta$ -  $C_{12}$  had only one weak peak at  $10.132^\circ$  ( $2\theta$ ), strong peaks at  $7.399^\circ$ ,  $512^\circ$  ( $2\theta$ ) and very strong peaks at  $19.452$  and  $22.601^\circ$  ( $2\theta$ ), shouldered by smaller, but very strong peaks at  $20.250$  and  $21.903^\circ$  ( $2\theta$ ).

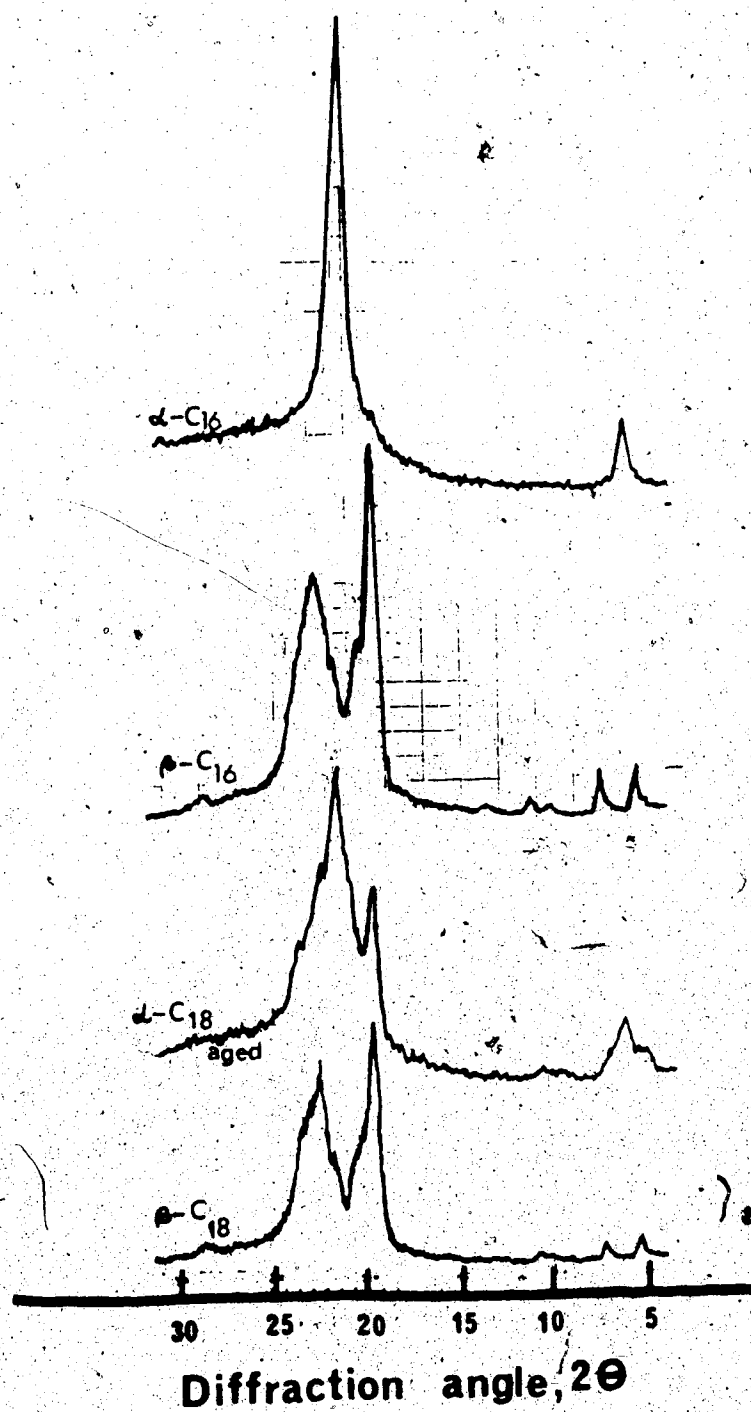


Figure 5.20. X-Ray Diffractograms of  $\alpha$ - and  $\beta$ -C<sub>16</sub> and C<sub>18</sub> Monoglycerides

Table 5.25. X-Ray Diffraction Patterns of  $\alpha$ - and  $\beta$ -Crystallinity Forms of  $C_{16}$  Monoglyceride

| $\alpha$ - $C_{16}$ Monoglyceride |                 |                  | $\beta$ - $C_{16}$ Monoglyceride |                 |                  |
|-----------------------------------|-----------------|------------------|----------------------------------|-----------------|------------------|
| $2\theta$                         | $d, \text{\AA}$ | Intensity<br>cps | $2\theta$                        | $d, \text{\AA}$ | Intensity<br>cps |
| 6.048                             | 14.6126         | 327              | 5.512                            | 16.0327         | 450              |
| 21.316                            | 4.1682          | 1957             | 7.399                            | 11.9474         | 529              |
| 24.569                            | 3.6233          | 338              | 10.132                           | 8.7301          | 197              |
| 25.890                            | 3.4413          | 329              | 11.156                           | 7.9313          | 280              |
|                                   |                 |                  | 13.505                           | 6.5562          | 212              |
|                                   |                 |                  | 19.452                           | 4.5633          | 3755             |
|                                   |                 |                  | 20.250                           | 4.3937          | 1777             |
|                                   |                 |                  | 22.601                           | 3.9341          | 2518             |
|                                   |                 |                  | 21.903                           | 4.0578          | 1481             |
|                                   |                 |                  | 28.479                           | 3.1341          | 359              |

In this and the following tables, the diffraction line intensity

strong (s) > cps 400  
 medium (m) 200-399  
 weak (w) < 200

Table 5.26. X-Ray Diffraction Patterns of  $\alpha$ - and  $\beta$ -Crystallinity Forms of  $C_{18}$  Monoglycerides

| $\alpha$ - $C_{18}$ Monoglyceride |         |               | $\beta$ - $C_{18}$ Monoglyceride |         |               |
|-----------------------------------|---------|---------------|----------------------------------|---------|---------------|
| $2\theta$                         | d, Å    | Intensity cps | $2\theta$                        | d, Å    | Intensity cps |
| 5.312                             | 16.6375 | 162           | 5.465                            | 16.1697 | 212           |
| 6.141                             | 14.3929 | 283           | 7.266                            | 12.1666 | 173           |
| 10.468                            | 8.4505  | 114           | 10.703                           | 8.2655  | 120           |
| 19.562                            | 4.5378  | 814           | 13.462                           | 6.5772  | 96            |
| 21.357                            | 4.1604  | 1261          | 15.990                           | 5.5426  | 131           |
| 23.151                            | 3.8419  | 624           | 19.718                           | 4.5023  | 2308          |
| 26.469                            | 3.3674  | 292           | 22.643                           | 3.9269  | 1812          |
| 29.182                            | 3.0601  | 239           | 26.879                           | 3.3169  | 255           |
|                                   |         |               | 28.699                           | 3.1105  | 252           |

$\beta$ -C<sub>18</sub> X-ray diffractogram had more weak peaks than C<sub>18</sub> located at 7.266, 10.703 and 13.462° (2 $\theta$ ). It had two strong peaks at 19.718 and 22.643° (2 $\theta$ ) without the existence of shoulder peaks as observed for  $\beta$ -C<sub>18</sub>.

### *Amylose Complexing Indices*

Monoglyceride complexing with starches, expressed as percentage, is presented in Figures 5.21 and 5.22 for  $\alpha$ - and  $\beta$ -C<sub>18</sub>, and for C<sub>18</sub> with arrowroot starch. The corresponding data are provided in Tables 5.27 and 5.28.

The percent complexing indices of a monoglyceride for starch increased from ungelatinized to gelatinized and solubilized starches. Monoglycerides in their  $\alpha$ -crystallinity were more reactive than in their  $\beta$ -crystallinity forms. The differences between the two were, however, small for ungelatinized starch, but widened for the gelatinized and even further for the solubilized starch. Palmitic acid monoglycerides tended to complex more with starch than stearic acid monoglyceride.

Data for cassava, sweet potato, taro, yam and wheat ( cvs. Fielder and Neepawa) starches were similar to arrowroot, but of different magnitude. They are presented in Appendices 5-16. Optimum complexing observed in the presence of about 0.5% monoglycerides on DWB of starch (Figures 5.21 and 5.22).

### *X-Ray Diffraction Analysis of Starch-Monoglyceride Clathrates*



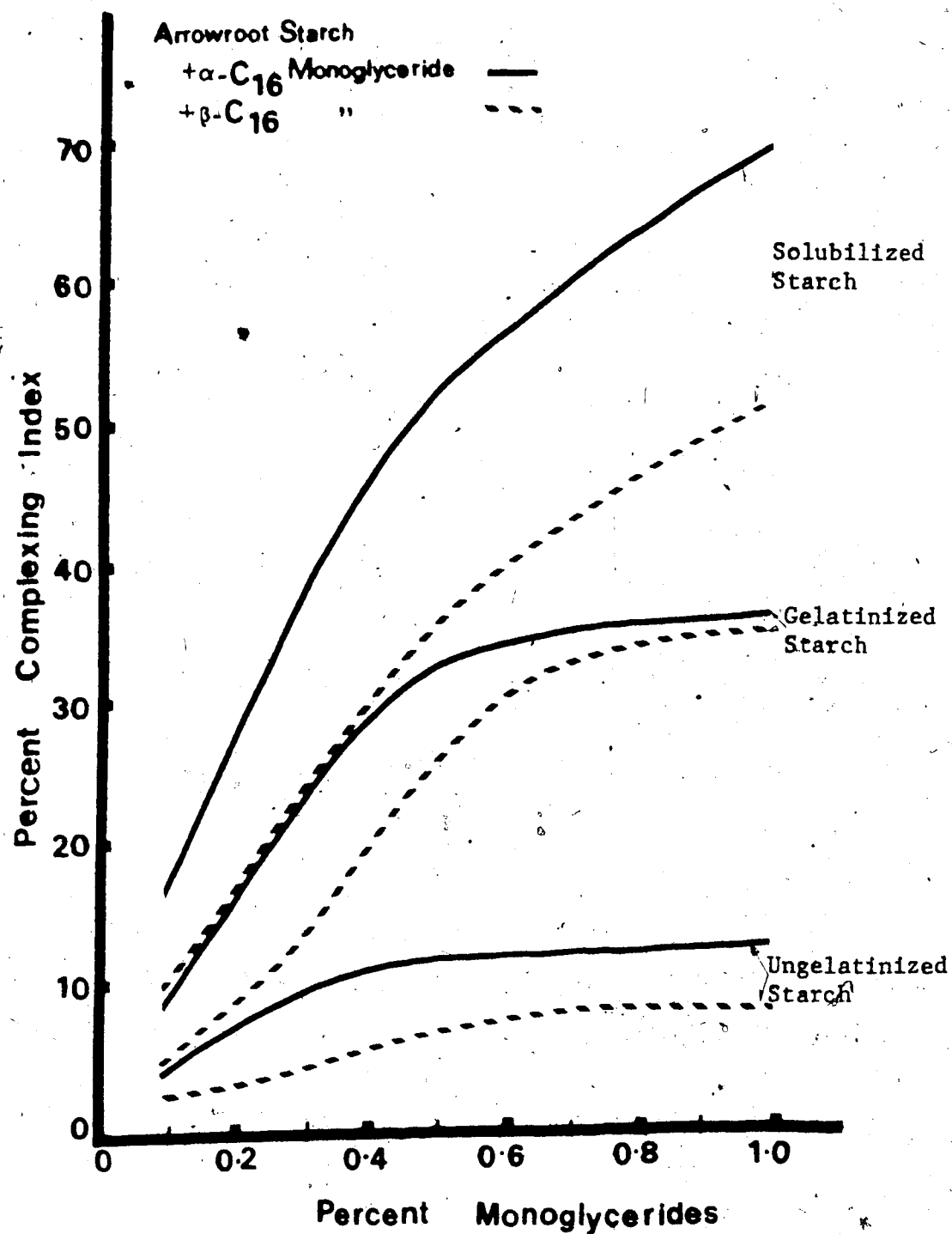


Figure 5.21, Arrowroot Starch Interaction with Type C<sub>16</sub> Monoglycerides

Table 5.27. Percent Complexing Indices for the Interaction of the  $\alpha$ -Crystallinity Forms of C<sub>16</sub> and C<sub>18</sub> Monoglycerides with Arrowroot Starch

| % MG*** | Arrowroot Starch* |                 |                 |                 |                 |                 |
|---------|-------------------|-----------------|-----------------|-----------------|-----------------|-----------------|
|         | Ungelatinized     |                 | Gelatinized     |                 | Solubilized     |                 |
|         | C <sub>16</sub>   | C <sub>18</sub> | C <sub>16</sub> | C <sub>18</sub> | C <sub>16</sub> | C <sub>18</sub> |
| 0.1     | 3.97±0.58**       | 3.77±0.41       | 8.48±0.17       | 5.86±0.08       | 16.96±2.13      | 13.33±0.41      |
| 0.2     | 7.16±1.36         | 6.96±0.82       | 16.95±0.30      | 12.53±2.13      | 29.17±1.39      | 27.60±0.78      |
| 0.3     | 10.43±1.14        | 10.15±0.40      | 23.10±0.77      | 16.23±0.82      | 37.50±0.97      | 37.02±0.94      |
| 0.4     | 11.13±1.44        | 11.59±0.01      | 29.82±1.03      | 21.02±1.03      | 47.73±1.93      | 44.50±0.21      |
| 0.5     | 11.72±1.25        | 12.20±0.04      | 33.09±1.54      | 24.38±0.86      | 52.18±0.25      | 49.71±0.61      |
| 0.8     | 12.03±1.36        | 12.46±0.00      | 35.64±1.03      | 31.45±0.21      | 64.32±2.64      | 53.94±0.45      |
| 1.0     | 12.25±1.36        | 12.73±0.04      | 37.42±0.57      | 34.20±0.82      | 70.18±1.03      | 55.75±0.96      |

\*In this and following tables: the starch was previously lintnerized with 7.5% HCl at 40°C for 72 hrs.

$$**\text{Complexing Index (A}_{680\text{ nm}}), \text{ percent} = \frac{A_{TA} - A_{RA}}{A_{TA}} \times 100$$

where A<sub>TA</sub>, absorbance<sub>680nm</sub> of starch amylose complex with iodine, before starch interaction with monoglycerides, and A<sub>RA</sub>, absorbance of residual amylose complexed with iodine after starch interaction with monoglycerides, per gram dry matter of ungelatinized, gelatinized or solubilized starch samples.

\*\*\* MG, Monoglyceride

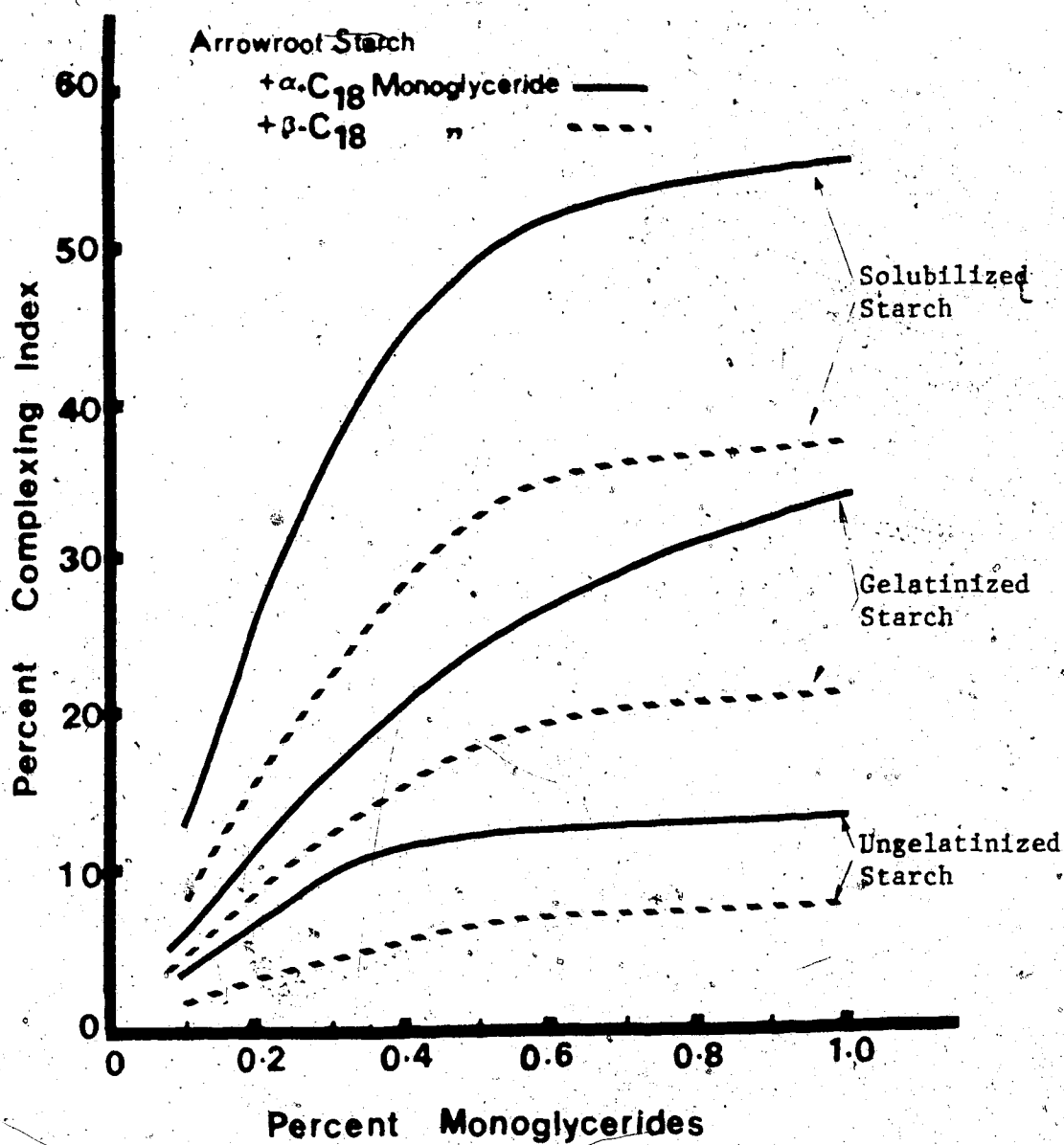


Figure 5.22. Arrowroot Starch Interaction with Type C<sub>18</sub> Monoglyceride

Table 5.28. Percent Complexing Indices for The Interaction of the  $\beta$ -Crystallinity Forms of  $C_{16}$  and  $C_{18}$  Monoglycerides with Arrowroot Starch

| mg  | Arrowroot Starch |                 |                  |                  |                  |                  |
|-----|------------------|-----------------|------------------|------------------|------------------|------------------|
|     | Ungelatinized    |                 | Gelatinized      |                  | Solubilized      |                  |
|     | $C_{16}$         | $C_{18}$        | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         |
| 0.1 | 1.67 $\pm$ 0.58  | 1.37 $\pm$ 0.01 | 4.37 $\pm$ 0.24  | 3.60 $\pm$ 0.91  | 10.24 $\pm$ 0.69 | 7.78 $\pm$ 1.06  |
| 0.2 | 2.87 $\pm$ 0.81  | 2.67 $\pm$ 0.39 | 9.04 $\pm$ 1.15  | 8.05 $\pm$ 0.06  | 15.36 $\pm$ 0.45 | 15.91 $\pm$ 0.87 |
| 0.3 | 4.06 $\pm$ 1.06  | 4.17 $\pm$ 0.05 | 12.81 $\pm$ 1.26 | 12.08 $\pm$ 0.90 | 22.87 $\pm$ 0.55 | 23.38 $\pm$ 1.30 |
| 0.4 | 5.25 $\pm$ 1.31  | 5.14 $\pm$ 0.06 | 19.64 $\pm$ 2.18 | 15.25 $\pm$ 0.60 | 29.70 $\pm$ 0.45 | 29.59 $\pm$ 2.56 |
| 0.5 | 5.90 $\pm$ 1.29  | 6.13 $\pm$ 0.16 | 26.47 $\pm$ 1.94 | 18.43 $\pm$ 0.90 | 36.53 $\pm$ 0.14 | 32.84 $\pm$ 2.41 |
| 0.8 | 7.64 $\pm$ 0.82  | 7.29 $\pm$ 0.07 | 34.47 $\pm$ 2.11 | 20.55 $\pm$ 0.09 | 51.35 $\pm$ 0.09 | 35.36 $\pm$ 1.54 |
| 1.0 | 8.33 $\pm$ 0.81  | 7.44 $\pm$ 0.09 | 42.21 $\pm$ 3.32 | 21.82 $\pm$ 0.90 | 61.43 $\pm$ 0.88 | 39.36 $\pm$ 0.05 |

Gallant *et al.* (1982) stated that native starches have either an A, B, or C X-ray diffraction pattern. Pattern A is commonly given by cereal starches and is referred to as the 'cereal type'. The B pattern occurs mainly in tuber starches and is referred to as the 'tuber type'. The C pattern is intermediate between A and B.

The A pattern has two peaks between  $16$  and  $18^\circ (2\theta)$  and one peak at  $24^\circ (2\theta)$ . Type B pattern has one peak at  $17^\circ (2\theta)$  and two peaks at  $23$  and  $24$ , and one at  $5^\circ 44' (2\theta)$ .

If fat-amylose complexes are present, they give an X-ray pattern with a strong peak at  $4.4 \text{ \AA}$  d spacing (Varriano-Marston *et al.* 1980). Amylose interaction with surfactants or monoglycerides give the v-type X-ray diffraction pattern. It is characterised by another strong peak at d spacing of  $6.8 \text{ \AA}$  (Ghiasi *et al.* 1982).

X-ray diffractograms of ungelatinized, gelatinized, and solubilized arrowroot starch with  $\alpha\text{-C}_{12}$  monoglyceride are presented in Figure 5.23. The corresponding data for the X-ray diffraction patterns are provided in Table 5.29.

Similar patterns for wheat (cv. Neepawa) starch are provided in Figure 5.24 and Table 5.30 for comparison.

X-ray diffractograms to compare wheat (cv. Neepawa) and yam starch clathrates with  $\alpha\text{-C}_{12}$  and  $\beta\text{-C}_{12}$  monoglycerides are given in Figure 5.25 and Tables 5.31 and 5.32.

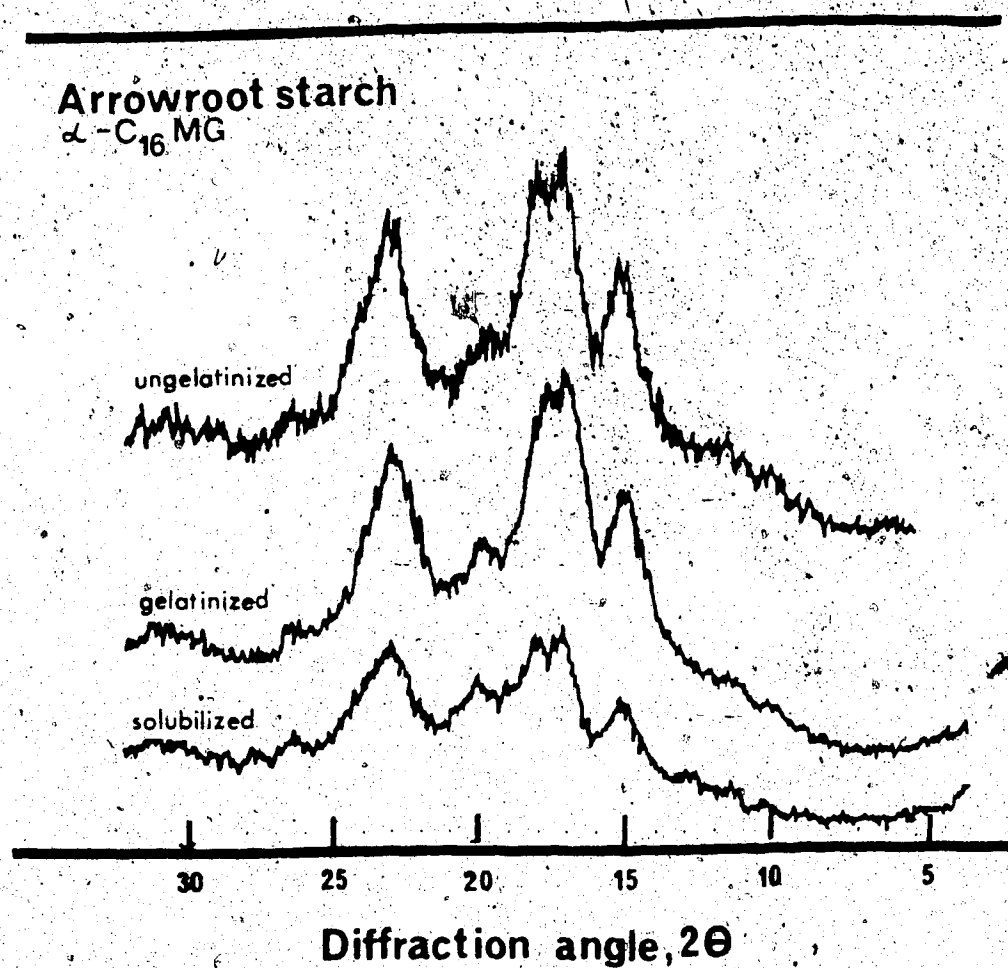


Figure 5.23. X-Ray Diffractograms of Arrowroot Starch  
Clathrates with  $\alpha$ -C<sub>16</sub> Monoglyceride

Table 5.29. X-Ray Diffraction Patterns of Arrowroot Starch.  
Clathrates with  $\alpha$ -C<sub>16</sub> Monoglyceride

| Ungelatinized      |        |                  | Arrowroot Starch   |        |                  | Solubilized        |        |                  |
|--------------------|--------|------------------|--------------------|--------|------------------|--------------------|--------|------------------|
|                    |        |                  | Gelatinized        |        |                  |                    |        |                  |
| $\theta_{2\theta}$ | d, Å   | Intensity<br>cps | $\theta_{2\theta}$ | d, Å   | Intensity<br>cps | $\theta_{2\theta}$ | d, Å   | Intensity<br>cps |
| 9.932              | 8.9057 | 148              | 14.997             | 5.9073 | 389              | 10.190             | 8.6808 | 87               |
| 11.572             | 7.6469 | 191              | 17.140             | 5.1731 | 524              | 14.936             | 5.9312 | 199              |
| 14.983             | 5.9127 | 404              | 17.633             | 5.0295 | 522              | 17.135             | 5.1746 | 299              |
| 17.046             | 5.2015 | 538              | 19.832             | 4.4766 | 329              | 18.046             | 4.9156 | 277              |
| 19.620             | 4.5246 | 330              | 26.326             | 3.3853 | 403              | 20.060             | 4.4262 | 247              |
| 22.918             | 3.8803 | 448              | 23.646             | 3.7625 | 326              | 23.373             | 3.8058 | 260              |
| 26.227             | 3.3978 | 243              | 26.535             | 3.3591 | 224              | 26.425             | 3.3728 | 171              |
| 28.835             | 3.0961 | 219              | 31.188             | 2.8678 | 230              |                    |        |                  |
| 30.352             | 2.9447 | 224              |                    |        |                  |                    |        |                  |

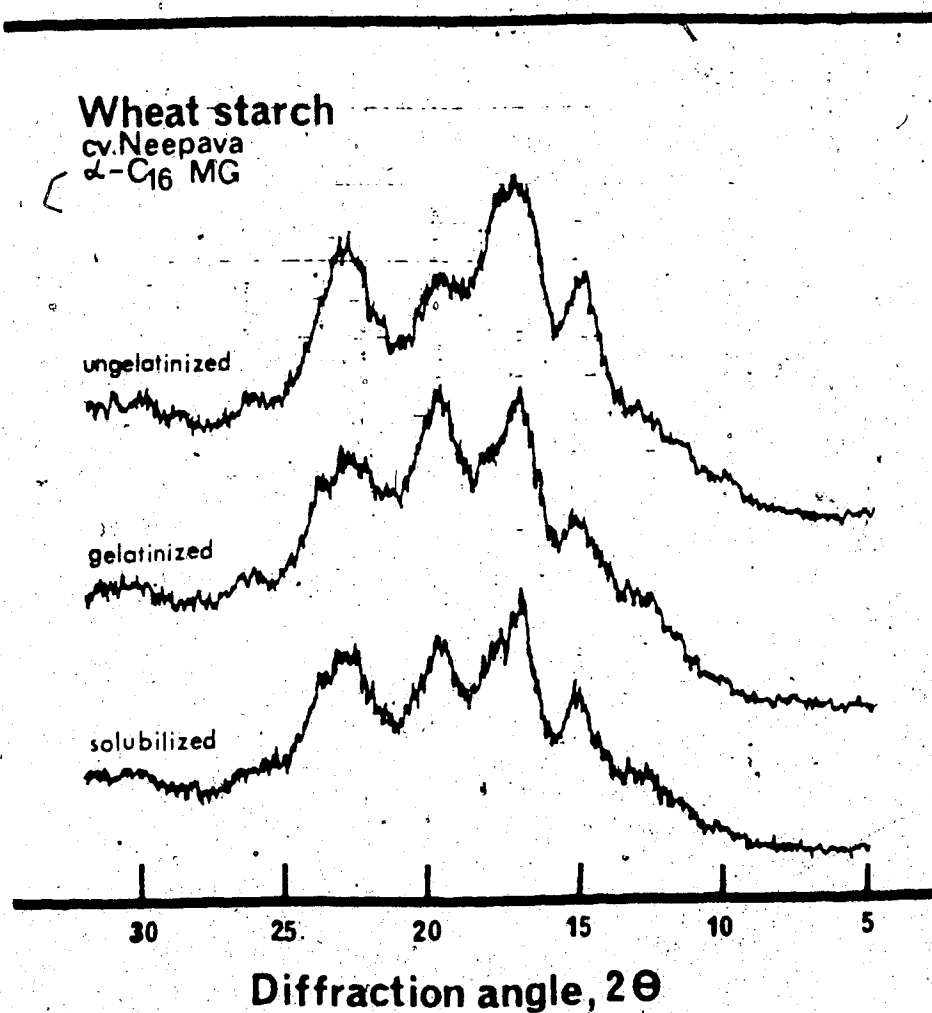


Figure 5.24. X-Ray Diffractograms of Wheat, cv. Neepawa  
Starch Clathrates with  $\alpha$ -C<sub>16</sub> Monoglycerides



Table 5.30. X-Ray Diffraction Patterns of Wheat, cv. Neepawa Starch Clathrates with  $\alpha$ -C<sub>16</sub> Monoglycerides

| Wheat, cv. Neepawa Starch |        |               |             |        |               |             |        |               |
|---------------------------|--------|---------------|-------------|--------|---------------|-------------|--------|---------------|
| Ungelatinized             |        |               | Gelatinized |        |               | Solubilized |        |               |
| $2\theta$                 | d, Å   | Intensity cps | $2\theta$   | d, Å   | Intensity cps | $2\theta$   | d, Å   | Intensity cps |
| 9.939                     | 8.8989 | 116           | 11.687      | 7.5720 | 158           | 13.216      | 6.6989 | 185           |
| 11.549                    | 7.6621 | 155           | 12.614      | 7.0176 | 195           | 14.968      | 5.9187 | 262           |
| 15.063                    | 5.8817 | 353           | 15.274      | 5.8007 | 301           | 16.997      | 5.2163 | 383           |
| 17.082                    | 5.1908 | 446           | 17.136      | 5.1744 | 462           | 17.710      | 5.0079 | 348           |
| 19.804                    | 4.4830 | 359           | 19.769      | 4.4908 | 458           | 19.584      | 4.5328 | 325           |
| 22.964                    | 3.8727 | 388           | 22.440      | 3.9619 | 376           | 22.584      | 3.9370 | 320           |
| 26.233                    | 3.3970 | 206           | 23.706      | 3.7532 | 348           | 23.033      | 3.8612 | 318           |
| 28.794                    | 3.1005 | 193           | 26.041      | 3.4217 | 234           | 23.785      | 3.7408 | 299           |
| 29.929                    | 2.9854 | 214           | 31.391      | 2.8496 | 221           | 28.206      | 3.1637 | 163           |
| 31.009                    | 2.8839 | 218           |             |        |               |             |        |               |

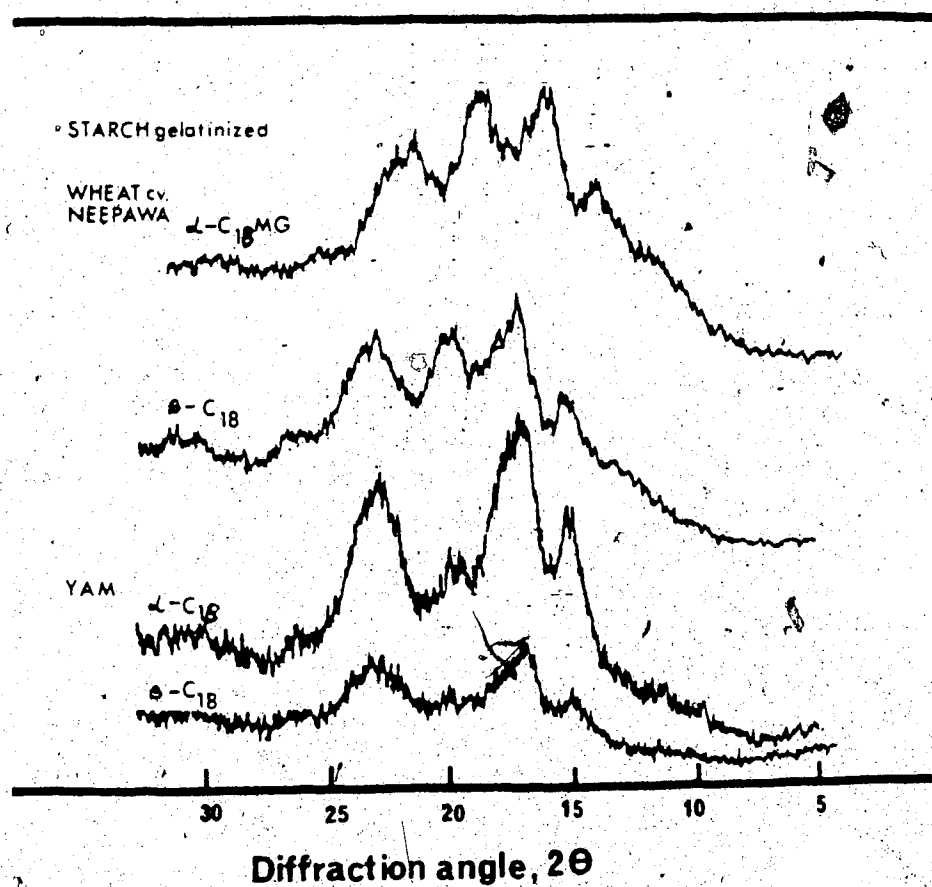


Figure 5.25. X-Ray Diffractograms of Gelatinized Wheat, cv. Neepawa and Yam Starches Clathrates with  $\alpha$ -C<sub>18</sub> and  $\beta$ -C<sub>18</sub> Monoglycerides

Table 5.31. X-Ray Diffraction Patterns of Gelatinized Wheat,  
cv. Neepawa Starch Clathrates with  $\alpha$ - and  $\beta$ -C<sub>18</sub>  
Monoglycerides

| $\alpha$ -C <sub>18</sub> Monoglyceride |        |                  | $\beta$ -C <sub>18</sub> Monoglyceride |        |                  |
|---|--------|------------------|--|--------|------------------|
| $2\theta$                               | d, Å   | Intensity<br>cps | $2\theta$                              | d, Å   | Intensity<br>cps |
| 11.664                                  | 7.5866 | 166              | 13.253                                 | 6.6805 | 180              |
| 12.822                                  | 6.9041 | 214              | 15.222                                 | 5.8206 | 273              |
| 14.231                                  | 6.2235 | 262              | 17.103                                 | 5.1844 | 411              |
| 15.116                                  | 5.8610 | 331              | 17.881                                 | 4.9606 | 362              |
| 17.233                                  | 5.1454 | 452              | 19.692                                 | 4.5081 | 376              |
| 19.734                                  | 4.4988 | 452              | 22.929                                 | 3.8785 | 376              |
| 22.480                                  | 3.9549 | 397              | 26.420                                 | 3.3734 | 228              |
| 23.182                                  | 3.8368 | 361              | 30.133                                 | 2.9656 | 225              |
| 26.238                                  | 3.3964 | 225              | 31.121                                 | 2.8738 | 220              |

Table 5.32. X-Ray Diffraction Patterns of Gelatinized Yam Starch Clathrates with  $\beta$ -C<sub>16</sub> and C<sub>18</sub> Monoglycerides

| Yam Starch, Gelatinized                |        |               |  |        |               |
|--|--------|---------------|--|--------|---------------|
| $\beta$ -C <sub>16</sub> Monoglyceride |        |               | $\beta$ -C <sub>18</sub> Monoglyceride |        |               |
| $2\theta$                              | d, Å   | Intensity cps | $2\theta$                              | d, Å   | Intensity cps |
| 15.395                                 | 5.7553 | 305           | 15.174                                 | 5.8386 | 123           |
| 17.350                                 | 5.1112 | 448           | 17.055                                 | 5.1988 | 190           |
| 19.730                                 | 4.4996 | 283           | 20.095                                 | 4.4186 | 143           |
| 23.228                                 | 3.8292 | 373           | 23.353                                 | 3.8091 | 191           |
| 26.471                                 | 3.3671 | 206           | 24.069                                 | 3.6973 | 171           |
| 31.073                                 | 2.8781 | 216           |  |        |               |

Clathrates obtained from ungelatinized, gelatinized and solubilized cassava, sweet potato, taro and yam starches with  $\alpha$ -C<sub>16</sub> were found similar to arrowroot or wheat starch clathrates. Their X-ray diffractograms and patterns are presented in Appendices 17-24.

The X-ray diffraction patterns as a whole showed that the diffractogram peaks became more defined when interaction with monoglyceride was preceded by gelatinization or solubilization of the starch. Some peaks originally present in the ungelatinized starch disappeared or became diminished in the gelatinized and solubilized starch. This was observed in both root and wheat starch clathrates with the monoglycerides.

#### 5.2.6 Starch Affinity for Gluten

The affinities of ungelatinized and gelatinized starch for gluten in systems simulating the dough, early and fully baked stages of bread making for wheat ( cvs. Fielder and Neepawa), and for arrowroot, cassava, sweet potato, taro and yam starches are presented in Table 5.33.

Wheat starches were observed to have lower affinities for gluten in the dough than root starches. The affinity values of the wheat starches for gluten however tripled (CWRSW) or more than trippled (SWSW) in early baking, but declined to about 1.5 times of their initial values in the fully baked systems.

Table 5.33 Starch Affinity for Gluten

| Starch                | Percent Affinity               |                          |                          |
|-----------------------|--------------------------------|--------------------------|--------------------------|
|                       | Undenatured Gluten             |                          | Denatured Gluten         |
|                       | + Ungelatinized Starch (A) *** | + Gelatinized Starch (B) | + Gelatinized Starch (C) |
| Wheat (cv. Neepawa)*  | 31.82 $\pm$ 3.99               | 88.61 $\pm$ 1.79         | 45.11 $\pm$ 0.78         |
| Wheat (cv. Fielder)** | 24.91 $\pm$ 0.81               | 89.57 $\pm$ 1.75         | 41.29 $\pm$ 0.39         |
| Arrowroot             | 50.27 $\pm$ 2.02               | 83.57 $\pm$ 2.86         | 41.01 $\pm$ 1.33         |
| Cassava               | 48.46 $\pm$ 7.46               | 82.05 $\pm$ 2.9          | 49.04 $\pm$ 2.99         |
| Taro                  | 41.37 $\pm$ 4.02               | 89.95 $\pm$ 1.60         | 40.99 $\pm$ 1.48         |
| Yam                   | 40.79 $\pm$ 2.47               | 91.49 $\pm$ 2.74         | 28.46 $\pm$ 1.53         |
| Sweet Potato          | 39.92 $\pm$ 2.19               | 89.12 $\pm$ 1.40         | 43.99 $\pm$ 0.86         |

\*CWRSW - Canadian Western Red Spring Wheat

\*\*SWSW - Soft White Spring Wheat

\*\*\*A represents the dough, B the early baking, and C the fully baked stages of breadmaking.

Root starches doubled their dough affinity for gluten values in early baking, hence equalling the wheat starches, but declined to about their initial values in fully baked stage. Exceptions were arrowroot and yam, whose affinities for vital gluten were lower in the fully baked system than in the dough.

### 5.3 B. BREAD

#### 5.3.1 Dough Rheological Properties

Farinographic rheological data for the root starch/vital gluten composite doughs are presented in Table 5.34. Similar data for wheat (cvs. Neepawa and Fielder) flours and their starch/vital gluten composite doughs are provided in Table 5.35. Representative farinograms for starch/vital gluten doughs of arrowroot, cassava and wheat (cv. Neepawa) are given in Figures 5.26, 5.27, and 5.30 respectively. For comparison, farinograms for wheat (cvs. Neepawa and Fielder) flours are also given in Figures 5.28 and 5.29. Corresponding farinograms for sweet potato, taro, yam and wheat (cv. Fielder) starch/vital gluten composite doughs are provided in Appendices 25-28. .

##### 5.3.1.1 Water Absorption

Percent water absorptions of the various flours are included in Tables 5.34 and 5.35. Arrowroot starch/vital gluten composite flour had the highest water absorption (80.5%) while SWSW (cv. Fielder) starch/vital gluten

Table 5.34 Farinographic Data of Doughs from Tropical Root  
Starches with Vital Gluten Composite Flours

| Sample   | Percent<br>Moisture<br>Content | Percent*<br>Absorption | Arrival<br>Time in<br>Minutes | Peak<br>Time in<br>Minutes | Dough<br>Stability<br>in Minutes | Departure<br>Time in<br>Minutes | Mixing**<br>Tolerance<br>Index (MTI) | 20 Min Drop |
|--|--------------------------------|------------------------|-------------------------------|----------------------------|----------------------------------|---------------------------------|--------------------------------------|-------------|
| Arrowroot<br>Starch/Gluten<br>Flour Composite    | 9.23±0.71                      | 80.50±0.10             | 3.25±0.18                     | 7.00±0.20                  | 7.75±0.30                        | 11.00±0.25                      | 60.0±0.5                             | 120.0±10.0  |
| Cassava<br>Starch/Gluten<br>Flour Composite      | 14.23±0.41                     | 64.70±0.10             | 2.00±0.16                     | 4.50±0.25                  | 8.25±0.20                        | 10.25±0.30                      | 60.0±0.7                             | 130.0±10.0  |
| Sweet Potato<br>Starch/Gluten<br>Flour Composite | 8.96±0.26                      | 68.80±0.20             | 2.50±0.10                     | 3.50±0.10                  | 4.20±0.10                        | 6.70±0.20                       | 145.0±5.0                            | 190.0±10.0  |
| Taro<br>Starch/Gluten<br>Flour Composite         | 13.51±0.26                     | 68.4±0.15              | 3.00±0.15                     | 3.80±0.18                  | 1.80±0.20                        | 4.80±0.20                       | 120.0±5.0                            | 190.0±10.0  |
| Yam<br>Starch/Gluten<br>Flour Composite          | 10.35±0.22                     | 68.50±0.10             | 2.30±0.20                     | 4.00±0.10                  | 10.20±0.10                       | 12.50±0.10                      | 48.4±3.0                             | 80.0±5.0    |

\*All farinographic data were based on a flour of 14% moisture.

\*\*5 min after peak time.



Table 5.35 Farinographic Data of Doughs, from Wheat Flours,  
and Their Starch-Vital Gluten Composites

| Sample                                      | Percent<br>Moisture<br>Content | Percent*<br>Absorption | Arrival<br>Time in<br>Minutes | Peak<br>Time in<br>Minutes | Dough<br>Stability<br>in Minutes | Departure<br>Time in<br>Minutes | Mixing**<br>Tolerance<br>Index (MTI) | 20 Min Drop |
|---|--------------------------------|------------------------|-------------------------------|----------------------------|----------------------------------|---------------------------------|--------------------------------------|-------------|
| Neepawa<br>Wheat Flour                      | 8.91±0.13                      | 68.40±0.20             | 3.80±0.15                     | 6.00±0.20                  | 6.70±0.10                        | 10.50±0.20                      | 26.0±5.0                             | 45.0±5.0    |
| Neepawa<br>Starch/gluten<br>Flour composite | 9.82±0.38                      | 60.40±0.10             | 1.75±0.10                     | 6.50±0.10                  | 11.75±0.15                       | 13.50±0.15                      | 25.0±3.0                             | 45.0±5.0    |
| Fielder<br>Wheat Flour                      | 11.67±1.55                     | 56.80±0.49             | 0.80±0.10                     | 1.20±0.10                  | 0.90±0.10                        | 1.70±0.10                       | 120.0±5.0                            | 150.0±10.0  |
| Fielder<br>Starch/gluten<br>Flour composite | 11.03±0.99                     | 54.0±0.10              | 1.00±0.10                     | 3.50±0.15                  | 7.50±0.10                        | 8.50±0.10                       | 40.0±2.0                             | 60.0±5.0    |

\*All farinographic data were based on a flour of 14% moisture.

\*\*5 min after peak time.

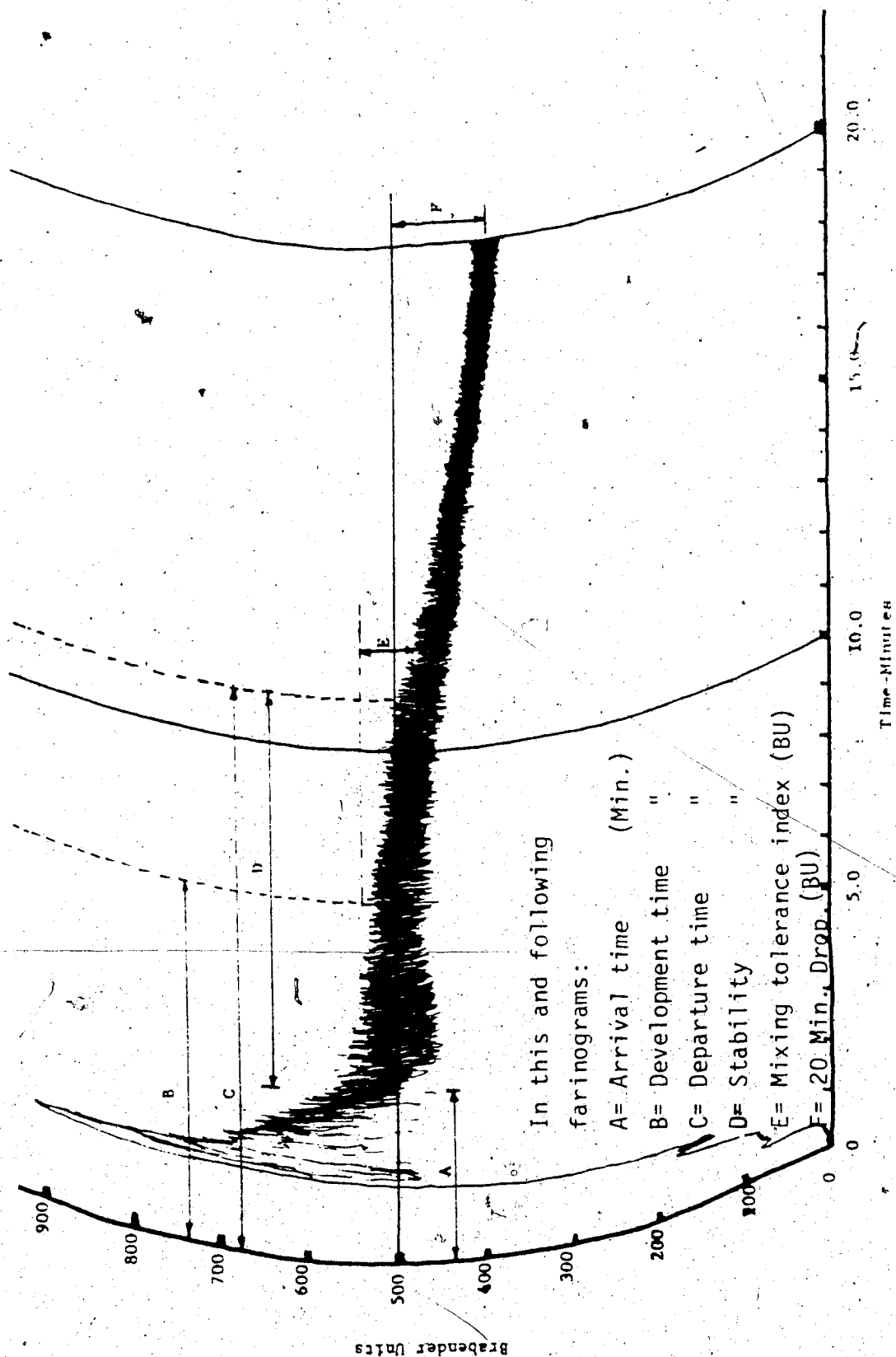


Figure 5.26: Arrowroot Starch-85%; Vital Gluten-15%, Composite Flour Dough Farinogram.

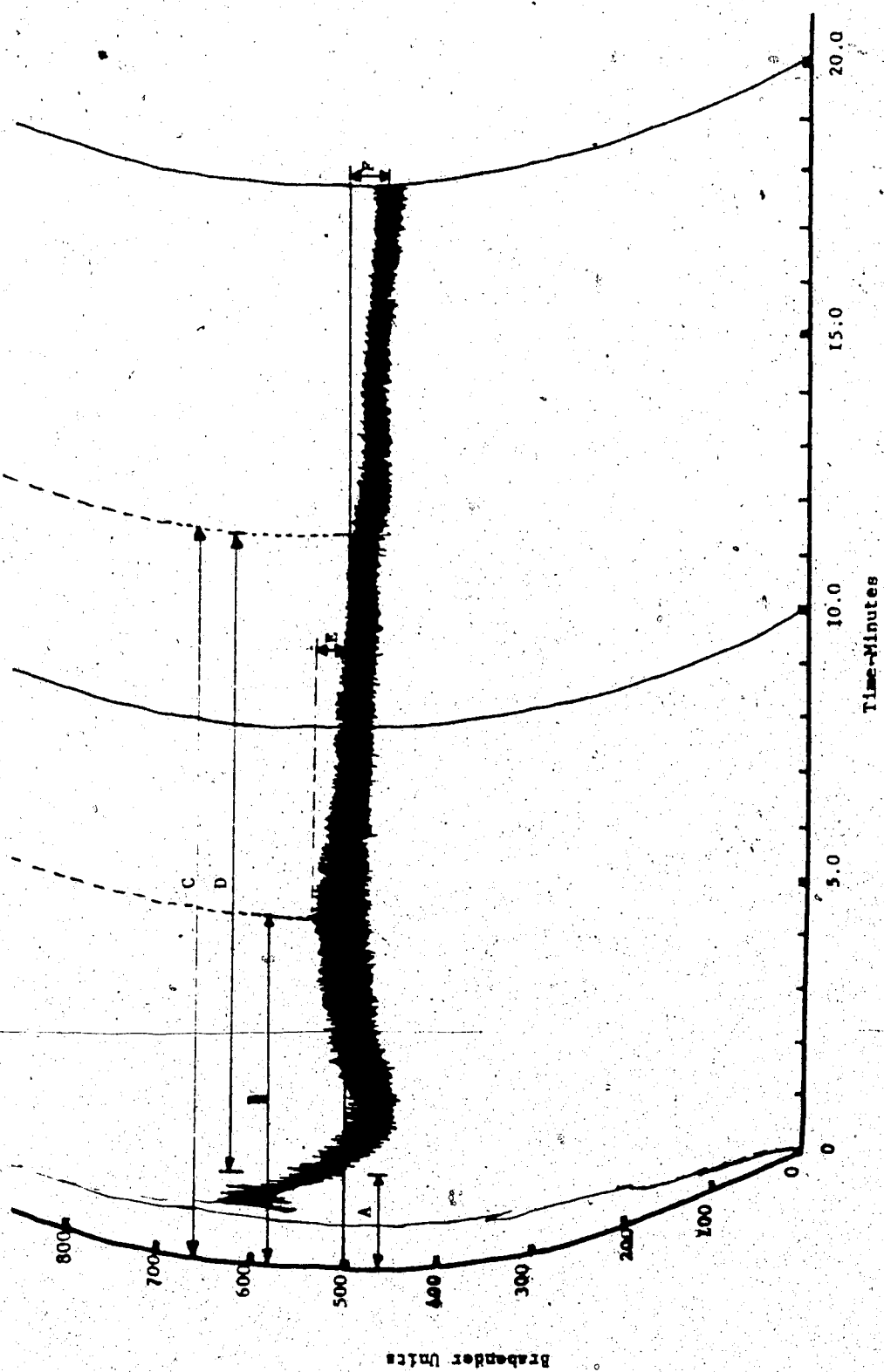


Figure 5.27. Cassava Starch-85%; Vital Gluten-15%, Composite  
Flour Dough Farinogram

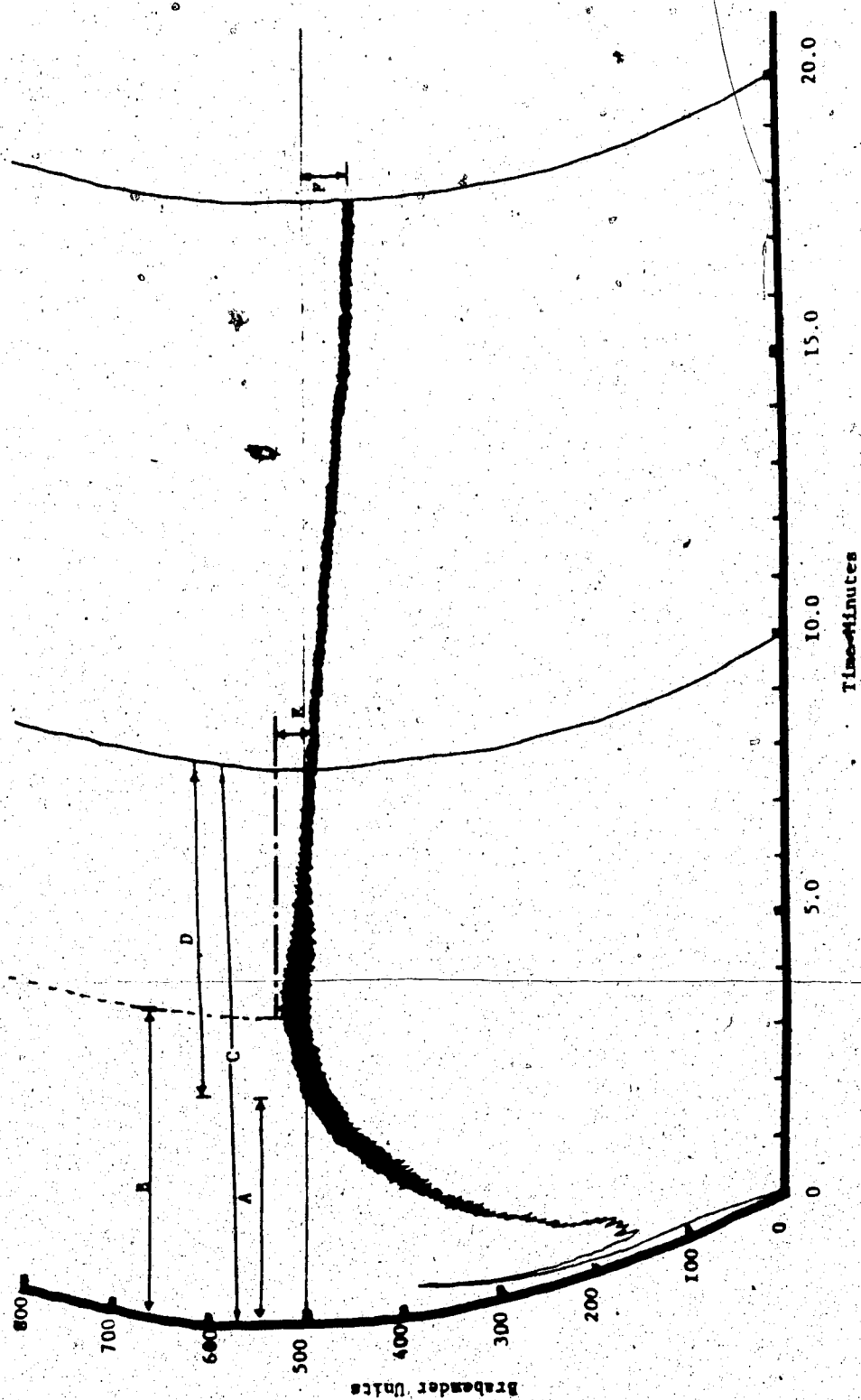


Figure 5.28. Wheat, cv. Neepawa, Flour Dough Farinogram

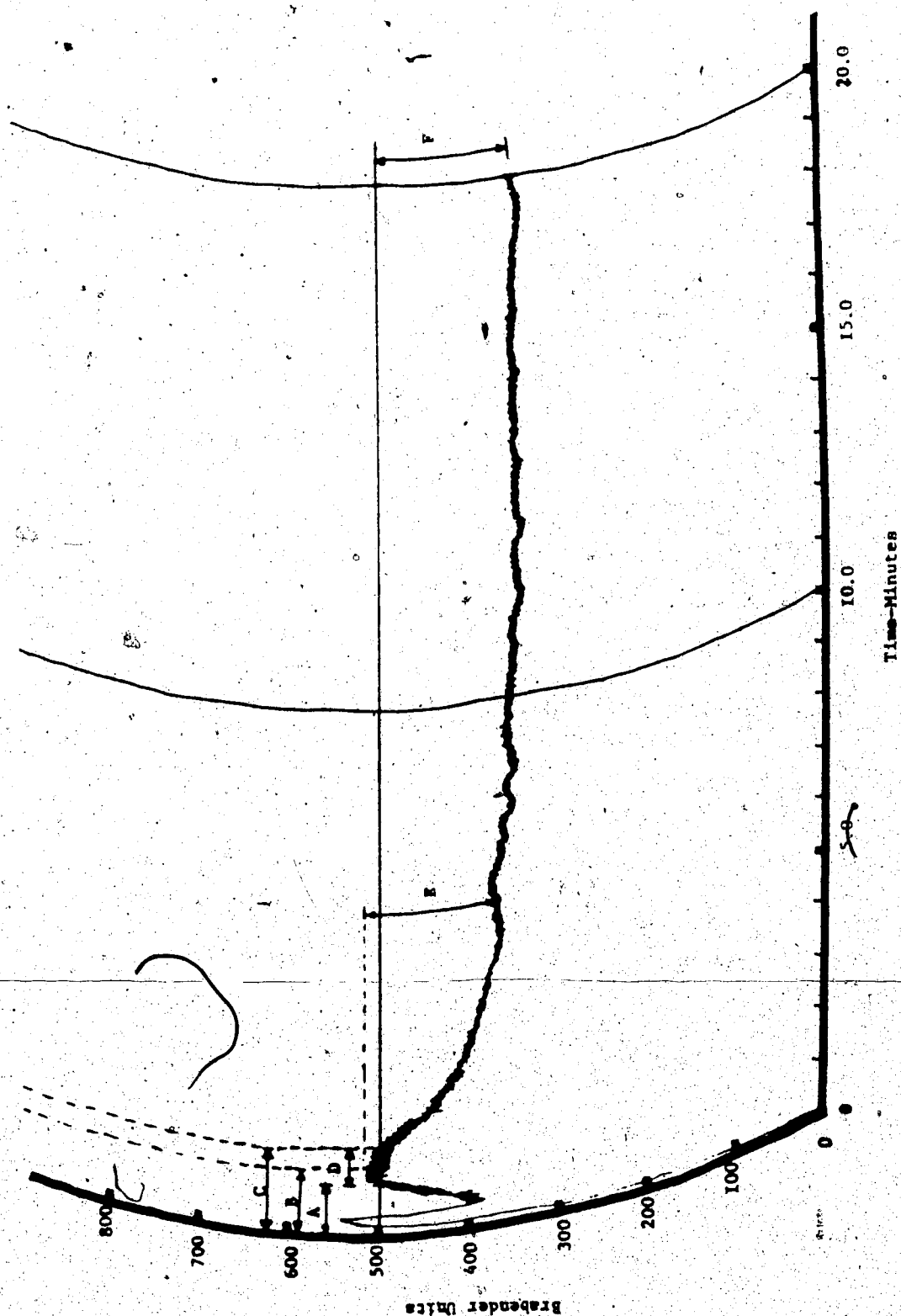


Figure 5.29 Wheat, cv. Fielder, Flour Dough Farinogram

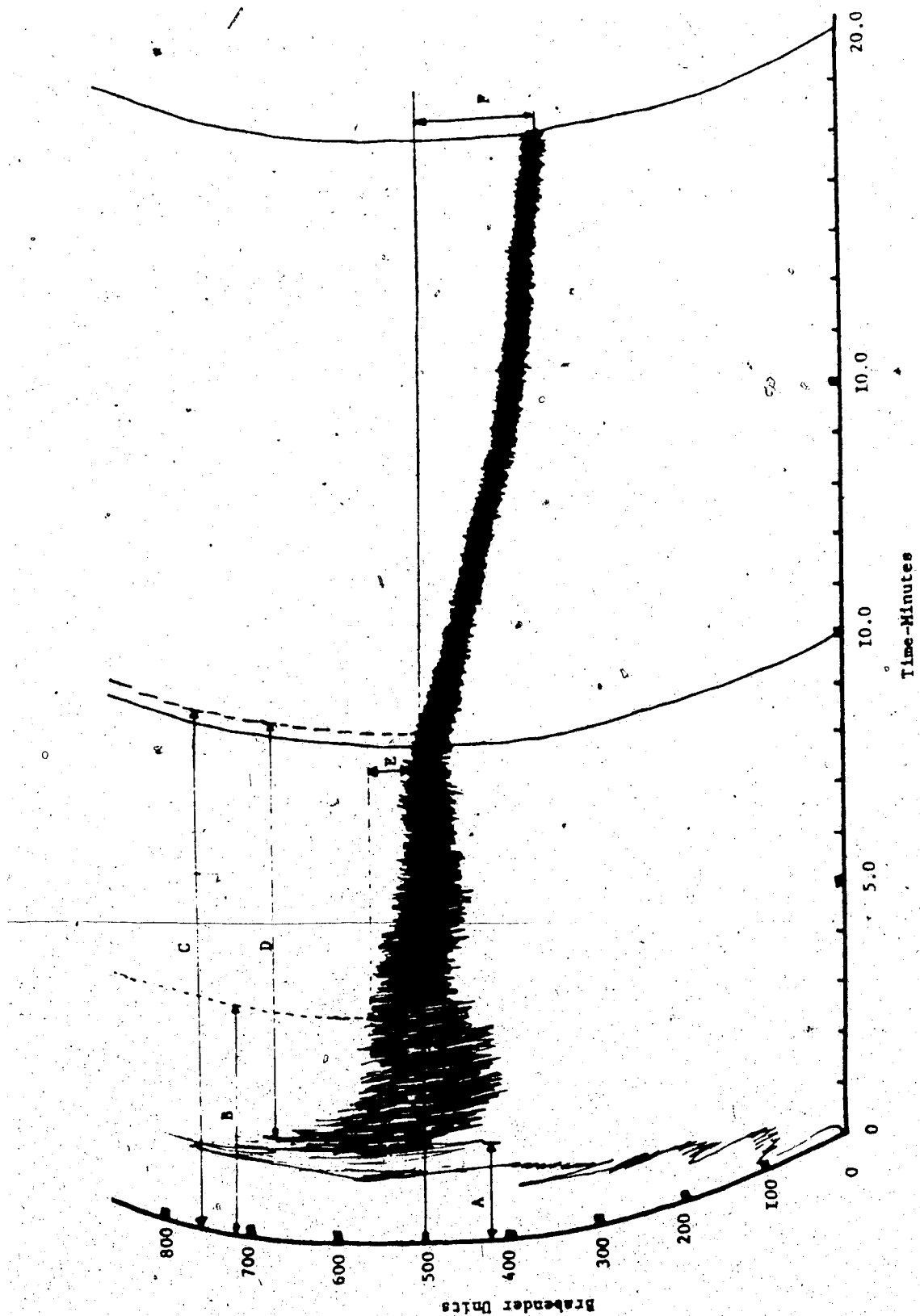


Figure 5.30. Wheat, cv. Neepawa Starch-85%; Vital Gluten-15%,

Composite Flour Dough Farinogram

composite flour had the lowest (54.0%). Similar composite flours with sweet potato, taro, and yam starches had practically the same water absorption (68.4-68.8%), which was similar to absorption by CWRSW (cv. Neepawa) flour (68.4%). Cassava starch/vital gluten composite flour had intermediate water absorption of 64.7%. Data in Table 5.35 show that the wheat flours had higher absorptions than their corresponding starch/vital gluten composites, but cv. Fielder flour and its starch composite with vital gluten had lower water absorption than their CWRSW (cv. Neepawa) counterparts.

#### 5.3.1.2 Dough Physico-Mechanical Properties

CWRSW (cv. Neepawa) flour had the longest arrival time of 3.80 min., compared to the shortest arrival time of 0.80 min. for SWSW (cv. Fielder) flour. Root starch/vital gluten composite flours had longer arrival times (2.0-3.25 min.) than soft wheat flour, but shorter than the corresponding time for the hard wheat flour. The data in Table 5.35 show further that, whereas CWRSW starch composite with vital gluten had inferior arrival times in comparison to the flour, the opposite was true for SWSW.

Dough development times for CWRSW flour (6.0 min.) and its starch/gluten composite (6.5 min.) were found to be practically the same. There was however significant difference between the development times of SWSW flour (1.20 min.) and its starch/gluten composite (3.50 min.). Root starch/vital gluten composites required less time

(3.50-4.50 min.) than CWRSW flour or its starch/vital gluten composite to reach the same consistency. Arrowroot starch/vital gluten composite was the exception, with a development time of 7.0 min. Whereas Fielder flour dough required a shorter time to reach similar consistencies as the other doughs, its starch/vital gluten composite was close to similar composites involving the root starches.

It was further observed that starch/gluten composite dough of wheat starches had better stability than the respective flour doughs. The dough stability change of 0.9 min. for SWSW flour to 7.5 min. for its starch composite with gluten was large. A similar change from 6.70 min. for flour to 11.75 min. for starch/gluten composite dough, was observed for CWRSW.

Arrowroot and cassava starch composites with vital gluten doughs were close in stability to SWSW starch/vital gluten composite and CWRSW flour doughs. SWSW flour dough had the least stability, as shown in Table 5.35. CWRSW starch/vital gluten composite had the longest stability (11.75 min). Yam starch/vital gluten composite was close to CWRSW with a stability of 10.20 min. Of the root starch composites with vital gluten doughs, taro starch had the least stability (1.80 min), followed by sweet potato starch (4.20 min.).

In relation to the mixing tolerance, root starch/vital gluten doughs broke down faster in their consistencies than the CWRSW flour or its starch or SWSW starch/vital gluten



composite dough. Composite doughs of vital gluten with sweet potato and taro starches had very poor stabilities (Table 5.34), as did SWSW flour dough (Tables 5.35).

### 5.3.2 Dough and Crumb Hydration Capacities

The dough and crumb hydration capacities are given in Table 5.36. All dough hydration capacities were very low as compared to their crumb hydration capacities.

Arrowroot and taro/vital gluten doughs had negative hydration capacities. The other doughs and all their bread crumbs had positive hydration capacities.

Wheat (cv. Fielder) and yam starch composites with gluten doughs had the highest hydration capacities. It was observed that wheat flour doughs had lower hydration capacities than their respective starch/vital gluten composite doughs. Furthermore, SWSW flour dough had higher hydration capacity than the CWRSW flour dough. A similar relation was evident in the case of their starch vital/gluten doughs.

CWRSW (cv. Neepawa) flour bread crumb had lower hydration capacity than corresponding crumb from SWSW. The reverse relationship was observed for their starch/vital gluten composite bread crumbs. Although yam starch/vital gluten dough had higher hydration capacity than all others, except SWSW starch/vital gluten dough, its crumb had the lowest hydration capacity (2.65g/g DMB). The highest hydration capacity of 6.557g/g DMB was obtained for taro

Table 5.36 Dough and Crumb Hydration Capacities - Water, g/g

## Dry Solids

|                                    | Dough**                    | Crumb         | Gelatinization**<br>Hydration |
|------------------------------------|----------------------------|---------------|-------------------------------|
| Wheat flour, cv. Neepawa           | 0.061 ± 0.010              | 3.640 ± 0.060 | 3.579.0                       |
| Wheat flour, cv. Fielder           | 0.092 ± 0.003              | 4.480 ± 0.120 | 4.388.0                       |
| Wheat starch cv. Neepawa + gluten* | 0.125 ± 0.025              | 5.370 ± 0.120 | 5.245.0                       |
| Wheat starch cv. Fielder + gluten  | 0.269 ± 0.025              | 4.030 ± 0.020 | 3.761.0                       |
| Arrowroot starch + gluten          | 0.083 ± 0.012 <sup>†</sup> | 3.290 ± 0.260 | 3.373.0                       |
| Cassava starch + gluten            | 0.043 ± 0.004              | 6.600 ± 0.143 | 6.557.0                       |
| Sweet potato starch + gluten       | 0.083 ± 0.008              | 4.120 ± 0.070 | 4.037.0                       |
| Taro starch + gluten               | 0.100 ± 0.001 <sup>†</sup> | 3.140 ± 0.200 | 3.240.0                       |
| Yam starch + gluten                | 0.253 ± 0.049              | 2.650 ± 0.140 | 2.397.0                       |

\*Starch and vital gluten were used at 85% and 15%, respectively.

\*\*Dough formulation: Flour 100% (or 85% starch + 15% vital gluten); sugar 7%; salt 1.5%; yeast 3.0%; water - variable to give doughs of same consistency.

\*\*\*Gelatinization hydration = crumb hydration capacity - dough hydration capacity.

<sup>†</sup>Negative hydration capacity.

starch/vital gluten bread crumb. In all cases, high increases in hydration capacity from a change of dough to the crumb system, were observed.

### 5.3.3 Fractional Volume Increases

Volume changes ( $\text{cm}^3$ ) from the dough to bread, expressed as a fraction of the original dough volume, are presented in Table 5.37.

Bread from CWRSW flour had the highest fractional volume increase ( $1.68 \text{ cm}^3$ ) followed by SWSW flour ( $1.65 \text{ cm}^3$ ). The composite of CWRSW starch with vital gluten had a higher fractional volume increase ( $1.49 \text{ cm}^3$ ) than the increase of  $1.19 \text{ cm}^3$  observed for SWSW starch/vital gluten composite. Generally, wheat flours and starch composites with gluten showed higher volume changes from the dough to the bread than the composites of the root starches with vital gluten.

Of the composite involving the root starches, arrowroot had the highest fractional volume increase ( $0.96$ ), while taro had the smallest ( $0.49 \text{ cm}^3$ ). Cassava, sweet potato and yam starch/vital gluten composites were close to each other in the extent of their fractional volume increases ( $0.64$ - $0.7 \text{ cm}^3$ ).

### 5.3.4 Dough and Bread Crumb Microstructure

#### 5.3.4.1 Dough Microscopy

Table 5.37 Fractional Volume Increases, cm<sup>3</sup>

| Type of Dough-Bread System          | $\frac{V-V_0}{V_0}$ cm <sup>3</sup> |
|-------------------------------------|-------------------------------------|
| Wheat flour, cv. Neepawa            | 1.68 $\pm$ 0.09                     |
| Flour, cv. Fielder                  | 1.65 $\pm$ 0.09                     |
| Neepawa starch 85% + gluten 15%*    | 1.49 $\pm$ 0.23                     |
| Fielder starch 85% + gluten 15%     | 1.19 $\pm$ 0.19                     |
| Tropical starches 85% + gluten* 15% |                                     |
| Arrowroot                           | 0.96 $\pm$ 0.12                     |
| Cassava                             | 0.70 $\pm$ 0.12                     |
| Sweet potato                        | 0.64 $\pm$ 0.21                     |
| Taro                                | 0.49 $\pm$ 0.13                     |
| Yam                                 | 0.67 $\pm$ 0.04                     |

\*Vital gluten.

\*\*V<sub>0</sub> = Volume of 50g of dough.

V = Volume of the bread loaf.

The microscopic surface properties of doughs derived from the various tropical root starch/vital gluten composites are presented in Plates 5.7, 5.8, and 5.9. In Plate 5.10 are portrayed dough surface properties of wheat (cvs. Neepawa and Fielder) flour doughs, while related wheat starch/gluten composite dough surfaces are given in Plate 5.11.

For each sample, micrographs of typical undisturbed and fractured surfaces are provided. The fractured surface micrographs elucidated the internal association between the gluten films and the starch granules (also see Plate 5.16).

#### *5.3.4.2 Bread Crumb Microscopy*

The tropical root starch/vital gluten composite bread crumb surfaces, as shown by SEM, are presented in Plates 5.12 and 5.13. Plates 5.14 and 5.15 show similarly related features of bread crumb surfaces of wheat, cv. Fielder starch/vital gluten, and cv. Neepawa starch/vital gluten; and their pure flours.

#### *5.3.4.3 Transmission Electron Microscopy of Dough and Bread Crumb*

Transmission electron micrographs of arrowroot starch/vital gluten dough and bread crumb are presented in Plate 5.16 as an example elucidating the internal association between gluten film and starch granules in the dough and bread crumb. Unswollen starch granules in the dough and swollen gelatinized granules in the bread crumb

Plate 5.7. SE-Micrographs of Arrowroot Starch/Vital Gluten  
(a, b), and Taro Starch/Vital Gluten (c, d)

Composite Dough Surfaces. Typical Surfaces( a, x4,940;  
c, x1,260). Fractured Surfaces (b, x4,940; d, x1,210).

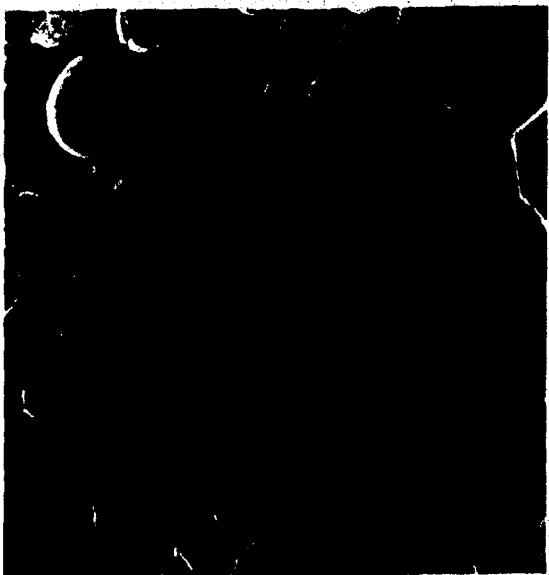
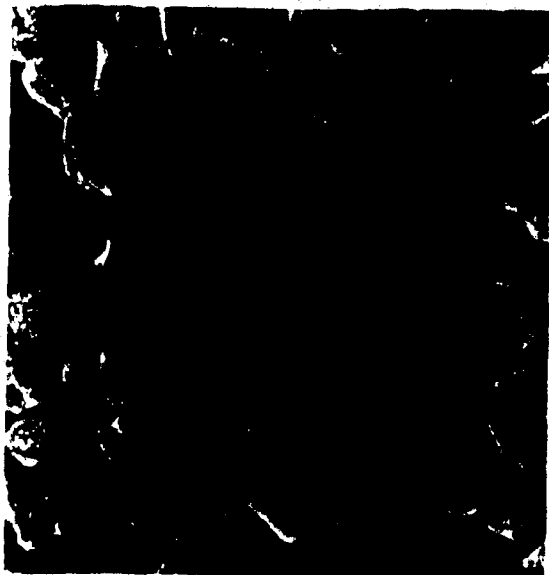


Plate 5.8. SE-Micrographs of Cassava Starch/Vital Gluten  
(a, b), and Sweet potato Starch/Vital Gluten  
(c, d) Dough Surfaces. Typical Surfaces (a, x1,260;  
c, x5,110). Fractured Surfaces(b, x1,280; d, x4,940).



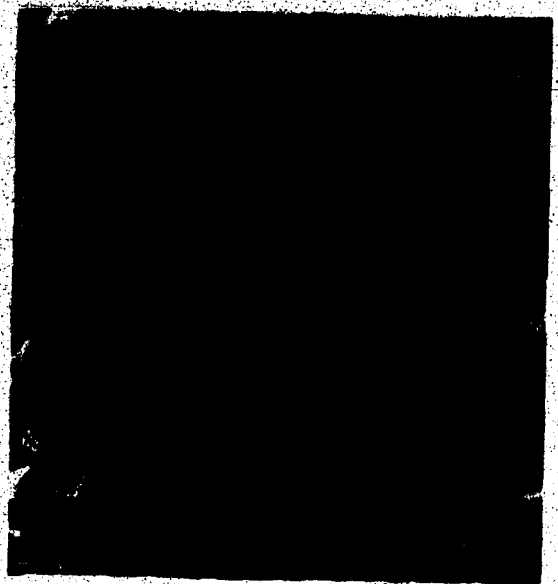
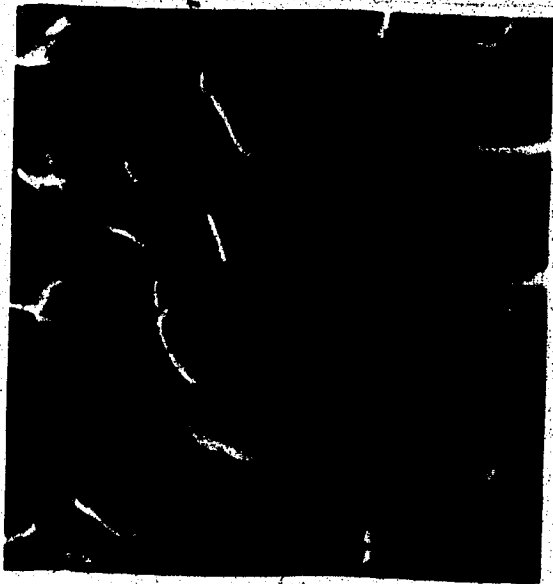
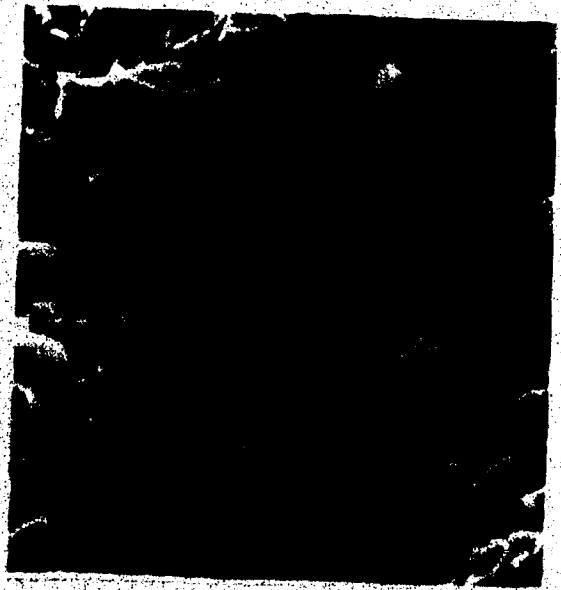
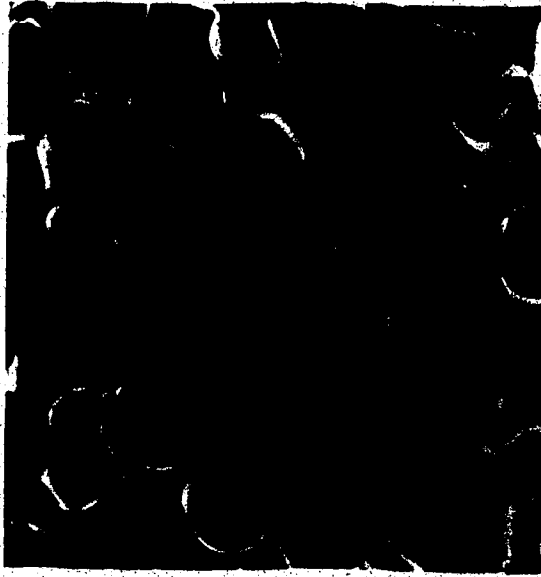


Plate 5.9. SE-Micrographs of Yam Starch/Vital Gluten Dough Surfaces.  
Typical Surfaces (a, x1,160; b, x3,010). Fractured  
Surfaces (b, x1,240; d, x5,040).

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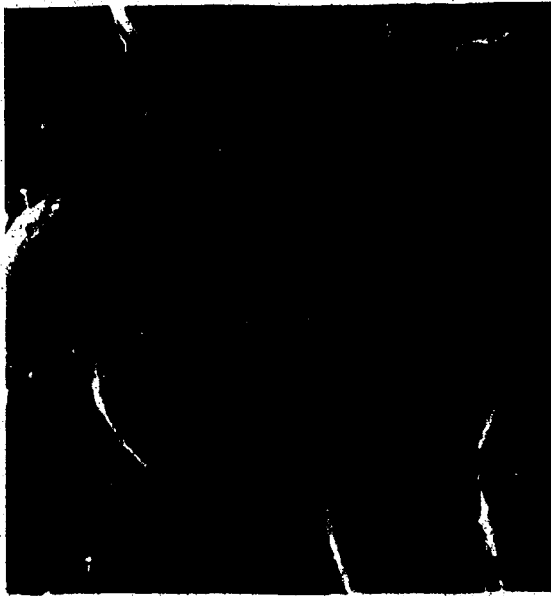


Plate 5.10. SE-Micrographs of Wheat, cvs. Neepawa (a, b), and Fielder  
(c, d) Flour dough Surfaces. Typical Surfaces (a, x2,510;  
c, x1,280). Fractured Surfaces (b, x2,420; d, x4,940).

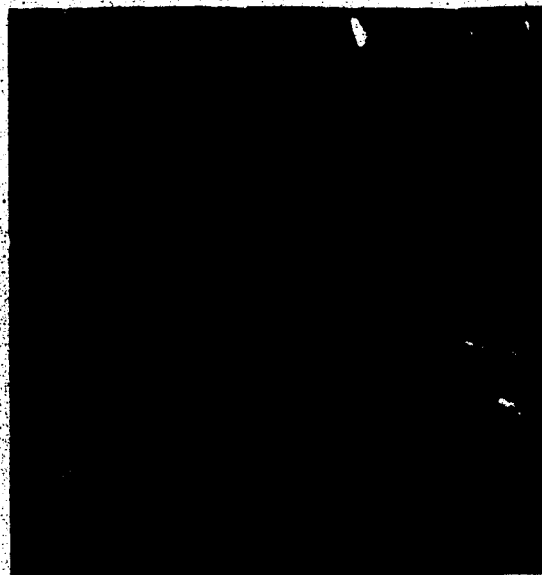


Plate 5.11. SE-Micrographs of Wheat, cv. Fielder (a, b, and c), and  
cv. Neepawa (d) Starch/Vital Gluten Dough Surfaces.  
Typical Surfaces (a, x1,260; c, x5,110). Fractured  
Surfaces (b, x1,250; d, x5,040).

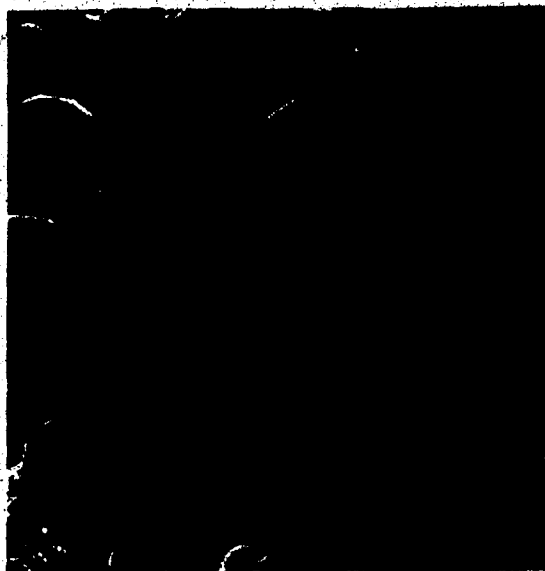


Plate 5.12. SE-Micrographs of Bread Crumb Surfaces of Arrowroot/Vital  
Gluten Composite -a, x6,900; b, x6,400; c, x2,100 and d, x770.



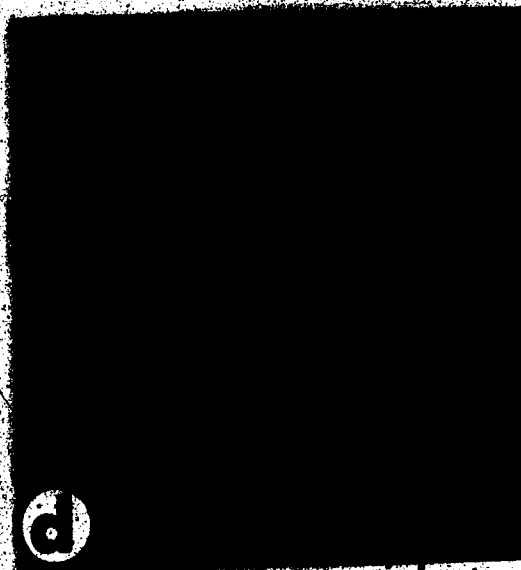
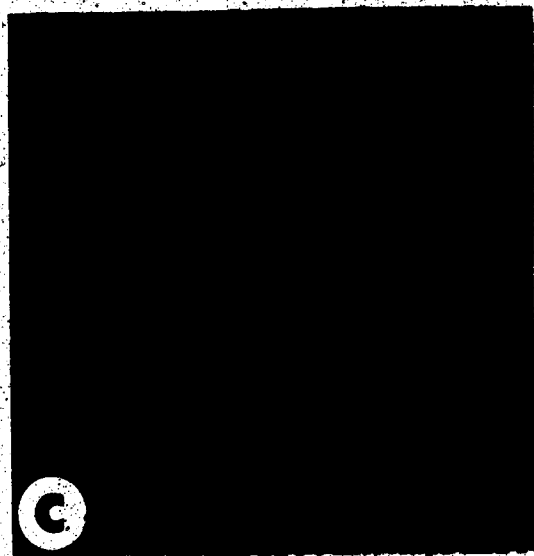


Plate 5.13. SE-Micrographs of Bread Crumb Surfaces of Srach/Vital Gluten Composites of Cassava- a, x1,700; Sweet potato- b, x1,700; Taro-c, x8000; and Yam- d, x960.

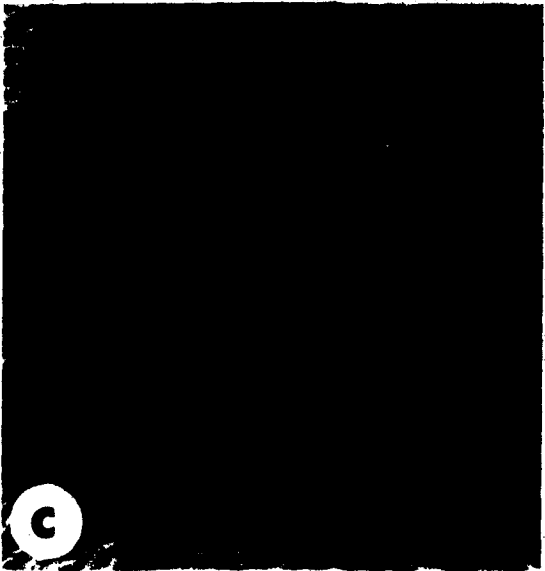
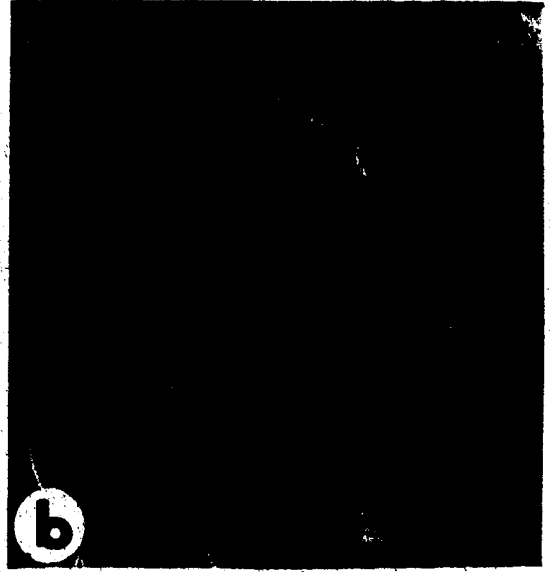
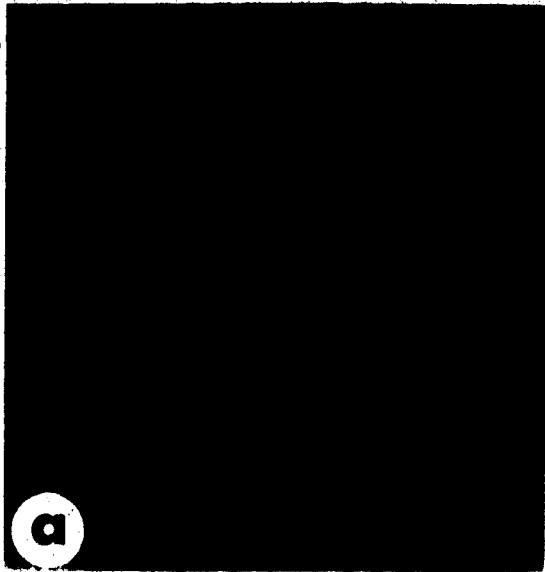


Plate 5.14. SE-Micrographs of Bread Crumb Surfaces of Wheat, cv. Fielder  
Starch/Vital Gluten Composite-a, x1,600; b, x870; and its  
Flour- c, 450; d, x1,600.



Plate 5.15. SE-Micrographs of Bread Crumb Surfaces of Wheat,  
cv. Neepawa Starch/Vital Gluten Composite- a,  
x1,700; b, x920; and its Flour-c, x930, d, x1,700.



Plate 5.16 TE- Micrographs of Arrowroot Starch/Vital Gluten  
Dough (c and d, each x1000); and Bread Crumb  
(a and b, each x550).





were observed to be embedded in the gluten matrix.

### 5.3.5 Bread Keeping Properties

#### 5.3.5.1 Bread Crumb Compressibility

Bread crumb compressibility curves and data in the presence and absence of monoglyceride are presented in Figure 5.31 and Table 5.38 for composite crumb consisting of vital gluten and starches from arrowroot cassava, sweet potato, taro and yam. Corresponding curves and data for the wheat starch flours and their starch/vital gluten composites are given in Figure 5.32 and Table 5.39.

Crumb compressibility was observed to differ from one type of bread to the other. In each case compressibility decreased as a function of storage time towards a limit reached after about 4 days storage in bread containing no monoglyceride and latter than 8 days storage in bread containing monoglycerides. Monoglyceride used at 0.5% level on flour basis reduced significantly the rate and extent of firming in the bread crumbs, as shown in Figures 5.32 and 5.33.

In general, increases in compressibility as a result of the presence of monoglyceride in the crumb were more significant in the composites of vital gluten with root starches than in the crumbs of wheat flours and their starch/vital gluten composites. In presence of monoglycerides, crumb of yam starch had the smallest compressibility, followed by arrowroot, sweet potato, taro

Figure 5.31. Bread Crumb Compressibilities of Tropical Root Starch/Vital Gluten Composite Flours in Presence and Absence of Monoglyceride

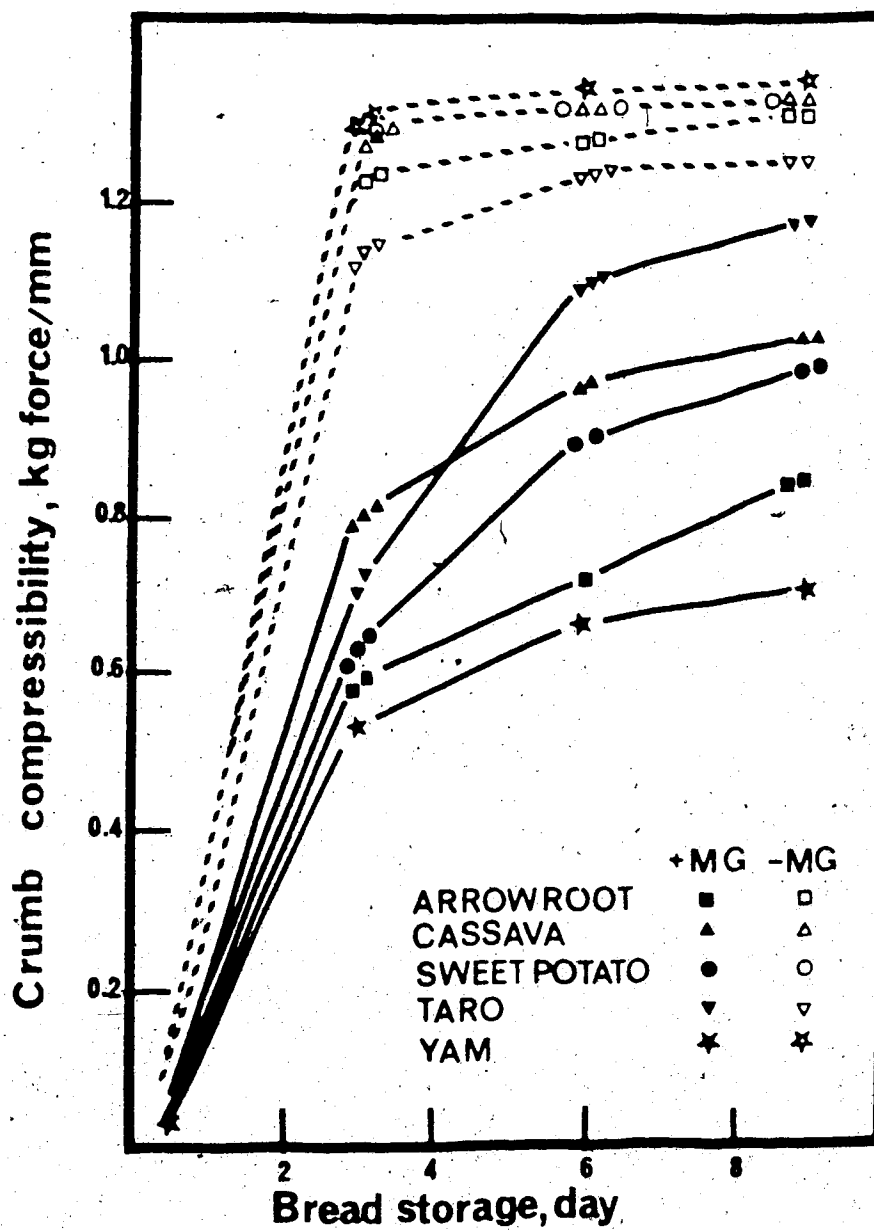


Table 5.38 Bread Crumb Compressibilities of Tropical Root  
Starch/VitalGluten Composite Flours in Presence and Absence  
of Monoglyceride

| Storage, Days                | Arrowroot       |                 | Cassava         |                 | Sweet Potato    |                | Taro            |                 | Yam             |                 |
|------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|----------------|-----------------|-----------------|-----------------|-----------------|
|                              | +MG*            | -MG             | +MG             | -MG             | +MG             | -MG            | +MG             | -MG             | +MG             | -MG             |
| Fresh Bread<br>(1/2 Day Old) | 0.020<br>+0.004 | 0.040<br>+0.008 | 0.040<br>+0.010 | 0.100<br>+0.012 | 0.080<br>+0.010 | 0.10<br>+0.002 | 0.090<br>+0.020 | 0.110<br>+0.005 | 0.080<br>+0.01  | 0.100<br>+0.004 |
| 3                            | 0.600<br>+0.040 | 1.240<br>+0.07  | 0.810<br>+0.070 | 1.290<br>+0.060 | 0.640<br>+0.210 | 1.300<br>+0.04 | 0.720<br>+0.070 | 1.140<br>+0.090 | 0.540<br>+0.090 | 1.310<br>+0.04  |
| 6                            | 0.730<br>+0.110 | 1.280<br>+0.05  | 0.970<br>+0.050 | 1.300<br>+0.060 | 0.900<br>+0.06  | 1.300<br>+0.05 | 1.100<br>+0.07  | 1.230<br>+0.070 | 0.660<br>+0.060 | 1.350<br>+0.07  |
| 9                            | 0.850<br>+0.090 | 1.320<br>+0.060 | 1.030<br>+0.090 | 1.320<br>+0.050 | 0.990<br>+0.100 | 1.330<br>+0.08 | 1.180<br>+0.07  | 1.250<br>+0.060 | 0.710<br>+0.08  | 1.360<br>+0.050 |

\*MG, Monoglyceride ( $\alpha$ -glycerol monopalmitate)

Figure 5.32. Bread Crumb Compressibility of Wheat Starch/Vital Gluten Composite Breads in Presence and Absence of Monoglycerides

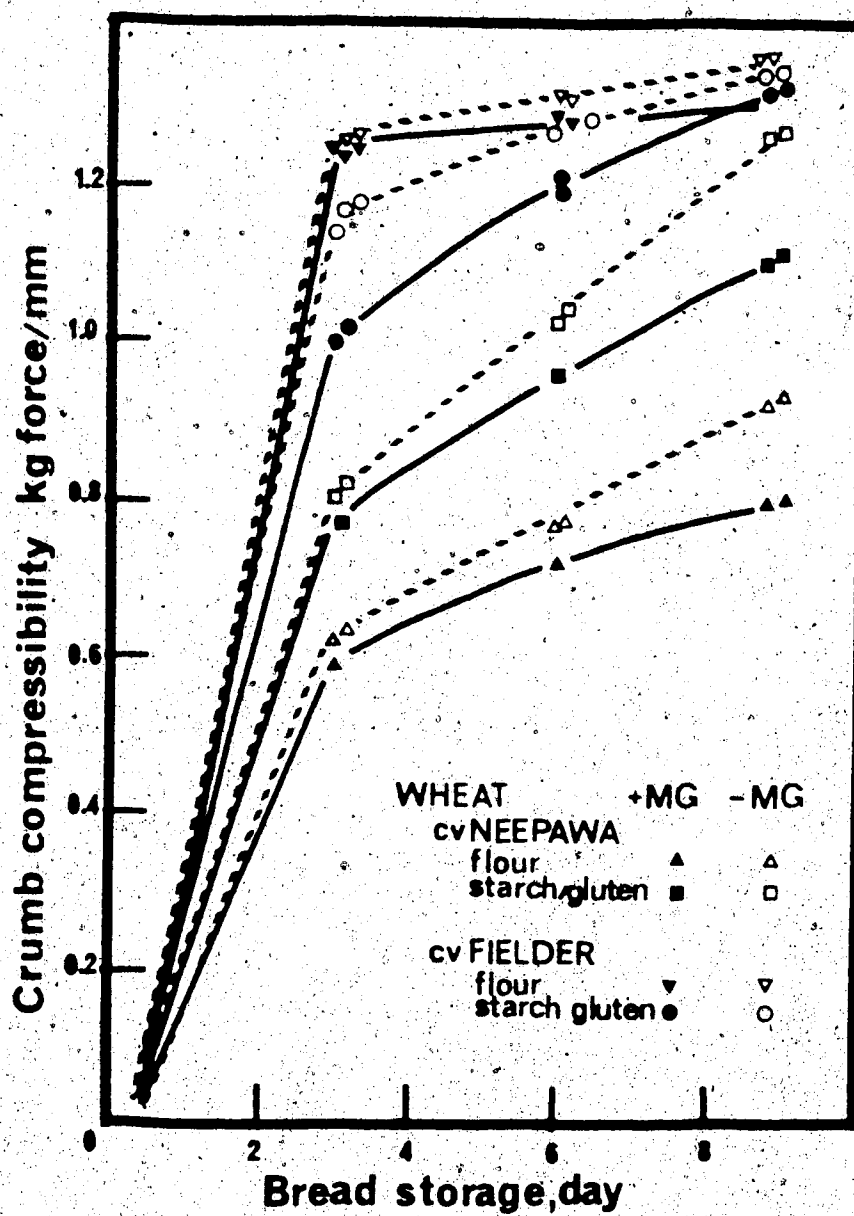


Table 5.39 Bread Crumb Compressibilities of Wheat and Wheat  
Starch/Vital Gluten Composite Flours in Presence and Absence  
of Monoglycerides

| Storage, Days | Neepawa |        |               |        | Fielder |        |               |        |
|---------------|---------|--------|---------------|--------|---------|--------|---------------|--------|
|               | Flour   |        | Starch/Gluten |        | Flour   |        | Starch/Gluten |        |
|               | +MG*    | -MG    | +MG           | -MG    | +MG     | -MG    | +MG           | -MG    |
| Fresh Bread   | 0.030   | 0.060  | 0.040         | 0.050  | 0.060   | 0.110  | 0.06          | 0.070  |
| (1/2 Day Old) | +0.004  | +0.030 | +0.006        | +0.010 | +0.001  | +0.005 | +0.008        | +0.007 |
| 3             | 0.590   | 0.620  | 0.770         | 0.800  | 1.000   | 1.160  | 1.250         | 1.250  |
|               | +0.080  | +0.040 | +0.090        | +0.035 | +0.080  | +0.040 | +0.070        | +0.080 |
| 6             | 0.720   | 0.750  | 0.950         | 1.020  | 1.210   | 1.270  | 1.270         | 1.320  |
|               | +0.060  | +0.010 | +0.007        | +0.140 | +0.060  | +0.070 | +0.030        | +0.150 |
| 9             | 0.790   | 0.930  | 1.100         | 1.270  | 1.320   | 1.340  | 1.290         | 1.340  |
|               | +0.060  | +0.221 | +0.040        | +0.03  | +0.060  | +0.05  | +0.050        | +0.040 |

\*MG,  $\alpha$ -glycerol monopalmitate.

(after 3 days), and cassava. In the absence of monoglyceride, yam bread had the firmest crumb, followed equally by sweet potato and cassava starch/vital gluten composites. Taro bread crumb, followed by arrowroot bread crumbs, had the least compressibility.

In absence or presence of monoglycerides, SWSW flour had firmer crumb than CWRWSW. A similar observation was made for wheat starch/vital gluten composites.

#### 5.3.5.2 Crumb Penetration Resistance

Plots for crumb penetration resistance to a standard probe and constant force as a function of storage time for the various tropical root starch/vital gluten bread composites are presented in Figure 5.33. The corresponding data are provided in Table 5.40. Similar plots and data for wheat flour breads and their starch/vital gluten bread composites are given in Figure 5.34 and Table 5.41.

The presence of monoglyceride in the crumb reduced the resistance to penetration of all crumbs as a function of time. The reduction was significant in all cases, as illustrated in Figures 5.33 and 5.34.

Among breads from the root starches, arrowroot bread was the most tender, followed by yam, taro, cassava and sweet potato in presence of monoglycerides. When no monoglycerides were incorporated, arrowroot bread still remained the most tender, followed by bread from taro, sweet potato, yam and cassava.

Figure 5.33. Bread Crumb Penetration Resistances of Tropical Root Starch/vital Gluten Composites in Presence and Absence of Monoglyceride

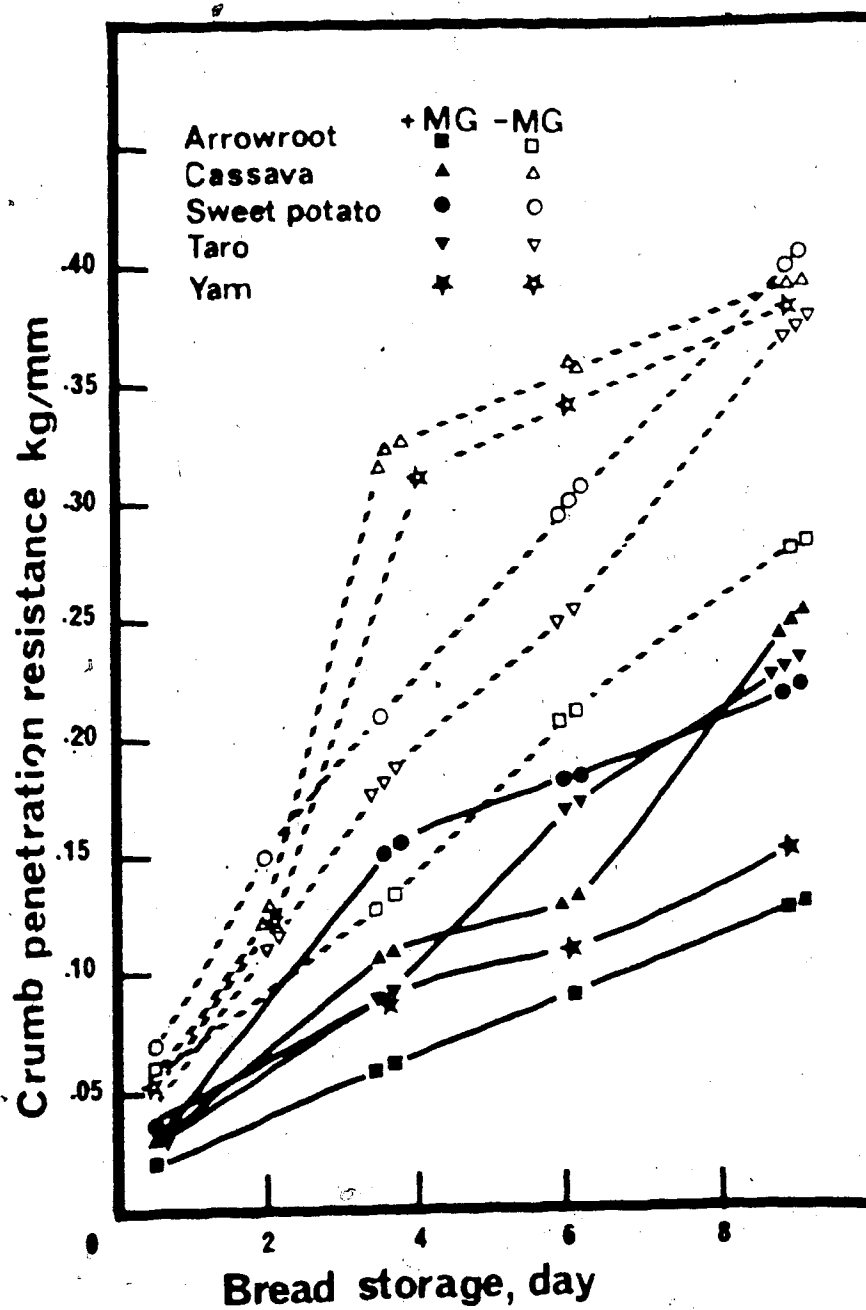




Table 5.40. Bread Crumb Penetration Resistance (kg-force/mm) of Tropical Root Starch /Vital Gluten Composites in Presence and Absence of Monoglycerides.

| Storage, Days             | Arrowroot       |                 | Cassava         |                 | Sweet Potato    |                 | Taro            |                 | Yam             |                 |
|---------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
|                           | +MG **          | -MG             | +MG             | -MG             | +MG             | -MG             | +MG             | -MG             | +MG             | -MG             |
| Fresh Bread (1/2 Day Old) | 0.020<br>+0.001 | 0.060<br>+0.002 | 0.030<br>+0.006 | 0.040<br>+0.005 | 0.030<br>+0.003 | 0.070<br>+0.004 | 0.040<br>+0.008 | 0.050<br>+0.006 | 0.03<br>+0.002  | 0.040<br>+0.003 |
| 2                         | -<br>-          | 0.110<br>+0.011 | -<br>-          | 0.130<br>+0.010 | -<br>-          | 0.15<br>+0.050  | -<br>-          | 0.110<br>+0.010 | -<br>-          | 0.120<br>+0.003 |
| 3 1/2                     | 0.060<br>+0.020 | 0.12<br>+0.010  | 0.110<br>+0.005 | 0.320<br>+0.030 | 0.15<br>+0.008  | 0.18<br>+0.030  | 0.090<br>+0.008 | 0.180<br>+0.020 | 0.090<br>+0.010 | 0.310<br>+0.040 |
| 6                         | 0.090<br>+0.020 | 0.210<br>+0.040 | 0.130<br>+0.020 | 0.360<br>+0.030 | 0.180<br>+0.020 | 0.30<br>+0.020  | 0.170<br>+0.010 | 0.250<br>+0.020 | 0.110<br>+0.020 | 0.340<br>+0.008 |
| 9                         | 0.130<br>+0.010 | 0.280<br>+0.030 | 0.250<br>+0.030 | 0.390<br>+0.020 | 0.220<br>+0.010 | 0.400<br>+0.030 | 0.230<br>+0.010 | 0.380<br>+0.050 | 0.150<br>+0.020 | 0.380<br>+0.020 |

\*Starch and vital gluten were used at 85 and 15%, respectively. For dough formulation see Table .

\*\*MG,  $\alpha$ -glycerol monopalmitate.

Figure 5.34 Bread Crumb Penetration Resistances of Wheat and Wheat Starch/Vital Gluten composites Flours in Presence and Absence of Monoglyceride

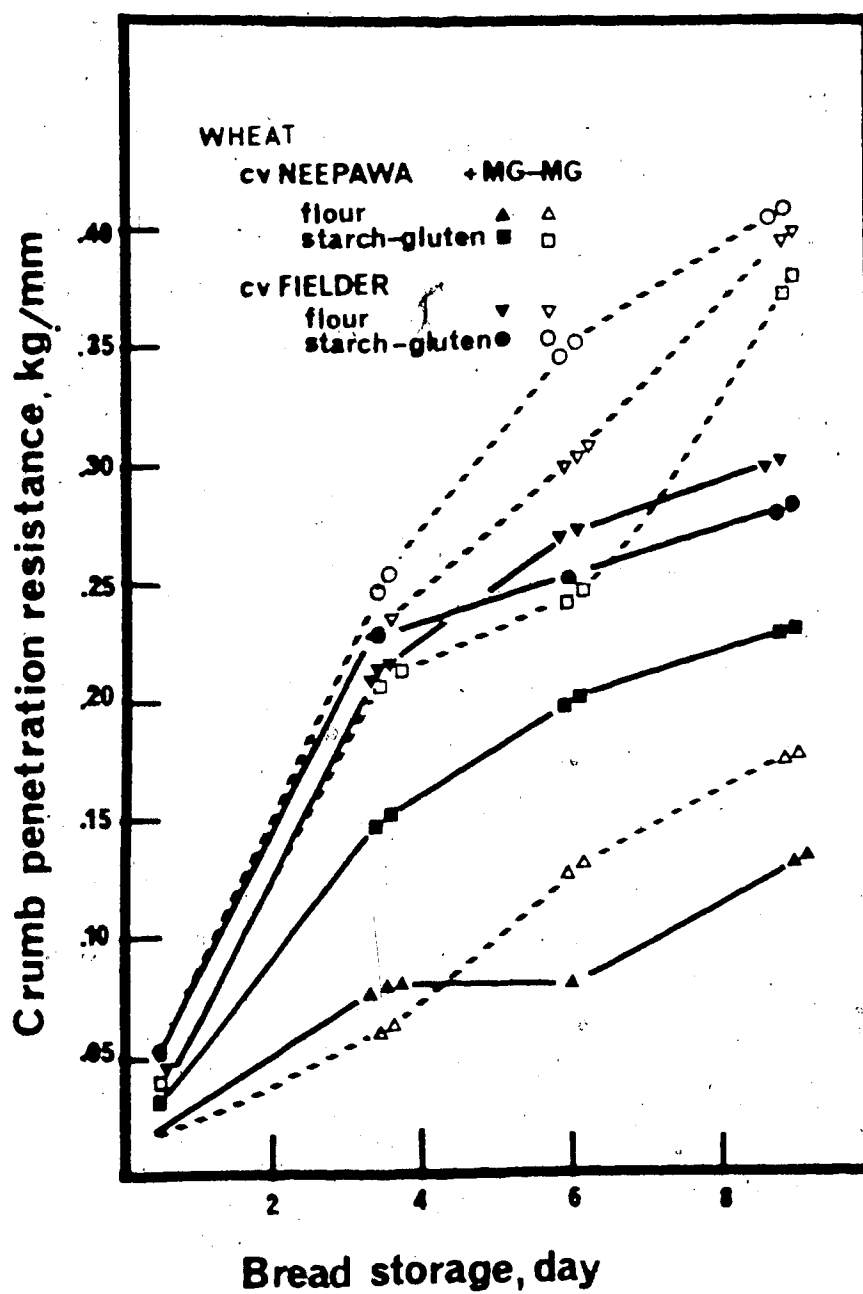


Table 5.41. Bread Crumb Penetration Resistances of Wheat Flour, and Wheat Starch /Vital Gluten Composite in Presence and Absence of Monoglycerides.

| Storage, Days | Neepawa |        |               |        | Fielder |        |               |        |
|---------------|---------|--------|---------------|--------|---------|--------|---------------|--------|
|               | Flour   |        | Starch/Gluten |        | Flour   |        | Starch/Gluten |        |
|               | +MG**   | -MG    | +MG           | -MG    | +MG     | -MG    | +MG           | -MG    |
| Fresh Bread   | 0.020   | 0.018  | 0.030         | 0.030  | 0.030   | 0.035  | 0.040         | 0.050  |
| (1/2 Day Old) | +0.002  | +0.003 | +0.002        | +0.003 | +0.004  | +0.005 | +0.005        | +0.007 |
| 3 1/2         | 0.080   | 0.060  | 0.150         | 0.210  | 0.210   | 0.230  | 0.230         | 0.250  |
|               | +0.010  | +0.006 | +0.010        | +0.020 | +0.020  | +0.006 | +0.040        | +0.030 |
| 6             | 0.08    | 0.13   | 0.200         | 0.240  | 0.270   | 0.300  | 0.250         | 0.350  |
|               | +0.010  | +0.040 | +0.030        | +0.030 | +0.030  | +0.002 | +0.040        | +0.01  |
| 9             | 0.130   | 0.160  | 0.230         | 0.380  | 0.300   | 0.400  | 0.280         | 0.410  |
|               | +0.013  | +0.010 | +0.030        | +0.030 | +0.010  | +0.040 | +0.006        | +0.02  |

\*Starch and vital gluten were used at 85 and 15%, respectively. For dough formulation see Table .

\*\*MG  $\alpha$ -glycerol monopalmitate.

For the wheat cases, CWRSW (cv. Neepawa) flour bread containing monoglycerides, was more tender than bread from the corresponding SWSW (cv. Fielder) flour. Similarly, composite bread of starch and vital gluten involving CWRSW was more tender than corresponding bread from SWSW. The effect of the presence of monoglycerides in the crumbs was more pronounced in the case of SWSW flour and starch/vital gluten composites, than in the corresponding cases with CWRSW.

With the exception of the CWRSW (cv. Neepawa) wheat flour, the presence of monoglyceride produced more pronounced effects in the root starch composites with gluten than in the other cases involving the CWRSW starch, SWSW flour and starch.

#### 5.3.5.3 X-Ray Diffraction Analysis

Retrogradation in bread crumbs as a function of storage time, followed by X-ray diffraction crystallinity analysis of starch isolated from the crumbs during storage is portrayed in Figures 5.35-5.38 for composite bread from vital gluten and starch from arrowroot, cassava, and wheat (cvs. Fielder and Neepawa). Figure 5.39 shows, for comparison, similar X-ray diffractograms for wheat (cv. Neepawa) flour. Corresponding data for the X-ray diffraction patterns are given in Tables 5.42-5.45.

Similar X-ray diffractograms and patterns for starch isolated from aging bread crumbs made from taro, sweet potato, and yam starches and from wheat (cv. Fielder) flour

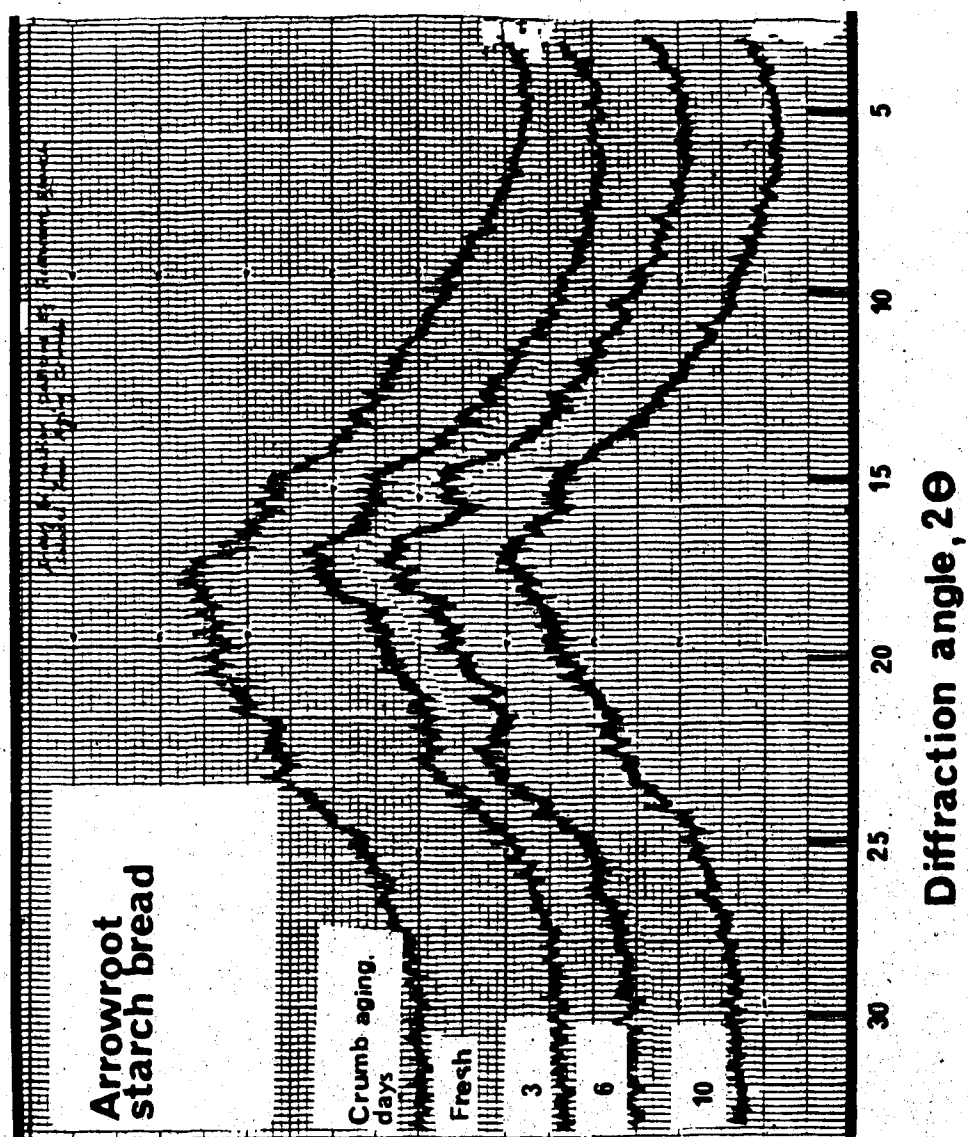


Figure 5.35. X-Ray Diffractograms of Starch Isolated From Aging Arrowroot Bread Crumbs.

Table 5.42. X-Ray Diffraction Patterns of Starch Isolated from Aging Arrowroot Bread Crumbs.

| Bread Storage, Days |        |                  |           |        |                  |           |        |                  |           |        |                  |
|---------------------|--------|------------------|-----------|--------|------------------|-----------|--------|------------------|-----------|--------|------------------|
| 1                   |        |                  |           | 3      |                  |           |        | 6                |           |        |                  |
|                     |        |                  |           |        |                  |           |        |                  |           |        |                  |
|                     |        |                  |           |        |                  |           |        |                  |           |        |                  |
| $2\theta$           | d, Å   | Intensity<br>cps | $2\theta$ | d, Å   | Intensity<br>cps | $2\theta$ | d, Å   | Intensity<br>cps | $2\theta$ | d, Å   | Intensity<br>cps |
| 15.163              | 5.8430 | 726              | 15.059    | 5.8831 | 674              | 10.322    | 8.5699 | 352              | 14.935    | 5.9318 | 657              |
| 17.455              | 5.0806 | 889              | 17.069    | 5.1945 | 831              | 11.407    | 7.7567 | 389              | 17.135    | 5.1746 | 772              |
| 20.100              | 4.4176 | 848              | 16.486    | 5.3770 | 772              | 14.929    | 5.9340 | 708              | 23.147    | 3.8426 | 496              |
| 21.235              | 4.1840 | 798              | 22.734    | 3.9114 | 562              | 17.070    | 5.1942 | 835              | 26.599    | 3.3511 | 336              |
| 23.154              | 3.8413 | 700              |           |        |                  | 17.888    | 4.9585 | 795              |           |        |                  |
| 31.642              | 2.8276 | 390              |           |        |                  | 21.201    | 4.1905 | 631              |           |        |                  |
|                     |        |                  |           |        |                  | 22.643    | 3.9269 | 626              |           |        |                  |
|                     |        |                  |           |        |                  | 26.424    | 3.3729 | 370              |           |        |                  |
|                     |        |                  |           |        |                  | 30.077    | 2.9710 | 343              |           |        |                  |

\*Arrowroot starch composite flours had 15% vital gluten.

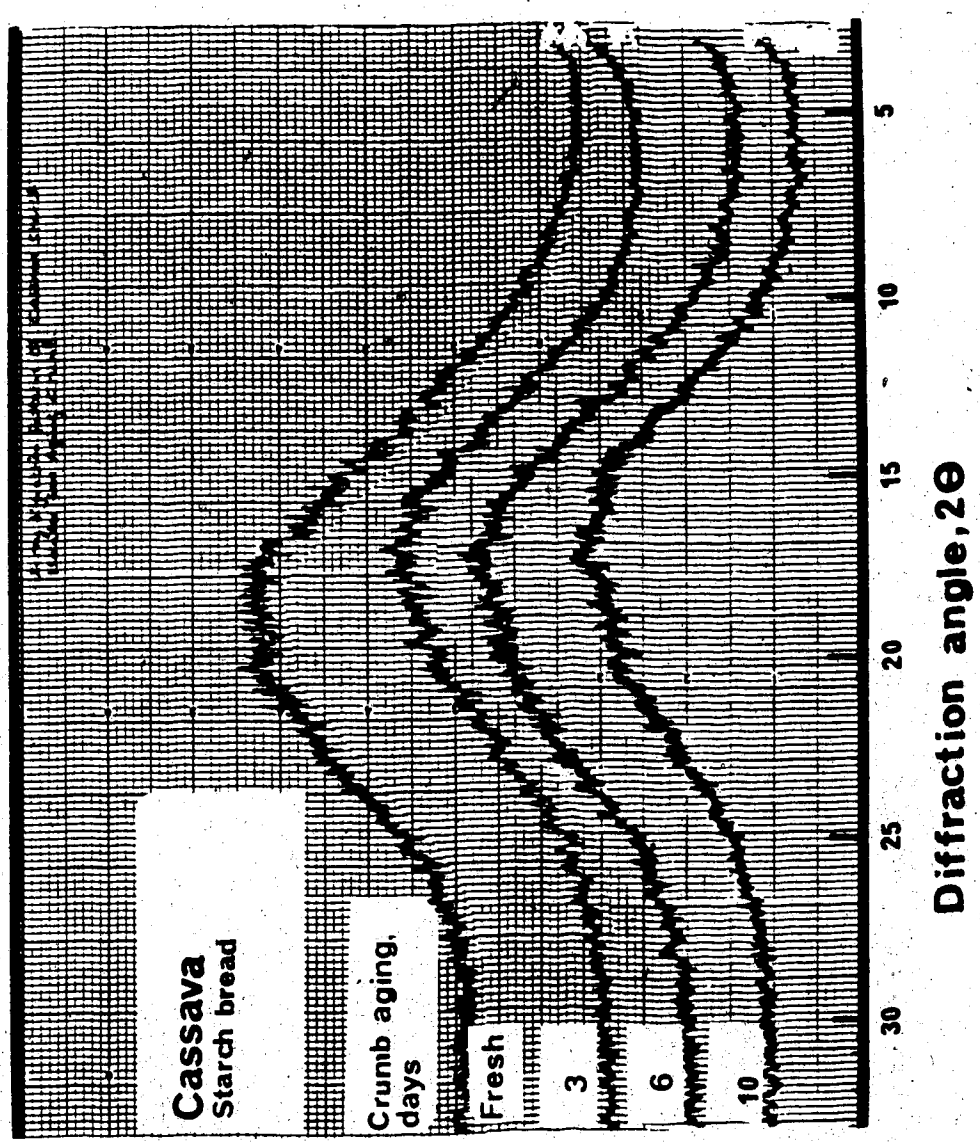


Figure 5.36 X-Ray Diffractograms of Starch Isolated from Aging Cassava Bread Crumbs

Table 5.43. X-Ray Diffraction Patterns of Starch Isolated from Aging Cassava Bread Crumbs.

| Bread Storage, Days |        |     |                   |           |     |                   |        |           |                   |        |     |                   |      |     |                   |      |     |
|---------------------|--------|-----|-------------------|-----------|-----|-------------------|--------|-----------|-------------------|--------|-----|-------------------|------|-----|-------------------|------|-----|
| 1                   |        |     |                   | 3         |     |                   |        | 6         |                   |        |     | 10                |      |     |                   |      |     |
|                     |        |     |                   |           |     |                   |        |           |                   |        |     |                   |      |     |                   |      |     |
| Intensity           |        |     |                   | Intensity |     |                   |        | Intensity |                   |        |     | Intensity         |      |     |                   |      |     |
| $^{\circ}2\theta$   | d, Å   | cps | $^{\circ}2\theta$ | d, Å      | cps | $^{\circ}2\theta$ | d, Å   | cps       | $^{\circ}2\theta$ | d, Å   | cps | $^{\circ}2\theta$ | d, Å | cps | $^{\circ}2\theta$ | d, Å | cps |
| 9.466               | 9.3431 | 244 | 12.489            | 7.0875    | 416 | 13.314            | 6.6499 | 507       | 13.732            | 6.4485 | 473 |                   |      |     |                   |      |     |
| 12.128              | 7.2977 | 402 | 14.561            | 6.0832    | 496 | 15.825            | 5.6000 | 720       | 14.892            | 5.9487 | 538 |                   |      |     |                   |      |     |
| 13.339              | 6.6374 | 497 | 14.728            | 6.0143    | 593 | 17.196            | 5.1565 | 775       | 17.231            | 5.1461 | 633 |                   |      |     |                   |      |     |
| 16.806              | 5.2753 | 802 | 15.527            | 5.7067    | 689 | 27.997            | 3.1869 | 333       | 18.479            | 4.8012 | 592 |                   |      |     |                   |      |     |
| 17.465              | 5.0778 | 830 | 19.289            | 4.6015    | 656 |                   |        |           | 20.235            | 4.3885 | 542 |                   |      |     |                   |      |     |
| 18.390              | 4.8243 | 827 | 20.203            | 4.3952    | 633 |                   |        |           | 22.678            | 3.9209 | 404 |                   |      |     |                   |      |     |
| 20.010              | 4.4373 | 821 | 22.542            | 3.9442    | 500 |                   |        |           |                   |        |     |                   |      |     |                   |      |     |
| 21.176              | 4.1954 | 748 | 24.384            | 3.6503    | 379 |                   |        |           |                   |        |     |                   |      |     |                   |      |     |
| 25.045              | 3.5555 | 500 |                   |           |     |                   |        |           |                   |        |     |                   |      |     |                   |      |     |
| 26.544              | 3.3580 | 417 |                   |           |     |                   |        |           |                   |        |     |                   |      |     |                   |      |     |
| 27.566              | 3.2357 | 419 |                   |           |     |                   |        |           |                   |        |     |                   |      |     |                   |      |     |
| 29.544              | 3.0234 | 389 |                   |           |     |                   |        |           |                   |        |     |                   |      |     |                   |      |     |



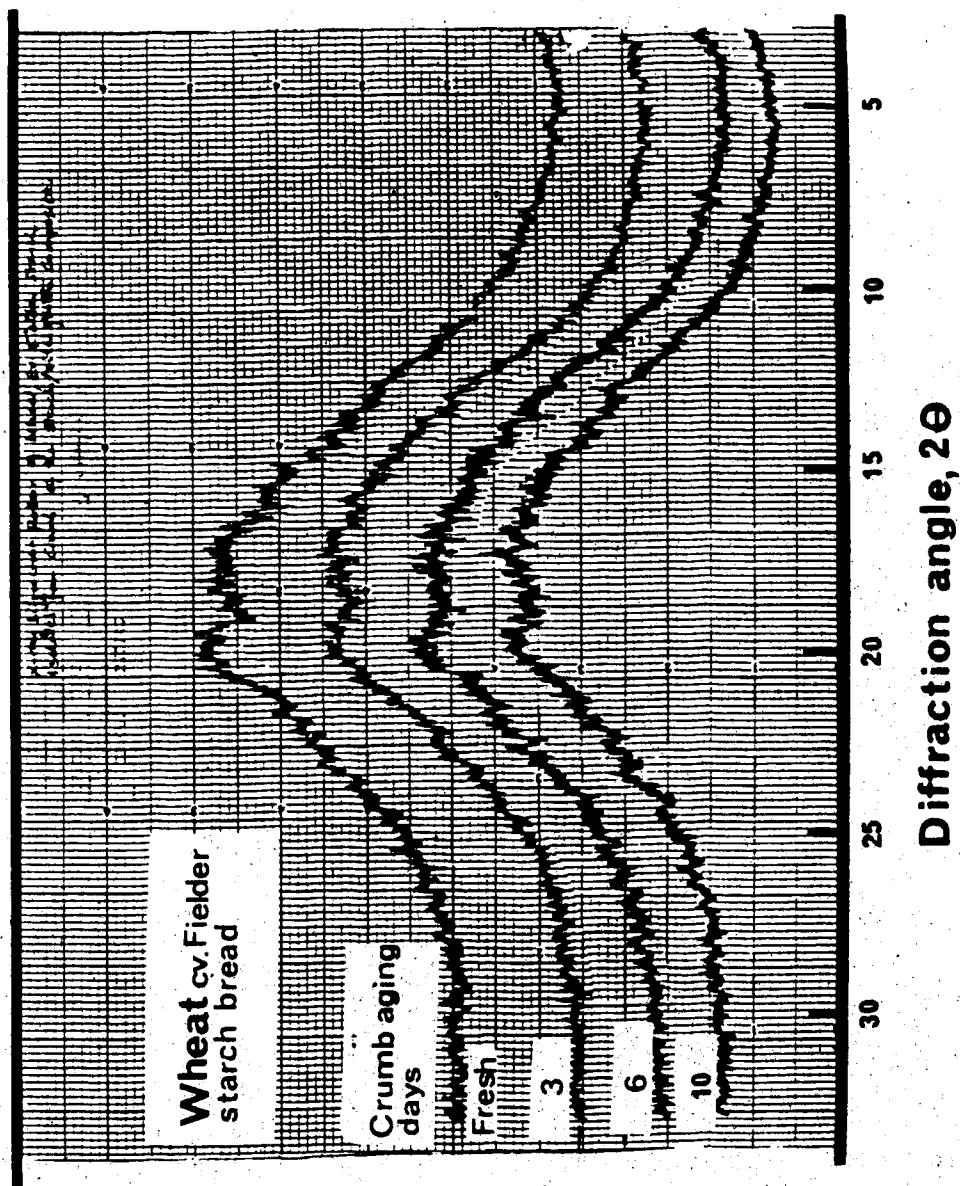


Figure 5.37. X-Ray Diffractograms of Starch Isolated From Aging Bread Crumbs of Wheat Starch, cv. Fielder.

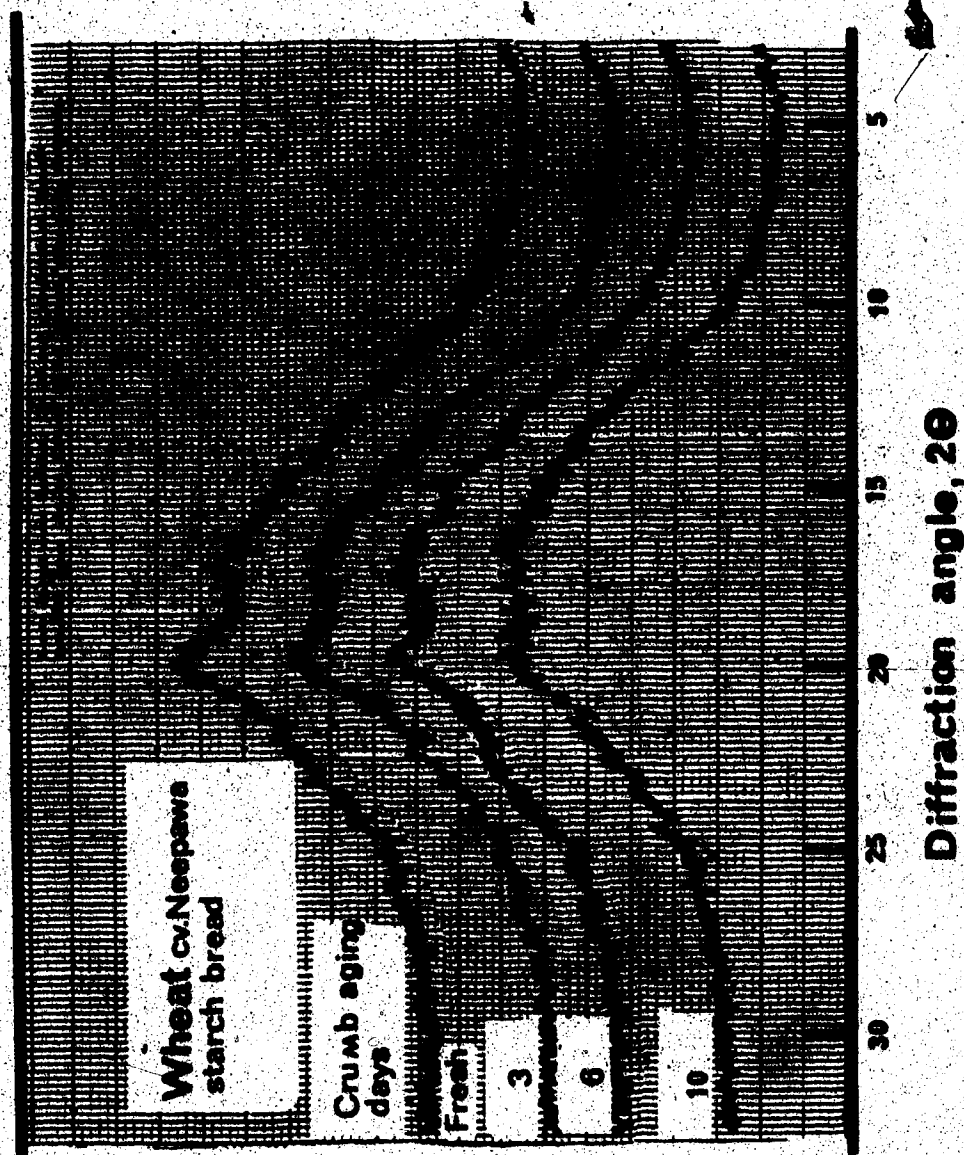


Figure 5.38. X-Ray Diffractograms of Starch Isolated From Aging Bread Crumbs of Wheat Starch, cv. Neepawa.

Table 5. 44. X-Ray Diffraction Patterns of Starch Isolated from Aging Bread Crumbs of Wheat, cvs. Fielder and Neepawa Starches.

|                          |                 | Bread Storage, Days |                 |           |                 |           |                 |           |                 |
|--------------------------|-----------------|---------------------|-----------------|-----------|-----------------|-----------|-----------------|-----------|-----------------|
|                          |                 | 3                   |                 | 6         |                 | 8         |                 | 10        |                 |
| $2\theta$                | $d, \text{\AA}$ | Intensity           |                 | Intensity |                 | Intensity |                 | Intensity |                 |
|                          |                 | cps                 | $d, \text{\AA}$ | cps       | $d, \text{\AA}$ | cps       | $d, \text{\AA}$ | cps       | $d, \text{\AA}$ |
| Wheat Starch cv. Fielder |                 |                     |                 |           |                 |           |                 |           |                 |
| 15.685                   | 5.6495          | 804                 | 15.798          | 5.6094    | 803             | 12.760    | 6.9372          | 516       | 12.931          |
| 17.184                   | 5.1601          | 900                 | 16.945          | 5.2324    | 853             | 13.417    | 6.5993          | 606       | 15.138          |
| 18.062                   | 4.9113          | 901                 | 17.984          | 4.9322    | 851             | 15.319    | 5.7837          | 730       | 17.496          |
| 19.436                   | 4.5671          | 930                 | 19.776          | 4.4892    | 855             | 16.773    | 5.2855          | 792       | 19.771          |
| 22.964                   | 3.8724          | 648                 | 22.411          | 3.9670    | 624             | 17.692    | 5.0131          | 803       | 21.691          |
|                          |                 |                     |                 |           |                 | 19.919    | 4.4574          | 849       | 23.483          |
|                          |                 |                     |                 |           |                 | 21.763    | 4.0837          | 853       | 24.626          |
|                          |                 |                     |                 |           |                 | 22.754    | 3.9380          | 812       | 26.257          |
|                          |                 |                     |                 |           |                 |           |                 |           | 335             |
| Wheat Starch cv. Neepawa |                 |                     |                 |           |                 |           |                 |           |                 |
| 21.381                   | 4.1857          | 920                 | 15.788          | 5.6301    | 723             | 15.154    | 5.8463          | 702       | 16.741          |
| 14.893                   | 5.9488          | 888                 | 17.332          | 5.1164    | 811             | 16.966    | 5.3511          | 787       | 19.753          |
| 15.382                   | 5.7486          | 780                 | 19.639          | 4.5303    | 859             | 17.369    | 5.1026          | 797       | 23.689          |
| 17.187                   | 5.1578          | 813                 | 22.359          | 3.9783    | 805             | 19.785    | 4.8877          | 811       | 24.580          |
| 19.311                   | 4.6061          | 919                 | 26.751          | 3.5324    | 505             | 20.149    | 4.6003          | 135       |                 |
| 21.213                   | 4.1882          | 782                 |                 |           |                 | 26.977    | 3.8668          | 759       |                 |
| 23.987                   | 3.7584          | 888                 |                 |           |                 |           |                 |           |                 |
| 25.147                   | 3.5503          | 819                 |                 |           |                 |           |                 |           |                 |

Wheat starch samples were prepared by the usual gluten.

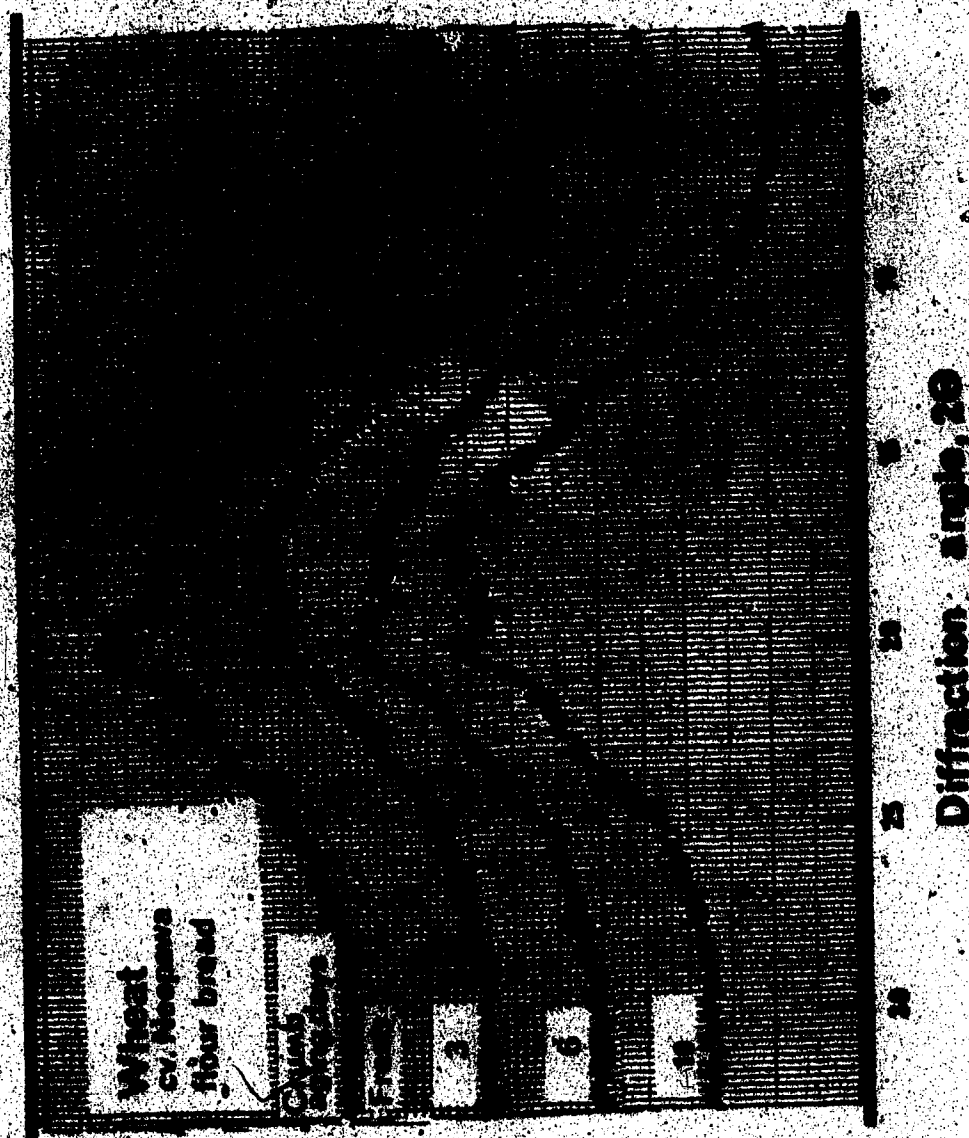


Figure 5.39. X-Ray Diffractograms of Starch Isolated from Aging Bread Crumbs made from Wheat Flour, cv. Neepawa.

Table 5.45 X-Ray Diffraction Patterns of Starch Isolated  
from Aging Bread Crumbs Made from Wheat Flour, cv. Neepawa

| Bread Storage, Days |        |     |           |        |     |           |        |     |           |        |     |
|---------------------|--------|-----|-----------|--------|-----|-----------|--------|-----|-----------|--------|-----|
| 1                   |        |     | 3         |        |     | 6         |        |     | 10        |        |     |
| Intensity           |        |     | Intensity |        |     | Intensity |        |     | Intensity |        |     |
| $2\theta$           | d, Å   | cps | $2\theta$ | d, Å   | cps | $2\theta$ | d, Å   | cps | $2\theta$ | d, Å   | cps |
| 13.045              | 6.7965 | 526 | 13.054    | 6.7918 | 505 | 12.748    | 6.9400 | 543 | 13.473    | 6.5721 | 574 |
| 14.005              | 6.4577 | 555 | 14.109    | 6.4073 | 753 | 15.109    | 5.7853 | 687 | 15.509    | 5.7000 | 823 |
| 15.000              | 6.3200 | 561 | 15.017    | 6.3254 | 739 | 16.054    | 5.2651 | 783 | 16.500    | 5.3249 | 845 |
| 17.200              | 6.2000 | 525 | 17.045    | 6.4516 | 745 | 18.180    | 5.0791 | 791 | 17.503    | 5.0515 | 903 |
| 18.000              | 6.1667 | 507 | 18.184    | 6.0827 | 599 | 19.050    | 4.8800 | 859 | 19.918    | 4.4679 | 888 |
| 20.000              | 6.0000 | 503 | 20.000    | 6.0000 | 500 | 20.000    | 6.0000 | 866 | 22.000    | 4.0720 | 883 |
| 22.000              | 5.4545 | 544 | 22.000    | 5.4545 | 500 | 22.000    | 5.4545 | 866 | 23.715    | 3.7657 | 905 |
| 24.000              | 5.2083 | 530 | 24.000    | 5.2083 | 500 | 24.000    | 5.2083 | 866 |           |        |     |
| 25.000              | 5.1680 | 534 | 25.000    | 5.1680 | 500 | 25.000    | 5.1680 | 866 |           |        |     |



are given in appendices 29-35.

In all cases the X-ray diffractograms of the starches became more defined the longer the crumb was stored. Characteristic strong intensity X-ray diffraction peaks were observed between 15-25° (2 $\theta$ ) angle of diffraction in all cases.

### 5.3.5 Bread Quality Evaluation

#### 5.3.5.1 Loaf Volume, Mass and Specific Volume

Loaf volumes, masses and specific volumes of the different types of bread are presented in Table 5.46.

Canadian western red spring wheat (cv. Neepawa), bread, used as the standard, had the largest loaf volume. Its specific volume was also the largest. Neepawa wheat starch/vital gluten bread was second to the standard in these properties.

Sweet potato starch/vital gluten bread had the largest loaf volume amongst the root starch/vital gluten composites. It was closely followed by taro starch/vital gluten bread. Arrowroot starch/vital gluten bread had the smallest volume followed by yam starch/vital gluten bread. Cassava starch/vital gluten composite bread had an intermediate volume which was greater than that of the internal standard wheat (cv. Fielder) flour bread. Fielder wheat starch/vital gluten composite bread, however, had a larger volume, but a smaller specific volume than the cassava composite bread.

Table 5.46 Bread Volume, Mass and Specific Volume

| Type of Bread        | Volume<br>cm <sup>3</sup> | Mass<br>g   | Specific Volume<br>cm <sup>3</sup> /g |
|----------------------|---------------------------|-------------|---------------------------------------|
| Neepawa flour        | 1532.0 ± 3.0              | 435.0 ± 3.0 | 3.52                                  |
| Neepawa starch*      | 1302.0 ± 5.0              | 443.0 ± 4.0 | 2.94                                  |
| Sweet potato starch* | 1192.0 ± 4.0              | 426.0 ± 3.0 | 2.80                                  |
| Taro starch*         | 1172.0 ± 3.0              | 426.0 ± 3.0 | 2.75                                  |
| Fielder starch*      | 1122.0 ± 2.0              | 457.0 ± 3.0 | 2.46                                  |
| Cassava*             | 1112.0 ± 6.0              | 476.0 ± 4.0 | 2.34                                  |
| Fielder flour        | 1051.0 ± 7.0              | 522.0 ± 3.0 | 2.01                                  |
| Yam*                 | 1022.0 ± 2.0              | 522.0 ± 3.0 | 1.96                                  |
| Arrowroot*           | 922.0 ± 3.0               | 476.0 ± 4.0 | 1.94                                  |

\*plus vital gluten.

In general, the composite breads from the root starches with vital gluten were closer to the internal standard in their volumes and specific volumes than the actual CWRS wheat bread.

#### 5.3.6.2 Internal and External Bread Qualities

The panelists' mean and total scores for the external (volume excluded), and internal properties of bread loaves from the wheat flours of cvs. Neepawa and Fielder, and from their starches as well as root/starch composites with vital gluten are presented in Table 5.47.

Bread from wheat (cv. Neepawa) flour was arbitrarily given the perfect score for each property investigated, so that its total score was 90 since the score 10 for volume was not included due to volume having been given a separate analysis. This was done for convenience in the statistical analyses and comparisons. The total mean scores were found to be a suitable index for assessing the total preference of the composite breads in comparison to the standard.

The analysis of variance amongst the quality properties of the different bread loaves, also excluding volume, and the panelists' perceptions of the degree of difference between the various characteristics in different breads are presented in Table 5.48.

The analysis of variance showed that the variability amongst the panelists' perceptions of differences in the color of the crust, break and shred, character of the crust, grain and chewability were significant at both 95



Table 5.47 Bread Quality Evaluation<sup>a</sup>

| Type of Bread   | Panelists' Mean Scores <sup>b</sup> |                |                  |                 |                        |                    |       |       |       |             | Total Score |
|-----------------|-------------------------------------|----------------|------------------|-----------------|------------------------|--------------------|-------|-------|-------|-------------|-------------|
|                 | Color of the Crust                  | Crust Symmetry | Evenness of Bake | Break and Shred | Character of the Crust | Color of the Crumb | Grain | Aroma | Taste | Chewability | Texture     |
| White           | 8.00                                | 3.00           | 1.00             | 1.00            | 1.00                   | 10.00              | 10.00 | 10.00 | 15.00 | 10.00       | 15.00       |
| Whole Wheat     | 8.00                                | 2.25           | 2.30             | 1.50            | 1.80                   | 8.90               | 8.31  | 9.40  | 12.89 | 7.70        | 11.55       |
| Whole Rye       | 5.75                                | 2.50           | 1.50             | 1.50            | 2.00                   | 9.66               | 9.28  | 5.62  | 12.07 | 7.82        | 13.69       |
| Whole Oat       | 6.50                                | 1.50           | 1.75             | 1.25            | 2.00                   | 9.35               | 7.95  | 6.75  | 9.28  | 6.78        | 9.52        |
| Whole Corn      | 4.25                                | 1.00           | 1.50             | 1.75            | 1.75                   | 7.61               | 9.17  | 8.82  | 9.45  | 5.29        | 9.17        |
| Whole Barley    | 4.25                                | 1.25           | 0.75             | 1.50            | 0.50                   | 8.41               | 7.67  | 6.39  | 8.32  | 4.37        | 6.19        |
| Whole Speltz    | 4.00                                | 1.50           | 1.13             | 1.50            | 0.50                   | 7.03               | 8.43  | 9.35  | 8.09  | 4.37        | 6.19        |
| Whole Triticale | 4.25                                | 0.75           | 0.50             | 0.50            | 0.50                   | 4.50               | 3.00  | 2.82  | 8.67  | 3.00        | 3.00        |
| Whole Emmer     | 4.25                                | 0.50           | 0.75             | 0.50            | 0.50                   | 1.30               | 1.50  | 1.50  | 3.39  | 1.50        | 1.50        |

<sup>a</sup>Based on 100-point scale for each attribute.

<sup>b</sup>Panelists were trained to use this scale.

Table 5.48 Analysis of Variance in Scores for Loaf Characteristics\* and Panelists' Perceptions of Characteristic Differences in Different Brands

| Source of Variation        | Sum of Squares | Degrees of Freedom | Mean of Squares (Variance) | F Value    |           |
|----------------------------|----------------|--------------------|----------------------------|------------|-----------|
|                            |                |                    |                            | Calculated | 95% 99%   |
| 1. Color of the crust:     |                |                    |                            |            |           |
| Loaves**                   | 496.51         | 8                  | 62.31                      | 70.81      | 2.05 2.74 |
| Panelists***               | 49.38          | 11                 | 4.49                       | 5.10       | 1.91 2.48 |
| Interaction                | 77.34          | 88                 | 0.88                       |            |           |
| 2. Symmetry of form:       |                |                    |                            |            |           |
| Loaves                     | 59.99          | 8                  | 7.50                       | 19.32      | 2.05 2.74 |
| Panelists                  | 10.27          | 11                 | 0.93                       | 2.40       | 1.91 2.48 |
| Interaction                | 34.16          | 88                 | 0.39                       |            |           |
| 3. Breeness of bake:       |                |                    |                            |            |           |
| Loaves                     | 82.83          | 8                  | 10.35                      | 27.80      | 2.05 2.74 |
| Panelists                  | 6.21           | 11                 | 0.56                       | 1.52       | 1.91 2.48 |
| Interaction                | 32.77          | 88                 | 0.37                       |            |           |
| 4. Break and shred:        |                |                    |                            |            |           |
| Loaves                     | 52.13          | 8                  | 6.52                       | 14.44      | 2.05 2.74 |
| Panelists                  | 21.80          | 11                 | 1.98                       | 4.39       | 1.91 2.48 |
| Interaction                | 39.71          | 88                 | 0.45                       |            |           |
| 5. Character of the crust: |                |                    |                            |            |           |
| Loaves                     | 75.64          | 8                  | 9.46                       | 42.51      | 2.05 2.74 |
| Panelists                  | 9.90           | 11                 | 0.90                       | 4.05       | 1.91 2.48 |
| Interaction                | 19.57          | 88                 | 0.22                       |            |           |
| 6. Color of the crumb:     |                |                    |                            |            |           |
| Loaves                     | 375.4          | 8                  | 46.93                      | 13.89      | 2.67 2.77 |
| Panelists                  | 56.23          | 9                  | 6.25                       | 1.85       | 2.01 2.67 |
| Interaction                | 243.27         | 72                 | 3.38                       |            |           |



and 99% levels of significance. These characteristics were found to differ significantly amongst bread loaves and between each test loaf and the standard. The significance in difference was highest for color of the crust and decreased significantly through the character of the crust, break and shred and grain, to lowest in chewability.

Insignificant variability in the panelists' abilities to detect differences in evenness of bake, color of the crust, aroma and texture was observed at both 95 and 99% levels. Different breads were found to differ significantly in these characteristics. The significance in difference diminished progressively from the evenness of bake to crumb color, aroma, and texture.

Panelists differed less significantly at the 95% level in their abilities to perceive differences in the symmetry of form and taste between the different breads. The difference became insignificant at the 99% level. Significant differences were observed in all breads in these characteristics, less significant differences being observed for taste than for symmetry of form.

Calculated F-values for the bread quality attributes available in Table 5.48 showed that the magnitude of difference amongst the test loaves or test loaves and the standard wheat flour bread was contributed to by varying amounts from the various bread characteristics.

The list of the quality attributes and their corresponding calculated F-values in Table 5.49, in



Table 5.49 Bread Loaf Quality Attributes and Their Calculated  
P Values

| Quality Attribute               | Calculated P Value |
|---------------------------------|--------------------|
| <b>External Loaf Properties</b> |                    |
| Color of the crust              | 70.81              |
| Character of the crust          | 42.51              |
| Evenness of bake                | 27.89              |
| Symmetry of form                | 19.32              |
| Break and shred                 | 14.44              |
| <b>Internal Loaf Properties</b> |                    |
| Color of the crumb              | 13.85              |
| Grain                           | 12.08              |
| Chewability                     | 11.41              |
| Texture                         | 11.23              |
| Taste                           | 9.09               |
| Aroma                           | 8.41               |

\*The greater the P value for a quality attribute, the greater was found to be the contribution of that attribute to the quality differences amongst the different composite breads and between composite breads and the standard wheat bread.

conjunction with panelists' mean scores in Table 5.47 and acceptability mean scores in Table 5.50, indicated that bread quality attributes with greater calculated F-values contributed more extensively to the quality differences amongst the test loaves and between such loaves and the standard wheat flourbread.

The acceptability scores provided in Table 5.50 were derived from data in Table 5.47. In Table 5.51 are summarized the means of the scores accorded to the different breads studied. The grand mean totals in the last row show the order of preference of the starch/vital gluten composite breads by the panelists.

Table 3.50 Bread Quality Attributes Acceptability Rating in Percent

| Type of Bread Crust | Acceptability Mean Scores As Judged by the Taste Panelists |               |                    |                 |                        |              |        |        |        |                     |
|---------------------|--|---------------|--------------------|-----------------|------------------------|--------------|--------|--------|--------|---------------------|
|                     | Color of the Crust   | Loaf Symmetry | Evenness of Baking | Break and Shred | Character of the Crust | Color of the | Grain  | Aroma  | Taste  | Chewability Texture |
| Sheet               |  |               |                    |                 |                        |              |        |        |        |                     |
| CR. Napoleon        | 100.00   | 100.00        | 100.00             | 100.00          | 100.00                 | 100.00       | 100.00 | 100.00 | 100.00 | 100.00              |
| CR. Napoleon        | 71.88  | 66.00         | 64.00              | 64.00           | 80.00                  | 96.66        | 92.80  | 54.20  | 80.47  | 91.27               |
| CR. Fielder         | 76.00  | 75.00         | 75.67              | 66.00           | 62.67                  | 89.80        | 83.10  | 94.00  | 85.93  | 77.00               |
| CR. Fielder         | 79.13  | 61.33         | 59.67              | 60.33           | 68.00                  | 95.50        | 79.50  | 57.20  | 62.53  | 63.47               |
| CR. Fielder         | 51.13  | 62.67         | 46.00              | 58.33           | 57.00                  | 76.10        | 91.70  | 85.20  | 63.27  | 61.13               |
| CR. Fielder         | 28.13  | 51.00         | 24.00              | 53.33           | 17.00                  | 84.10        | 74.70  | 63.90  | 55.57  | 61.13               |
| CR. Fielder         | 27.13  | 33.33         | 12.00              | 30.67           | 25.67                  | 65.20        | 80.20  | 48.20  | 57.80  | 57.93               |
| CR. Fielder         | 33.36  | 52.67         | 37.67              | 52.67           | 30.67                  | 70.50        | 84.30  | 67.20  | 51.93  | 41.27               |
| CR. Fielder         | 17.88  | 8.33          | 5.67               | 12.67           | 14.00                  | 31.80        | 33.70  | 47.00  | 35.93  | 35.73               |

CR. stands for CRust and 100 stands for 100 percent.

CRust stands for CRust and 100 stands for 100 percent.

Table 5.51 Bread Quality Mean Sensory Tasting Scores

| Bread Quality Attributes | No. of Tasters (Standard) | Type of Bread                           |                   |                   |         |        |              |        |           |  |  |
|--------------------------|---------------------------|---|-------------------|-------------------|---------|--------|--------------|--------|-----------|--|--|
|                          |                           | Type of Starch + Vital Gluten           |                   |                   |         |        |              |        |           |  |  |
|                          |                           | Fielder Wheat Flour (Internal Standard) | Wheat cv. Neepawa | Wheat cv. Fielder | Cassava | Yam    | Sweet Potato | Taro   | Arrowroot |  |  |
| Crust color              | 8                         | 6.0833                                  | 5.7500            | 6.3333            | 4.2500  | 2.4546 | 2.9091       | 2.3636 | 2.1500    |  |  |
| Symmetry of bake         | 3                         | 2.2500                                  | 1.9750            | 1.8417            | 1.8750  | 1.6727 | 1.5833       | 1.1125 | 1.0000    |  |  |
| Evenness of bake         | 3                         | 2.3000                                  | 1.9167            | 1.7917            | 1.3750  | 1.0750 | 1.3500       | 0.8600 | 1.0000    |  |  |
| Break and shred          | 3                         | 2.1636                                  | 1.9167            | 1.3273            | 1.7500  | 1.9200 | 1.7273       | 1.3750 | 1.1250    |  |  |
| Character of crust       | 3                         | 1.9167                                  | 2.6817            | 2.1167            | 1.2778  | 1.200  | 1.5000       | 1.0000 | 1.7500    |  |  |
| Crumbs color             | 10                        | 7.9000                                  | 8.5000            | 8.4000            | 6.7000  | 7.4000 | 6.2000       | 4.0000 | 3.9000    |  |  |
| Grain                    | 10                        | 6.9000                                  | 7.7000            | 6.6000            | 7.6000  | 6.2000 | 7.0000       | 5.0000 | 3.8000    |  |  |
| Aroma                    | 10                        | 7.8000                                  | 5.0000            | 5.6833            | 7.4000  | 5.8889 | 5.6000       | 4.0000 | 3.9000    |  |  |
| Taste                    | 15                        | 11.000                                  | 10.3000           | 8.6889            | 8.1000  | 5.8889 | 6.9000       | 7.4000 | 5.1100    |  |  |
| Texture                  | 15                        | 9.7000                                  | 11.5000           | 9.0000            | 7.7000  | 5.2000 | 5.2000       | 7.3000 | 5.6300    |  |  |
| Chewability              | 10                        | 6.7000                                  | 6.8000            | 5.2000            | 4.6000  | 3.8000 | 3.8000       | 5.3000 | 3.8800    |  |  |
| Grand Mean Totals        | 90                        | 64.81                                   | 64.05             | 55.91             | 52.63   | 44.70  | 43.77        | 39.71  | 32.55     |  |  |



## 6. DISCUSSION

### 6.1 Granule Size Distribution and Morphology

As reported by earlier investigators (Shoch and Maywald, 1956; D'Appolonia et al., 1971) it was found that starch granules were distinguishable in their size and morphology by which they could be identified.

Arrowroot and taro starch granules were the smallest ( $<5\mu\text{m}$ ) and polygonal in shape. Some were compounded. Taro starch granules were the largest ( $>10\mu\text{m}$ ) and oval in shape. Cassava and sweet potato starch granules were intermediate in size, with rounded edges and corners. Cassava starch contained a mixture of round and truncated granules. The truncated surfaces were concave with a point at the center, and some granules were compounded.

Starch granule sizes and morphologies were found comparable to similar results reported by Bradley, 1940; Amin, 1955; Matwejwe, 1958; Seidemann, 1963, 1964; Wivinia and Maywald, 1967; Shigashihara et al., 1975; Ciacco and D'Appolonia, 1977; and Onwueme, 1978. Some of the investigators found larger size distributions for arrowroot and taro. The differences observed in all starches were attributed to genetic and environmental influences.

Existence of large lenticular and small spherical granules in wheat starch (Kerr, 1950) was confirmed. However, different sizes of lenticular and spherical starch granules were observed. Based on this observation, it appeared that

no size continuum existed between the two kinds of wheat starch granules, as asserted by MacMasters and Waggle (1963).

## 6.2 Amylose Content

The amylose contents for soft white and hard red spring wheat starches did not deviate from the ranges of 23.4-26.9% reported by Ciacco and D'Appolonia (1977) and 23.4-27.5% reported by Medcalf and Gilles (1955).

The amylose contents found for the root starches differed from those reported in the literature by Greenwood et al. (1955); Ciacco and D'Appolonia (1977) and Onwueme (1978). This might have been due to starches from different cultivars and environments being used in the investigations. It was found in this study, however, that starches with predominantly, small sized granules such, as arrowroot and taro, had lower amylose content than those with large sized granules, such as yam. Cassava and sweet potato starches, with intermediate granule size distributions, had intermediate amylose contents.

Soft white spring wheat starch, with a higher proportion of large starch granules than hard red spring wheat starch was, however, found to have a lower amylose content. This implied that granule size was not the only factor controlling amylose content of starch granules. Genetic and environmental backgrounds probably exert significant influences.

### 6.3 Mineral Contents

Removal of lipid-phosphate complexes from wheat starch granules as recommended by Morrison et al. (1975) reduced their phosphate content from about 0.05% to 0.01% and 0.01% for cvs. Neepawa and Ender starches respectively. The starches had higher phosphate contents. This treatment increased their swelling power by repulsion of the phosphate groups carried by individual amylopectin molecules within the starch granules.

The above observation appeared to be supported by very low contents or no  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$  in the starches. High levels of these cations would react with phosphate on amylopectin and hence reduce starch granule swelling power by removing the repulsive charges partly responsible for excessive swelling power (Haydar et al. 1980).

Swelling power was however not proportional to phosphate content. Cassava starch, for example, with low phosphorus content had the higher swelling power at all temperatures than taro starch with the highest phosphate content. As expected, wheat starches with less phosphate content, swell less than the root starches as expected.

The above observations tended to suggest that other internal associations, such as the number of micelles and their strength within starch granules, were more important in controlling starch hydration and swelling power than the repulsion within the granule due to the ionized phosphate groups. The influence of  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$

not clear.

#### 4.4 Solubility and Swelling Power

It was observed that the solubility of the starches depended on the starch granule swelling power. Granule size and amylose content did not appear to have significant influence on the solubility.

Arrowroot and taro starches, with the smallest granules and close in amylose content, had widely different solubilities at their gelatinization midpoints. Starch from CWSW, with a higher amylose content, was found to be less soluble than starch from SWSW, which is lower in amylose content and contains a higher proportion of large sized granules. Starch from CWRW, with more amylose, and from taro, with less amylose content, were both equally poor in solubility at their gelatinization midpoints.

Since solubility was found to be directly correlated with swelling power, the observations made above did not fully support Leach et al. (1959); Leach (1965); Madcalf (1969) and Kulp (1973) in stating that starches high in amylose content were more resistant to swelling. It appeared that starch solubility and swelling power were more controlled by the degree of starch granule internal association and structural changes in the starch granule surface to permit free amylose to diffuse more easily from the granules.



Solubility curves showed rapid increase in solubility of taro and wheat starches after 70°C, indicating rapid breakdown in granule structure resulting in extensive leakage of amylose from the granules. The process was faster in taro, and became very extensive in wheat starches above 80°C. The change was gradual in sweet potato and yam starches, faster in arrowroot and cassava in cassava starch. These changes were closely correlated with swelling power.

Based on these observations, it seemed that swelling of hydrated starch granules during heating depended on the surfaces of starch granules, the process being characteristic of a particular starch. Amylose leached from the starch granules followed this process very closely according to the type of starch.

Rate and extent of disintegration in the starch granules forces within starch granules were different for each starch as judged by their solubility and swelling power characteristics during pasting.

Low initial solubility and swelling power for taro and wheat starches below 70 and 80°C, respectively, suggested a lag period during which energy input was necessary before accelerated changes in the granule structure. Solubility and swelling power increased rapidly after this lag period. Arrowroot, cassava, sweet potato and yam starches had a shorter lag in swelling and solubility. These results gave an indication of differences in the nature of the bonding of the starches.

### 6.5 Viscosity

It was found that the viscosity of gelatinized starch in water depended on the degree of granule swelling and the strength of the network formed by dissolved amylose interconnecting the granules. This observation was in agreement with similar observations reported by Miller *et al.* (1973) and Hoover and Hadziyev (1981).

The viscosity was found to be a function of temperature with a negative correlation. Numerous hydrogen bonds between the dissolved amylose chains rendered viscosity high at lower temperatures. Increase in temperature weakened the hydrogen bonding within the amylose network, as was shown by the drop in viscosity in spite of granule swelling.

The claim by D'Appolonia *et al.* (1971) that some swollen starch granules are disintegrated by the stirrer, hence partly accounting for decreasing viscosity with increasing temperature, may be true at higher temperatures.

Cassava starch, with the highest swelling power and solubility at all temperatures, exhibited the highest viscosities, as was also observed by Ciacco and D'Appolonia (1977). Arrowroot starch, next to cassava starch in swelling power and solubility, had surprisingly lower viscosity than taro starch, both of which had small starch granules ( $<4\mu\text{m}$ ). This might be explained by differences in molecular sizes of the dissolved amylose and its tendency to form a firm or weak network through hydrogen bonding. Similar observations were made for yam and sweet potato starches.

The wheat starches, due to their lower swelling power, had lower viscosities in comparison to the root starches. Lower viscosity in wheat starch slurries implied weaker hydrogen bonding than in the slurries of root starches.

## 6.6 DSC-Gelatinization Characteristics

Different starches were found to have specific onset, peak, and the end of gelatinization temperatures for the  $\Delta H$  endotherm for water volume fractions of  $v_1 = 0.3-0.6$ .

Root starches had higher onset, peak, and end of gelatinization temperatures than the wheat starches. Taro starch had exceptionally higher onset ( $74.5^\circ\text{C}$ ), peak ( $83.5^\circ\text{C}$ ) and end ( $83.7^\circ\text{C}$ ) gelatinization temperatures, which implied very firm associative forces within the taro starch granules. This was corroborated by the highest endotherm of fusion of  $6.38 \text{ cal/g starch (DWB)}$  observed for taro and  $2.59-5.09 \text{ cal/g starch (DWB)}$  for the other root starches and only  $0.20$  and  $0.66 \text{ cal/g}$  for SUSW and CWSW, indicating weaker bonds or association existing within wheat starch granules.

All  $\Delta H$  endotherms became shouldered by  $M_1$  endotherms representing the melting of crystalline order within starch granules. The  $M_1$  endotherm became greater at the expense of the  $\Delta H$  endotherm as  $v_1$  decreased, and hence confirmed the findings by Donovan (1979) that gelatinization was a function of the water present in the starch.



Wheat starches, but not root starches, had an additional  $M_n$  endotherm near  $100^{\circ}\text{C}$ , revealing the presence of lipid-starch clathrates. Low enthalpy of fusion for the clathrates suggested the existence of only few clathrates within the wheat starch granules.

### 6.7 Gel Aging

Results indicated that root starch gels with 60% moisture retrograded faster than similar gels from wheat starches at  $24^{\circ}\text{C}$ . The existence of two sets of DSC-endotherms, the first at  $50-60^{\circ}\text{C}$  and the second at  $90-100^{\circ}\text{C}$  in arrowroot,  $80-85^{\circ}\text{C}$  in cassava and  $78-82^{\circ}\text{C}$  in sweet potato starches, revealed that two types of retrogradation were taking place in the gels.

The retrogradation with endotherm at lower temperature was weaker in nature by virtue of the lower temperature required to break it. The endotherm at higher temperature formed only after six days of gel storage. The second set of endotherms was absent in taro, yam and wheat starch gels.

These endotherms probably represented the small and large amylose molecular realignments, the smaller ones giving rise to endotherms at higher temperatures due to the tendency of smaller molecules to compact more firmly during aging. This might however also involve the realignment of long molecules, which is a time-dependent process.



X-ray diffraction crystallography confirmed DSC findings that crystallinity developed in aged starch gels. Diffracted line intensities and peak sharpness, especially at angles of diffraction ranges of  $17.075-17.781$  and  $19.361-20.071(2\theta)$  implied that cassava starch gel retrograded more firmly than wheat starch gels during prolonged aging.

## 6.8 Complexing with Monoglycerides

### 6.8.1 Infrared analysis of Monoglycerides

In spite of similarity in infrared spectra of  $\alpha$ - and  $\beta$ -crystallinity forms of  $C_{10}$  and  $C_{12}$  monoglycerides, it was possible to differentiate between the two crystallinity forms by C-O bond stretching band of primary -OH groups at  $1062\text{ cm}^{-1}$  in the  $\beta$ -crystallinity powder form. The absence of this band in the  $\alpha$ -crystallinity or the gel form might be due to formation of hydrogen bonds between the -OH groups and water.

Further identification between  $\alpha$ - and  $\beta$ -crystallinity was based on the C=O bond stretching at frequency range  $1730-1739\text{ cm}^{-1}$ , which was consistent in the  $\beta$ -crystallinity form, but accompanied by a strong band at  $1715\text{ cm}^{-1}$  in the  $\alpha$ -crystallinity form, probably due to the occurrence of hydrogen bonding in the  $\alpha$ , but not in the  $\beta$ -crystallinity form.

Hoover and Hadziyev (1981) noticed that aging the  $\alpha$ -gels promoted the reappearance of the  $1062\text{ cm}^{-1}$  band. This observation implied that the  $\beta$ -crystallinity form was the more stable.

### 6.9 X-Ray Diffraction Analysis of Monoglycerides

X-ray diffraction analysis showed that the  $\alpha$  and  $\beta$ -crystallinity forms of the  $C_{12}$  and  $C_{14}$  monoglycerides were characteristically distinguishable. The distinguishable peaks for strong, medium and weak intensity diffracted lines were similar to earlier work reported by Hoover and Hadziyev (1981).

Of particular interest were the bold strong diffracted lines at  $4.1682\text{ \AA}$  ( $21.316^\circ 2\theta$ ) and at  $4.1604\text{ \AA}$  ( $21.357^\circ 2\theta$ ) for  $\alpha$ - $C_{12}$  and  $\alpha$ - $C_{14}$ . While  $\alpha$ - $C_{12}$  had in addition only one medium peak at  $14.6126\text{ \AA}$  ( $6.048^\circ 2\theta$ ),  $\alpha$ - $C_{14}$  had two additional ones at  $14.3929\text{ \AA}$  ( $6.141^\circ 2\theta$ ) for the medium one, shouldered to the left by a weak one at  $16.6375\text{ \AA}$  ( $5.112^\circ 2\theta$ ).

The strong line at  $4.5378\text{ \AA}$  ( $19.562^\circ 2\theta$ ) in the X-ray diffractogram of  $\alpha$ - $C_{12}$  indicated that the gel was already in the process of reverting to the  $\beta$ - $C_{12}$  form, which had bold strong lines at  $4.5023\text{ \AA}$  ( $19.718^\circ 2\theta$ ) and at  $3.9262\text{ \AA}$  ( $22.643^\circ 2\theta$ ) corresponding to others in  $\beta$ - $C_{12}$ , occurring at  $4.5633\text{ \AA}$  ( $19.452^\circ 2\theta$ ) and at  $3.9341\text{ \AA}$  ( $22.601^\circ 2\theta$ ).

While the  $\beta$ -crystallinity form had additional strong lines at 4.0578, 4.3927, 11.9478, 16.0327 Å, two medium ones at 7.9313 and 3.1341 Å, and a weak one at 8.7301 Å.  $\beta$ -C<sub>12</sub> had only two additional medium, but less intense diffraction lines at 3.1105 Å and 16.1697 Å, and two weak ones, also less intense at 12.1666 and 8.2555 Å.

The  $\beta$ -crystallinity forms of C<sub>12</sub> and C<sub>14</sub> monoglycerides had more diffracted lines due to their higher level of crystallinity than their  $\alpha$ -crystallinity forms. Both  $\alpha$  and  $\beta$ -C<sub>12</sub> monoglycerides however proved to be more crystalline than their corresponding counterparts of C<sub>14</sub> monoglycerides. This implied that, shorter carbon chain fatty acids in C<sub>12</sub> monoglycerides were more capable of orientating themselves more compactly in crystalline monoglycerides than C<sub>14</sub> monoglycerides.

#### 6.10 Complexing with Monoglycerides

The effect of the physical state of starch on the interaction with monoglycerides was proved by low, high and consistently higher complexing indices obtained by interacting monoglycerides with ungelatinized, additionally gelatinized or solubilized lintnerized starches.

Little difference was observed between complexing indices of ungelatinized starch with  $\alpha$  or  $\beta$  crystalline forms of monoglycerides, implying that, although monoglycerides have been proposed to enter starch helices to form a complex with amylose inside the starch granules, the

1965), their ability to do so is limited at lower temperatures, such as 45°C.

In all other cases, higher complexing indices were obtained for all starches using C<sub>12</sub> instead of C<sub>10</sub>. This tended to support earlier reports of the superiority of C<sub>12</sub> over C<sub>10</sub> in clathrate formation (Krog, 1981; and Hoover and Hadziyev, 1982). Also in all cases  $\alpha$ - and not the  $\beta$ -crystallinity form was found to be more reactive with the starches. This again supported earlier claims of the superiority of the  $\alpha$ - over the  $\beta$ -crystallinity forms in complexing with starch (Krog, 1970; Lagendijk and Penning, 1970; Lonkhuisen and Blankestijn, 1974, 1976; and Hoover and Hadziyev, 1982).

The finding by Osman et al. (1961), and Lonkhuisen and Blankestijn (1974, 1976) that different starches complexed differently with monoglycerides, the process being modified by the physical state of the starch, were also supported.

For all the starches, 0.5% monoglycerides was found to be the optimal level for starch interaction with monoglycerides.

#### 6.11 Affinity for Vital Gluten

The affinity of the starches for vital gluten was observed to rise from the dough to the highest level (2-3 times dough values) in the early baking stages before declining to lower levels in the fully baked stages.



The rise in affinity of starch for vital gluten from the dough to early baking stages appeared to have been stimulated by the swelling of the starch granules in early and mid gelatinization stages to ensure optimum affinity development between starch and the vital gluten before the gluten became fully denatured in the fully baked stage. The above observation appeared to be supported by similar observations made by Dahle (1971), and the absence of affinity between ungelatinized starch and native gluten as reported by Dehnett and Sterling (1979).

Reduced affinity of starch for vital gluten in the fully baked stage appeared to be advantageous in producing looser crumbs, a factor related to good bread keeping qualities as latter proved by compressibility, resistance to penetration and chewability tests.

Low swelling power and starch granule size distribution of about 42.6% granules  $< 5 \mu\text{m}$  and about 57.7% granules in the range 5-25  $\mu\text{m}$ , as in wheat starches, promoted affinity of starch in baked systems, resulting in firmer crumbs, as opposed to the cases where all starch granules are small (as in arrowroot, and tapioca) or large ( $10 > \mu\text{m}$ , as in yam), all of which had greater swelling powers than the wheat starches.

Starch solubility enhanced starch gluten affinity, but total amylose content did not provide a consistent influence on the affinity.

### 6.12 Flour Water Absorption

Wheat flour (CWRSW, cv. Neepawa), with a high proportion of small sized starch granules and good quality gluten, had a higher percent water absorption than flour with a higher proportion of large sized starch granules and inferior gluten (SWSW, cv. Fielder). A similar observation was made for the respective flours made of only their starches and the same quality vital gluten. This observation implied that the granule size distribution of undamaged starch had a definite influence on the level of water absorption by a flour.

More cases in support of the above observation were provided by flours of arrowroot starch/vital gluten and yam starch/vital gluten, in which the former flour had a higher water absorption than the latter, the difference between them being that arrowroot starch grains were all small ( $<4\text{ }\mu\text{m}$ ) compared to the large yam starch granules ( $>10\text{ }\mu\text{m}$ ).

It also appeared that starch permeability to water played an important role since yam and taro composite flours had the same absorption yet the starches differed greatly in particle size. Furthermore, in support of this view, arrowroot and taro starches, both with small granules of  $<4\text{ }\mu\text{m}$ , had composite flours of different percent water absorption, arrowroot flour having the higher absorption. That arrowroot starch granules were less compact than taro starch granules although both consisted of tiny granules, all less than  $4\text{ }\mu\text{m}$ , was reflected in starch granule swelling

power, solubility and enthalpy of fusion studies.

### 6.13 Dough Rheological Properties

Farinographic rheological data revealed that these starch/vital gluten composite doughs had longer development times and stabilities than their pure flour doughs. No significant differences were noticed between the consistencies of CWSW pure flour and its starch/vital gluten dough consistencies during prolonged mixing. Large differences were however observed between the consistencies of similar doughs from pure flour and starch/vital gluten composite flours from SWSW.

The above observations indicated that the initial dough strength was not only due to quantity and quality of the gluten, but that starch/gluten surface interactions also played a significant role. Large mixing tolerance index (MTI) 20 min drop for SWSW starch/vital gluten seemed to support the opinion that starch from poor baking quality flours had an inherent inability to combine well with gluten. This was in support of observations by Harris and Smith (1962) that different wheat starches behaved differently in dough formulations with the same sample of gluten. Starch particle size distribution also appeared to be an important factor influencing dough behavior.

Similar dough farinographic data of pure and composite starch/vital gluten proved that these doughs could be made to reach similar consistencies at the same mixing time.



dough in shorter times, with the exception of the composite dough with arrowroot starch, which took longer to develop. Furthermore, the composite doughs with root starches had better stabilities than the CWRSW flour dough (except taro and sweet potato starch composite doughs). This behavior might be explained by the higher affinities for gluten possessed by the root starches than the wheat starches. The deviant behavior of arrowroot starch composite dough in taking longer to develop might be explained by some other reason such as its large surface area requiring more time at constant mixing rate to be fully coated with gluten. Shorter dough stabilities in sweet potato and taro composite doughs might also be explained by differences in granule sizes and morphology in relation to gluten film coating stability against rupture during mixing.

In spite of the root starch/vital gluten composite doughs being more stable, they deteriorated faster in their consistency than pure or starch/vital gluten doughs from CWRSW. This suggested that, although root starches had more affinity for gluten, they formed weaker doughs when subjected to prolonged mixing.

Yam starch/vital gluten composite dough withstood prolonged mixing better than any other root starch composite doughs. This suggested that large and oval, apparently smooth granules with small surface area to volume ratio, as in yam, promoted coherence within the dough.



Findings which can be related to the above have been reported by Suren and Eiden (1975), Akinola (1975), Chinn and D'Amico (1975) and et al. (1981).

#### 6.14 Dough and Crumb Hydration Capacities

In general, higher amylose content appears to have a negative effect on dough and crumb capacities, thus confirming findings of Sterling (1978). The presence of large amounts of the flour was, however, observed to have a positive effect on dough hydration capacity (Sterling and Fielder).

Galatinization initiated crumb hydration several times over their counterparts in the arrowroot and taro, with negative effects on positive crumb hydration capacities of the crumb. Starch granule swelling power was found to have directly positive influence on capacities, which again confirmed the findings of soluble amylose in the crumb hydration capacity reported by Dey and Dey (1975).

#### 6.15 Fractional Volume Increase

When flour was added to the crumb, the crumb hydrated more than the flour alone. The crumb hydrated more than the flour alone over a period of 24 hours.

The differences observed in fractional volume increases of the various breads could not be related to starch granule size, amylose content, starch swelling power or solubility. Relation with starch affinity for gluten was not fully conclusive, but it appeared that the ability of a dough to retain air or proofing gas might have been more influential.

## 6.16 Dough and Crumb Microstructure

### 6.16.1 Dough Microstructure

Scanning electron (SE) micrographs of fermented and proofed dough surfaces prior to placement into the oven for baking indicated that starch granules, irrespective of source, were capable of being coated with a thin film of gluten. Bolder granule outlines retained by the root starches in their doughs appeared to suggest that root starches were only thinly coated with a film of gluten in comparison to the wheat starch granules in pure or starch composite flour doughs. This was supported further by the presence of thicker gluten coatings observed around wheat starch than root starch granules in fractured dough surfaces.

Doughs from SWSW pure or starch/vital gluten flours exhibited lower fractional volume increases than corresponding doughs from CWRSW when baked to bread. This might have been due to the presence of more pores observed in the surfaces of SWSW than in CWRSW doughs, as revealed by

SE-microscopy. The same technique also revealed that the SWSW starch was less cohesively associated with the gluten matrix than the CWRSW starch. This might also have facilitated more loss of gases from the the SWSW doughs, resulting in reduced fractional volume increases for SWSW than CWRSW based dough-bread systems.

Arrowroot and taro fractured dough surface SE-micrographs showed the existence of gluten spindle bridges between starch granules. The granules were barely covered with a thin film of gluten. Aggregates of starch granules were observed to be better coated with gluten than others. This might be explained by the large surface area possessed by the small starch granules (all  $<4\mu\text{m}$ ) in arrowroot and taro starch doughs. These could not be equally coated with gluten, thus sometimes resulting in gluten film shredding and formation of spindle bridges between some starch granules as observed.

Cassava and sweet potato starches were better coated with gluten, as revealed by SEM of fractured surfaces of their composite doughs. Although yam starch granules were evenly coated externally, the gluten inside the dough appeared to have gathered mainly in the spaces between the starch granules. This observation seemed to be a response of overstrained gluten films to predominantly large granules of yam starch, as further supported by intergranular fissures observed exteriorly in the in the gluten film coating.

As a whole, starch granules were best coated with gluten in pure wheat flour doughs. Granule coating with gluten deteriorated in wheat starch/vital gluten doughs. Coating of the root starches with gluten was quite variable, being more strained by dominance of very small or very large starch granules.

#### 6.16.2 Crumb Microstructure

Scanning electron microscopy of the bread crumb surfaces showed that all root starches had gelatinized well in the denatured gluten matrix, forming good crumb structures comparable to wheat bread crumb.

Whereas a weak wheat flour or composite of its starch/vital gluten flour produced weakly structured doughs reflected in less desirable crumbs compared to doughs and crumbs from a strong wheat or its starch/vital gluten composite flour, as shown respectively by wheat, cvs. Fielder and Neepawa, the observation with respect to the root starch composites with vital gluten was different.

Weaknesses seen in the composite doughs of the root starches with vital gluten seemed to disappear from their bread crumbs, which were of good quality comparable to good wheat bread crumbs. In fact root starches produced better bread crumbs than bread crumbs based on wheat, cv. Fielder flour and starch/vital gluten composite. This was attributed to the higher swelling power of the root starches, which helped to strengthen the gluten matrix structures

surrounding the air-cells in the crumb. The amylose provided by the higher solubility of the root starches strengthened the crumbs by a cementing action on retrogradation.

The tiny pores observed in SE-micrographs of doughs (Evans *et al.* 1981) and in crumbs (Fleming and Sosulski, 1978) were also observed in this study. The pores were more prevalent in wheat flour or starch based doughs and crumbs than in those of root starch. This confirmed the observation that the root starches were more thinly coated with gluten in the dough, hence allowing more diffusion of the gases from the doughs, while the thicker gluten coatings observed on starch granules in wheat flour or starch/vital gluten doughs encouraged higher pressure development in the air cells in the doughs. The pores must have formed as a means of equalizing the pressure within the different air cells in the dough and latter within the developing crumb during baking. This appeared to be supported by the presence of more pores in the crumb than in the dough as a response to increased pressure within the bread during baking as the temperature rose.

Higher solubility of amylose in the root starches over longer temperature ranges than observed for the wheat starches and its cementing influence on the crumb on retrogradation might explain why composite crumbs with root starches had fewer pores than crumbs from the wheat bread. Some of the root starch composite crumbs, as in arrowroot starch/vital bread crumb, had even fewer, but larger pores,

indicating the presence of greater tenacity in the amylose-gluten matrix complex between the air cells, exploding only occasionally and leaving behind only a few large pores. This observation might also explain why arrowroot/vital gluten composite bread had the highest fractional volume increase amongst all the breads based on the root starches.

#### 6.17 Transmission Electron Microscopy of Dough and Crumb

Representative transmission electron micrographs were made from arrowroot starch/vital gluten dough and crumb (Plate 5.16).

Individual starch granules, as well as aggregates, surrounded with films of gluten, were observed both in the dough and in the crumb. Observations made in the TE-micrographs corroborated those made in the SE-micrographs of the fractured dough surface, already discussed above.

In addition, the TE-micrographs showed that, although the starch granules had been previously graded as all being less than  $4\mu\text{m}$ , they were of different sizes even in their gelatinized state. Sections through the air cell walls showed that they were lined with starch granules embedded in the gluten matrix, as previously observed by Burhams and Clapp (1942), Baker and Mize (1946), Sandstedt *et al.* (1954), and Hanssen (1957).

### 6.18 Bread Keeping Qualities

Bread keeping qualities in the presence and absence of monoglyceride, investigated through crumb compressibility, resistance to penetration and X-ray diffraction analysis of crystallinity development in starches isolated from aged crumbs, provided complementary results.

Incorporation of monoglyceride in the bread formulation increased crumb softness, resulting in increased crumb compressibility, but reduced resistance to penetration. No monoglyceride was included in the bread formulation for X-ray analysis of aged starch isolated from the bread crumbs. This was necessary in order to determine the relative crystallinity development in the different starches, followed by establishment of the role played by starch granule internal crystallinity in bread firming.

Solubility and behavior of the solubilized amylose appeared to be the main factors influencing the compressibility of the crumb and its resistance to penetration.

In the absence of monoglyceride, yam starch/vital gluten crumb required the highest compression force and had high penetration resistance, just below that for cassava starch/vital gluten bread. Cassava starch had the highest solubility. Retrogradation of the solubilized amylose rendered the crumb firm. Compressibility and crumb resistance to penetration exhibited by its crumb were in agreement with the views of Schoch (1965). Yam had

comparatively much lower solubility than cassava starch. Yam starch behavior revealed by its crumb compressibility and resistance to penetration tests implied that yam amylose retrograded more firmly, establishing a rigid network close in strength to a similar network made from a larger quantity of solubilized and retrograded amylose from cassava. This view was supported by X-ray diffraction patterns, which showed that yam starch retrograded relatively faster than cassava starch.

Yam starch bread became the softest in presence of monoglyceride, as shown by crumb compressibility (and next to the arrowroot starch bread according to the crumb penetration resistance test), suggesting that yam starch was more readily complexed by monoglyceride. Application of  $C_{18}$  at 0.5% tended to support this observation. Larger granule size in yam starch might also have enabled more monoglyceride to penetrate the granules and hence immobilize more amylose to a greater extent.

Arrowroot starch granules, although as small as taro starch granules were more porous and soluble. Their ability to give soft crumb in the presence of monoglyceride (but firmer than taro starch bread crumbs in the absence of monoglycerides) may be due to the higher solubility of arrowroot starch, providing more amylose, which on retrogradation resulted in firmer crumb for arrowroot than for taro bread crumbs. A similar argument for cassava and sweet potato starch bread crumbs appeared to be valid.



X-ray diffraction patterns showed that relative rate of crystallinity development in the starch granules during crumb aging was highest in yam, followed by sweet potato starches. It was slightly slower in arrowroot than in sweet potato starches, dropping for taro, and being least in cassava starch. Compressibility and crumb penetration tests proved however that crystallinity development in the starch granules within the crumbs was less significant in contributing to crumb firmness during aging than retrogradation of solubilised amylose in the intergranular gluten matrix in the crumb.

Crumb microstructure and affinity of starch for gluten in the crumb revealed that CWRWSW flour and starch/vital gluten crumbs should be firmer than similar crumbs based on SWSW flour and its starch/vital gluten composite. These observations were contradicted by crumb compressibility and resistance to penetration tests. The contradiction could be explained in a similar manner as presented above for the root starch/vital gluten composite crumbs, since SWSW starch had higher solubility than CWRWSW starch.

Medcalf (1968) and Kulp (1973) reported that starches high in amylose content were more resistant to gelatinization, hence had smaller swelling power. This was observed to be true for CWRWSW starch, which had a higher amylose content and a lower swelling power than SWSW starch, with a lower amylose content. This might explain further the fact that crumbs from CWRWSW flour and starch were more

tender than bread crumbs from SWSW flour and starch.

X-ray diffraction analysis again showed that gelatinized CWRWSW starch developed crystallinity faster during aging than gelatinized SWSW starch. Such crystallinity development in wheat starch granules was also found to contribute much less towards crumb firming than retrograded amylose, as revealed by extent of starch solubility and its influence on crumb compressibility and resistance to penetration.

## 6.19 Bread Quality Evaluation

### 6.19.1 Loaf Volume

Breads from CWRWSW pure, and starch/vital gluten composite flours had larger volumes than similar breads from SWSW pure, and starch/vital gluten composite flours. This was attributed to superior gluten quality and quantity in CWRWSW flour, and also due to low solubility associated with its starch, resulting in formation of less rigid dough and crumb structures more prone to expansion by gases during fermentation and oven rise stages of bread making than in those cases involving SWSW flour and starch based breads.

It was similarly observed for root starches that starch solubility and extent of rigidity associated with soluble amylose on retrogradation had influence on loaf volumes. Starch size distribution did not appear to have any influence on loaf volume. Swelling power did not give a

It seemed that a low score for a characteristic did not indicate non-acceptability, but that the panelists viewed that characteristic in the composite bread to be different from the standard wheat bread. Composite bread internal properties were however viewed as being close to wheat bread. Since these are important properties of bread, it appeared that root starches, especially from cassava, have a good potential of being used to produce composite breads similar in quality to wheat bread. This finding would probably be of greater significance where wheat availability is limited, and food products from root crops are common in the diet. Color of the crust and its character would certainly need further improvement to make the composite breads more attractive.

Further development might mean establishing the proper formulations and baking conditions for composite breads, which appear not to be identical with those established for wheat bread. Proper formulations might require supplementation of the composite starch flours with a fraction of damaged starch, a higher level of sugar or addition of  $\alpha$ -amylase. Fermentation period, baking temperature and time may also require proper adjustment.

Table 5.52 is provided as a final summary of all the results.

Table 5.52 Comparison of Starch Characteristics

| Characteristic                                    | CMS<br>Neepawa       | SWS<br>Fielder       | Cassava                                  | Yam                      | Sweet Potato                     | Taro                     | Arrowroot                   |
|---|----------------------|----------------------|--|--------------------------|----------------------------------|--------------------------|-----------------------------|
| 1. <i>Triticum vulgare</i>                        | Triticum vulgare     | Triticum vulgare     | Manihot utilissima                       | Dioscorea cayenensis     | Ipomoea batatas                  | Colocasia esculenta      | Maranta arundinacea         |
| 2. <i>Cereal</i>                                  | Cereal               | Cereal               | Tuberous root (root cluster)             | Deep tuber               | Tuberous root                    | Corn (under-ground stem) | Philome (under-ground stem) |
| 3. Granule size, $\mu$ m                          | 76% 10               | 77% 10               | 80%, 5-15                                | 95%, 16-35               | 83% 10                           | all 4                    | all 4                       |
| 4. Granule Morphology                             | Spherical Lenticular | Spherical Lenticular | Round Smooth Some concave Some truncated | Round Ellipsoidal Smooth | Polygonal Rounded Corners Smooth | Polygonal                | Polygonal                   |
| 5. % Amylose                                      | 27.3                 | 23.0                 | 19-21                                    | 25.0                     | 20.0                             | 15.0                     | 17.0                        |
| 6. % Phosphorus                                   | 0.015                | 0.013                | 0.011                                    | 0.006                    | 0.013                            | 0.026                    | 0.023                       |
| 7. % (Ca <sup>++</sup> + Mg <sup>++</sup> )       | 0.005                | 0.005                | 0.010                                    | 0.003                    | 0.014                            | 0.006                    | 0.015                       |
| 8. % (Na <sup>+</sup> + K <sup>+</sup> )          | 0.041                | 0.047                | 0.062                                    | 0.035                    | 0.060                            | 0.118                    | 0.126                       |
| 9. Water binding capacity (g/g starch - DMS) *#   | 0.45                 | 0.34                 | 0.50                                     | 0.70                     | 0.55                             | 0.65                     | 0.50                        |
| 10. Swelling power (g paste/g dry starch) at 95°C | 19.0                 | 24.5                 | 73.4 (Kenyan cv.)                        | 34.0                     | 33.5                             | 33.0                     | 40.02                       |
| 11. % solubility at 95°C                          | 29.6                 | 28.8                 | 30.4                                     | 23.3                     | 18.7                             | 22.4                     | 25.0                        |

\*# See appendix 37.

|   |              |               |       |       |            |       |      |
|---|--------------|---------------|-------|-------|------------|-------|------|
| 12. Peak gelatinization temperature °C                                    | 55.2         | 50.1          | 58.8  | 65.7  | 58-66.5%   | 78.6  | 67.5 |
| 13. Viscosity at 70°C. 1% w/v slurry. 10 min of Hake rotoviscometer (cP). | 0.67         | 0.83          | 2.8   | 3.0   | 1.75-2.65* | 3.0   | 2.4  |
| 14. Enthalpy of fusion cal/g dry starch                                   | 0.66         | 0.20          | 4.90  | 4.56  | 3.10       | 6.38  | 4.93 |
| 15. Affinity for gluten (%)   |              |               |       |       |            |       |      |
| Raw   | 31.8         | 24.8          | 48.5  | 40.80 | 40.0       | 41.7  | 50.3 |
| Early baking  | 88.6         | 89.6          | 82.1  | 91.50 | 89.10      | 90.0  | 83.6 |
| Baked   | 45.1         | 41.3          | 49.0  | 28.50 | 44.0       | 41.0  | 41.0 |
| 16. % Moisture  | 9.82         | 11.03         | 14.23 | 10.35 | 8.96       | 13.51 | 9.23 |
|   | 8.91 (flour) | 11.67 (flour) |       |       |            |       |      |
| 17. % Absorption  | 60.4         | 54.0          | 64.7  | 68.5  | 68.8       | 68.4  | 80.5 |
|   | 68.4 (flour) | 56.8 (flour)  |       |       |            |       |      |
| 18. Arrival time (min)  | 1.75         | 1.0           | 2.0   | 2.3   | 2.5        | 3.0   | 3.25 |
|   | 3.8 (flour)  | 0.8           |       |       |            |       |      |
| 19. Peak time (min)   | 6.5          | 3.5           | 4.5   | 4.0   | 3.5        | 3.8   | 7.0  |
|   | 6.0 (flour)  | 1.20 (flour)  |       |       |            |       |      |
| 20. Dough stability (min)   | 11.8         | 7.5           | 8.3   | 10.2  | 4.2        | 1.8   | 7.8  |
|   | 6.7 (flour)  | 0.9 (flour)   |       |       |            |       |      |
| 21. MTI (BU)  | 25.0         | 40.0          | 60.0  | 48.0  | 145.0      | 120.0 | 60.0 |
|   | 26.0 (flour) | 120.0 (flour) |       |       |            |       |      |

|                                  |                |                |        |        |        |         |
|----------------------------------|----------------|----------------|--------|--------|--------|---------|
| 22. 20 min drop                  | 45.0           | 130.0          | 80.0   | 190.0  | 190.0  | 120.0   |
|                                  | 45.0 (flour)   | 150.0 (flour)  |        |        |        |         |
| 23. Dough HC                     | 0.125          | 0.043          | 0.253  | 0.083  | - 0.10 | - 0.083 |
| g H <sub>2</sub> O/g dry solids  | 0.061 (flour)  | 0.092 (flour)  |        |        |        |         |
| 24. Crumb HC                     | 5.37           | 6.6            | 2.65   | 4.12   | 3.14   | 3.29    |
| g H <sub>2</sub> O/g dry solids  | 3.64 (flour)   | 4.48 (flour)   |        |        |        |         |
| 25. Gelatinization               | 5.25           | 6.56           | 2.40   | 4.04   | 3.24   | 3.32    |
| hydration                        | 3.58 (flour)   | 4.39 (flour)   |        |        |        |         |
| 26. Loaf vol. (cm <sup>3</sup> ) | 1302.0         | 1112.0         | 1022.0 | 1192.0 | 1172.0 | 922.0   |
|                                  | 1432.0 (flour) | 1051.0 (flour) |        |        |        |         |
| 27. Loaf mass (g)                | 443.0          | 426.0          | 435.0  | 426.0  | 426.0  | 419.0   |
|                                  | 435.0 (flour)  | 421.0 (flour)  |        |        |        |         |
| 28. Loaf sp. vol.                | 2.94           | 2.61           | 2.26   | 2.80   | 2.75   | 2.20    |
| cm <sup>3</sup> /g               | 3.52 (flour)   | 2.50 (flour)   |        |        |        |         |
| 29. Fractional vol.              | 1.49           | 0.7            | 0.67   | 0.64   | 0.49   | 0.96    |
| increase cm <sup>3</sup>         | 1.68 (flour)   | 1.65 (flour)   |        |        |        |         |
| (based on 50 g dough)            |                |                |        |        |        |         |
| 30. Crumb compressibility        |                |                |        |        |        |         |
| (kg force/cm <sup>2</sup> )      |                |                |        |        |        |         |
| fresh crumbs                     | 0.04           | 0.04           | 0.08   | 0.08   | 0.09   | 0.02    |
| + Mg                             | 0.03 (flour)   | 0.06 (flour)   |        |        |        |         |
| - Mg                             | 0.05           | 0.07           | 0.10   | 0.10   | 0.11   | 0.04    |
|                                  | 0.08 (flour)   | 0.811 (flour)  |        |        |        |         |
| 6 day old crumbs                 | 0.95           | 0.97           | 0.66   | 0.90   | .10    | 0.73    |
| + Mg                             | 0.72 (flour)   | 1.21 (flour)   |        |        |        |         |
| - Mg                             | 1.02           | 1.32           | 1.35   | 1.30   | 1.23   | 1.28    |
|                                  | 0.75 (flour)   | 1.27 (flour)   |        |        |        |         |







conclusive direct effect. Some available evidence indicated that decreasing amylose content was accompanied by an increase in loaf volume.

#### 6.19.2 Sensory Panel Tasting and Statistical Evaluation

Composite breads of root starches with vital gluten appeared to be similar to those studied and reported by Kim and Ruiters (1969), Ciacco and D'Appolonia (1977), and Olatunji and Akinrele (1978).

Those considered in this study received lower sensory tasting scores than the standard wheat bread, especially in their external properties, of which the color of the crust scored the least. Their internal characteristics received more than average acceptability ratings. Cassava starch bread scored consistently higher than any other root starch breads, in both its external and internal loaf properties.

Analysis of variance showed that loaves of different breads actually differed in their characteristics. The organoleptic tests were, however, subject to the panelists' subconscious bias, as the majority of them were foreign to edible products from root crops. It is possible that results of the same sensory tasting might be significantly different if performed by people to whom the composite breads might mean improvement or attractive alternatives in their usual diet.

## 7. CONCLUSIONS

All starch granules were found to be characteristically distinguishable in their size and morphology, amylose and mineral contents, swelling power and solubility, viscosity and gelatinization properties, as well as in their ability to retrograde in aged gels and bread crumbs.

Retrogradation of leached out amylose was more responsible than crystallinity development in starch grains for crumb firming during aging. The presence of monoglycerides at the optimal level of 0.5% on starch dry weight basis produced a softening effect in all cases. The effect varied significantly with the type of starch.

Monoglycerides in their  $\alpha$ - but not  $\beta$ -crystallinity form were found to be more reactive with the starches. Glycerol monopalmitate ( $C_{16}$ ) was found to clathrate more than glycerol monostearate ( $C_{18}$ ) with the starches. Solubilized starches reacted most while ungelatinized one reacted least with the monoglycerides. The reaction with gelatinized starches was intermediate under the same conditions.

Highest affinity of starch for gluten in early baking was advantageous in ensuring maximum association between the starch and the gluten before the gluten became fully denatured at higher temperatures during baking. Low affinity of starch for gluten in fully baked systems was found to encourage the development of looser crumbs, which was found to be advantageous in bread keeping qualities.

Predominantly large or small starch granule size distribution in the flour promoted water absorption by the flour. This was found to be related to the high water binding capacities possessed by starches predominated by either large or small starch granules. Very close association between the starch granules and the gluten reduced water absorption by the flour.

Vital gluten/starch doughs reached similar consistencies as Canadian western red spring wheat flour dough, but in shorter times, and with better stabilities. This was found to be in agreement with the higher starch affinity for gluten observed for root starches than wheat starches in the dough. Root starch doughs, however, disintegrated faster on continued mixing due to root starches being only thinly coated with gluten film, and inherent weaknesses existing at the root starch granules-gluten film interfaces.

Root starch/vital gluten doughs had better mixing properties than Fielder wheat flour. Dough strength was found to depend on the nature of the gluten as well as the starch-gluten film surface interactions.

Dough and crumb microstructure were found to be significantly influenced by starch granule size, swelling power, solubility, gas formation ability and the capacity of the dough to retain the gases during mixing, fermentation, proofing and oven rise. The absence of damaged starch in the root starch/vital gluten composite flours and weaker dough

structure in the presence of root starches resulted in smaller loaf volumes observed in their composite breads as compared to wheat flour or starch/vital gluten composite breads.

Dough and crumb hydration capacities were negatively influenced by increasing amylose content of the starch. Predominance of large granule size promoted dough hydration capacity. The extent of gelatinization, starch swelling power, and solubility had positive influences on crumb hydration capacity..

CWRS wheat starch with vital gluten produced the best composite bread, followed by the composite bread containing SWSW starch. This appeared to prove that starch from good baking quality wheat had an inherent ability to combine better with gluten and produce better quality bread than starch from poor baking quality wheat. Next to the wheat starch composites, cassava starch/vital gluten bread was judged organoleptically the best amongst the composite breads containing root starches. Following in preference were breads containing yam, sweet potato, taro and lastly arrowroot starches.

Bread loaf external properties were the most limiting factors in the organoleptic evaluation of the composite breads containing the root starches. Color of the crust and its character were the most limiting. Since conditions established for baking wheat flour bread were used in baking these composite breads, it appeared, especially for the

composites with root starches, that investigations to establish proper formulations and baking conditions that would improve their sensory properties would be necessary. Nevertheless, root starches, especially cassava, yam, sweet potato and taro, have a good potential of being used as dilutants of strong wheat flours, or being used along with vital gluten to produce good quality composite breads. This development would probably be of greater significance in countries where wheat availability is limited, or food products from root crops are already popular. It would be much so especially if the availability of composite breads would lead to improvement in diet at reasonably affordable cost.

This study was concerned with model systems involving only native starches instead of flours obtained from tropical root crops. It has provided fundamental data which can only be attributed to these starches, and vital gluten in baking. There would be interferences from the other chemical compounds such as proteins, gums, sugars, fibre, oxidants, pigments, and flavor substances native to the tropical tubers, and hence present in their flours, were thus avoided. Since the flours are predominantly starch (75-85), their use in baking studies with for example strong wheat flours, or with vital gluten, guided by the findings of this study would be worthwhile. Changes in dough rheological properties and bread organoleptic properties would be expected.

## 8. REFERENCES

- American Association of Cereal Chemists (1982) AACC Approved methods. Method 26-95. St. Paul Minnesota.
- Alf, D. (1975) Flour Milling Technology. In: Grains and Oilseeds, Handling, Marketing and Processing. 2nd ed. Canadian International Grains Institute, Winnipeg. Manitoba.
- Alsberg, C.L. and Griffing, E.P. (1952). Effect of fine grinding on flour. Cereal Chem. 2:325.
- Amin, S. (1955). The polysaccharides of (*Colocassia antiquorum*). J. Chem. Soc. 2441. London.
- Anderson, R.A. (1967). Manufacture of wheat starch. In: Starch: Chemistry and Technology. Vol. II. Industrial Aspects. Whistler R. L. and Paschall, E.F. (eds). Academic Press. New York. London.
- Anker, C. A. and Geddes, W. F. (1944) Gelatinization Studies upon wheat and other starches with the amylograph. Cereal Chem. 21:335
- Atkinson, F. M. and Fuehrer, F. D. (1960). Effects of overgrinding on flour characteristics. Bakers Digest 4951, 80.
- Axford, D. W. E. and Colwell, K. H., Cornford, S.J. and Elton, G. A. H. (1968). Effect of bread specific volume on the rate and extent of staling in bread. J. Sc. Food Agric. 19:95.
- Badenhuizen, N. P., and Dutton, R. W., (1956). Growth of <sup>14</sup>C-Labelled starch granules in Potato tubers as revealed by autoradiographs. Protoplasma 47:156.
- Badenhuizen, N. P. (1959). Chemistry and Biology of the starch granule. Springer Vienna (cited in Carbohydrates, by D'Appolonia, B. L., Gilles, K. A., Osman E. M., and Pomeranz, Y. In : Wheat Chemistry and Technology. Pomeranz. Ed. AACC Inc. St. Paul, Minnesota.
- Badenhuizen, N. P. (1965). Occurrence and development of starch in Plants. In: Starch : Chemistry and Technology, Vol. 1, P. 65. Whistler, R. L, and Paschall, E. F. (eds). Academic Press. New York.
- Bailey, H. L. (1930). A simple apparatus for measuring the compressibility of baked products. Cereal Chem. 7:340.
- Bailey, H. L. (1932). The use of certain constituents in

bread making with particular reference to the problem of staling. Cereal Chem. 9:65.

Baker, J. C. and Mize, M. D. (1946). Gas occlusion during dough mixing. Cereal Chem. 23:39.

Bates, F. L., French, D., and Rundle, R. E. (1943). Amylose and amylopectin content of starches determined by their iodine complex formation. J. Am. Chem. Soc. 65:142.

Bean, M. L. and Osman, E. M. (1959). Behaviour of starch during food preparation. II. Effects of different sugars on the viscosity and gel strength of starch pastes. Food Res. 24:665.

Bechtel, W. G. (1959). Staling studies of bread made from flour fractions. V. Effect of heat stable amylase and a cross-linked starch. Cereal Chem. 36:368.

Bechtel, W. G. Geddes, W. F. and Gilles, K. A. (1964). Carbohydrates, P. 277. In : Wheat: Chemistry and Technology, (ed). by Hlynka, I. Am. Ass. of Cereal Chemists. St. Paul, Minnesota.

Bechtel, W. G. and Meisner, D. F. (1952). Bakers Digest. 26(1):5. (Cited in Baking Science and Technology vol. 2. by Pyler, E. J. Siebel Publishing Comp. Chicago).

Bechtel, W. G. (1955). A review of bread staling research. Trans Am. Assoc. Cereal Chem. 8:108.

Bennet, R. and Ewart, J. A. D. (1962). The reaction of acids with dough proteins. J. Sci. Food Agric. 13:15.

Bennet, R. and Cappock, J. B. M. (1956) Flour testing. I. A Comparison of Brabender, extensograph, Chopin Alveograph and Simmon extensometer methods of testing bread flours with particular reference to effect of various forms of flour treatments. J. Sci. Food Agric. 7:754.

Biliaderis, C. O., Maurice, T. J. and Vose, J. R. (1980). Starch gelatinization phenomena by differential scanning calorimetry. J. Food Sci. 45 1664.

Binger, H. P. (1965). Current Studies on Flour Composition and baking quality. Bakers Digest 39(6):24.

Birnbaum, H. (1955): Emulsifiers and regulators of labile water distribution between protein and starch. Bakers Digest 29(10):46.

Birnbaum, H. (1965). Pan release agents. Bakers Digest 39 (6):40.

Birnbaum, H. (1971). The relation of the physical state of emulsifiers and their efficacy in bread making. Bakers Digest 45(6):22.

Blaise, B. S., and Okezie, B. O. (1980). Baking and organoleptic quality of composite flour bread with winged bean, triticale and wheat. Bakers Digest 52(12):22.

Bloksma, A. H. (1964). Rheologie von Brotteig bei langsamen Deformationen. Prog Gebaeck 18:173.

Bloksma, A. H. (1971). Rheology and Chemistry of dough. In: Wheat Chemistry and Technology ed. by Pomeranz, Y. AACC. St. Paul Minnesota.

Boussingault, J. B. (1852) Experiences ayant pur but de determinen la cause de transportation due pain tendre in pain rassis. Ann. Chim. Phys.(3)36:490,

Brachfeld, B. A., Geminder, J. J. and Hetzel, C. P. (1966) Sodium Stearyl fumarate, a new bread improver. Bakers Digest. 40(4):53.

Bradbury, D., MacMasters, M. M., and Cull, I. M. (1956) Structure of the mature wheat kernel. 1. Gross anatomy and relationships of parts. Cereal Chem 33:329.

Brandbury, D., MacMasters, M. M., and Cull, I. M. (1956) II. Structure of mature wheat kernel. Microscopic structure of pericarp, seed coat, and other coverings of endosperm and germ of hard red spring wheat. Cereal Chem. 33:342.

Brandbury, D., MacMasters M. M. and Cull, I. M. (1956) III. Structure of the mature wheat kernel. Microscopic structure of the endosperm of hard red spring wheat. Cereal Chem. 33:361.

Brandbury, D., MacMasters, M. M. and Cull, I. M. (1956). Structure of the mature wheat kernel. IV. Microscopic structure of the germ of hard red winter wheat cereal chem 33:373.

Brehmer, W. V. (1928). In: J. V. Wiesner: Die Rohstoffe des Pflanzenreiches. Verlag W. Engelmann, Leipzig Bd. 2. S. 1953.

Brennan, J. G., and Sodah-Ayemnor, G. (1973). A study of the kinetics of retrogradation in a starch based dough made from dehydrated yam (*Dioscorea rotunda* L. Poir). Starke 25:276.

Britton, D. K. (1969). Cereals in the United Kingdom. Production, Marketing and Utilization. Pergamon Press. Oxford. London. Edinbough. New York. Toronto, Sydney,



Paris, Braunsschweig.

- Brokaw, G. Y. and Lyman, W. C. (1958). The behavior of distilled monoglycerides in the presence of water. J. Am. Oil Chemists' Soc. 35:49.
- Burham, M. E. and Clapp, J. A. (1942). A microscopic study of bread and dough. Cereal Chem. 19:196.
- Bushuk, W. (1961) Accessible Sulfhydryl groups in dough. Cereal Chem. 38:438.
- Bushuk, W. and Hlynka, I., (1961). The bromate reaction in dough. IV. Effect of reducing agents. Cereal Chem. 38:309.
- Bushuk, W., Briggs, R. G. and Shebeski, L. H. (1968). Protein quantity and quality as factors in the evaluation of bread wheats. Can. J. Plant. Scie. vol. 49:113.
- Bushuk, W., Tsen, C. C., and Hlynka, I. (1968). The function of mixing. Bakers Digest 42(4):36.
- Bushuk, W., (1975). Development, Licencing and distribution of New Varieties of grain in Canada. In: Grains and Oilseeds, handling, marketing and utilization 2nd. ed. By Canadian International Grains Institutes Winnipeg. Manitoba.
- Buttrose, M. S. (1962). The influence of environment on the shell structure of starch granules. J. Cell Biology, 14:159.
- Carlin, G. T. (1947). Fat in bread dough. Bakers Digest 21:78.
- Cathcard, W. H. and Lubber, S. V. (1939). Freezing as a means of retarding bread staling. Ind. Eng. Chem. 31:362.
- Carthcart, W. H. (1941). Further studies on the retrogradation of the staling of bread by freezing. Cereal Chem. 18:771.
- Chapman, D. (1955). Infrared spectra and Polymorphism of glycerides. Nature 176:216.
- Chapman, D. (1965). In: "The structure of lipids by Spectroscopic and X-ray techniques" P. 287. John Wiley and Sons Inc. N.Y.
- Ching-Shen, M. (1955). Morphologische Untersuchungen uber die in Chinesischen Arznei- und Nutz Pflanzen vorkommenden Staerkearten. Acta Pharmac. Sinica 3:105 (cited in Starch Atlas by Seidemann, 1966).

- Chopin, M. A. (1957). A propos de l'article de mm. E. Mae et al. A. Pirrotte relatif a L'hydratation des pates dans l'alveographie. Bull. Ecole Meunerie Belge 19:43.
- Christianson, D. D., Bakes, F. L., Loffredo, A. R. and Bagley, E. B. (1982). Correlation of the microstopic structure of corn starch granules with rheological properties of cooked pastes. In: Food Microstructure, vol. I. PP. 13-24. SEM Inc. AMF O'hare. Chicago.
- Chung, O. K., Pomeranz, Y., Hubbard, J. D. and Shogren, M. D. (1977) Defatted and reconstituted wheat flours. I. Effects of solvent and soxhlet types on functional (Bread making) Properties. Cereal Chem. 54(3):454.
- Chung, O. K., Pomeranz, Y., Hubbard, J. D. and Shogren, M. D. (1977). Defatted and reconstituted preheated flours. II. Effects of solvent type and extracting conditions on flours varying in breadmaking quality. Cereal Chem. 54:484.
- Chung, I., and Hadziyev, D. (1980). Tuber and Starch characteristics of Alberta grown potatoes. Can. Inst. Food Sci. Technol. J. 13:143.
- Chung, O. K., Pomeranz, Y. and Finney, K. F. (1978) Wheat flour lipids in bread making. Cereal Chem. 55:598.
- Ciacco, C. F., and D'Appolonia, B.L. (1977). Characterization of Starches from various tubers and their use in bread making. Cereal Chem. 54(5):1096-1107.
- Ciacco, C.F., and D'Appolonia, B.L. (1978). Baking studies with cassava and yam flour. I. Biochemical composition of cassava and yam flour. Cereal chem. 55(93):402-411.
- Ciacco, C.F.; and Fernandes, J.L.A. (1979). Effects of various ions on the kinetics of retrogradation of concentrated wheat starch gels. Stärke 31:51
- Collison, R. (1968). Starch retrogradation. In: "Starch and its derivatives", P.168. Radley, J.A. (ed) Chapman and Hall, London U.K.
- Colwell, K.H.; Axford, D.E., Chamberlain, N., and Elton, G.A.H. (1969). Effect of storage temperature on the aging of concentrated wheat starch gels J. Sc. Food Agric. 20:550
- Cornford, S. J. Axford, D. E. and Elton, G. A. H. (1964). The elastic modulus of bread crumb in linear compression in relation to staling. Cereal Chem. 57:310.
- Coursey, A. G. (1968). The Edible Aroids, WLD Crops, 20:25.
- Dahle, L. K., (1971) Wheat protein - Starch Interaction. I.

- L. and Paschall, E.F. (eds). Academic Press. New York. London.
- Knight, J. W. (1969). The Starch Industry. Pergamon Press. Oxford. London. Edinburg. New York. Toronto, Sydney. Paris. Braunschweig.
- Knightly, W. H. (1968). The surfactants in baked foods. S.C.I. Monograph 32:131, London. UK.
- Knightly, W. H. (1977). Bread staling - a review. Bakers Digest 51:52.
- Krog, N. (1981). Theoretical aspects of surfactants in relation to their use in bread making. Cereal Chem. 58:158.
- Krog, N. and Jenssen, N. B. (1970). Interaction of Monoglycerides in different physical states with amylose and their antifirming effects in bread. J. Food Technol. 5:77.
- Kulp, K. (1972). Physico-chemical properties of starches of wheats and flours cereal chem. 49:697.
- Kulp, K. (1973). Physico-chemical properties of wheat starch as related to bread. Bakers Digest 47(5):34.
- Kulp, K. and Lorenz, K. (1981). Starch functionality in white pan breads - New developments. Bakers Digest 55:24.
- Lagendijk, J. and Penning, H. J. (1970). Relation between complex formation of starch with monoglycerides and the firmness of bread. Cereal Sci. Today 15:265.
- Lamond, E. (1970). Methods for sensory evaluation of foods. Publ. No. 1284 Canadian Dept. of Agriculture, Ottawa.
- Lamour, R. K., Working, E. B. and Ofelt, C. W. Quality tests on bread red winter wheats. Cereal Chem. 16:733.
- Lampitt, L. H., Fulter, C. H. F. and Goldenberg, N. (1984). The fractionation of wheat starch. VII. Retrogradation of starch solutions. J. Soc. Chem. Ind. 60:175.
- Lansky, S., Schoch, T. J. and Williams, C. B. (1949). Properties of the fractions and linear subfractions from various starches. J. Am. Chem. Soc. 71:4066.
- Lauridsen, J. B. (1976). Food emulsifiers, surface activity, edibility, Manufacture, Composition and Application. J. Am. Oil Chem. Soc. 53:400.
- Leach, H. W. McCowen, L. D., and Schoch, T. J. (1959). Structure of Starch granule. I. Swelling and Solubility

- Johnson, J. A., Shellenberger, J. A., and Swanson, C. O. (1946) Farinograms and Mixograms as a means of evaluating flours for specific uses. *Cereal Chem.* 23:388.
- Johnson, J. A., Shellenberge, J. A. and Swanson, C. O. (1946). Extensograph studies of commercial flours and their relation to certain other physical dough tests. *Cereal Chem.* 23:400.
- Jongh, G. (1961). The formation of dough and bread structures. I. The ability of starch to form structures and the improving effect of glyceryl monostearate. *Cereal Chem.* 38:140.
- Kalb, A. J. and Sterling, C. (1961). Temperature and retrogradation of starch. *J. Food. Sci.* 26:587.
- Katz, J. R. (1928). Gelatinization and retrogradation of starch in relation to the problem of bread staling. In: comprehensive survey of starch chemistry; Walton, R. P. (ed). chem. Catalog. Co., Inc., NY.
- Katz, J. R. and Van Itallie, T. B. (1930). Abhandlungen zur physikalischen chemie der Starke und der Brotbereitung. V. Alle starkearten haben das gleiche retrogradationsspektrum. *Z. physikal. Chemie. (Leipzig)*. A150:90.
- Katz, J. R. and Rientsma, L. M. (1930). Physical and Chemistry of Starch and bread baking. III first and second stages of paste formation. *Z. Physik. Chem. (Leipzig)* A150:67.
- Kay, D. H. (1965). The techniques for electron microscopy 2nd ed. Blackwell Scientific Publications. Oxford. U.K.
- Keitt, T. E. (1912). Sweet Potato Investigations. S. Carolina Agric. exp. Sta. Bulletin 146.
- Kerr, R. W. (1950). Chemistry and Industry of starch, 3rd ed. Academic Press. New York.
- Kim, S. K. and D'Appolonia, B. L. (1977). Bread Staling studies. I. Effect of protein content on staling rate and bread crumb pasting properties. *Cereal Chem.* 54(2):207.
- Kim, J. C., and Ruiter, D. (1969). Bakery Products with non-wheat flours. *Bakers Digest* 43(6):58.
- Knight, J. W. (1965) The chemistry of wheat starch and gluten and their conversion products. Leonard Hill. London.
- Knight, J. W. (1967) Modification and uses of wheat starch. In: Starch: Chemistry and Technology 2nd ed. By Whistler R.

potato starch and its monoglyceride complexes. *Stärke* 33:290.

Hoover, R., and Hadziyev, D. (1982). Effect of monoglycerides on some rehydrating properties of potato starch granules. *Stärke* 34:152.

Hopkins, C. Y. and Graham, R. P. (1935). Starch content of some samples of Canadian wheat. *Can. J. Research* 12:820.

Hoseney, R. C., Finney, K. F. and Pomeranz, Y. (1970). Functional (bread making) and biochemical properties of wheat flour components. VI. Gliadin - lipid - gluten interactions in wheat gluten. *Cereal Chem.* 47:135.

Hoseney, R. C., Finney, K. F., Pomeranz, Y., and Shogren, M. S. (1971) Functional (bread making) and biochemical properties of wheat flour component VIII. *Starch. Cereal Chem.* 48:191.

Hough, L. and Jones, J. K. N. (1953). The Chemical evidence for the structure of starch. In: *Starch and its derivatives* (3rd ed). Vol. 1. Radley, J. A. Radley (ed) Chapman and Hall. London.

Hoyer, O. (1911) Die Korngroesse der Weizen und Kartoffelskaerke. *Z. Aug. Oesterr. Apotheken ver.* 49:227.

Hudson, J. F. and Ogunsua, A. O. (1976). The effects of fibre, starch damage and surfactants on baking quality of wheat/cassava composite flour. *J. Food technol.* 11:129.

Hussein, L., Gabriel, G. N. and Morias, S. R. (1974). Nutritional value of mixtures of Baladi bread and broad beans. *J. Sci. Food Agri.* 25:1433.

Hutchinson, J. B. (1936). The staling and keeping quality of bread. *Research association Brit. Flour Millers, Spec. Rept.* No.15.

Hylka, I., and Channin, W. G. (1957). Effect of pH on bromated and unbromated doughs. *Cereal Chem.* 34:371.

Jeffers, H. C. and Rubenthaler, G. L. (1978) High-yielding European wheats: determination of pastry end use properties. *Bakers Digest* 52(1):39.

Jeffers, H. C. Rubenthaler, G. C., Finney, P. L., Anderson, P. D. and Bruinsma, B. L. (1978). Pea : A high functional fortifier in wheat flour blends. *Bakers Digest* 52(3):36.

Jenkins, S. M. (1975). *Bakery Technology Book I. Bread.* Lester and Orpen Ltd. Toronto.

- Halton, P. (1961). Amylose activity, damaged starch, and water absorption. *Milling* 136:146.
- Hanssen, E., Dodt, E. and Niemann, E. G. (1953). Determination of Grain size, Surface, and weight of plant starches. *Kolloid Z.* 130:19.
- Hanssen, E. (1957). Fluoreszenzmikroskopische methode zur Beobachtung der Kleberausbildung in getreidemehlteigen. *Z. Lebensm. Untersuch. Forsch.* 106:196
- Harris, R. H. and Sibbit, L. O. (1941) The comparative baking qualities of starches prepared from different wheat varieties. *Cereal Chem.* 18:585.
- Harris, R. H. and Sibbit, L. D. (1942) The comparative baking qualities of hard red spring wheat starches and glutens as prepared by gluten - starch blend baking method. *Cereal Chem.* 19:763.
- Haydar, M. Moledina, K., Oorikul, B. and Hadziyev, D. (1980). Effect of Calcium and Magnesium on Cell wall and starch of dehydrated potato granules. *J. Agric. Food. Chem.* 28:383.
- Hellendoorn, E. W. 1971. Aspects of retrogradation in some dehydrated starch containing precooked food products. *Stärke* 23:63
- Hellman, N. N., Fairchild, B. and Senti, F. R. (1954). The bread staling problems: Molecular organization of starch upon aging of concentrated starch gels of various moisture levels. *Cereal Chem.* 31:495.
- Herd, C. W., and Kent-Jones, D. W. (1931). The determination of starch in cereal products. *J. Soc. Chem. Ind.* 50:15
- Herz, K. O. (1965). staling of bread - a review. *Food Technol.* 19:1828.
- Higashihara, M., Umeki, M., and Yamamoto, T. (1975). Isolation and some properties of taro root starch. *Denpun Kagaku (J. Japan Soc. Starch Sci)* 22:61.
- Holme, J. B. (1966). A review of wheat flour proteins and their functional properties. *Bakers Digest* 45(6):38.
- Hollo, J., Szejtli, J. and Gunter, G. S. (1960). Retrogradation of amylose. *Stärke* 12:73.
- Hollo, J. and Gunter, G. S. (1960<sub>2</sub>). The mechanism of the retrogradation of amylose. *Stärke* 12:106.
- Hoover, R., and Hadziyev, D. (1981). Characterization of

aspects of some tropical tuber starches. *Stärke* 34(8):255-262.

Gemeinder, J. J., Thomas, P. D. and Hetzel, C. P. (1965) Sodium Stearyl fumarate : Effects in continuous process bread. *Cereal Sci. Today*. 10:425

Gilbert, G. A. and Spragg, S. P. (1964). Iodometric determination of amylose iodine sorption. "Blue Value" In: *Methods in carbohydrate chemistry*. Vol. IV. P. 168. Whistler, R. L. (eds). Academic. Press, NY.

Ghiasi, K., Varriano-Marston, E. and Hoseney, R. C (1982). Gelatinization of wheat starch. II. Starch - surfactant interaction. *Cereal Chem.* 59(2):86.

Goldstein, S. (1957) Sulfhydryl and Disulfidgruppen der Klebereiweisse und ihre Beziehung zur Backfähigkeit der Brotmehle. *Mitt Gebiete Lebensmittel Hyg.* (Bern) 48:87.

Gray, V. M. and Schoch. T. J. (1962). Effect of Surfactants and Fatty adjuncts on swelling and solubilization of granular starches. *Stärke*. 14:239.

Greenwood, C. T., (1956). Aspects of the physical chemistry of starch. *Advances in Carbohydrate Chem.* 11:355.

Greenwood, C. T. and Houston, B. J. (1967). Physico-chemical studies on starches XXXIII. Some physico-chemical properties of hydroxy ethyl starch used as a volume - extender for blood plasma. *Stärke*, 19:243.

Greenwood, C. T. and Muirhead, H. E. (1967). "Thermal degradation of Starch VII. DTA of Maltodextrins and of starch and its derivatives. *Stärke*. 19:281.

Grewe, E. and Bailey, C. H. (1927). The concentration of gluten and other proteins in various types of wheat flour. *Cereal Chem.* 4:230.

Griffin, W. C. (1949). Classification of surface active Agents by HLB. *J. Soc. Cos. Chem.* 1:331.

Grosskreutz, J. C. A. (1961). Lipoprotein Model for wheat gluten structure. *Cereal Chem.* 38:336.

Hahn, P. P. and Rasper, V. P. (1974). Effect of non-starchy polysaccharides from yam, sorghum, and millet flours on rheological behavior of wheat doughs. *Cereal Chem.* 51:734.

Halton, P. (1949). Significance of load-extension tests in assessing the baking quality of wheat flour doughs. *Cereal Chem.* 26:24.

57(5):310.

Duckworth, R. B. and Smith, G. M. (1963). Diffusion of solutes at low moisture levels. *Recent Adv. Food Sci.* 3:250.

Edmond, J. B. and Ammerman, G. R. (1971). *Sweet Potatoes : Production Processing and Marketing*. AVI. Westport, Connecticut.

Elton, G. A. H. (1969) Some quantitative aspects of bread staling. *Bakers Digest* 43(3):24.

Evans, L. G., Pearson, A. M. and Hooper, G. R. (1981) Scanning electron microscopy of flour-water doughs treated with oxidising and reducing agents. In: "Studies of Food Microstructure" by Cohen, S. H., Davis, E. A. Holcomb, D. N. and Kalab, M. (eds) P. 313. Scanning Electron Microscopy, Inc. AMF O'Hare - Chicago.

Farrand, E. A (1972). The influence of particle size and starch damage on the characteristics of bread flours. *Bakers Digest* 46(1):22.

Farrand, E. A. (1972). Controlled levels of starch damage in a commercial United Kingdom bread flour and effects on absorption sedimentation value and loaf quality. *Cereal Chem.* 49:479

Fleming, S. E. and Sosulski, F. W. (1977). Nutritive value of bread fortified with concentrated plant proteins and lysine. *Cereal Chem.* 54:238.

Foster, J. F. (1965) physical properties of amylose and amylopectin in solution. In: *Starch. Chemistry and Technology*. Whistler R. L. and Paschall E. F. (eds).

Foster, J. F. and Paschall, E. P. (1953). Effect of disaggregation of amylose on the properties of the Iodine Complex. *J. Am. Chem. Soc.* 75:1181.

Fraser, J. R. and Holmes, D. C. (1956). The proximate analysis of wheat flour. *Carbohydrates II*. The analysis of carbohydrate fractions of different flour types. *J. Sci. Food Agric.* 7:589.

Fruton, J. S. and Simmonds, S. (1958). *General Biochemistry*. 2nd ed. John Wiley and Sons, New York.

Gage, J. W. (1978). Fortification of bread products. *Bakers Digest* 52(1):26.

Gallant, D. H., Bewa, H., Buy, Q. H., Bouchet, B., Szylił, O. and Sealy L. (1982). On ultrastructural and nutritional



Some Starch binding effects of wheat flour Proteins Cereal Chem. 48:706.

D'Appolonia, B. L., Gilles, K. A., Osman, E. M., and Pomeranz, Y. (1971). Carbohydrates. Chapter 7. In: Wheat Chemistry and Technology, 2nd ed. by Pomeranz. AACC. St. Paul. Minnesota.

D'Appolonia, B.L. and Gilles, K.A. (1971) Effect of various starches in baking. Cereal Chem. 48:625

D'Appolonia, B. C. (1978). Use of untreated, and roasted navy beans in bread making. Cereal chem 55:898

D'Appolonia B. L. and MacArthur, L. A., (1979). Utilization of Sunflower flour derived from untreated and roasted sunflower meats in bread making.. Bakers Digest 53(2):32.

D'Appolonia, B. L., and Morad. M. M. (1981). Bread Staling. Cereal Chem. 58(3):186.

Deathrage, W. L., MacMasters, M. M. and Rist, C. E. (1955). A partial survey of amylose content in starch in domestic and foreign varieties of corn, wheat and sorghum, and from some other starch - bearing plants. Trans. Ame Assoc. Cereal Chem 13:31.

Del Vecchio, A. J. (1975). Emulsifiers and their use in soft - wheat products. Bakers Digest 44(8):28.

Dendy, D.V.A.; Clerke, P.A. and James, A.W. (1970). The use of blends of wheat and non-wheat flours in bread making. Tropical Sciences 12:13

Dennett, K. and Sterling, C., Davis, Calif. (1979). The role of starch in Bread formation. Starke 31:209.

Dimler, R. J., Davis, H. A., Rist, C. E. and Hilbert, G. E. (1944). Production of starch from wheat and other cereal flours. Cereal Chem. 12:430.

Dimler, R. J. (1963) Gluten - the key to wheat quality. Bakers Digest 37(1):52.

Donovan, J. W. (1979). Phase transition of the starch - water system. Biopolymers 18:263.

Donovan, J. N. and Mapes, C. J. (1980). Multiple phase transitions of starches and nageli amyloextrins. Starke 32:190.

Dragsdorf, R. D. and Varriano-Marston, E. (1979). Bread staling: X-ray diffraction studies on bread supplemented with  $\alpha$ -amylase from different sources. Cereal Chem.

- patterns of various starches. *Cereal Chem.* 36:534.
- Leach, H. W. (1965). Gelatinization of starch. In: *Starch: Chemistry and Technology* Vol. 1. P. 289. Whistler, R. L. and Paschall, E. F. (ed). Academic Press. NY>
- Leach, H. W. and Schoch, T. J. (1961) Structure of the starch granule. II Action of various amylases on granular starches. *Cereal Chem.* 38:34.
- Leach, H. W. and Schoch, T. J. (1962). Structure of the starch granule. III. Solubility of starches in Dimethyl Sulfoxide. *Cereal Chem.* 39:318
- Lee, E. Y. C., Mercier, C., and Whelan, W. J. (1968). The fine structure of amylopectin. *Arch. Biochem. Biophysics.* 125:1028.
- Lindet, L. (1902). On the condition of starch in fresh and in state bread. *Bull. Soc. Chem. France. Ser. 3*, 27:63.
- Lentner, C. (1956). Beitrag zur Kenntnis des Staerkeanteils verschiedener Getreidearten und sorten. Dis. Wien (cited in *Starch Atlas* by Seidemann, 1966)
- Locken, L., Loska, S. and Shuey, W. (1960). *The Farinograph hand book*. Am. Assoc. Cereal Chemists. St. Paul, Minn.
- Loewus, F. A. and Briggs, D. R. (1957). A Potentiometric study of the Change in iodine binding capacity of amylose while retrograding in dilute solution. *J. Am. Chem. Soc.* 79:1494.
- Longley, R. W. and Miller, B. S. (1971). Note on the relative effect of monoglyceride. On the gelatinization of wheat starch. *Cereal Chem.* 8:81.
- Lonkhuysen, V. H. and Blankestijn, J. (1974). Interaction of Monoglycerides with starches. *Stärke* 26(10):337.
- Lonkhuysen, V. H. and Blankestijn, J. (1976). Influence of monoglycerides on the gelatinization and enzymatic breakdown of wheat and cassava starch. *Stärke* 28(7):277.
- Lorenz, K. (1978). 'Non-Oilseed' Sunflower products in bakery foods. *Bakers Digest.* 52(8):28.
- Lorenz, K., Dilsaver, W. and Wolt, M. (1979). Fababean flour and protein concentrate in baked goods and in baked goods and in pasta products. *Bakers Digest* 53(6):39.
- Lowry, O. H., Rosebrough, N. J., Farr, A. L. and Randall, R. J. (1951). Protein Measurement with the folin phenol reagent. *J. Biol. Chem.* 193:265.

- MacConnell, L. M., Simmonds, D. H., and Bushuk, W. (1974). High Protein bread from Wheat-Fababean Composite flour. *Cereal Science Today* 19:517.
- MacDonald, I. A., (1964). The functional properties of various surface-active agents. *Bakers Digest* 38(1):56.
- MacDonald, I. A. and Lensack, G. P. (1967). Fluid-liquid shortenings. Formation and evaluation in baker's Cakes. *Cereal Sci. Today* 12:10.
- MacMasters, M. M. (1961). Starch Research and Baking. *Bakers Digest* 36(5):42.
- MacMasters, M. M., Hinton, J. J. C. and Bradbury, D. (1971). Microscopic Structure and Composition of wheat Kernel. In: *Wheat chemistry and Technology*, 2nd ed. Edited by Pomeranz. Y. AACC. St. Paul. Minn.
- MacMasters, M. M. and Waggle, D. H. (1963). The importance of starch on the identification of cereal grains in feeds. *Stärke* 25:7.
- MacMasters, M. M., Wolf, M. J. and Seckinger, H. L. (1957). Microscopic Characteristics of starches in identification of ground cereal grains. *J. Agric. Food. Chem.* 5:455.
- Maes, E., and Pirotte, A. (1956). L'alveographe de chopin : Hydration constante on variable? *Bull. Ecole Mennerie Belge* 18:118.
- Maga, J. A. and Ponte, J. G. (1975). Bread staling. CRC critical review. *Food Technol.* 4:443.
- Maleki, M., Vetter, J. L. and Hoover W. J. (1981). The effect of emulsifiers, Sugar, Shortening and soya flour on staling of Barbari flat bread. *J. Sci. Food Agric.* 32:1209.
- Mangels, C. E. and Bailey, C. H. (1933). Relative Viscosities of Wheat Starches. *Ind. Eng. Chem.* 25:456.
- Mangels, C. E. (1934). Varietal and regional variation in properties of wheat starchs. *Cereal Chem.* 11:571.
- Marnett, L. F. and Tenney, R. J. (1961). Calcium Stearyl-2 lactylate - a new and versatile baking ingredient. *Bakers Digest* 35(6):52.
- Marston, P. E. and Short, A. L. (1969). Factors involved in storage of bread. *Food Technol. Anst.* 21:154.
- Martz, S. A. (1960). *Bakery Technology and Engineering*. AVI Pub. Co, Inc. Westport. Connecticut.

Matwejew, W. P. (1958). On Sweet Potato starch granule size distribution; cited by Seidemann, J. in "Starch Atlas" Paul Parey in Berlin and Humburg (1966).

McCreedy, R. M., and Hassid, W. Z. (1943). The Separation and quantitative estimation of amylose and amylopectin in potato starch. J. Am. Chem. Soc. 65:1154.

McIver, R. S. Axford, D. W. E., Colwell, K. H., and Elton, G. A. H. (1968). Kinetic Study of retrogradation of gelatinized starch. J. Sci. Food Agric. 19:560.

Mecham, D. K., Sokol, H. A., Pence, J. W. (1962) Extractable Protein and Hydration Characteristics of flours and doughs in dilute acid. Cereal Chem. 39:81.

MedCalf, D. S., and Gilles, K. A. (1968). Structural Characterization of a pentosans from the water insoluble portion of durum wheat endosperm. Cereal Chem. 45:550.

MedCalf, D. G. and Gilles, K. A. (1965). Wheat Starches. I. Comparison of physico-chemical properties. Cereal Chem. 42:558.

Meisner, D. (1969). Succinylated Monoglyceride: a new acid for bakers. Bakers Digest 43(3):38.

Meyer, A. (1895). Untersuchungen uber die starke korner, G. Fischer Verlag, Jena, Germany.

Meyer, K. H. (1942). Recent developments in starch chemistry. In : Advances in colloid Science, ed. by E. O. Kraemer. Vol. 1. pp. 143-182. Interscience. NY.

Meyer, K. H. (1950). "Natural and Synthetic high polymers" p. 456. Interscience.

Miller, B. S. Derby, R.I., and Trimbo, H. B. (1973). A pictorial explanation for the increase in viscosity of heated wheat starch water suspension. Cereal Chem. 50:271.

Miller, M., (1974). Protein fortified Cereal Foods for Developing countries. Cereal Sci. Today 19:509.

Montgomery, E. M., and Senti, F. R. (1958). Separation of amylose from amylopectin of starch by an extraction - Sedimentation procedure. J. Polymer. Sci. 28:1.

Morrison, W. R., Mann, D. L., Soon, W., and Coventry A. M. (1975) Selective Extraction and quantitative analysis of non-starch and starch lipids from wheat flour. J. Sci. Fd. Agric. (26).507-521.

Moy, J. H., and Nip, W. K. (1983). In processed food,

- Chapter 10, In : Taro: A review of colocasia esenlenta and its potentials by Jaw-Kai Wang (ed). University of Hawai Press. Honolulu.
- Nikuni, Z. (1957). Fine structure of starch granules. Studies with electron microscope and X-ray diffraction. Kagaku (Tokayo) 27:283.
- Nikuni, Z. (1977). Studies on starch granules. Stärke30(4):105.
- Noznick, P. P. and Geddes, W. F (1943). Application of the baker compressimeter to cake studies. Cereal Chem. 20:463.
- Ohashi, K., Goshima, G., Kusuda, H., and Tsuge, H. S. (1979). Effect of embraced lipids on the geletanization of rice starch. Stärke 32:54.
- Olatunji, O. and Akinrele, I. A. (1975). Comparative rheological properties and bread qualities of wheat flour diluted with tropical tuber and breadfruit flours. Cereal Chem. 55:1.
- Onwneme, I. C. (1978). The tropical tuber crops. John Wiley and Sons. Chichester. New York. Brisbane. Toronto.
- Osborne, T. B. (1907). The proteins of wheat Kernel. Carnegie Inst. Wash. Publ. No. 84.
- Osman, E. M. (1967). Starch in the food industry. In: Starch, Chemistry and techhnology, Vol. II, P. 163, R. L. Whister and Paschall E. F. (eds). Academic Press. NY.
- Osman E. M. and Cummisford, P. D. (1959). Some factors affecting the stability of frozen white sources. Food Res. 24:595.
- Oyenuga, V. A. (1968). Nigeria's Foods and Feeding Stuffs, University of Ibadam Press, Ibadan, Nigeria.
- Payne, J. H., Ley, G. J., and Akau, G. (1941) Processing and chemical investigations of taro. Hawaii Agri. Exp. Sta. Bull 86.
- Pence, J. W., Standridge, N. N., Mecham, D. K., Lubisich, T. M. and Okott, H. S. (1956). Moisture distribution in fresh, frozen and frozen-defrosted bread. Food Technol. 10:76.
- Pence, J. W. and Nimmo, C.C. and Hepburn, F. N. (1964) Proteins. In: Wheat Chemistry and Technology Edited by Hlynka, I. AACC. St. Paul. Minn.
- Phillips, K. L., and Sallans, H. R. (1966). Ammonia Process for separating starch and gluten from wheat flour. Cereal

Science Today II. 61.

Phillips, T. P. (1974). World Market prospects for cassava and its products. P. 13-19. In : Cassava Processing and Storage. Proceedings of an inter disciplinary workshop, Pattaya, Thailand. 17-19.

Pitt, J. S. (1971). Surfactant in food stuffs. Process Biochem. 6:11.

Planchion, L. et. Juillet, A. (1909). Etude de quelques fecules coloniales. Ann. Musee Colon. Marseille, Ser. 2 7, 411. (cited in Starch Atlas by Seidemann, 1966).

Platt, W. and Powers, R. (1940). Compressibility of bread crumb. Cereal Chem. 17:601.

Pomeranz, Y., Rubenthaler, G. L. and Finney, K. F. (1966). Studies on the mechanisms of the bread-improving effects of lipids. Food Technol. 20:1485.

Pomeranz, Y., and Finney, K. F. (1969). Glycolipids, their distribution, chemistry and role in bread making Cereal Sci. Today 14:172.

Pomeranz, Y. (1980). Molecular approach to bread making. An update and new perspectives. Bakers Digest 4.

Pomeranz, Y. and Chung, O. K. (1979). Proc. Inter. symp. Protein utilization Guelph, Canada.

Pomeranz, Y., and Shellenbeger, J. A. (1971). Bread science and technology Westport. Connecticut. AVI Pub. Comp. Inc.

Ponte, J. G. Jr. Titcomb, S. T. Cerning, J. and Cotton, R. H. (1963). Studies of the high-protein fines and coarse fractions of a southwest and an intermountain flour. Cereal Chem. 40:601.

Potter, J. G. Jr. Titcomb, S. T. and Cotton, R. H. (1962). Flour as a factor in bread firming. Cereal Chem. 39:437.

Potter, A. L., and Hassid, W. Z. (1948). Starch, I. End group determination of amylose and amylopectin by periodate oxidation. J. Am. Chem. Soc. 70:3488.

Pratt, D. B. Jr. (1971). Criteria of flour quality. In: Wheat: Chemistry and technology (2nd ed). AACC Monograph Series III. Chap. 5, P. 201. American Association of Cereal Chemists : St. Paul., Minn.

Prentice, N., Cuendet, L. S. and Geddes, W. F. (1954). Studies on bread making. V. Effect of flour fractions and various starches on the firming of bread crumb. Cereal

Chem. 31:188.

Pringle, W., Williams, A., and Hulse, J. H. (1969). Mechanically developed doughs from composite flours. Cereal sci. Today 14:114.

Pulkki, L. H. (1938). Particle size in relation to flour characteristics and starch cells of wheat. Cereal Chem. 15:749.

Purseglove, J. W. (1968). Tropical Crops. Dicotyledons. I. Longmans Green and Co, London. PP 332.

Pyler, E. J. (1979). Baking Science and Technology Vol. 1, Siebel publishing Company. Chicago.

Radley, J. A. (1940). Starch and its derivatives. Chapman and Hall. London.

Radley, J. A. (1953). Starch and its Derivatives 3rd ed. Chapman and Hall. London.

Rao, P. S., and Beri, R. M. (1955). Dioscorea starches. Science and Cult, (India) 20:397.

Rask, O. S., and Alsberg, C. L. A. (1924). Viscometric Study of wheat starches. Cereal Chem. 1:7.

Rask, O. S., and Aslberg, C. L. A. (1924). On Gelatinization by heat of wheat and corn starch. Cereal Chem. 1:107

Rasper, V., Rasper, J. and Mabey, G. L. (1974). Functional properties of non-wheat flour substitutes in composite flours. I. The effect of non-wheat starches in composite doughs. Can. Inst. Food. Sci. Technol. J. 7(2):86.

Reichert, E. T. (1913). The differentiation and specificity of sterches in realtion to genera, species etc. "Carnegie Institution Washington, Washington, D. C. Publication 173. Part 1.

Remenovsky, E. (1921). Erdmann-Konigs Grundriss der allegemeinen Warenkunde unter Berucksichtigung der technologie und Mikroskopie. Verlag, J. A. Berth, Leipzig. 16 Auft, Bd.I.S.317.

Roberts, J. D. and Caserio, M. C. (1965). Basic principles of organic chemistry. W. A. Benjamin, Inc. New York.

Roewe, P., Lorenz, K. and Kulp, K. (1982). Staling of variety breads. Lebensm, - Wiss. u. - Technol. 15:263.

Rundle, R. E., and French. D, (1943). The centrifugation of starch in starch iodide complex. III. X-ray diffraction

studies of the starch iodide complex. J. Am. Chem. Soc. 65:1707.

Sandstedt, R. M., Jolitz, C. E., and Blish, M. L. (1959). Starch in relation to some baking properties of flour. Cereal Chem. 16:780

Sandstedt, R. M. (1955). Photomicrographic studies of wheat starch: IV. Enzymatic digestion and granule structure. Cereal Chem. 32(Suppl):17.

Sandstedt, R. M., and Schroeder, H. (1960). A photographic study of mechanically damaged wheat starch. Food Technol 14:257.

Sandstedt, R. M. (1961). The function of starch in the baking of bread. Bakers Digest 35(3):36.

Sandstedt, R. M. and Gates, R. L. (1954). Raw Starch digestion: a comparison of the raw starch digesting capabilities of the amylase systems from four alpha-amylase sources. Food Research 19:190.

Sathe, S. K., Ponte, J. G. Jr. Rangnekar, P. D. and Salunkhe, D. K. (1981) Effects of addition of Great Northern bean flour and protein concentrates on rheological properties of dough and baking quality of bread. Cereal Chem. 58(2):97.

Schoch, T. J. (1941). Physical aspects of starch behaviour. Cereal Chem. 18:121.

Schoch, T. J. (1942). Fractionation of starch with selective precipitation with butanol. J. Am. Chem. Soc. 64:2957.

Schoch, T. J. (1961) Starches and analyses. Proc. Ann. Mech. Ann. Soc. Brewing Chemists, P. 83.

Schoch, T. J. (1964). Swelling Power and Solubility of granular starches. In: "Methods in Carbohydrate Chemistry", Vol. IV, P. 106, Whistler R. L. (ed) Academic Press. NY.

Schoch, T. J. (1964). Iodometric determination of amylose. Potentiometric titration: Standard method. In: "Methods in Carbohydrate Chemistry", Vol. IV, P. 157. Whistler, R. L. (ed). Academic Press. NY.

Schoch, T. J. (1965) Swelling Power and Solubility of granular starches. Page 106. In: Whistler, R. L. (ed). Methods in Carbohydrate chemistry, Vol. 5, Academic Press. New York and London.

Schoch, T. J. (1945). The fractionation of starch. In : Advances in Carbohydrate Chemistry, Vol. 1. , Academic



Press. New York.

Schoch, T. J. and Maywald, E. C. (1956). Microscopic examination of modified starches. *Anal. Chem.* 28:382.

Schoch, T. J. and French, J. (1947). Studies on bread staling. 1. The role of starch. *Cereal Chem.* 24:231.

Seckinger, H. L. and Wolf, M. J. (1970). Electron Microscopy of endosperm protein from hard and soft wheats. *Cereal Chem.* 47:236.

Seidemann, J. (1963). Untersuchungen zur mikroskopischen unterscheidung von Manioka und Bataten Starke. *Starke* 15:326.

Seidemann, J. (1964). Mikroskopische Untersuchungen verschiedener Dioscoreastärken. *Starke* 16:246.

Seidemann, J. (1966). Staerke-Atlas. Grundlagen der Staerke-Mikroskopie und Beschreibung der wichtigsten Staerkearten. Paul Parey in Berlin und Hamburg.

Selman, R. W. (1949). New recording consistometer for commerical mixers. *Bakers Digest* 23:102.

Shehata, N. A. and Fryer, B. A. (1979). Effect on protein quality of supplementing wheat flour with chickpea flour. *Cereal Chem.* 47(6):663.

Shewfelt, M. and Kidman, F. C. (1975). Starches of non-wheat flours, their effect on bread quality. *Bakers Digest*.

Shewfelt, L. (1967). Manufacture of tapioca, arrowroot and sago starches, In: *Starch Chemistry and Technology*, Vol. 1. Industrial Aspects Whistler (ed) PP. 103-119.

Shewfelt, W. C., and Gilles, K. A. (1966). Effect of spring softening and absorption on mixograms for measuring dough characteristics. *Cereal Chem.* 43:94.

Sokol, H. A., Mecham, D. K. and Pence, J. W. (1960). Sulfhydryl losses during mixing of doughs: Comparison of flours having different mixing characteristics. *Cereal Chem.* 37:739. Sokol, R. K. and Rohlf, J. F. (1969). *Biometry*. Freeman and Company. San Francisco.

Sosulski, F. W. and Fleming, S. E. (1979) Sensory evaluation of bread prepared from composite flours. *Bakers digest* 53(6): 20.

Stamberg, O. E., (1959). Starch as a factor in dough formation. *Cereal Chem.* 16:769.

Sterling, C. (1957). Retrogradation in starch jelly candy. Food Research 22:184.

Sterling, C. (1960). Molecular association of starch at high temperature. Starke 12:28.

Sterling, C. and Panghorn, J. (1960). Fine structure of potato starch. Ann. J. Bot.

Sterling, C. (1974). Fibrillar Structure of Starch. Evidence for crossed fibrils from incipient gelatinization. Starke 26:105.

Sterling, C. (1976). Fibrils of starch in potato and curcuma. Starke 28:39.

Sterling, C. (1978). Textural qualities and molecular structure of starch products. J. Texture Studies. 9 :225.

Stevens, D. J. and Elton, G. A. H. (1971). Thermal properties of starch-water system. 1. Measurement of heat of geletimization by DSC. Stärke 23:8.

Sullivan, Betty, Near, Cleo and Foley, G. H. (1936). The role of lipids in relation to flour quality. Cereal Chem. 13:318.

Swanson, C. O., and Working, E. B. (1933). Testing the quality of flour by recording dough mixer. Cereal Chem. 10:1.

Swortfiguer, M. J. (1971). Improving the shelf life of bread. Bakers Digest, 45(5):46.

Takuechi, I. (1969). Interaction between protein and starch. Cereal Chem. 46:570.

Taylor, N. W., Cluskey, J. E. and Senti F. R. (1958). Moisture content and relative humidity of bread and its components, wheat starch and gluten, in relation to staling. Abstr., 43rd Ann. Meeting, Am. Ass. Cereal Chem. Cincinnati, Ohio.

Thurber, F. H. (1933). Chemical and physical properties of sweet potato starch. Ind. Eng. Chem. 25:565.

Tipples, K. H. (1969). The relation of starch damage to the baking performance of flour. Paper No. M-84 of the Board of Grain Commissioners, Grain Research Laboratory. Winnipeg. Manitoba.

Tipples, K. H., and Kilborn, R. H. (1975). "Unmixing"- The disorientation of developed bread doughs by slow speed

- mixing. Cereal Chem. 52:248.
- Tsen, C. C. (1970). Chemical dough development. Bakers Digest. 44(4):28.
- Tsen, C. C., Peters E M, Shaffer, T. and Hoover, W. J. (1973). High protein cookies. 1. Effect of soy fortification and surfactants. Bakers Digest. 47:(4):30.
- Tsen, C. C., Tang, R. T. (1971). K-State process for making high protein breads. 1. Soy flour bread. Bakers Digest. 45(5):26.
- Tsen, C. C. and Hoover W. J. Higher protein bread from wheat flour fortified with full fat soy flour. Cereal Chem. 50:7 (1973).
- Tu, C. C., Nip, W. K., and Nakayama, T.O.M. (1979). Starch and flour from taro. In : Small scale processing and storage of tropical root crops, ed. Donald, L. Pucknett, PP. 249-259. Westview trop. Agric. Series No. 1. Westview press. Boulder. Colorado.
- Varriano-Marston, E., Ke, V., Huang, G., and Ponte, D. Jr. (1980) Comparison of methods to determine starch gelatinization in bakery products. Cereal Chem. 57(4):242.
- Voisey, M. and Unrau, A. M. (1964). Flour composition and Chemical constituents of flour from cytologically synthesized and natural cereal species. J. Agri. Food Chem. 12:84.
- Voisey, P. W., Miller, H. and Kloeck, M. (1966). An electronic recording dough mixer. II. An experimental evaluation. Cereal Chem. 43:420.
- Voisey, P. W., Miller, H. and Kloeck, M. (1966). An electronic recording mixer. IV. Application in Farinography. Cereal Chem. 43:438.
- Volz, F. E. and Ramstad, P. E. (1952). Effect of emulsifiers on the enzyme susceptibility of starch during staling of bread. Cereal Chem. 28:118.
- Von Bibra, E. (1861). Getreidearten und das Brot, Nuremberg.
- Waltdt, L. (1968). The problem of staling, its possible solution. Bakers Digest. 42(5):64.
- Wang, J. K. (1983). Introduction (Chapter 1). In: Taro: A review of *Colocasia esculenta* and its potential uses by Jaw-Kai Wang (ed). University of Hawai: Press. Honolulu.
- Watson, S. A. (1964). Determination of starch gelatinization

- temperature. In : "Methods in Carbohydrate Chemistry", Vol. IV, P. 240, Whistler, R. L. (ed). Academic Press. NY.
- Wehrli, H. P. and Pomeranz, Y. (1969). The role of chemical bonds in dough. *Bakers Digest*. 43:22.
- Wehrli, H. P. and Pomeranz, Y. (1970). A note on autoradiography of tritium labelled galactolipids in dough and bread. *Cereal Chem*. 47:221.
- Whistler, R. L. and Johnson, C. (1948). Effect of acid hydrolysis on retrogradation of amylose. *Cereal Chem*. 25:418.
- Whistler, R. L., Byrd, J. D. and Thornburg, W. L., (1955). Surface structure of starch granules. *Biochem. Biophys. Acta*. 18:146.
- Whistler, R. L. and Turner, E. S. (1955) Fine structure of starch granule sections. *J. Polymer Sci*. 18:153.
- Whistler, R. L. (1965). Starch retrogradation. In : "Starch : Chemistry and Technology", Whistler, R. L. and Paschall, E. F (eds). Academic Press, NY.
- Wijeratne, W. B. (1974). Cultivation, Processing and Utilization of Cassava in Sri Lanka. In : Cassava Processing and storage. Proceeding of an Interdisciplinary Workshop. Pattaya, Thailand, 17-19. April. 1974. Araullo- (ed). IDRC-031e.
- Willhoft, E. M. A. (1971). Bread staling. 1. Experimental study. II. Theoretical study. *J. Sci. Food Agric*. 22:176.
- Willhoft, E. M. A. (1973). Recent developments on the bread staling problem. *Bakers Digest*. 47(6):14.
- Wivinis, G. P., and Maywald, E. C. (1967). Photographs of starches. In : "Starch : Chemistry and Technology. Vol. II. P.649. Whistler, R. L. and Paschall. E. F. (eds). Academic Press; New York.
- Wolfom, M. L., and Elkhadem, H. (1965). Chemical evidence for the structure of starch. In : "Starch Chemistry and Technology", Vol.I, P.251. Whistler, R. L. and Paschall, E. F. (ed). Academic Press. NY.
- Wootton, M. and Bamunuaracchchi, A. (1979). Application of differential Scanning Calorimetry to starch gelatinization. I. Commercial native and modified starches. *Starke* 6:201.
- Wren, J. J. (1968). Importance of physical state in the applications of fat-derived emulsifiers. S.C.I. Monograph 32:158. London. UK.

Wurtzburg, O. B. and Szymanski (1970). Modified Starches for the food industry. J. Agric. Food Chem., Vol. 18(6):997.

Yasunaga, T., Bushuk, W. and Irvine, G. N. (1968). Gelatinization of Starch during bread-making. Cereal Chem. 45:269.

Yoshino, D. and Matsumoto, H. (1966). Colloid titration of wheat proteins, dough and flour. Cereal Chem. 43:187.

Ziegler, E., and Green, E. N. (1971). Principles of milling. In : Wheat Chemistry and Technology. Pomeroy, Y. (ed). AACC. St. Paul. Minn.

Zobel, H. F. (1964). X-ray analysis of starch granules. In : "Methods in carbohydrate chemistry"; Vol. IV, P.109, Whistler, R. L. (ed). Academic Press, NY.

Zobel, H. F. (1973). A review of bread staling. Bakers Digest. 47:52.

Zobel, H. F., and Senti, F. R. (1959). The bread staling problem. X-ray diffraction studies on breads containing cross-linked starch and heat stable amylases. Cereal Chem. 36:441.

## 9. APPENDICES

# APPENDIX 1.

## Gelatinization Characteristics of Cassava Starch - Fijian Cultivar

| Water<br>Volume<br>Fraction<br>(V <sub>1</sub> ) | Endotherms         |             |            |                   |                  |                               |             |             |                   |                  |
|--|--------------------|-------------|------------|-------------------|------------------|-------------------------------|-------------|-------------|-------------------|------------------|
|  | Gelatinization (g) |             |            |                   |                  | Crystallite (m <sub>1</sub> ) |             |             |                   |                  |
|  | t°C<br>Onset       | t°C<br>Peak | t°C<br>End | cm <sup>2</sup> * | ΔH <sup>**</sup> | t°C<br>Onset                  | t°C<br>Peak | t°C<br>End  | cm <sup>2</sup> * | ΔH <sup>**</sup> |
| 0.8  | 62.88±1.10         | 67.25±1.32  | 72.50±1.80 | 3.50±0.63         | 4.23±0.76        | -                             | -           | -           | -                 | -                |
| 0.7  | 60.88±1.70         | 67.13±1.18  | 72.50±0.71 | 4.21±0.67         | 5.09±0.81        | 73.50±0.00                    | 80.70±2.55  | 86.38±3.10  | 1.92±0.38         | 2.32±0.46        |
| 0.6  | 59.50±1.08         | 66.38±0.95  | 78.17±1.04 | 4.22±0.68         | 4.22±0.82        | 72.20±1.52                    | 85.50±2.30  | 93.00±1.50  | 3.68±1.05         | 4.45±1.27        |
| 0.5  | 59.20±1.44         | 65.48±1.11  | 71.40±2.04 | 1.15±0.52         | 1.39±0.63        | 72.33±1.04                    | 93.25±2.10  | 101.70±3.27 | 3.81±0.05         | 4.61±0.06        |
| 0.4  | 56.50±0.35         | 63.75±0.41  | 77.17±2.59 | 0.99±0.23         | 1.20±0.28        | 76.30±1.60                    | 97.00±1.170 | 115.57±9.29 | 6.07±0.88         | 6.12±0.77        |
| 0.3  | 56.00±0.00         | 62.50±0.00  | 75.50±0.00 | -                 | -                | 83.50±8.41                    | 122.75±1.77 | 125.86±3.80 | 12.59±0.00        | 15.23±0.00       |

\* Endotherm area.

\*\* ΔH Enthalpy of Fusion, Cal/g Starch.

## APPENDIX 2.

## Gelatinization Characteristics of Sweet Potato Starch - Californian Centennial Cultivar

| Water<br>Volume<br>Fraction<br>(V <sub>1</sub> ) | Endotherms   |             |            |                   |             |              | Crystallite (m <sub>1</sub> ) |             |                   |           |
|--|--------------|-------------|------------|-------------------|-------------|--------------|-------------------------------|-------------|-------------------|-----------|
|  | t°C<br>Onset | t°C<br>Peak | t°C<br>End | cm <sup>2</sup> * | ΔH**        | t°C<br>Onset | t°C<br>Peak                   | t°C<br>End  | cm <sup>2</sup> * | ΔH**      |
| 0.8  | 54.50±0.71   | 64.50±0.50  | 74.67±0.58 | 8.64±0.85         | 3.49±0.34   | -            | -                             | -           | -                 | -         |
| 0.7  | 54.75±0.35   | 61.34±0.76  | 69.00±0.00 | 6.40±0.50         | 2.59±0.20   | 72.50±0.71   | 85.25±0.35                    | 94.25±0.35  | 2.62±0.60         | 1.07±0.24 |
| 0.6  | 52.50±0.50   | 61.17±0.38  | 70.00±0.00 | 3.41±0.06         | 1.38±0.02   | 71.67±2.10   | 81.33±2.50                    | 90.33±4.30  | 1.72±0.08         | 0.69±0.03 |
| 0.5  | 52.75±1.50   | 60.38±0.48  | 68.63±0.48 | 2.43±0.47         | 0.98±0.19   | 73.50±1.80   | 88.13±5.54                    | 99.17±3.40  | 1.42±0.33         | 0.57±0.13 |
| 0.4  | 53.67±0.76   | 60.50±0.50  | 68.50±0.50 | 2.56±0.11         | 1.03±0.04   | 87.00±2.00   | 105.54±1.54                   | 121.17±1.00 | 6.42±0.50         | 2.59±0.20 |
| 0.3  | -            | -           | -          | -                 | 79.67±12.40 | 97.50±27.3   | -                             | -           | -                 | -         |

\* Endotherm area.

\*\* ΔH Enthalpy of fusion, Cal/g Starch.



## APPENDIX 3.

## Gelatinization Characteristics of Sweet Potato Starch - Porto Rico Cultivar

| Water<br>Volume<br>Fraction<br>( $V_1$ ) | Endotherms                 |                           |                          |                            |                           |                          |
|--|----------------------------|---------------------------|--------------------------|----------------------------|---------------------------|--------------------------|
|  | Gelatinization (g)         |                           |                          | Crystallite ( $m_1$ )      |                           |                          |
|  | $t^\circ\text{C}$<br>Onset | $t^\circ\text{C}$<br>Peak | $t^\circ\text{C}$<br>End | $t^\circ\text{C}$<br>Onset | $t^\circ\text{C}$<br>Peak | $t^\circ\text{C}$<br>End |
|  |                            |                           |                          | $\Delta H^{**}$            | $\text{cm}^2$             | $\Delta H^{**}$          |
| 0.7                                      | 51.50 $\pm$ 0.00           | 57.50 $\pm$ 0.71          | 66.00 $\pm$ 0.00         | 2.88 $\pm$ 0.04            | 3.49 $\pm$ 0.05           | 77.0 $\pm$ 8.50          |
| 0.6                                      | 50.88 $\pm$ 0.63           | 58.33 $\pm$ 0.29          | 65.00 $\pm$ 0.00         | 2.26 $\pm$ 0.50            | 2.74 $\pm$ 0.61           | 72.25 $\pm$ 2.47         |
| 0.5                                      | 55.00 $\pm$ 0.00           | 74.00 $\pm$ 0.00          | 85.50 $\pm$ 0.00         | 1.60 $\pm$ 0.42            | 1.94 $\pm$ 0.51           | 91.00 $\pm$ 2.83         |
| 0.4                                      | 53.50 $\pm$ 0.00           | 75.00 $\pm$ 0.00          | 86.50 $\pm$ 0.71         | 1.46 $\pm$ 0.13            | 1.77 $\pm$ 0.16           | 94.00 $\pm$ 0.00         |
| 0.3                                      | -                          | -                         | -                        | -                          | -                         | 65.50 $\pm$ 0.71         |
|  |                            |                           |                          |                            |                           | 89.25 $\pm$ 1.10         |
|  |                            |                           |                          |                            |                           | 101.50 $\pm$ 0           |
|  |                            |                           |                          |                            |                           | 1.53 $\pm$ 0.09          |
|  |                            |                           |                          |                            |                           | 1.84 $\pm$ 0.03          |
|  |                            |                           |                          |                            |                           | 2.26 $\pm$ 1.85          |
|  |                            |                           |                          |                            |                           | 1.32 $\pm$ 0.50          |
|  |                            |                           |                          |                            |                           | 2.04 $\pm$ 0.65          |
|  |                            |                           |                          |                            |                           | 2.47 $\pm$ 0.79          |

\* Endotherm area.

\*\*  $\Delta H$  Enthalpy of Fusion, Cal/g Starch.

APPENDIX 4.  
Gelatinization Characteristics of Some Canadian Wheat Starches

| Endotherms         |                 |           |          |         |                           |                        |          |         |                           |    |
|--------------------|-----------------|-----------|----------|---------|---------------------------|------------------------|----------|---------|---------------------------|----|
| Gelatinization (g) |                 |           |          |         |                           | Crystallite ( $\eta$ ) |          |         |                           |    |
| Type of Starch     | Volume fraction | t°C Onset | t°C Peak | t°C End | $\Delta H/\text{Cal/g}^*$ | t°C Onset              | t°C Peak | t°C End | $\Delta H/\text{Cal/g}^*$ |    |
| cv. Fielder        | 0.8             | 50.50     | 56.00    | 62.50   | 0.89                      | --                     | --       | --      | --                        | -- |
|                    |                 | +0.72     | +1.30    | +1.80   |                           |                        |          |         |                           |    |
|                    | 0.7             | 50.08     | 55.18    | 60.90   | 0.66                      | 61.50                  | 70.30    | 76.50   | 0.320                     |    |
|                    |                 | +0.91     | +0.82    | +2.02   | +0.10                     | +0.41                  | +0.20    | +2.30   | +0.030                    |    |
|                    | 0.6             | 49.50     | 55.50    | 62.45   | 1.20                      | 62.80                  | 77.50    | 92.50   | 0.920                     |    |
|                    |                 | +0.65     | +0.70    | +0.90   | +0.44                     | +1.10                  | +0.50    | +1.40   | +0.070                    |    |
|                    | 0.5             | 49.0      | 54.50    | 58.50   | 0.63                      | 78.50                  | 89.00    | 105.50  | --                        |    |
|                    |                 | +1.10     | +1.50    | +1.70   | +0.15                     | +1.60                  | +3.00    | +3.50   |                           |    |
|                    | 0.4             | 50.00     | 53.50    | 57.00   | --                        | 86.50                  | --       | --      | --                        |    |
|                    |                 | +1.50     | +1.30    | +2.00   |                           | +1.30                  |          |         |                           |    |
|                    | 0.3             | --        | --       | --      | --                        | --                     | --       | --      | --                        |    |

\* Enthalpy of Fusion.

## APPENDIX 5.

Complexing Indices (%) for the Interaction of the  $\alpha$ -crystallinity Form of  $C_{16}$  and  $C_{18}$  Monoglycerides With Cassava Starch

| % MG | Cassava Starch   |                  |                  |                  |                  |                  |
|------|------------------|------------------|------------------|------------------|------------------|------------------|
|      | Ungelatinized    |                  | Gelatinized      |                  | Solubilized      |                  |
|      | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         |
| 0.1  | 4.75 $\pm$ 0.26  | 1.24 $\pm$ 0.22  | 11.69 $\pm$ 2.77 | 8.66 $\pm$ 0.38  | 28.72 $\pm$ 0.23 | 14.62 $\pm$ 0.80 |
| 0.2  | 6.24 $\pm$ 0.18  | 2.33 $\pm$ 0.06  | 24.09 $\pm$ 1.50 | 16.71 $\pm$ 2.89 | 47.93 $\pm$ 1.11 | 26.84 $\pm$ 0.19 |
| 0.3  | 7.58 $\pm$ 0.20  | 6.69 $\pm$ 0.91  | 38.07 $\pm$ 2.27 | 23.37 $\pm$ 2.31 | 58.02 $\pm$ 1.16 | 46.65 $\pm$ 2.47 |
| 0.4  | 9.67 $\pm$ 0.15  | 8.47 $\pm$ 0.06  | 43.31 $\pm$ 0.79 | 26.53 $\pm$ 0.80 | 63.30 $\pm$ 1.85 | 60.84 $\pm$ 1.51 |
| 0.5  | 9.97 $\pm$ 0.18  | 8.74 $\pm$ 0.11  | 46.19 $\pm$ 3.27 | 30.64 $\pm$ 0.08 | 70.73 $\pm$ 0.73 | 70.41 $\pm$ 1.37 |
| 0.8  | 11.05 $\pm$ 0.07 | 10.11 $\pm$ 0.56 | 4.09 $\pm$ 4.55  | 35.19 $\pm$ 0.49 | 78.76 $\pm$ 0.52 | 75.10 $\pm$ 1.70 |
| 1.0  | 12.09 $\pm$ 0.74 | 11.89 $\pm$ 0.30 | 49.24 $\pm$ 6.54 | 37.30 $\pm$ 0.47 | 83.12 $\pm$ 0.62 | 76.36 $\pm$ 2.86 |

## APPENDIX 6.

Complexing Indices (%) for the Interaction of the  $\beta$ -crystallinity Form of  $C_{16}$  and  $C_{18}$  Monoglycerides With Cassava Starch

| % MG | Cassava Starch  |                 |                  |                  |                  |                  |
|------|-----------------|-----------------|------------------|------------------|------------------|------------------|
|      | Ungelatinized   |                 | Gelatinized      |                  | Solubilized      |                  |
|      | $C_{16}$        | $C_{18}$        | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         |
| 0.1  | 1.85 $\pm$ 0.09 | 1.28 $\pm$ 0.24 | 3.80 $\pm$ 0.01  | 1.75 $\pm$ 0.25  | 9.50 $\pm$ 0.88  | 4.62 $\pm$ 0.45  |
| 0.2  | 5.08 $\pm$ 0.01 | 2.70 $\pm$ 0.27 | 6.75 $\pm$ 0.13  | 3.65 $\pm$ 0.75  | 13.40 $\pm$ 0.85 | 8.30 $\pm$ 1.17  |
| 0.3  | 6.96 $\pm$ 0.19 | 4.10 $\pm$ 0.34 | 8.23 $\pm$ 0.06  | 5.53 $\pm$ 1.61  | 15.40 $\pm$ 0.85 | 11.28 $\pm$ 0.74 |
| 0.4  | 7.83 $\pm$ 0.08 | 5.47 $\pm$ 0.23 | 9.88 $\pm$ 0.01  | 7.03 $\pm$ 1.52  | 17.79 $\pm$ 0.92 | 13.70 $\pm$ 1.31 |
| 0.5  | 8.50 $\pm$ 0.17 | 6.51 $\pm$ 0.34 | 10.76 $\pm$ 0.11 | 7.92 $\pm$ 1.61  | 18.46 $\pm$ 0.95 | 15.98 $\pm$ 0.18 |
| 0.8  | 9.50 $\pm$ 0.11 | 8.36 $\pm$ 0.23 | 11.94 $\pm$ 0.24 | 11.73 $\pm$ 1.53 | 19.41 $\pm$ 0.13 | 19.12 $\pm$ 1.72 |
| 1.0  | 9.95 $\pm$ 0.06 | 8.72 $\pm$ 0.17 | 12.22 $\pm$ 0.19 | 12.00 $\pm$ 1.43 | 20.38 $\pm$ 0.44 | 19.94 $\pm$ 1.72 |

## APPENDIX 7.

Complexing Indices (%) for the Interaction of the  $\alpha$ -crystallinity Form of  $C_{16}$  and  $C_{18}$  Monoglycerides With Sweet Potato Starch

| % MG | Sweet Potato Starch |                 |                  |                  |                  |                  |
|------|---------------------|-----------------|------------------|------------------|------------------|------------------|
|      | Ungelatinized       |                 | Gelatinized      |                  | Solubilized      |                  |
|      | $C_{16}$            | $C_{18}$        | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         |
| 0.1  | 2.99 $\pm$ 0.21     | 0.99 $\pm$ 0.08 | 14.17 $\pm$ 0.72 | 9.00 $\pm$ 0.77  | 37.82 $\pm$ 4.19 | 30.13 $\pm$ 3.57 |
| 0.2  | 5.24 $\pm$ 0.86     | 2.34 $\pm$ 0.15 | 27.21 $\pm$ 3.00 | 17.65 $\pm$ 1.24 | 51.64 $\pm$ 4.20 | 47.80 $\pm$ 2.38 |
| 0.3  | 7.35 $\pm$ 0.21     | 3.57 $\pm$ 0.43 | 38.00 $\pm$ 0.70 | 25.15 $\pm$ 2.55 | 59.93 $\pm$ 3.63 | 56.20 $\pm$ 2.40 |
| 0.4  | 7.80 $\pm$ 0.14     | 4.71 $\pm$ 0.61 | 50.13 $\pm$ 1.41 | 34.30 $\pm$ 1.51 | 65.06 $\pm$ 1.95 | 62.86 $\pm$ 2.50 |
| 0.5  | 8.51 $\pm$ 0.25     | 5.72 $\pm$ 0.37 | 60.60 $\pm$ 3.08 | 40.84 $\pm$ 1.78 | 68.42 $\pm$ 2.80 | 68.01 $\pm$ 2.38 |
| 0.8  | 9.74 $\pm$ 0.07     | 7.50 $\pm$ 0.47 | 69.70 $\pm$ 0.22 | 56.42 $\pm$ 1.20 | 72.37 $\pm$ 5.58 | 73.90 $\pm$ 1.20 |
| 1.0  | 10.36 $\pm$ 0.14    | 8.23 $\pm$ 0.07 | 70.17 $\pm$ 0.18 | 60.62 $\pm$ 1.28 | 75.33 $\pm$ 4.19 | 75.58 $\pm$ 1.19 |

## APPENDIX 8.

Complexing Indices (%) for the Interaction of the  $\beta$ -crystallinity Form of  $C_{16}$  and  $C_{18}$  Monoglycerides With Sweet Potato Starch

| % MG | Sweet Potato Starch |                 |                  |                  |                  |                  |
|------|---------------------|-----------------|------------------|------------------|------------------|------------------|
|      | Ungelatinized       |                 | Gelatinized      |                  | Solubilized      |                  |
|      | $C_{16}$            | $C_{18}$        | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         |
| 0.1  | 0.49 $\pm$ 0.09     | 0.44 $\pm$ 0.03 | 10.75 $\pm$ 0.49 | 7.40 $\pm$ 1.47  | 13.70 $\pm$ 2.69 | 10.38 $\pm$ 1.49 |
| 0.2  | 0.91 $\pm$ 0.15     | 0.89 $\pm$ 0.06 | 23.66 $\pm$ 0.43 | 18.05 $\pm$ 1.43 | 25.09 $\pm$ 1.44 | 22.25 $\pm$ 5.04 |
| 0.3  | 1.19 $\pm$ 0.20     | 1.10 $\pm$ 0.06 | 29.69 $\pm$ 0.42 | 25.52 $\pm$ 1.26 | 32.56 $\pm$ 0.19 | 30.26 $\pm$ 6.29 |
| 0.4  | 1.43 $\pm$ 0.24     | 1.24 $\pm$ 0.03 | 32.70 $\pm$ 0.95 | 29.63 $\pm$ 0.15 | 35.36 $\pm$ 1.36 | 35.51 $\pm$ 7.40 |
| 0.5  | 1.65 $\pm$ 0.18     | 1.73 $\pm$ 0.06 | 36.90 $\pm$ 0.23 | 33.74 $\pm$ 0.00 | 38.93 $\pm$ 0.16 | 38.32 $\pm$ 8.20 |
| 0.8  | 2.08 $\pm$ 0.13     | 1.92 $\pm$ 0.03 | 42.14 $\pm$ 0.21 | 37.74 $\pm$ 0.33 | 47.91 $\pm$ 0.96 | 40.75 $\pm$ 8.94 |
| 1.0  | 2.20 $\pm$ 0.06     | 2.15 $\pm$ 0.06 | 44.42 $\pm$ 0.42 | 38.90 $\pm$ 1.65 | 52.51 $\pm$ 0.16 | 43.07 $\pm$ 8.94 |

## APPENDIX 9.

Compositional Indices (%) for the Interaction of the  $\alpha$ -Crystallinity Form of  $C_{16}$  and  $C_{18}$  Monoglycerides With Taro Starch

|      | Taro Starch      |                  |                  |                  |                  |                  |
|------|------------------|------------------|------------------|------------------|------------------|------------------|
|      | Un gelatinized   |                  | Gelatinized      |                  | Solubilized      |                  |
| % ME | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         |
| 0.1  | 4.59 $\pm$ 0.97  | 3.48 $\pm$ 0.71  | 10.02 $\pm$ 0.74 | 9.75 $\pm$ 0.88  | 36.70 $\pm$ 1.51 | 29.75 $\pm$ 0.77 |
| 0.2  | 6.93 $\pm$ 1.4   | 5.97 $\pm$ 1.41  | 15.29 $\pm$ 0.75 | 14.38 $\pm$ 0.53 | 44.40 $\pm$ 0.81 | 38.82 $\pm$ 0.62 |
| 0.3  | 8.2 $\pm$ 1.72   | 8.47 $\pm$ 1.54  | 20.77 $\pm$ 1.17 | 18.30 $\pm$ 0.42 | 47.64 $\pm$ 0.58 | 45.51 $\pm$ 0.44 |
| 0.4  | 10.89 $\pm$ 1.75 | 10.44 $\pm$ 2.11 | 26.89 $\pm$ 0.74 | 21.87 $\pm$ 0.57 | 51.26 $\pm$ 0.40 | 49.53 $\pm$ 0.42 |
| 0.5  | 11.89 $\pm$ 1.82 | 10.68 $\pm$ 2.46 | 29.99 $\pm$ 2.32 | 25.22 $\pm$ 0.85 | 54.33 $\pm$ 0.30 | 52.03 $\pm$ 0.38 |
| 0.8  | 14.08 $\pm$ 1.72 | 11.93 $\pm$ 2.81 | 41.96 $\pm$ 0.49 | 31.02 $\pm$ 1.25 | 56.49 $\pm$ 0.73 | 55.03 $\pm$ 0.22 |
| 1.0  | 14.72 $\pm$ 1.56 | 12.43 $\pm$ 2.81 | 44.52 $\pm$ 0.16 | 35.37 $\pm$ 2.71 | 55.81 $\pm$ 0.61 | 55.81 $\pm$ 0.61 |

## APPENDIX 10.

Complexing Indices (%) for the Interaction of the  $\beta$ -Crystallinity Form of  $C_{16}$  and  $C_{18}$  Monoglycerides With Taro Starch

| % MG | Taro Starch      |                 |                  |                  |                  |                  |
|------|------------------|-----------------|------------------|------------------|------------------|------------------|
|      | Ungelatinized    |                 | Gelatinized      |                  | Solubilized      |                  |
|      | $C_{16}$         | $C_{18}$        | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         |
| 0.1  | 2.35 $\pm$ 0.47  | 1.19 $\pm$ 0.00 | 4.76 $\pm$ 0.84  | 2.52 $\pm$ 0.15  | 7.65 $\pm$ 0.78  | 6.57 $\pm$ 1.73  |
| 0.2  | 4.69 $\pm$ 0.94  | 2.23 $\pm$ 0.21 | 8.63 $\pm$ 0.42  | 4.76 $\pm$ 0.52  | 10.52 $\pm$ 0.58 | 9.88 $\pm$ 2.10  |
| 0.3  | 5.69 $\pm$ 1.41  | 3.42 $\pm$ 0.21 | 11.01 $\pm$ 0.42 | 7.04 $\pm$ 0.46  | 13.39 $\pm$ 1.16 | 13.00 $\pm$ 1.90 |
| 0.4  | 7.03 $\pm$ 1.16  | 3.82 $\pm$ 1.34 | 13.28 $\pm$ 1.53 | 8.82 $\pm$ 0.45  | 15.71 $\pm$ 0.96 | 15.23 $\pm$ 2.11 |
| 0.5  | 8.71 $\pm$ 0.95  | 5.36 $\pm$ 0.84 | 14.58 $\pm$ 0.42 | 10.17 $\pm$ 0.57 | 17.76 $\pm$ 0.38 | 17.02 $\pm$ 1.27 |
| 0.8  | 10.72 $\pm$ 1.89 | 7.59 $\pm$ 0.77 | 18.00 $\pm$ 0.21 | 16.06 $\pm$ 0.60 | 21.32 $\pm$ 0.77 | 19.70 $\pm$ 0.84 |
| 1.0  | 12.05 $\pm$ 1.90 | 8.33 $\pm$ 1.68 | 19.19 $\pm$ 0.21 | 18.74 $\pm$ 1.26 | 22.68 $\pm$ 0.39 | 20.86 $\pm$ 1.65 |



## APPENDIX 11.

Complexing Indices (%) for the Interaction of the  $\alpha$ -crystallinity Form of  $C_{16}$  and  $C_{18}$  Monoglycerides With Yam Starch

| % MG | Ungelatinized    |                 | Yam Starch<br>Gelatinized |                  | Solubilized      |                  |
|------|------------------|-----------------|---------------------------|------------------|------------------|------------------|
|      | $C_{16}$         | $C_{18}$        | $C_{16}$                  | $C_{18}$         | $C_{16}$         | $C_{18}$         |
| 0.1  | 2.13 $\pm$ 0.13  | 1.21 $\pm$ 0.53 | 13.97 $\pm$ 0.39          | 10.62 $\pm$ 0.11 | 19.46 $\pm$ 0.97 | 18.62 $\pm$ 0.65 |
| 0.2  | 3.46 $\pm$ 0.07  | 2.25 $\pm$ 0.40 | 27.95 $\pm$ 0.52          | 23.48 $\pm$ 0.35 | 34.86 $\pm$ 1.45 | 29.86 $\pm$ 0.59 |
| 0.3  | 4.88 $\pm$ 0.10  | 2.83 $\pm$ 0.64 | 45.96 $\pm$ 0.45          | 30.98 $\pm$ 0.27 | 46.38 $\pm$ 1.61 | 44.63 $\pm$ 1.85 |
| 0.4  | 6.22 $\pm$ 0.17  | 3.19 $\pm$ 0.67 | 52.60 $\pm$ 0.16          | 45.24 $\pm$ 1.10 | 54.82 $\pm$ 0.65 | 48.54 $\pm$ 1.52 |
| 0.5  | 7.27 $\pm$ 0.32  | 3.77 $\pm$ 0.27 | 59.66 $\pm$ 0.45          | 47.00 $\pm$ 1.84 | 61.21 $\pm$ 0.00 | 51.31 $\pm$ 0.91 |
| 0.8  | 10.89 $\pm$ 2.27 | 4.08 $\pm$ 0.00 | 63.87 $\pm$ 0.03          | 50.45 $\pm$ 0.00 | 67.13 $\pm$ 1.92 | 60.33 $\pm$ 0.61 |
| 1.0  | 11.90 $\pm$ 1.92 | 4.08 $\pm$ 0.00 | 65.28 $\pm$ 0.35          | 55.45 $\pm$ 0.32 | 69.20 $\pm$ 1.62 | 63.22 $\pm$ 0.80 |

## APPENDIX 12.

Complexing Indices (%) for the Interaction of the  $\beta$ -crystallinity Form of  $C_{16}$  and  $C_{18}$  Monoglycerides With Yam Starch

| % MG | Yam Starch      |                 |                  |                  |                  |                  |
|------|-----------------|-----------------|------------------|------------------|------------------|------------------|
|      | Ungelatinized   |                 | Gelatinized      |                  | Solubilized      |                  |
|      | $C_{16}$        | $C_{18}$        | $C_{16}$         | $C_{18}$         | $C_{16}$         | $C_{18}$         |
| 0.1  | 0.21 $\pm$ 0.03 | 0.17 $\pm$ 0.01 | 16.17 $\pm$ 2.93 | 7.24 $\pm$ 1.49  | 17.53 $\pm$ 4.03 | 13.80 $\pm$ 2.88 |
| 0.2  | 0.37 $\pm$ 0.00 | 0.26 $\pm$ 0.01 | 37.70 $\pm$ 0.17 | 12.99 $\pm$ 1.66 | 42.99 $\pm$ 0.26 | 20.78 $\pm$ 1.43 |
| 0.3  | 0.53 $\pm$ 0.05 | 0.50 $\pm$ 0.07 | 40.65 $\pm$ 0.11 | 17.60 $\pm$ 1.76 | 46.20 $\pm$ 0.10 | 26.84 $\pm$ 0.69 |
| 0.4  | 0.67 $\pm$ 0.16 | 0.59 $\pm$ 0.06 | 43.48 $\pm$ 0.25 | 20.61 $\pm$ 0.44 | 49.36 $\pm$ 0.12 | 30.51 $\pm$ 0.59 |
| 0.5  | 0.77 $\pm$ 0.06 | 0.73 $\pm$ 0.13 | 45.88 $\pm$ 5.67 | 22.74 $\pm$ 0.78 | 56.18 $\pm$ 4.91 | 33.21 $\pm$ 1.98 |
| 0.8  | 0.93 $\pm$ 0.13 | 0.83 $\pm$ 0.11 | 56.39 $\pm$ 1.65 | 26.46 $\pm$ 2.30 | 58.71 $\pm$ 3.87 | 38.09 $\pm$ 1.49 |
| 1.0  | 1.05 $\pm$ 0.20 | 0.92 $\pm$ 0.11 | 55.24 $\pm$ 8.59 | 27.08 $\pm$ 2.40 | 62.00 $\pm$ 6.19 | 39.59 $\pm$ 1.77 |

## APPENDIX 13

Percent Complexing Indices of the Interaction Between Lintnerized Wheat  
cv. Neepawa Starch With  $\alpha$ -C<sub>16</sub> and  $\alpha$ -C<sub>18</sub> Monoglycerides

| Lintnerized Wheat cv. Neepawa Starch* |                           |                           |                           |                           |                           |                           |
|---------------------------------------|---------------------------|---------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
| % MG***<br>Added                      | Ungelatinized             |                           | Gelatinized               |                           | Solubilized**             |                           |
|                                       | $\alpha$ -C <sub>16</sub> | $\alpha$ -C <sub>18</sub> | $\alpha$ -C <sub>16</sub> | $\alpha$ -C <sub>18</sub> | $\alpha$ -C <sub>16</sub> | $\alpha$ -C <sub>18</sub> |
| 0.1                                   | 8.47±0.36                 | 3.75±0.82                 | 17.80±0.54                | 11.32±0.55                | 29.66±0.23                | 16.56±0.77                |
| 0.2                                   | 13.55±0.46                | 7.26±0.74                 | 36.00±0.00                | 27.78±0.11                | 43.24±0.88                | 27.66±3.25                |
| 0.3                                   | 15.37±1.17                | 8.59±0.73                 | 46.21±1.07                | 35.17±0.06                | 51.32±2.85                | 39.06±3.23                |
| 0.4                                   | 16.09±1.75                | 9.32±0.77                 | 52.27±1.06                | 47.22±0.55                | 57.46±1.55                | 48.97±2.09                |
| 0.5                                   | 16.33±1.93                | 9.56±0.84                 | 56.06±0.00                | 51.39±0.71                | 66.35±2.32                | 56.80±1.17                |
| 0.8                                   | 16.70±1.70                | 9.94±0.91                 | 66.50±2.12                | 55.56±0.40                | 68.62±3.16                | 69.37±2.35                |
| 1.0                                   | 16.94±1.73                | 10.05±0.44                | 70.46±1.07                | 55.56±0.36                | 71.23±2.94                | 73.49±2.41                |

\*Starch lintnerization was done by treatment of dry starch with 7.5% HCl at 40°C for 72 hrs.

\*\*Starch solubilization was done by dispersing the ungelatinized starch in aqueous 1 N KOH at 4°C for 30 min.

\*\*\*MG, Monoglyceride.

## APPENDIX 14.

Percent Complexing Indices of the Interaction Between Lintnerized Wheat  
cv. Neepawa Starch With  $\beta$ -C<sub>16</sub> and  $\beta$ -C<sub>18</sub> Monoglycerides

| Lintnerized Wheat cv. Neepawa Starch* |                          |                          |                          |                          |                          |                          |
|---------------------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| % MG***<br>Added                      | Ungelatinized            |                          | Gelatinized              |                          | Solubilized**            |                          |
|                                       | $\beta$ -C <sub>16</sub> | $\beta$ -C <sub>18</sub> | $\beta$ -C <sub>16</sub> | $\beta$ -C <sub>18</sub> | $\beta$ -C <sub>16</sub> | $\beta$ -C <sub>18</sub> |
| 0.1                                   | 4.03 $\pm$ 0.23          | 2.25 $\pm$ 0.39          | 8.75 $\pm$ 0.58          | 5.64 $\pm$ 0.71          | 12.88 $\pm$ 0.86         | 7.11 $\pm$ 1.07          |
| 0.2                                   | 8.06 $\pm$ 1.81          | 3.84 $\pm$ 1.19          | 13.30 $\pm$ 1.01         | 8.53 $\pm$ 0.69          | 19.80 $\pm$ 2.30         | 14.21 $\pm$ 2.14         |
| 0.3                                   | 12.90 $\pm$ 0.29         | 5.42 $\pm$ 1.73          | 17.86 $\pm$ 0.25         | 11.46 $\pm$ 1.03         | 26.80 $\pm$ 2.51         | 22.57 $\pm$ 2.05         |
| 0.4                                   | 13.71 $\pm$ 0.01         | 6.88 $\pm$ 2.09          | 21.14 $\pm$ 0.38         | 12.81 $\pm$ 2.96         | 30.42 $\pm$ 0.26         | 29.47 $\pm$ 1.00         |
| 0.5                                   | 15.32 $\pm$ 0.59         | 7.94 $\pm$ 1.34          | 23.23 $\pm$ 0.91         | 12.85 $\pm$ 0.64         | 33.41 $\pm$ 0.96         | 31.26 $\pm$ 0.88         |
| 0.8                                   | 16.13 $\pm$ 2.25         | 9.39 $\pm$ 0.16          | 26.43 $\pm$ 0.96         | 19.81 $\pm$ 0.89         | 45.89 $\pm$ 0.42         | 36.40 $\pm$ 2.10         |
| 1.0                                   | 16.93 $\pm$ 2.20         | 9.66 $\pm$ 0.86          | 29.62 $\pm$ 0.87         | 20.21 $\pm$ 0.52         | 54.25 $\pm$ 0.71         | 39.79 $\pm$ 0.55         |

\*Starch lintnerization was done by treatment of dry starch with 7.5% HCl at 40°C for 72 hrs.

\*\*Starch solubilization was done by dispersing the ungelatinized starch in aqueous 1N KOH at 4°C for 30 min.

\*\*\*MG, Monoglyceride.

## APPENDIX 15.

Percent Complexing Indices of the Interaction Between Lintnerized Wheat  
cv. Fielder Starch With  $\alpha$ -C<sub>16</sub> and  $\alpha$ -C<sub>18</sub> Monoglycerides

| Lintnerized Wheat cv. Fielder Starch* |                           |                           |                           |                           |                           |                           |
|---------------------------------------|---------------------------|---------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
| % MG***<br>Added                      | Ungelatinized             |                           | Gelatinized               |                           | Solubilized**             |                           |
|                                       | $\alpha$ -C <sub>16</sub> | $\alpha$ -C <sub>18</sub> | $\alpha$ -C <sub>16</sub> | $\alpha$ -C <sub>18</sub> | $\alpha$ -C <sub>16</sub> | $\alpha$ -C <sub>18</sub> |
| 0.1                                   | 12.16 $\pm$ 0.58          | 11.84 $\pm$ 0.45          | 12.87 $\pm$ 0.52          | 13.05 $\pm$ 1.05          | 13.35 $\pm$ 0.57          | 11.24 $\pm$ 1.62          |
| 0.2                                   | 19.52 $\pm$ 2.18          | 15.04 $\pm$ 0.41          | 26.11 $\pm$ 0.52          | 27.54 $\pm$ 0.00          | 26.90 $\pm$ 1.79          | 24.53 $\pm$ 0.64          |
| 0.3                                   | 22.08 $\pm$ 0.46          | 17.92 $\pm$ 0.72          | 38.61 $\pm$ 1.56          | 39.13 $\pm$ 2.05          | 38.51 $\pm$ 1.27          | 38.74 $\pm$ 0.64          |
| 0.4                                   | 23.36 $\pm$ 0.68          | 19.33 $\pm$ 0.74          | 47.43 $\pm$ 0.52          | 47.83 $\pm$ 4.09          | 49.81 $\pm$ 2.14          | 48.58 $\pm$ 1.51          |
| 0.5                                   | 24.17 $\pm$ 0.48          | 20.16 $\pm$ 1.92          | 53.31 $\pm$ 0.36          | 50.73 $\pm$ 4.09          | 55.38 $\pm$ 3.01          | 54.19 $\pm$ 2.24          |
| 0.8                                   | 25.43 $\pm$ 0.49          | 20.88 $\pm$ 2.32          | 62.87 $\pm$ 0.82          | 50.90 $\pm$ 3.08          | 64.52 $\pm$ 3.57          | 63.68 $\pm$ 1.84          |
| 1.0                                   | 25.92 $\pm$ 0.49          | 21.44 $\pm$ 0.94          | 65.81 $\pm$ 0.32          | 54.90 $\pm$ 3.08          | 70.36 $\pm$ 3.64          | 68.82 $\pm$ 1.94          |

\*Starch lintnerization was done by treatment of dry starch with 7.5% HCl at 40°C for 72 hrs.

\*\*Starch solubilization was done by dispersing the ungelatinized starch in aqueous 1 M KOH at 4°C for 30 min.

\*\*\*MG, Monoglyceride.

## APPENDIX 16.

Percent Complexing Indices of the Interaction Between Lintnerized Wheat  
cv. Fielder Starch With  $\beta$ -C<sub>16</sub> and  $\beta$ -C<sub>18</sub> Monoglycerides

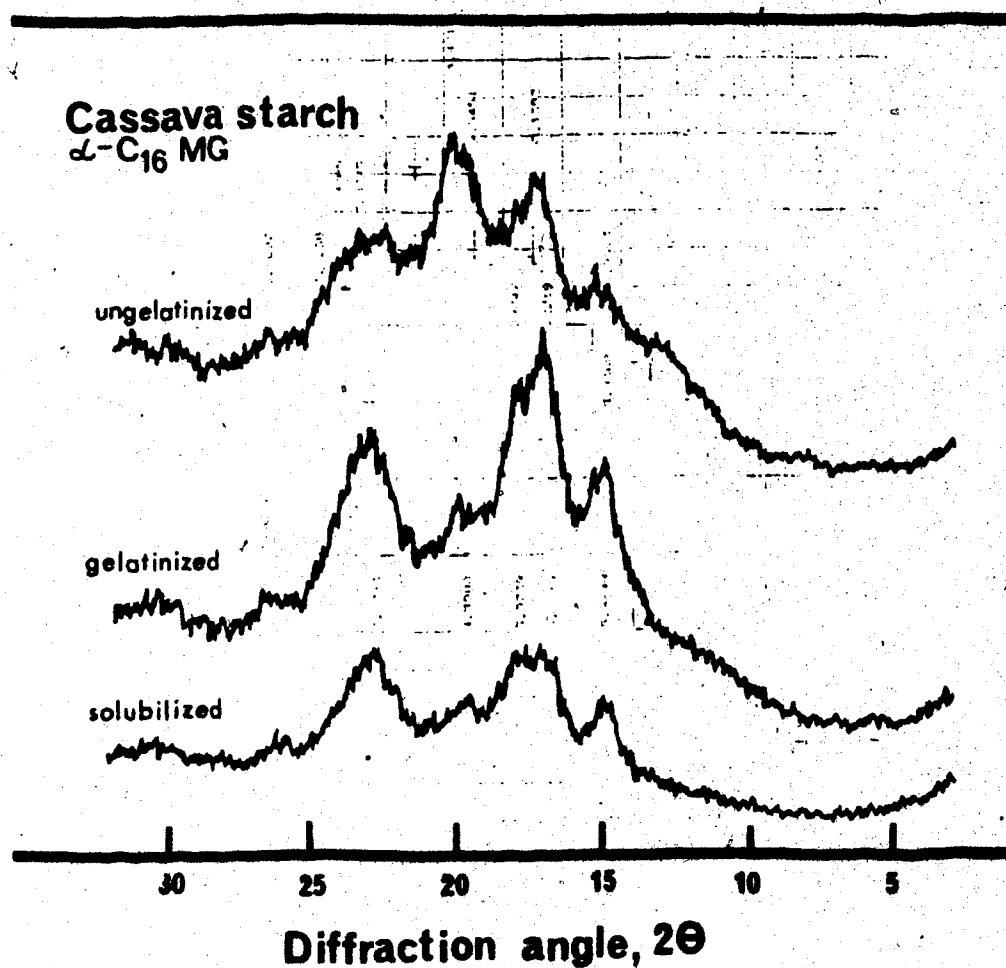
| Lintnerized Wheat cv. Fielder Starch* |                          |                          |                          |                          |                          |                          |
|---------------------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| % MG***<br>Added                      | Ungelatinized            |                          | Gelatinized              |                          | Solubilized**            |                          |
|                                       | $\beta$ -C <sub>16</sub> | $\beta$ -C <sub>18</sub> | $\beta$ -C <sub>16</sub> | $\beta$ -C <sub>18</sub> | $\beta$ -C <sub>16</sub> | $\beta$ -C <sub>18</sub> |
| 0.1                                   | 7.20 $\pm$ 0.58          | 5.83 $\pm$ 0.52          | 14.93 $\pm$ 0.66         | 8.09 $\pm$ 0.78          | 13.65 $\pm$ 0.66         | 12.34 $\pm$ 0.32         |
| 0.2                                   | 14.24 $\pm$ 2.18         | 10.29 $\pm$ 0.41         | 27.63 $\pm$ 0.88         | 18.70 $\pm$ 0.42         | 26.45 $\pm$ 0.50         | 22.72 $\pm$ 0.26         |
| 0.3                                   | 19.89 $\pm$ 0.46         | 13.38 $\pm$ 0.72         | 34.00 $\pm$ 1.41         | 25.43                    | 38.25 $\pm$ 0.01         | 30.98 $\pm$ 1.39         |
| 0.4                                   | 22.63 $\pm$ 0.68         | 15.43 $\pm$ 0.74         | 36.37 $\pm$ 1.23         | 28.73 $\pm$ 2.33         | 50.24 $\pm$ 0.05         | 35.79 $\pm$ 1.11         |
| 0.5                                   | 24.14 $\pm$ 0.48         | 17.14 $\pm$ 1.92         | 37.75 $\pm$ 0.35         | 34.18 $\pm$ 0.56         | 57.81 $\pm$ 0.07         | 42.56 $\pm$ 0.78         |
| 0.8                                   | 25.03 $\pm$ 0.49         | 19.87 $\pm$ 2.32         | 41.92 $\pm$ 1.29         | 38.23 $\pm$ 0.36         | 71.86 $\pm$ 0.42         | 48.10 $\pm$ 1.28         |
| 1.0                                   | 25.37 $\pm$ 0.49         | 20.23 $\pm$ 0.94         | 42.50 $\pm$ 0.71         | 38.91 $\pm$ 1.76         | 71.50 $\pm$ 0.25         | 51.30 $\pm$ 1.70         |

\*Starch lintnerization was done by treatment of dry starch with 7.5% HCl at 40°C for 72 hrs.

\*\*Starch solubilization was done by dispersing the ungelatinized starch in aqueous 1 N KOH at 4°C for 30 min.

\*\*\*MG, Monoglyceride.

## APPENDIX 17.



X-Ray Diffraction Patterns of Cassava Starch Clathrates With  
 $\alpha$ -C<sub>16</sub> Monoglyceride

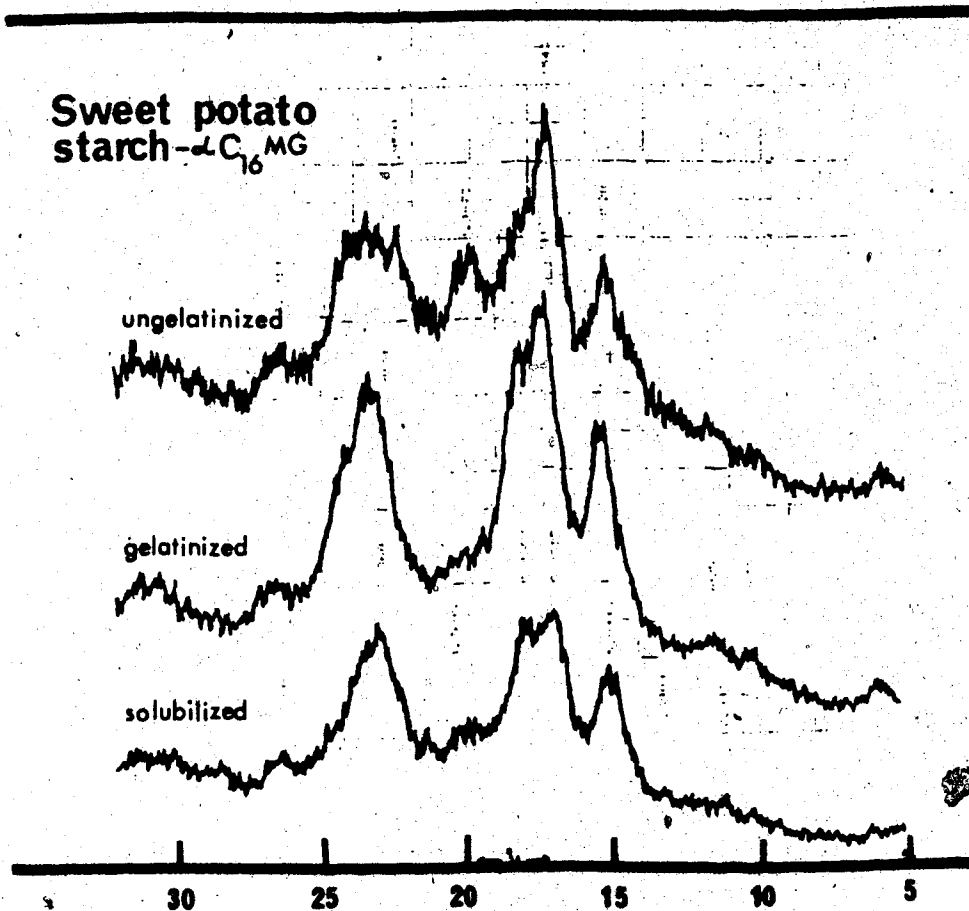
## APPENDIX 18.

X-Ray Diffraction Patterns of Cassava Starch Clathrates  
With  $\alpha$ -C<sub>16</sub> Monoglyceride

| Ungelatinized |        |                  | Cassava Starch<br>Gelatinized |        |                  | Solubilized |        |                  |
|---------------|--------|------------------|-------------------------------|--------|------------------|-------------|--------|------------------|
| $2\theta$     | d, Å   | Intensity<br>cps | $2\theta$                     | d, Å   | Intensity<br>cps | $2\theta$   | d, Å   | Intensity<br>cps |
| 14.885        | 5.9513 | 281              | 15.055                        | 5.8848 | 193              | 14.922      | 5.9368 | 390              |
| 17.254        | 5.1392 | 422              | 16.948                        | 5.2315 | 245              | 16.982      | 5.2210 | 543              |
| 19.531        | 4.5451 | 434              | 17.478                        | 5.0739 | 248              | 17.830      | 4.9747 | 490              |
| 20.081        | 4.4218 | 483              | 19.769                        | 4.4908 | 203              | 19.955      | 4.4492 | 351              |
| 22.432        | 3.9634 | 360              | 22.955                        | 3.8743 | 262              | 22.871      | 3.8883 | 443              |
| 23.196        | 3.8345 | 356              |                               |        |                  | 26.524      | 3.3605 | 237              |
| 22.680        | 3.9205 | 309              |                               |        |                  | 29.766      | 3.3605 | 237              |
| 24.570        | 3.6230 | 271              |                               |        |                  | 29.766      | 3.0014 | 237              |
| 26.450        | 3.3697 | 243              |                               |        |                  |             |        |                  |
| 29.953        | 2.9831 | 226              |                               |        |                  |             |        |                  |
| 30.928        | 2.8913 | 226              |                               |        |                  |             |        |                  |



## APPENDIX 19.



**Diffraction angle,  $2\theta$**

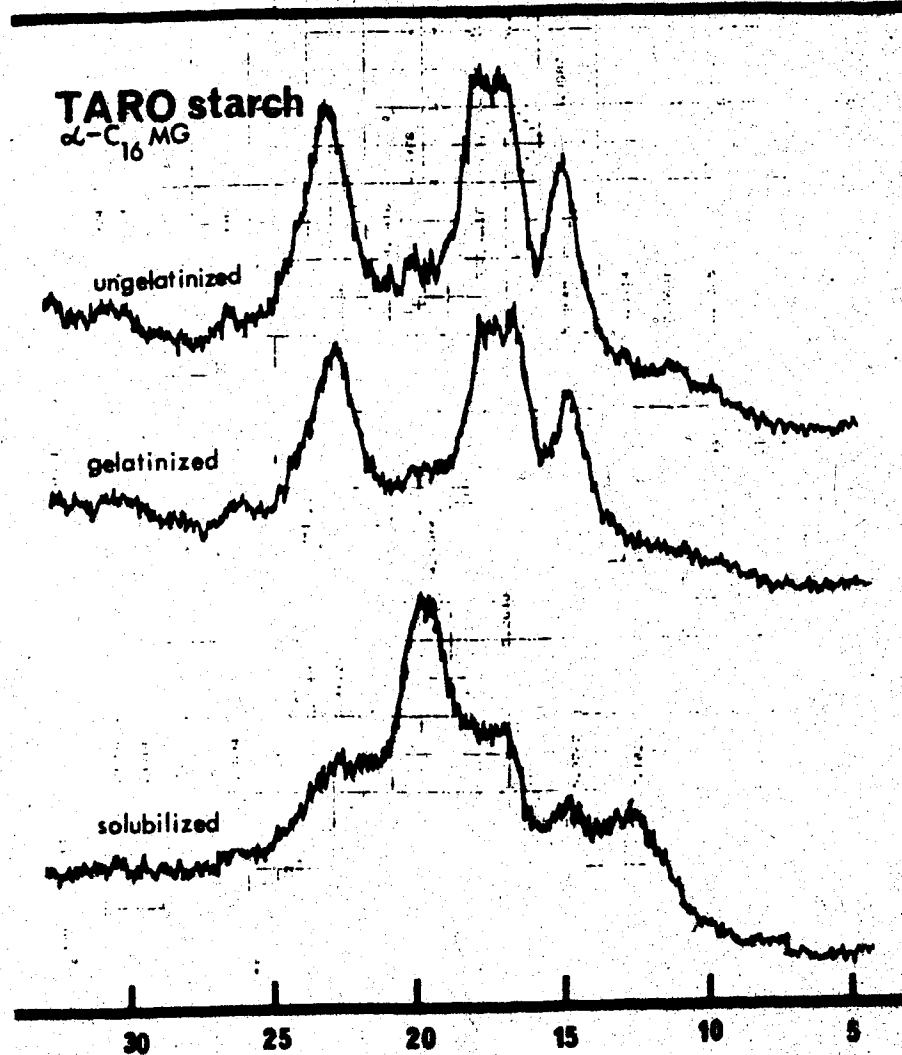
X-Ray Diffraction Patterns of Sweet Potato Clathrates with  $\alpha$ -C<sub>16</sub> Monoglyceride.

## APPENDIX 20.

X-Ray Diffraction Patterns of Sweet Potato Starch Clathrates  
With  $\alpha$ -C<sub>16</sub> Monoglyceride

| Ungelatinized |        |                  | Sweet Potato Starch<br>Gelatinized |        |                  | Solubilized |        |                  |
|---------------|--------|------------------|------------------------------------|--------|------------------|-------------|--------|------------------|
| $2\theta$     | d, Å   | Intensity<br>cps | $2\theta$                          | d, Å   | Intensity<br>cps | $2\theta$   | d, Å   | Intensity<br>cps |
| 11.669        | 7.5837 | 141              | 10.170                             | 8.6978 | 146              | 11.194      | 7.9039 | 108              |
| 15.137        | 5.8528 | 346              | 11.196                             | 7.9025 | 170              | 13.232      | 6.6909 | 126              |
| 17.203        | 5.1543 | 526              | 14.971                             | 5.9173 | 432              | 15.159      | 5.8445 | 248              |
| 19.874        | 4.4673 | 361              | 17.002                             | 5.2148 | 591              | 17.045      | 5.2018 | 341              |
| 22.454        | 3.9591 | 380              | 17.767                             | 4.9919 | 526              | 17.971      | 4.9358 | 326              |
| 23.272        | 3.8222 | 385              | 22.789                             | 3.9020 | 407              | 20.324      | 4.3695 | 189              |
| 26.281        | 3.3910 | 244              | 23.594                             | 3.7707 | 391              | 21.391      | 4.1538 | 183              |
| 30.199        | 2.9594 | 230              | 26.721                             | 3.3361 | 230              | 23.040      | 3.8601 | 326              |
| 31.085        | 2.8770 | 232              | 30.239                             | 2.9555 | 251              | 26.315      | 3.3866 | 176              |
|               |        |                  |                                    |        |                  | 28.458      | 3.1363 | 157              |
|               |        |                  |                                    |        |                  | 30.083      | 2.9705 | 168              |

## APPENDIX 21.

**Diffraction angle,  $2\theta$** 

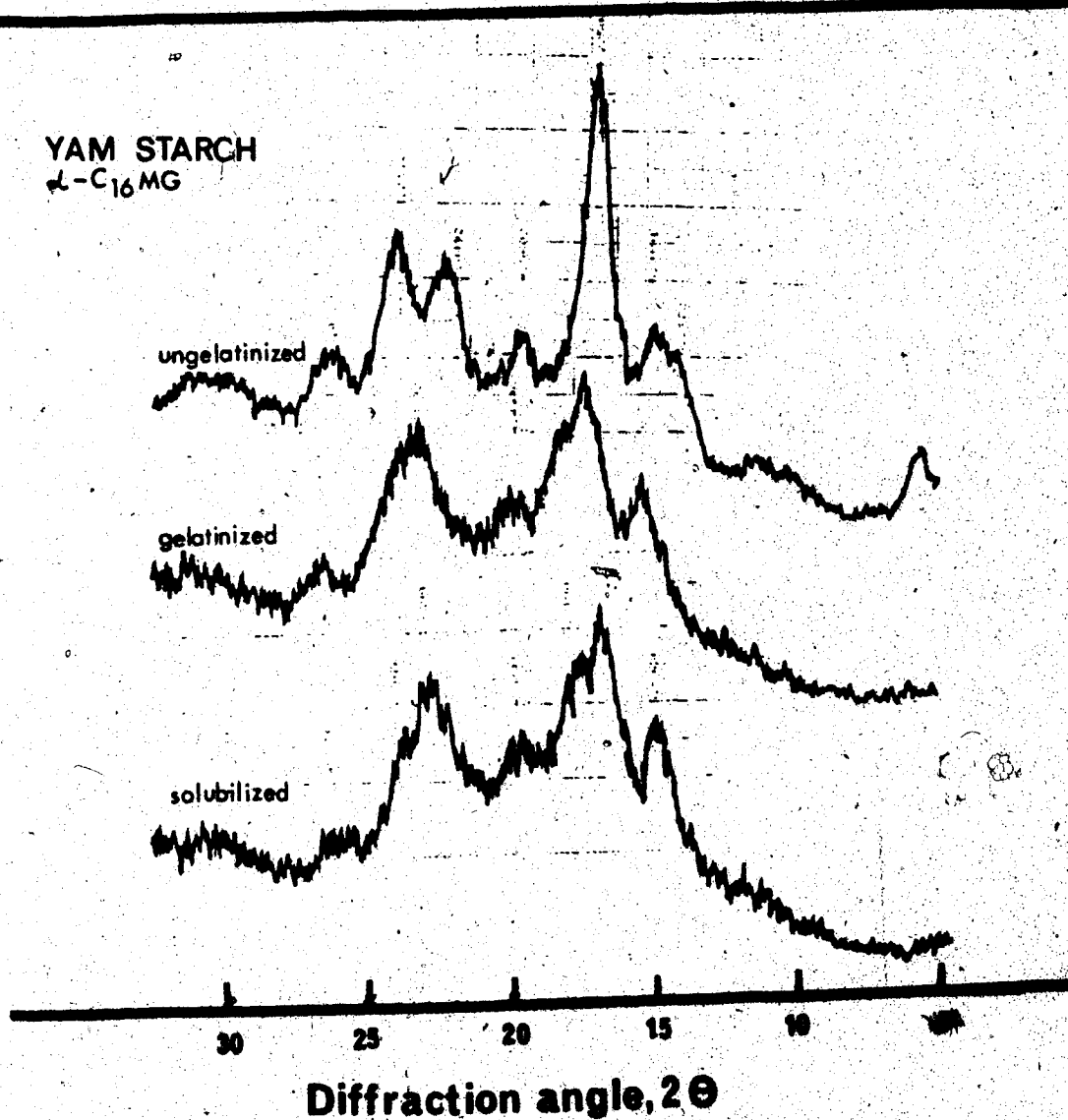
X-Ray Diffraction Patterns of Taro Starch Clathrates with  
 $\alpha$ -C<sub>16</sub> Monoglyceride.

## APPENDIX 22.

X-Ray Diffraction Patterns of Taro Starch Clathrates  
With  $\alpha$ -C<sub>16</sub> Monoglyceride

| Ungelatinized |        |                  | Taro Starch<br>Gelatinized |        |                  | Solubilized |        |                  |
|---------------|--------|------------------|----------------------------|--------|------------------|-------------|--------|------------------|
| $2\theta$     | d, Å   | Intensity<br>cps | $2\theta$                  | d, Å   | Intensity<br>cps | $2\theta$   | d, Å   | Intensity<br>cps |
| 9.895         | 8.9385 | 118              | 9.935                      | 8.9030 | 96               | 12.463      | 7.1022 | 258              |
| 11.391        | 7.7681 | 154              | 15.106                     | 5.8649 | 308              | 14.822      | 5.9767 | 261              |
| 12.940        | 6.8414 | 167              | 16.974                     | 5.2234 | 389              | 17.038      | 5.2040 | 378              |
| 15.045        | 5.8887 | 408              | 17.927                     | 4.9478 | 338              | 19.592      | 4.5309 | 537              |
| 16.958        | 5.2282 | 505              | 22.989                     | 3.8686 | 352              | 22.729      | 3.9122 | 331              |
| 17.932        | 4.9466 | 514              | 30.961                     | 2.8882 | 182              | 23.554      | 3.7769 | 296              |
| 20.191        | 4.3978 | 283              |                            |        |                  | 26.294      | 3.3894 | 222              |
| 20.943        | 4.2416 | 255              |                            |        |                  | 29.465      | 3.0314 | 198              |
| 23.058        | 3.8571 | 472              |                            |        |                  | 30.449      | 2.9356 | 207              |
| 26.522        | 3.3607 | 223              |                            |        |                  |             |        |                  |
| 30.173        | 2.9619 | 239              |                            |        |                  |             |        |                  |
| 30.915        | 2.8924 | 234              |                            |        |                  |             |        |                  |

## APPENDIX 23.



X-Ray Diffraction Patterns of Yam Starch Clathrates with  
 $\alpha$ -C<sub>16</sub> Monoglyceride.

## APPENDIX 24.

X-Ray Diffraction Patterns of Yam Starch Clathrates  
With  $\alpha$ -C<sub>16</sub> Monoglyceride

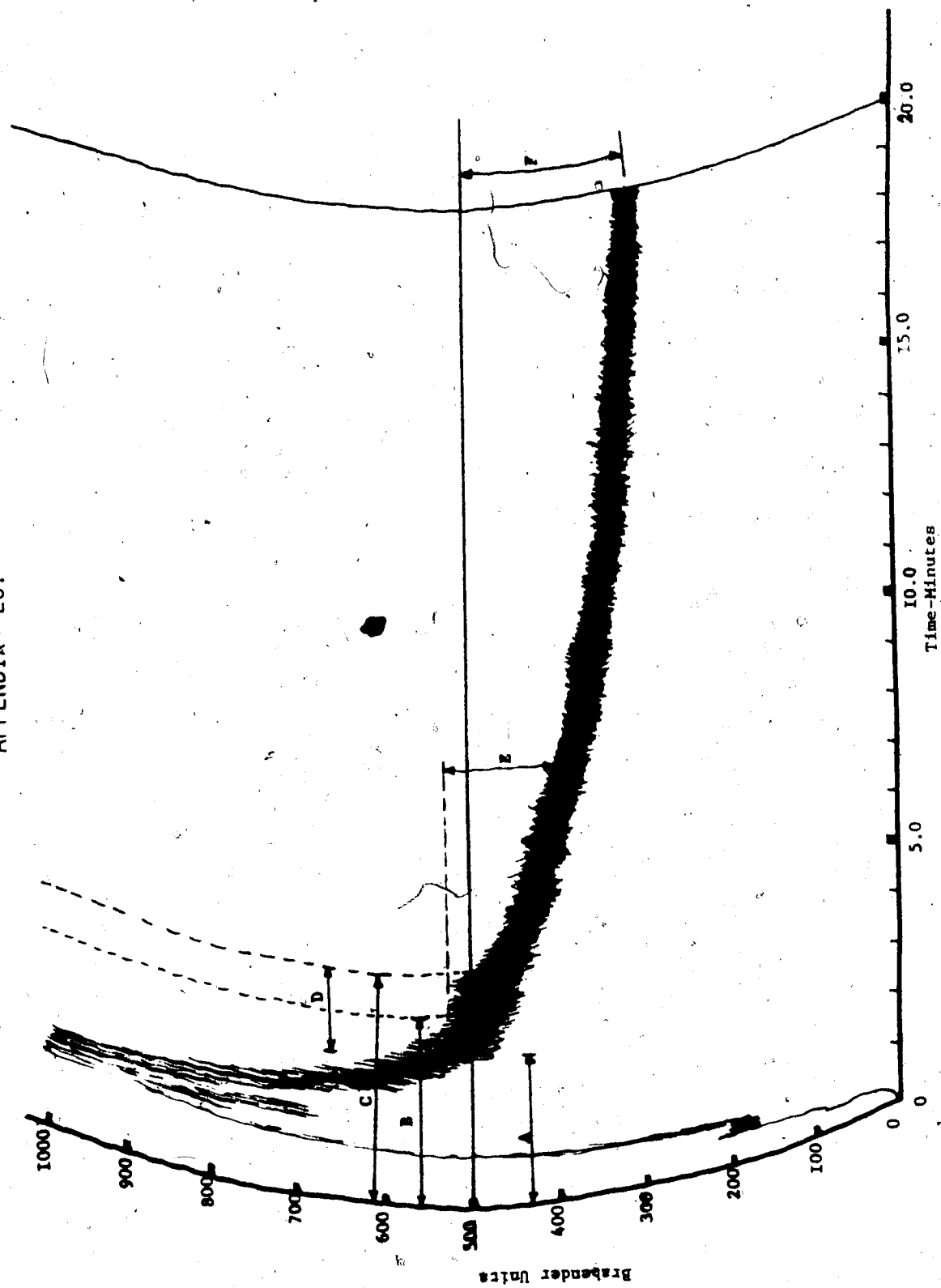
| Ungelatinized |         |                  | Yam Starch<br>Gelatinized |        |                  | Solubilized |        |                  |
|---------------|---------|------------------|---------------------------|--------|------------------|-------------|--------|------------------|
| $2\theta$     | d, Å    | Intensity<br>cps | $2\theta$                 | d, Å   | Intensity<br>cps | $2\theta$   | d, Å   | Intensity<br>cps |
| 5.482         | 16.1216 | 159              | 9.995                     | 8.8491 | 109              | 15.285      | 5.7964 | 352              |
| 14.245        | 6.2172  | 273              | 11.412                    | 7.7538 | 146              | 17.192      | 5.1576 | 472              |
| 14.967        | 5.9191  | 322              | 15.202                    | 5.8279 | 400              | 16.988      | 5.2190 | 394              |
| 16.969        | 5.2250  | 639              | 17.097                    | 5.1861 | 530              | 19.666      | 4.5141 | 321              |
| 19.465        | 4.5602  | 309              | 19.615                    | 4.5257 | 324              | 23.168      | 3.8390 | 395              |
| 21.950        | 4.0492  | 362              | 22.940                    | 3.8767 | 455              | 24.001      | 3.7076 | 321              |
| 23.819        | 3.7356  | 442              | 26.383                    | 3.3781 | 220              | 26.377      | 3.3788 | 210              |
| 26.075        | 3.4172  | 280              |                           |        |                  | 30.525      | 2.9285 | 213              |
|               |         |                  |                           |        |                  | 31.246      | 2.8626 | 225              |

# APPENDIX 25.



Sweet Potato Starch (85%)/Vital Gluten (15%) Composite Flour Parinogram

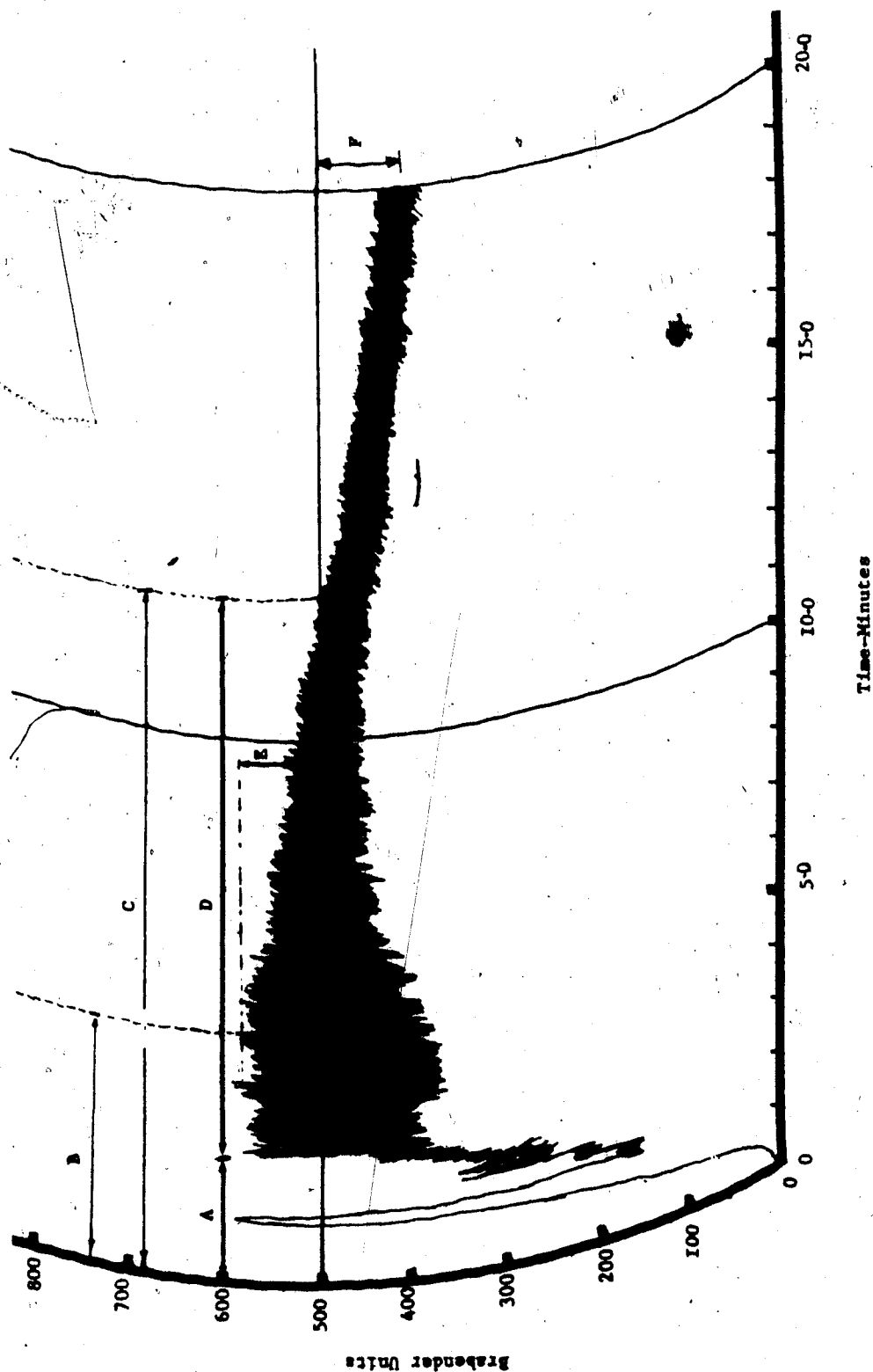
## APPENDIX 26.



Taro Starch (85%)/Vital Gluten (15%) Composite Flour Farinogram

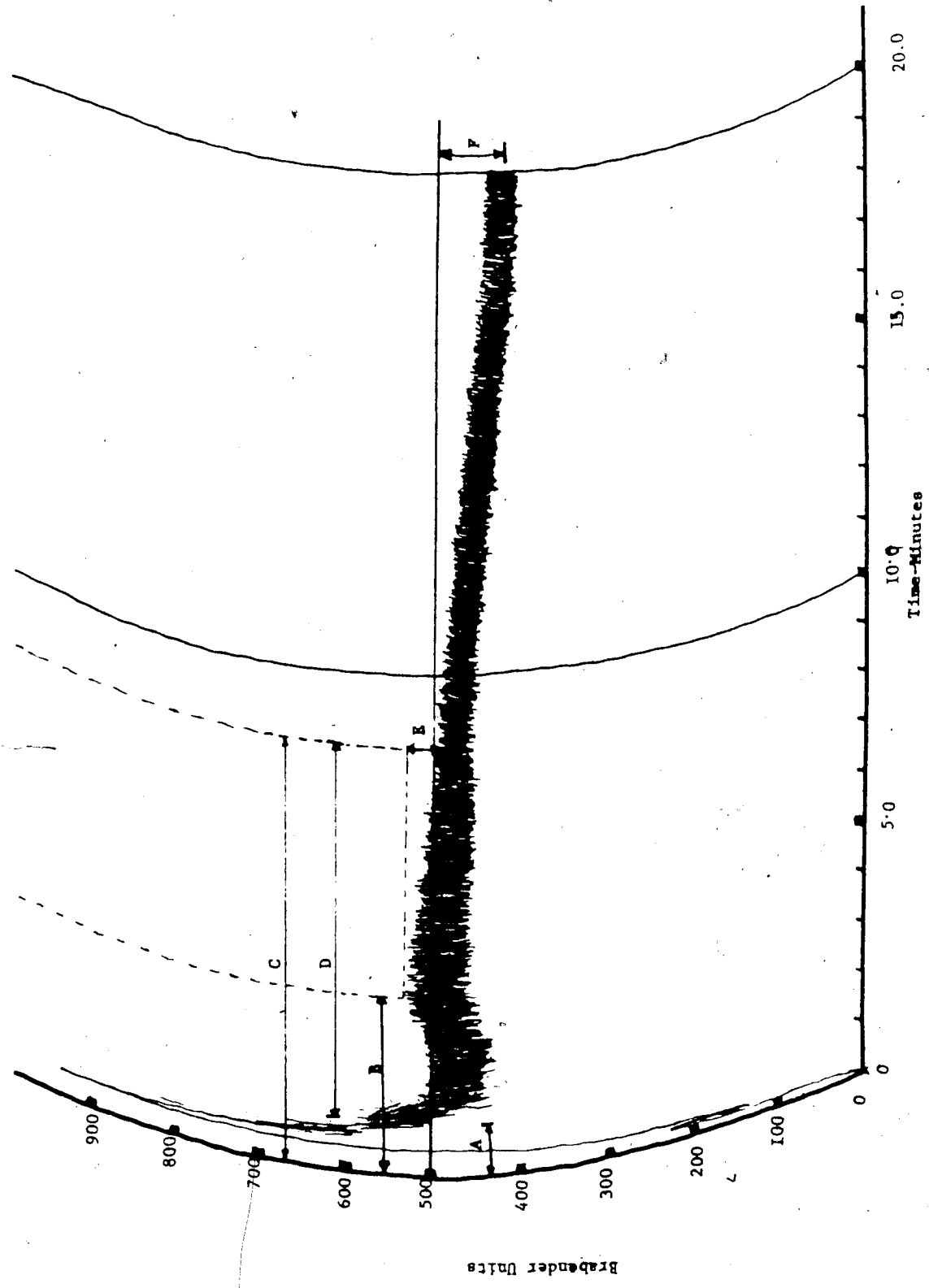


APPENDIX 27.

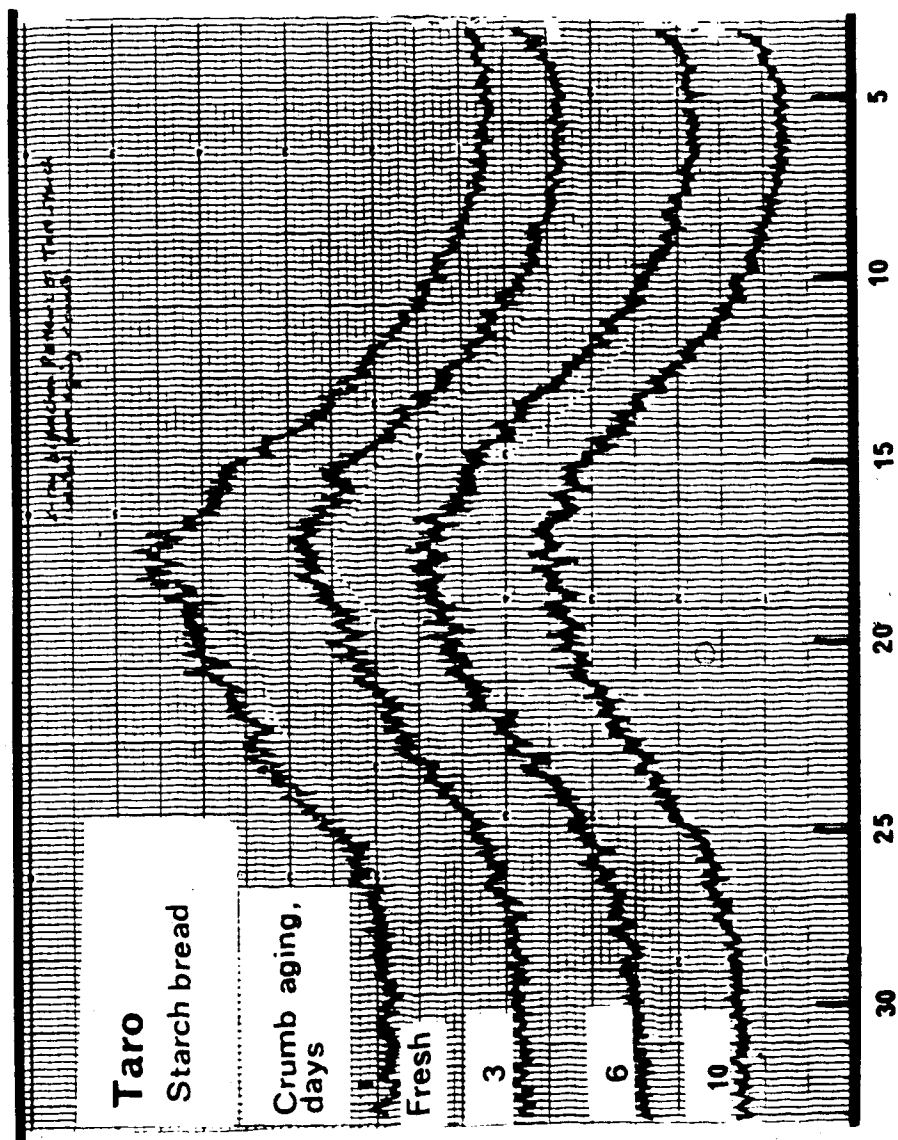


Yam Starch (85%)/Vital Gluten (15%) Composite Flour Farinogram

APPENDIX 28.



Wheat, cv. Fielder, Starch(85%)/Vital Gluten(15%) Composite Flour Farinogram



### Diffraction angle, $2\theta$

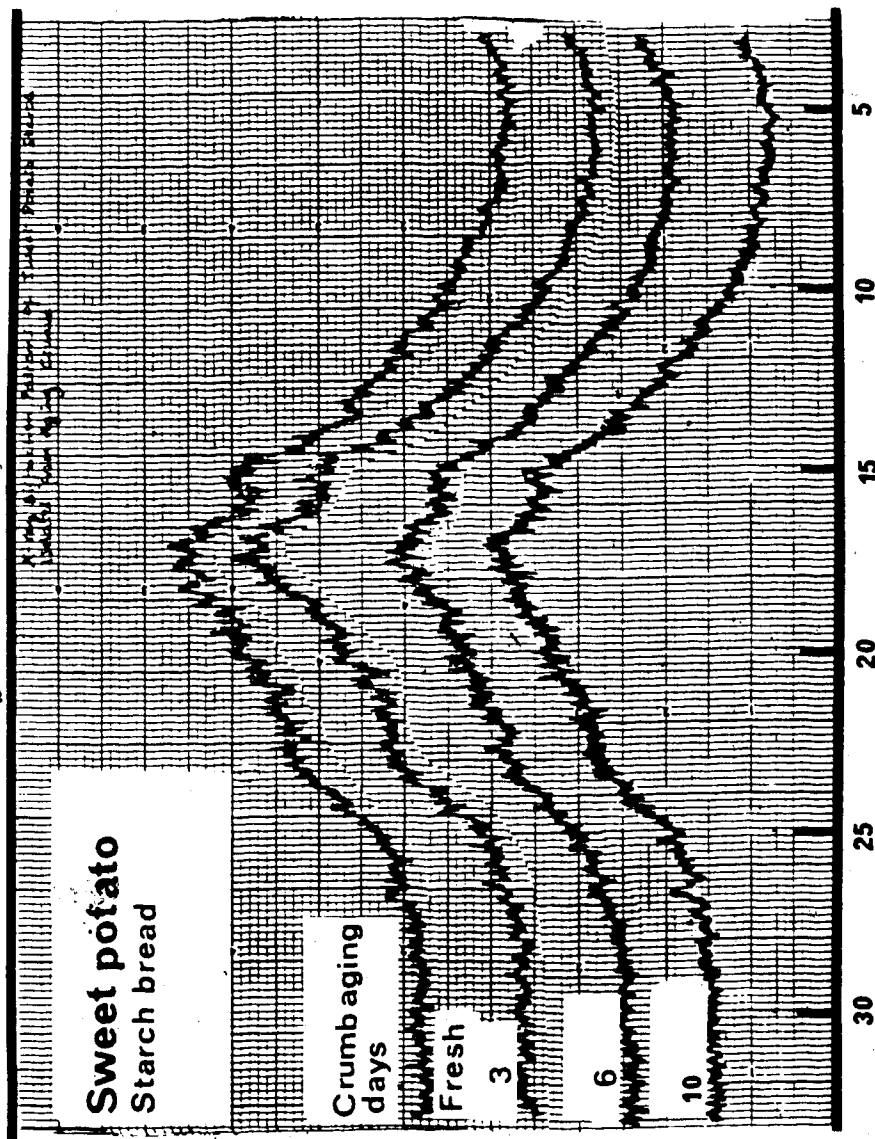
X-Ray Diffraction Patterns of Starch Isolated From Aging Taro Bread Crumbs.

APPENDIX 30.

X-Ray Diffraction Patterns of Starch Isolated From Aging Taro Bread Crumbs\*

| Bread Storage, Days |                 |     |           |                 |     |           |                 |           |           |                 |     |
|---------------------|-----------------|-----|-----------|-----------------|-----|-----------|-----------------|-----------|-----------|-----------------|-----|
| 1                   |                 |     |           | 3               |     |           |                 | 6         |           |                 |     |
|                     |                 |     |           |                 |     |           |                 |           |           |                 |     |
| Intensity           |                 |     |           | Intensity       |     |           |                 | Intensity |           |                 |     |
| $2\theta$           | $d, \text{\AA}$ | cps | $2\theta$ | $d, \text{\AA}$ | cps | $2\theta$ | $d, \text{\AA}$ | cps       | $2\theta$ | $d, \text{\AA}$ | cps |
| 14.569              | 6.0800          | 629 | 15.193    | 5.8313          | 688 | 14.724    | 6.0163          | 647       | 13.985    | 6.3324          | 625 |
| 15.244              | 5.8120          | 721 | 16.979    | 5.2220          | 730 | 16.365    | 5.4163          | 739       | 15.849    | 5.5916          | 648 |
| 17.923              | 4.9489          | 838 | 19.951    | 4.4502          | 644 | 18.361    | 4.8319          | 749       | 16.725    | 5.3000          | 702 |
| 17.999              | 4.9283          | 903 | 22.917    | 3.8806          | 517 | 19.783    | 4.4876          | 709       | 18.861    | 4.7050          | 682 |
| 19.965              | 4.4472          | 784 | 26.524    | 3.3604          | 324 | 21.208    | 4.1891          | 643       | 20.551    | 4.3216          | 620 |
| 20.709              | 4.2890          | 777 |           |                 |     | 25.835    | 3.4485          | 400       | 24.277    | 3.6661          | 406 |
| 21.690              | 4.0973          | 696 |           |                 |     | 27.811    | 3.2078          | 347       | 26.588    | 3.3526          | 324 |
| 22.588              | 3.9363          | 657 |           |                 |     | 28.766    | 3.1035          | 307       |           |                 |     |

\*Taro starch composite flours had 15% vital gluten.



### Diffraction angle, $2\theta$

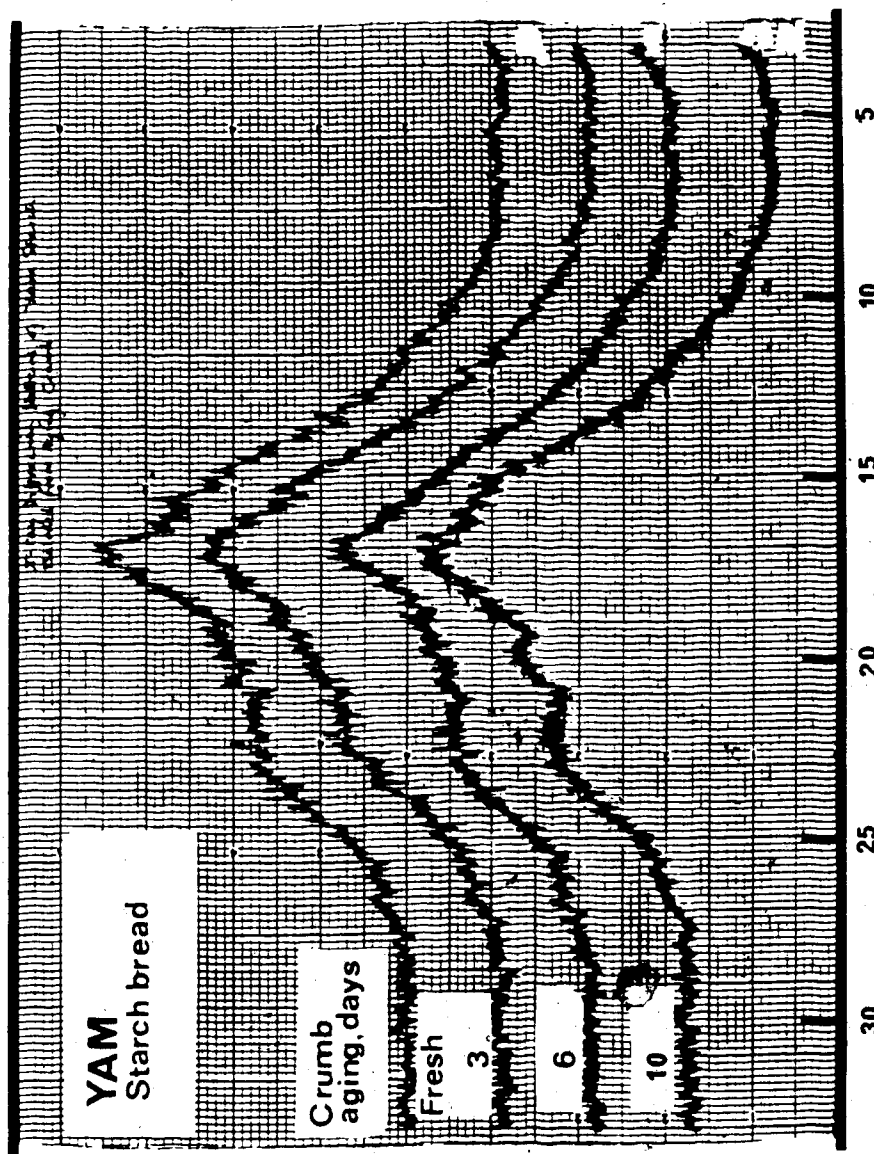
X-Ray Diffraction Patterns of Starch Isolated From Aging Sweet Potato Bread Crumbs.

# APPENDIX 32.

## X-Ray Diffraction Patterns of Starch Isolated From Aging Sweet Potato Bread Crumbs\*

| Bread Storage, Days |  |  |  |   |  |  |  |   |  |  |  |
|---------------------|--|--|--|---|--|--|--|---|--|--|--|
| 1                   |  |  |  | 3 |  |  |  | 6 |  |  |  |
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## APPENDIX 33.

**Diffraction angle,  $2\theta$** 

X-Ray Diffraction Patterns of Starch Isolated From Aging Yam Bread Crumbs.

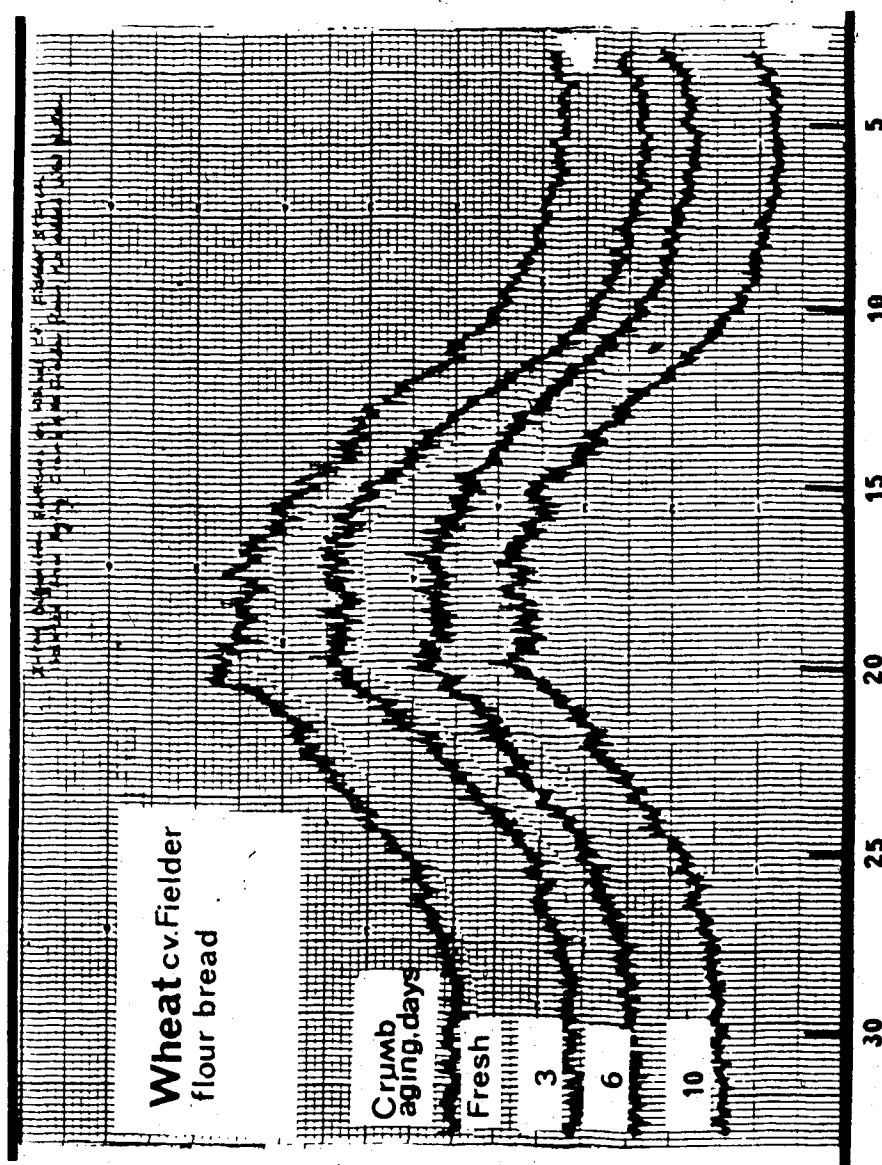
# APPENDIX 34.

## X-Ray Diffraction Patterns of Starch Isolated From Aging Yam Bread Crumbs\*

| Bread Storage, Days |        |      |           |           |      |        |           |           |        |        |           |
|---------------------|--------|------|-----------|-----------|------|--------|-----------|-----------|--------|--------|-----------|
| 1                   |        |      |           | 3         |      |        |           | 6         |        |        |           |
|                     |        |      |           |           |      |        |           |           |        |        |           |
| Intensity           |        |      |           | Intensity |      |        |           | Intensity |        |        |           |
| $2\theta$           | d, Å   | cps  | $2\theta$ | $2\theta$ | d, Å | cps    | $2\theta$ | $2\theta$ | d, Å   | cps    | $2\theta$ |
| 13.264              | 6.6750 | 538  | 15.352    | 5.7716    | 865  | 16.822 | 5.2702    | 919       | 11.519 | 7.6821 | 343       |
| 14.811              | 5.9811 | 793  | 16.735    | 5.2975    | 986  | 19.674 | 4.5124    | 730       | 15.067 | 5.8799 | 795       |
| 15.642              | 5.6651 | 938  | 17.942    | 4.9436    | 953  | 22.856 | 3.8907    | 656       | 16.565 | 5.3514 | 874       |
| 16.957              | 5.2287 | 1081 | 18.777    | 4.7258    | 880  |        |           |           | 19.799 | 4.4841 | 733       |
| 19.221              | 4.6175 | 800  | 22.319    | 3.9831    | 729  |        |           |           | 29.789 | 2.9991 | 361       |
| 20.316              | 4.3711 | 781  | 23.385    | 3.8039    | 647  |        |           |           |        |        |           |
|                     |        |      | 26.586    | 3.3527    | 443  |        |           |           |        |        |           |
|                     |        |      | 32.213    | 2.7788    | 380  |        |           |           |        |        |           |

\*Yam starch composite flours had 15% vital gluten.





### Diffraction angle, $2\theta$

X-Ray Diffraction Patterns of Starch Isolated From Aging Bread  
Crumbs made Wheat Flour, cv. Fielder.

**X-Ray Diffraction Patterns of Starch Isolated From Aging Bread Crumbs  
Made From Wheat Flour cv. Fielder**

[illegible]

## Appendix 37

## Water Binding Capacities of Some Wheat and Tropical Root Starches

| Type of Starch | Water Binding Capacity g H <sub>2</sub> O/g Starch DM |
|----------------|---|
| Yam            | 0.70  |
| Taro           | 0.65  |
| Sweet Potato   | 0.55  |
| Arrowroot      | 0.50  |
| Cassava        | 0.50  |
| Wheat          |   |
| cv. Neepawa    | 0.45  |
| cv. Fielder    | 0.35  |

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#### PUBLICATONS

1. Keya, E.L. and Hadziyev, D. Physico-chemical properties of some tropical root starches. Presented at the 27th Annual conference of Canadian Institute of Food Science and Technologists - Vancouver, B.C. 1984.
2. Keya, E.L. and Hadziyev, D. The use of tropical root starches in bread making. Presented at the 27th Annual Conference of Canadian Institute of Food Science and Technologists. Vancouver, B.C. 1984.  
(The two papers are currently pending publication).