University of Alberta

Development of a New Slump Test Method to Measure the Rheological Properties of Cement Paste

by

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Dedication

To my lovely mother, father, brothers and my girlfriend, who were behind me and encouraged me to stay strong during my research.

Abstract

A new and cost effective technique is developed to measure the rheological properties of cement paste and is validated through experiment and analytical analysis with the use of flow table. Instead of conventional methods, such as free flow test and modified mini slump test without drops, the technique of slump test (ASTM C1437) with impact force was utilized. To date there has been no study to measure viscosity from mini slump test with impact force. The yield stress and the plastic viscosity of cement pastes were determined using AR-G2 rheometer. Rheology tests were conducted on more than twenty cement paste specimens with different proportions of cellulose nano fibrillated and sucrose producing different yield stress and viscosity. The correlation between the rheological parameters and mini slump tests were investigated from the experimental graphs of shear stress and viscosity versus shear rate. Plots of viscosity versus drop data were acquired through both experiment and analytical calculations. Based on our experiment, the formulas have been found, indicating the effectiveness of mini slump test instead of rheological analysis for the specific cement paste case.

Keywords: Cement paste, workability, rheology, yield stress, viscosity, slump test

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Symbols – Latin characters

Symbols	Description	Unit
A	Area	(m^2)
D	Final Spread	(m)
g	Gravitational constant (9.81)	(m/s^2)
h	Height	(m)
v	Volume	(m^3)
r	radius	(m)
s	Slump	(m)
n	drops	#
ρ	Density	(kg/m^3)
τ	Shear stress	(Pa)
τ_0	Yield stress	(Pa)
$\dot{\gamma}$	Shear rate	(s^{-1})
μ	Plastic viscosity	(Pa.s)
ν	Velocity	(m/s)
π	Pi (3.1415)	
t	Time	(second)

Abbreviations and acronyms

Symbol	Description
HRWRA	High Range Water Reducing Admixture
CNF	Cellulose Nano Fiber
SEM	Scanning Electron Microscopic
ASTM	American Standard Test Method

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Chapter 1. Introduction

1-1. Background

Concrete is an important structural material, which is prepared with cement, water, sand and gravel (aggregates) and additional additives of a reasonable cost. The most important use of cement is to produce mortar and concrete. The significant role of cement in mortar and concrete is creating a bond between aggregates (cement paste), which turns the mixture to a strong form of building material. In simplified terms, concrete could be considered as aggregates glued together by hardened cement paste.

Due to high consumer demand for the concrete and cement production, it is important to create workable concrete with the high quality. The term workability can be defined as the ability of a fresh concrete/cement paste to be transported and placed, without reducing the concrete's quality.

The workability of cement paste is an important factor and is significantly related to the water/cement ratio, chemical admixtures and quality of concrete. In this research, the workability of fresh cement paste is measured with the mini slump test, where a sample is placed on a plate and it is raised and dropped 25 times during 15 seconds.

Yield stress and viscosity are two rheological properties of cement paste. The rheological properties are important because different properties of cement paste; such as workability, durability, and strength depend on its rheological properties.

In this research, yield stress and plastic viscosity of cement pastes with CNF and sucrose were determined using AR-G2 rheometer. The term yield stress is described as the stress necessary to initiate flow whereas viscosity is described as the resistance to flow once the fluid is in motion.

1-1-1. Recent development

According to the recent developments, most of the researchers agree on using at least two different empirical tests to create relationships between the rheological parameters and workability of fresh cement paste.

According to Roussel (2005) the result of a slump flow test is representative of concrete rheological behavior. In his research, he used the ASTM Abrams cone to measure the slump of fresh concrete and used viscometer test to measure the yield stress of fresh concrete [1]. In this test, Roussel has shown that for limited spreads, the yield Stress should be written as:

$$\tau_{\circ} = \frac{225\rho g v^2}{128\pi^2 D_f^{5}} - \lambda \frac{R^2}{v} \qquad [1]$$

Where ρ is the density, $\tau_{\circ}$ is the yield stress, g is the gravity, v is the volume, D_f is the final diameter, the λ coefficient is a function of both the unknown tested fluid surface tension and contact angle, R is and v is the viscosity of the cement paste.

According to Roussel (2006) the correlation between yield stress and slump was investigated by comparison between numerical simulations and concrete rheometers result [2]. In his research, the ASTM Abrams cone was used to measure the slump of fresh concrete and used BTRHEOM rheometer to measure the yield stress of fresh concrete. In this test, Roussel has shown that yield stress can be expressed as a function of slump:

$$s=25.5-17.6\,\frac{\tau_\circ}{\rho} \qquad [2]$$

Where ρ is the density, $\tau_\circ$ is the yield stress and s is the slump of fresh concrete.

Wallevik (2006) has found the relationship between Bingham parameters and Slump. In his research, he used the ConTec BML viscometer to measure the yield stress, and slump cone was used to measure the slump of fresh concrete.

$$s=300-0.347\,\frac{(\tau_\circ-212)}{\rho} \qquad [3]$$

Where s is the slump, $\tau_\circ$ is the yield stress and ρ is the density of the sample.

Tregger (2008) identified the yield stress and viscosity of cement paste from mini-slump-flow test. In his research, the mini-slump test was performed similar to ASTM C1611, but using the mini-cone. He found the final spread and time to final spread following the same procedure from ASTM C1611. And a rheometer with a concentric cylinder configuration (0.8 mm gap) was used.

$$\tau_\circ = 2.75 \times 10^{-9} D_f^{-5.81} \qquad [4]$$

$$\mu = \tau_\circ(6.41\times10^{-3} T_f - 1.94\times10^{-3}) \qquad [5]$$

The results show a correlation between the final diameter (D_f) and yield stress ($\tau_\circ$) also between the time to final spread (T_f) and yield stress ($\tau_\circ$) and the viscosity (μ).

1-1-2. Scope of research

In this research, the AR-G2 rheometer has been used to measure the rheological parameters of cement paste. This instrument is known as one of the most powerful rheometer nowadays. Mostly the AR-G2 has been used to measure the rheological parameters of nano materials. On the other hand to measure the workability of the fresh cement paste, the mini slump test according to ASTM C1437 was performed. The experiments were conducted in a laboratory at constant temperature and were based on the determination of both flow behavior of cement paste and yield stress of concrete using a single rheological test. The rheology parameters and flow table results were compared to establish the correlation between the rheology parameters and final spread from the mini flump test. The results indicated the use of new method to measure the yield stress and viscosity of fresh cement paste without using the rheology data anymore. In the past few years, the cost of using the rheometers to measure the rheological parameters of cement paste has been found to be very expensive, so the new method will help to measure the approximate parameters.

1-2. Structure of the thesis

This thesis consists of 5 chapters in addition to the list of references.

In chapter 1, a general description of the entire thesis is provided as an introduction. In chapter 2, the aim and scope of this research project are clarified. A comprehensive literature review was performed on the rheological properties and workability of fresh cement paste. In chapter 3, material, the mixer design, specimen preparation and test procedures are explained. In chapter 4, the results of the tests performed are reported and discussed. In chapter 5, the conclusion of the thesis is stated. Finally, the list of references is provided at the end of this thesis.

In this paper, rheology tests are conducted on 21 cement pastes specimens with and without addition of Cellulose Nano Fiber (CNF) and sucrose. CNF is an organic material composed of nano sized cellulose fibrils with a high aspect ratio (length to width ratio). Based on the experimental results, the influence of CNF and sucrose was studied individually on cement paste. Afterwards, both admixtures were added to the cement paste to investigate their combined effect on the viscosity and workability of the cement paste.

In the next step, workability of various cement paste specimens where measured. In addition, the slump tests were recorded by digital camera for all samples during the test. The purpose of recording the slump test was to measure step by step change in diameter when each drop happens. For instance, one sample has been chosen as a control sample to measure each spread after each drop.

As a result, the yield stress and viscosity are calculated from the rheology tests and final diameter is measured from the slump test. In this study, correlation between rheological parameters and slump test is proposed.

Chapter 2. Literature Review

This chapter covers the literature review on both workability behavior and rheological behavior of concrete and cement paste. And also the correlation between the rheological parameters (yield stresses and viscosity) and workability of cement paste are emphasized.

2-1. Introduction

Researchers have studied the rheology of concrete and cement paste [3–7]. Rheology of concrete and cement paste is very important to improve the mixture design. Moreover, simple test such as the slump-flow test is used recently to assess workability of concrete and cement paste [8]. Yield stress and viscosity are two main rheological parameters, which have effects on workability of concrete or cement paste [8].

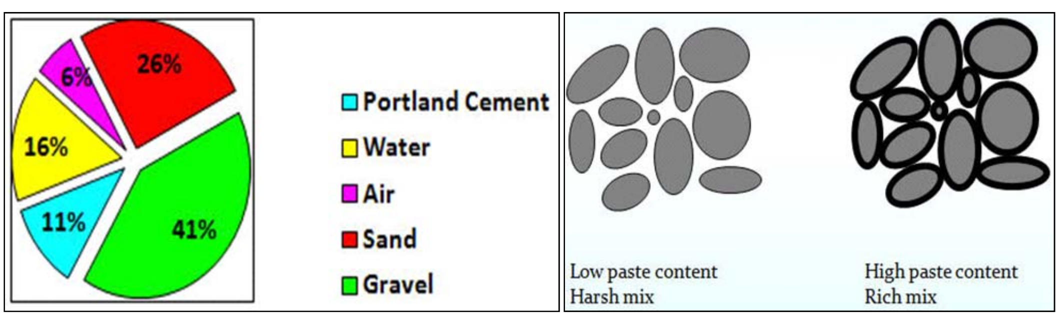
2-2. Concrete/Cement paste

Ordinary concrete is created using a mixture of aggregates and paste. The aggregate component generally contains sand and gravel. Sand and gravel are used to increase the ability to tolerate the pressure, which provide the better transpiration load force within the concrete [9].

"Cement paste is obviously the most complicated constituent of concrete, consisting of fine cement particles undergoing a chemical reaction with and within a water medium. A complete understanding of concrete workability, therefore, must include a thorough understanding of the cement paste that suspends the aggregate." (Bodenlos, Kainan David and Fowler, David W [7]).

2-2-1. Cement, Water and Aggregate

The paste component is generally comprised of cement, which is a dry powder (Portland cement), water and air. The function of paste is similar to glue, binding the aggregate together, through a chemical reaction between the cement and water which is called hydration reaction [10]. Tension between the aggregate in concrete can be tolerated by the cement paste. The structural component of concrete contains large amounts of aggregates, comprising about 60% to 75% of the resulting product, with the paste composing about 25% to 40% of the total volume of concrete. Air comprises about 4% to 8% of the volume of concrete (See Figure 2-1). If the paste and aggregates have good quality, the resulting concrete will be well made; however, the strength of the concrete depends on the bond between the paste and aggregates. In concrete, each part of the aggregate is coated with paste (See Figure 2-1).



a. b.

Figure 2-1. a. Components of concrete, b. Factors affecting paste content

Water is a polar molecule, which has the chemical formula H_2O, meaning that one molecule of water contains two hydrogen atoms and one oxygen atom [11]. The chemical formula of water has helped engineers to understand how water react with cement. As a result, knowing about the reaction between the molecules of water and cement makes it possible to use other material in this mixture to change its workability. The most commonly used cement is called Portland cement. Cement is a dry powder of very small particles. The size of particle is limited to lower size of 7.5 μm [12]. Cement is not a simple chemical compound; however, it is a mixture of five components such as Tricalcium Silicate (C3S), Dicalcium Silicate (C2S), Tricalcium Aluminate (C3A), Tetracalcium Aluminoferrite (C4AF) and Gypsum, therefore the formula of each of these minerals can be broken down into calcium, silicon, aluminum and iron oxides [13]. In this respect, the composition of cement shows that cement is not only a single particle, but is created by five fine particles. As a result, when using other substances within a mixture of cement and water, engineers should consider the reaction of the new materials with these five fine substances.

2-2-2. Workability

Cementitious mixtures made of Portland cement including mortars, grout and cement pastes are widely used in construction industry [12]. Their applications include bonding material in masonry construction, bonding different types of cladding and finishes to exterior and interior walls of buildings and making adhesion between reinforcement and surrounding concrete or soil in some construction activities [13][14]. The main disadvantages of ordinary Portland cementitious materials is its low tensile and shear strengths, which results in low adhesion properties [15]. To exhibit desirable structural performance, cementitious materials should have both sufficient workability and adhesive properties.

The American Concrete Institute defines workability as "that property of freshly mixed concrete or mortar which determines the ease and homogeneity with which it can be mixed, placed, consolidated, and finished." [16]. ASTM defines workability as "that property determining the effort required to manipulate a freshly mixed quantity of concrete with minimum loss of homogeneity."[17].

To achieve the acceptable hardened concrete and good mixture, concrete industry has realized that workability of concrete should be more monitor [18]. Several methods have been used to understand the workability of concrete or cement paste. One of the methods that has been used and it has not failed with the increase in knowledge of concrete rheology is slump test [18].

Workability of the cement paste improves with increasing water to cement (w/c) ratio. However, as water/cement ratio increases, the viscosity and adhesion properties, i.e. the tensile and shear strength, of the cement paste degrades [5], [19], [20]. An accurate test method to measure the rheological properties of the cement pastes including workability, viscosity and adhesion properties is rheology test conducted by a rheometer, in which shear stress is employed on fresh cement paste specimens at desired shear rates. An overview of the rheological features of fresh concrete and mortar is presented by Banfill (2006) [6].

2-2-3. Mini-slump flow test

The focus of workability measurement has changed many times over the years. The slump test was developed in the early 20th century; "concrete researchers were just beginning to recognize the importance of water content in predicting concrete strength" [18]. The slump test shows the relationship between the water content, and the hardened strength of concrete. Having less water in concrete can create much stronger concrete with high strength [9][18][21]. The slump test was quickly adopted in 1918 by Duff Abrams [9][18][22].

Slump test was first standardized into ASTM in 1922 [22][23]. "Even with the increase in knowledge of concrete rheology, the slump test remains the most commonly used test method for measuring concrete workability" (Koehler, Eric P and Fowler, David W (2004) [22]).

One of the research objectives is rheological study on superplasticizer/cement compatibility at high temperature. "When transportation of concrete by truck mixers from the mixing plant to the job site takes long time in the highly congested traffic of urbanized areas, especially with very high environmental temperatures even polycarboxylate superplasticizer may be ineffective in preventing slump-loss" it was conducted by F. Curto, S.Carrà, A. Lolli, F. Surico and R. Saccone (2008)[24]. The mini slump tests on the side of rheological measurements and concrete mixes showed the advantage of using superplasticizer even in high temperature to impart an excellent retention of workability in cementitious organizations [24].

The other research objective is to modeling and quantification of the effect of mineral additions on the rheology of fresh powder type self-compacting concrete which was conducted by Gert HEIRMAN [25]. In this research, the slump test was used to see the change in flow behavior by adding superplasticizer [25]. Also Pierre-Claude Aitcin (1994) had studied on superplasticizers in concrete and he said “superplasticizers are highly efficient dispersing admixtures when they are properly used” [26].

2-2-4. Vicat test

In 2008, M.S. Shetty investigated the theory and practice of concrete technology. He used the Vicat needle test for setting time. The Vicat instrument has been used for finding out initial setting time, final setting time and soundness of cement [13].

Ferraris, Chiara F, in 1999 investigated the measurement of the rheological properties of high performance concrete. He used the Vicat test to determine the setting time of concrete [27].

FrancŠvegla et al in 2008 studied the influence of aminosilanes on macroscopic properties of cement paste. They investigated the initial and final setting time, which was determined by Vicat apparatus equipped with appropriate steel needles [28].

2-3. Chemical Admixtures

Gert Heirman studied modeling and quantification of the effect of mineral additions on the rheology of fresh powder type self-compacting concrete (2011) [25]. He defined chemical admixtures as “materials added in small quantities related to the mass of cementitious binder during the mixing process of concrete in order to modify the fresh or hardened concrete properties” [25].

Accelerating admixture, retarding admixture and water-reducing admixture are three types of chemical admixtures for concrete [29][30]. According to ASTM C494/C494M, accelerating admixture is a chemical admixture to accelerate the setting time and accelerate the strength progress of concrete [29].

A lot of research has been conducted on different admixtures to improve rheological behavior of cement pastes and mortars D. M. Roy and K. Asaga (1979), they have found out that Ish-Shalom and Greenberg, Powers, Dimond and Tattersall, Jones, et al, Lapasin, et have been studied onthe rheological properties of fresh concrete or cement and they pointed out that the chemical composition changed the rheological properties of paste [31].

Chandra and Aavik (1987) experimentally studied the effect of proteins on mechanical properties of mortars and found that added protein improved air entrainment, adhesion and increased the setting time of the mortars [32].

Ghio et al. (1994) experimentally studied the effect of Poly-Saccharide Gums (PSG) and High Range Water Reducer (HRWR) on the rheological behavior of cement pastes, and observed that addition of PSG greatly increased viscosity of the cement pastes at low shear rates. But the water reducer was found to decrease the viscosity for low shear rates. However, the combined effect of adding both PSG and HRWR improved the viscosity for low and medium shear rates [4]. Brien, Joshua V and Mahboub, Kamyar C studied the influence of polymer type on adhesion performance of blended cement mortar (2013). They added 4 different types of polymer in mortar listed in bellow:

Table 2-1 different types of polymer in mortar

Polymer Type	Temperature
vinyl acetate / ethylene with	T_g=-7°C
acrylic	T_g=-10°C
styrenebutadienerubber	T_g=15°C
vinylacetate/ethylene	T_g=20°C

They found that polymers modified mortars displayed excellent adhesion characteristics [33].

2-3-1. Materials

Portland cement

Portland cement is a very fine powder with the particle size less than <50 μm [34]. The chemical composition of Portland cement was described in part 2.2.1, where it contains 5 materials. Additive materials in fresh concrete play an important role to change the rheological behavior of fresh concrete [29]. Viscosity and yield stress are two main rheological parameters for fresh concrete. Viscosity of Portland cement paste is a function of w/c ration [4][20]. Moreover, chemical additives and superplasticizers are the other factors to change these two main rheology parameters. On the other hand, yield stress is corresponding to viscosity, for instance, when viscosity gradually increase, yield stress gradually increase as well [6], [22], [27], [35], [36].

Physical properties of the cellulose

Neithalath, and Narayanan studied development and characterization of acoustically efficient cementitious materials (2004). They found that "good mechanical properties and high processability, as well as the lower production energy required are the two main criteria that establish the viability of using cellulose fibers in a cementitious matrix" as they said [37].

Influence of Cellulose Fibers on fresh concrete on the water requirement, workability and setting time of cellulose-cement composites has been studied by Neithalath, and Narayanan (2004) and Soroushian (1990). They observed that cellulose has Hydrophilic property, which means fibers absorb water from the mixture (reducing the workability) [37][38].

Also, they found with increasing the amount of fibers in mixture, the setting times increases as well. Neithalath, and Narayanan (2004) said "This could be because of the fact that some constituents of the fiber can act as set retarders". Water absorption is reported as the main physical properties of cellulose-cement composites [37]. The amount of fiber and the binder type are reported by Neithalath and Narayanan (2004 to be effective on the water absorption capacity of cellulose-cement composites [37]. Increases in amount of fiber in mixture, increases the water absorption, the reason is the tendency of the fibers to absorb water [37].

Physical Properties of the Sucrose

Changing the workability of fresh concrete is one of the physical properties of the sucrose. Sucrose is a Hydrophobic material, which does not absorb water in mixture and that is why it is known as water reducers [39], [30].Workability of the fresh concrete has been increased with adding some sucrose in fresh concrete/cement [39].

2-4. Rheology

2-4-1. Background

Concrete or cement paste rheology typically describes the workability terms of fresh concrete [6][22][40]. The concrete rheology is very important because it affects the workability of fresh concrete and the properties of the hardened concrete [41][31].

The rheological behavior of fresh concrete can be affected by the composition of mixtures such as w/c ratio, cement, mineral addition, etc [22][5].

Traditional experimental methods such as the V-funnel, slump flow, sieve stability test and the L-box have been used to measure the workability of fresh concrete [13][25][42].

A series of rheological tests were conducted on 80 concrete and mortar mixtures with and without HRWR by Ferraris (1998), and a model was established to correlate the yield stress and plastic viscosity of cement pastes to their material compositions. The plastic viscosity was found to be a function of the relative volumetric composition of the solids in the mixture independent of the amount of HRWR and mineral admixtures in the specimens. The yield stress was found to be dependent on the volumetric concentration, roughness and size of the present particles and their capacity to absorb HRWR [40].

Ferraris et al. (2001) used rheological tests on various mineral admixtures to find the suitable type and the optimum dosage of the admixture to improve concrete workability without increase in w/c ratio. Ultra-Fine Fly Ash (UFFA) was found to yield the best rheological improvement. The results were also validated through concrete slump tests [5].

Svegl et al. in 2008 experimentally studied the effect of aminosilanes on macroscopic properties of fresh cement pastes and mortars. Experimental investigations conducted by Svegl et al. (2008) showed that addition of aminosilanes can improve workability, hardening, compressive and flexural strengths of tested mortar samples [28].

Vieira et al. (2005) developed a new biodegradable dispersing agent for concrete and mortar with starch and cellulose to improve their workability and viscosity [43].

Leslie Struble and Guo-Kuang Sun have studied on viscosity of portland cement paste as a function of concentration (1995). They were able to found the viscosity values for dispersed cement paste from the concentration of portland cement [44] Papo et al. (2010) conducted rheological tests under continuous flow conditions on three super plasticizers based on melamine resin, modified lignosulphonate and modified polyacrylate. They showed that addition of superplasticizers improves the viscosity of ordinary Portland cement pastes if added at an optimum dosage and concentration [34].

Petit and Wirquin (2012) conducted 11 tests on ceramic tile adhesive mortars with cellulose ethers (CE), and observed improvement in the viscosity of the mortars due to added CE [45].

Another method that has found to change the rheological behavior of cement paste is mixing methods. M. Yang and H.M. Jennings have studied the influences of mixing method on the microstructure and rheological behavior of cement paste (1995). They have found the rheological behaviors of cement paste are changed during the first 2 hours [46].

2-4-2. Constitutive Equations for Fluid Flow

Koehler has studied the rheological behavior of the fresh concrete (2003). In his research, he mentioned that "Rheology is the study of the flow of a material including the deformation of hardened concrete and the flow of fresh concrete and measuring the rheological properties of fresh concrete provides a fundamental physical description of concrete flow behavior that can be standardized across the concrete industry" [47].

The rheological behavior of fluid usually characterized by at least two parameters, τ_0 and μ according to Bingham equation [27][22]:

$$\tau = \tau_0 + \mu\dot{\gamma} \qquad [6]$$

Where τ_0 (Pa) is the shear stress applied to the material, τ_0 (Pa) is the yield stress, μ (Pa.s) and $\dot{\gamma}$ (s-1) is the shear rate. As the equation states $\tau < \tau_0$ there is no flow, material behave solid and when $\tau > \tau_0$ material move

A combination of shear stress and shear rate, during the steady flow is shown in Figure 2-2. When the rheology behavior of material is measuring, it is important to find a realtionship between shear stress and shear rate [22].

To idealize flow curves, several models and equations have been developed. Six of the common constitutive relationships associated with concrete and cement paste are plotted in Figure 2-2 [15][36][36][47].

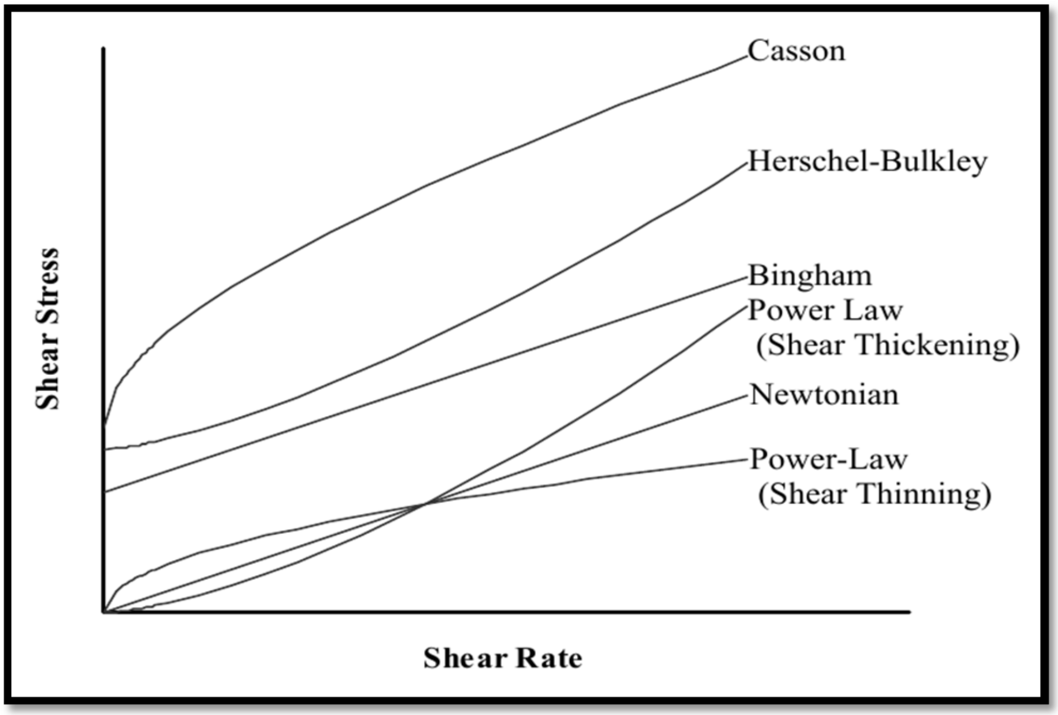


Figure 2-2 Basic Constitutive Relationships for Flow [22]

Newtonian fluid has the basic constitutive equation, where the linear relationship between shear stress and shear rate is given above as equation 2, applies for the whole range of shear rates [22]. Therefore, viscosity (μ) is a material constant:

$$\tau = \mu \times \dot{\gamma} \qquad [7]$$

The Bingham model includes a yield stress term (τ_0) however keeps a linear relationship between shear stress and shear rate.[22][48][47][49]:

$$\tau = \tau_0 + \mu\dot{\gamma} \qquad [8]$$

“Concrete possess a yield stress, or a minimum stress that must be exceeded to initiate flow. This property is readily seen in the ability of concrete to support its own weight” Koehler sad (2003) [47].

The power-law model shows nonlinear relationship between shear stress and shear rate, which yield stress assumes to be zero however, shows the flow curve shape as an exponential relationship:

$$\tau = a\dot{\gamma}^{b} \qquad [9]$$

Where, both (a) and (b) are material constants.

The Herschel-Bulkley model basically a combination of the Bingham equation and power-law equation to represent both a yield stress and a nonlinear flow relationship [10][22][25]:

$$\tau = \tau_0 + m\dot{\gamma}^{n} \qquad [10]$$

τ = shear stress applied to fresh concrete

γ = shear strain rate

τ_o = yield stress = initial shear stress

m,n = parameters determined from calibration

Casson model is another constitutive relationship to represent nonlinearity of flow in fluids with a yield stress [22]:

$$\tau^{\frac{1}{2}} = \tau_0^{\frac{1}{2}} + \mu^{\frac{1}{2}}\dot{\gamma}^{\frac{1}{2}} \qquad [11]$$

2-4-3. Rheological test

In 2012, JuditCanadell, Han Goossens, and Bert Klumperman investigated "a self-healing concept that is based on the use of disulfide links incorporated in a rubber network, which is able to fully restore its mechanical properties at moderate temperatures". In this research they used AR-G2 rheometer by using 8 mm parallel-plate geometry. Creep measurements were performed on a stress-controlled AR-G2 rheometer (TA Instruments) at a constant shear force of 100 Pa. Stress versus strain curves for three samples are plotted in their research from the AR-G2 rheometer [50].

In 2008, Florian J. Stadlera and Helmut Münstedt investigated the terminal viscous and elastic properties of linear ethane/α-olefin copolymers. The rheological data were acquired using a TA Instruments AR-G2 with a 25 mm parallel plate geometry operated at 150°C [51].

In 2009, A. Arocas, T. Sanz, S.M.Fiszman have investigated the clean label starches as thickeners in white sauces. They have studied a linear viscoelastic property in the freshly prepared sauces and after one freeze/thawcycle. A controlled stress rheometer (AR-G2) with 45 mm diameter was used. The rheometer AR-G2 was used to record the storage modulus (G'), loss modulus (G"), complex modulus (G"') and loss tangent (tgδ ¼ G"/G') values[52].

2-4-4. Parallel plate

Figure 2-3 shows varieties of geometry for rheology tests. The geometries are divided to two categories, drag flows and pressure-driven flow. The drag flows is similar to what researchers have done for fresh concrete and cement paste. Drag flows is categorized to four different types of geometries such as Sliding plate, Concentric cylinder, Cone-and-plate and Parallel-plate. The choice of geometry type depends on type of rheometer. For instance, the parallel plate geometry is used for BTRHEOM rheometer for fresh concrete [6][25][49]. However, Banfill (2006) studied that “the cone-and-plate cannot be used for cement pastes because particles jam in the zero gap under the apex of the cone and this led to the development of the truncated cone and the annular plate and cone geometries” he said [6].

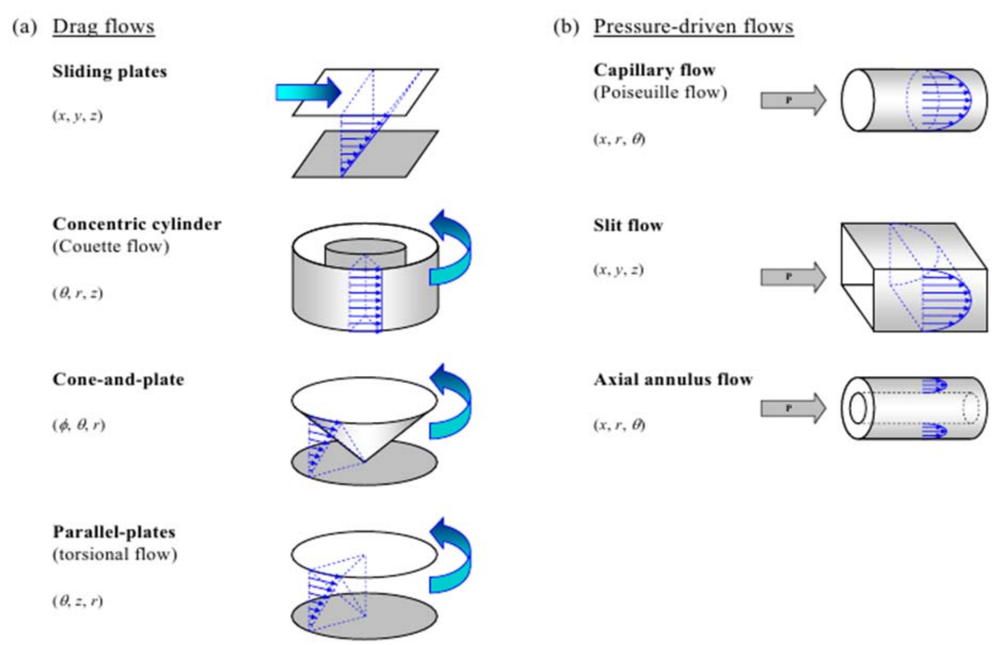


Figure 2-3 Shear flow geometries [25]

Figure 2-4 shows a time line of different rheometers that have been used [25][2]. According to the figure, BTRHEOM and IBB rheometer are the most recent rheometers that have been used by concrete industry researchers. However, the IBB rheometer was found in 1994, which is 19 years old [25].

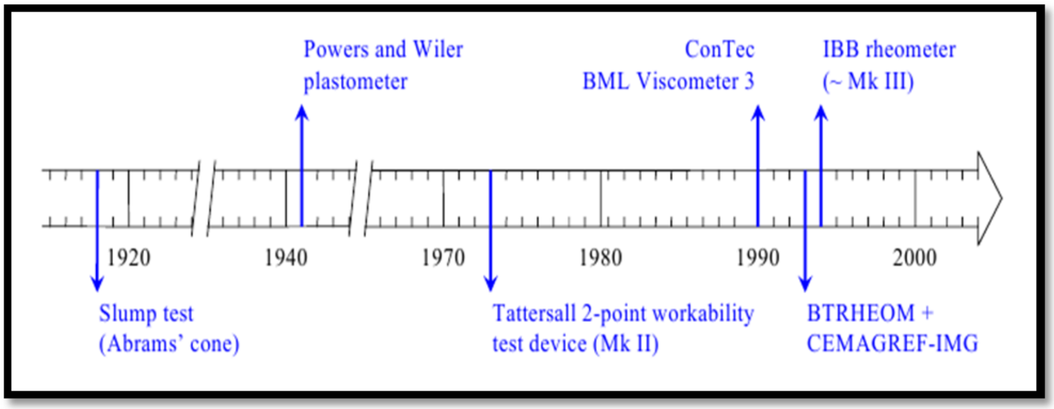


Figure 2-4 Timeline of today commercially available concrete rotational rheometers [25]

2-4-5. Rheometer for cement paste

As shown in Figure 2-5, higher shear rates can be used for describing the flow behavior of fresh concrete during pumping, and lower shear rates is needed for describing the flow behavior of cement paste for gravity leveling [25].

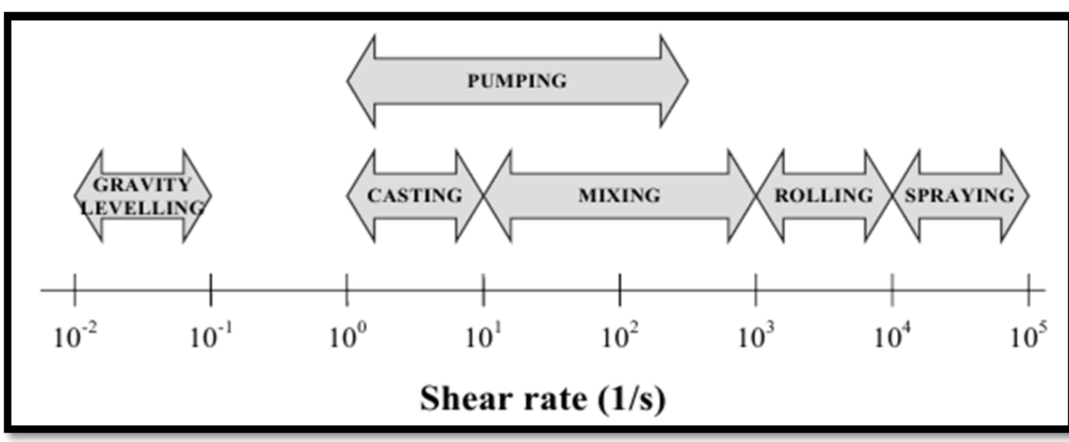


Figure 2-5 Shear rates for typical concrete processing operations [25]

2-5. Correlation between rheological parameters and flow test

Tattersall and Banfill were the first researchers to find out a linear relationship between slump values and yield stress [23], [53]. Moreover, Ferraris and de Larrard (1998) were the first researchers who proposed the "modified slump test" [23], [54]. They tried to find a relationship between the rheological parameters like yield stress and viscosity, and slump test.

In addition, Roussel and Le Roy used Marsh cone instead of flow test to determine both yield stress and viscosity of cement paste [8], [55].

It has been shown that the result of free flow test method, the slump value diameter, correlates well with the (Bingham) yield stress. It has been found that there is no standard test for fresh concrete to correlate directly to the plastic viscosity [25][56].

A modification of the mini-slump-flow test was developed by Tregger (2008) to allow the measurement of the viscosity of cement paste [8]. In his study, he used a power-law plot to relate the yield stress to final diameter. Then he measured the viscosity of samples from the rheology. Then he plotted viscosity and yield stress versus time to the end of the mini-slump flow test, where he created an indirect relationship between viscosity and time to the diameter of the sample at its final state [8].

As stated above, there is no standard test to directly correlate plastic viscosity μ to workability of fresh concrete [25]. However, Ferraris and de Larrard (1998) were the first who proposed the "modified slump test". They modified slump test to measure rheological parameters of fresh concrete. In their study, they used BTRHEOM rheometer to evaluate the yield stress and viscosity of the samples, and to measure the workability of the samples, they used the slump test. Therefore, they found a relationship between yield stress and viscosity with slump test. A modified slump test intended to assess the two Bingham parameters of fresh concrete [54].

Study on Modification of slump cone test was conducted by Ferraris (1999), to allow the measurement of the viscosity [25]. Roussel, N (2006) studied the correlation between yield stress and slump, comparing the numerical simulations and concrete rheometer results [2]. In this study, numerical correlation was compared to the experimental correlation between slump and yield stress using three different types of rheometer, BTRHEOM, the BML and the two-point test. He found that there is a good agreement between the theoretical prediction and BTRHEOM for the range of traditional concrete (50 mm < slump < 250 mm) [2].

In summary, the yield stress can be calculated from the slump test, however viscosity of cement paste cannot be calculated directly from the slump test. Recently, researchers have been tried to use the second parameter such as time to final spread to calculate the viscosity.

Chapter 3. Experiments

3-1. Materials and methods

In this research Cellulose Nano Fiber (CNF), Glucose and Sucrose were used as admixtures to cement paste to assess their effects on rheological behavior of the cement paste specimens. On the other hand, Rheology test and flow table test are two mainly methods that were significantly used during this research.

3-1-1. Materials

The selected admixtures are CNF, as an organic material and Sucrose and Glucose also organic material.

3-1-2. Cement

Cement is the adhesive material to bind the aggregates in concrete, mortar and grout. Lafarge Portland Cement is a high quality and inexpensive basic building material. It has been used in various forms of construction including, homes, bridges, tunnels and airports. Lafarge Products meets all applicable chemical and physical requirements of ASTM C 150 [57].

There are 12 different types of Portland Cements such as GU, GUL, MSs, MH, MHL, HE, HEL, LH, LHL and HS. Each type is required for a specific application of cement [13]. Type GU is one of the most commonly used cement type. It is suitable to use for all applications excepts where the special properties of any other type of Portland cement are required. The Chemical composition with weight percentage and formula is given in Table 3-1.

A type GU Portland cement was used for all of the rheology and flow table tests.

Table 3-1 Composition of Portland cement with chemical composition and weight percent

Cement Compound	Weight Percentage	Chemical Formula
Tricalcium Silicate	50%	Ca_3SiO_5 or $3CaO\ SiO_2$
Dicalcium Silicate	25%	Ca_2SiO_4 or $2CaO\ SiO_2$
Tricalcium Aluminate	10%	$Ca_3Al_2O_6$ or $3CaO\ Al_2O_3$
Tetracalcium Aluminoferrite	10%	$Ca_4Al_2Fe_2O_{10}$
Gypsum	5%	$CaSO_4\ 2H_2O$

3-1-3. Cellulose Nano Fiber (CNF)

Cellulose Nano Fiber (CNF) is an organic marital composed of Nano sized cellulose fibrils with a high aspect ratio (length to width ratio). Typical average diameter is 0.18 nanometers and longitudinal dimension is in a wide range from 1 to 2 of micrometers. The scanning electron microscope (SEM) picture of dimension of CNF is shown in Figure 3-1 to prove of the previse statues. CNF was prepared at Innventia AB (Stockholm, Sweden). They are two different types of CNF Innventia 2.5 w.t % and Innventia 10 w.t %. Innventia 2.5 w.t % was used in this research, which is shown in figure [2].

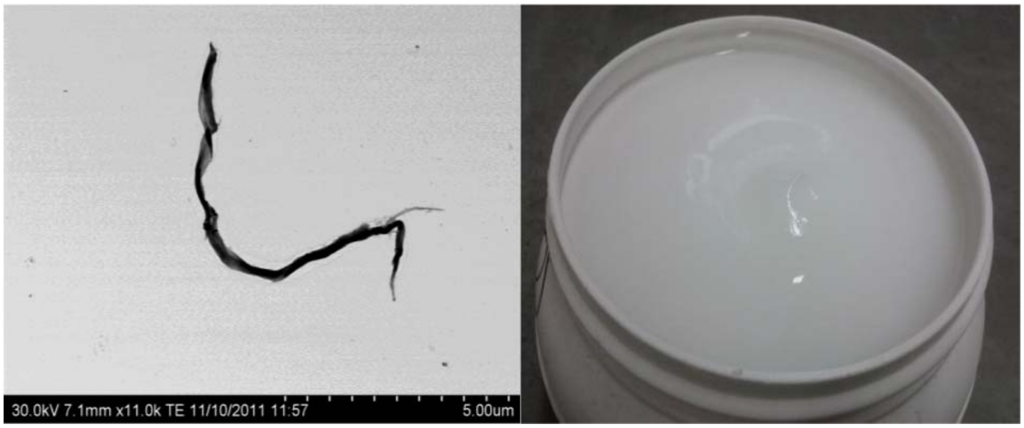


Figure 3-1 SEM of CNF Innventia 2.5 w.t %

CNF has been chosen as one of the additives in cement paste because of the following advantages:

- Manmade and Natural Cellulose
- High surface area
- –High interaction
- Light-weight
- Green: biodegradable, renewable
- Low cost/High value compared with other Nano fiber Technologies

3-1-4. Glucose

Glucose is a main source of energy for living organisms. Glucose with molecular formula $C_6H_{12}O_6$ and molecular weight 180.2 was used for this research which was supplied by Sigma-Aldrich. The molecular structure of glucose is shown in Figure 3-2.

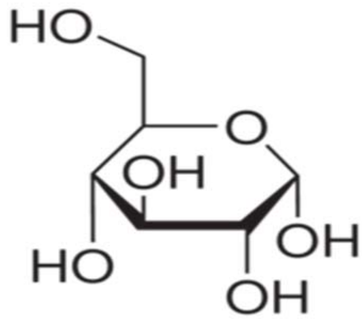


Figure 3-2 Molecular Structure of Glucose [Sigma Aldrich]

3-1-5. Sucrose

Sucrose or table sugar is obtained from sugar beets. Sucrose with molecular formula $C_{12}H_{22}O_{11}$ and molecular weight 342.3 was used for this research which was supplied by Sigma-Aldrich. The molecular structure of glucose is shown in Figure 3-3.

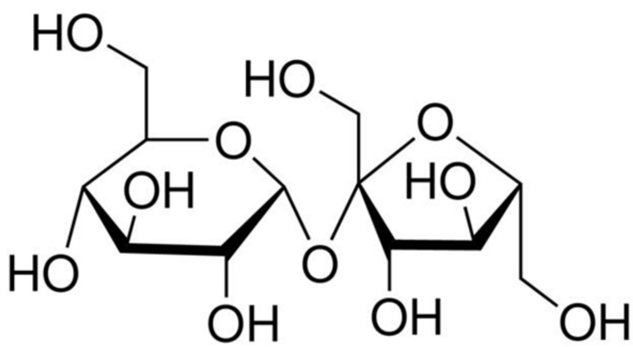


Figure 3-3 Molecular Structure of Sucrose [Sigma Aldrich]

3-2. Using the AR-G2 Rheometer Test

The experimental plan was designed to make a workable mixture of the chosen components. The viscosity of the samples was tested by TA Instruments AR-G2 rheometer as it is shown in Figure 3-4 [58].

Figure 3-4 AR-G2 rheometer

This test measures the viscosity and the shear stress of cement paste. It should be noted the rheology on cement paste is not very sensitive to temperature, however temperature was kept constant during the test. All tests reported here were performed at 25^0 Celsius room temperature.

The rheology of cement paste was measured during research to determine the effects of chemical admixture on cement paste rheology. “This is a laboratory test, which gives an indication of the quality of concrete with respect to consistency, cohesiveness and the proneness to segregation” by M.S. SHETTY [13].

3-2-1. Digital Vortex Mixer

The mixing method affects the rheological response of the cement paste; therefore a controlled speed mixer is the best method to ensure that the cement paste is always mixed in the same way [57]. In our tests the test speed was held constant at 1000 rpm for all experiments. Figure 3-5 shows the digital vortex mixer used in our experiments.

Figure 3-5 Digital Vortex Mixture

3-2-2. Scale

Scale model PL303 with 310g capacity and 0.001g precision was used for all the material weight measurments and it is shown in Figure 3-6.

Figure 3-6 Scale

3-2-3. Smart Swap Rheometer

The results of the rheology test are significantly affected by selected geometry. In these tests, parallel plate geometry was selected, which is recommended by Ferraris for rheology test on cement pastes [27]. This geometry has a flat rough surface which is shown in Figure 3-7.

Figure 3-7 Parallel plate geometry with rough surface

Sand Paper

A 60 grade sand paper is also laid under the specimen to prevent slippage of the specimen during the test. It was waterproof to avoid absorbing water from sample which is shown in Figure 3-8.

Figure 3-8 Sand paper

Test Setup and Measurement for Rheology

In this research, the viscosity of cement paste samples with CNF and sucrose was experimentally studied. The CNF/cement and sucrose/cement ratios of the test samples are summarized in Table 3-2. Test number 1 is the control sample, where the cement paste is prepared 15 grams of cement and w/c ratio of 0.35. This amount of cement and w/c ratio was kept constant throughout the test. The w/c ratio 0.35 was selected to prevent premature hardening of the cement paste due to water absorption by CNF. The experiments on cement pastes with CNF and sucrose were conducted in three phases. In the first phase, test numbers 2 to 8, CNF was added to the cement paste with CNF/c ratio from 0.083% to 0.625%. In the second phase, test numbers 9 to 14, sucrose was added to the samples with sucrose/c ratio from 5% to 50%. In the third phase, CNF /c ratio in cement pastes was kept constant at 0.625% and sucrose/c ratio was varied from 5% to 50%.

Table 3-2 Material proportions for the rheology tests

Test number	w/c	CNF/C	Sucrose/C		Test number	w/c	CNF/C	Sucrose/C
	%	%	%			%	%	%
1	35	0	0		12	35	0	30
2	35	0.625	0		13	35	0	40
3	35	0.5	0		14	35	0	50
4	35	0.375	0		15	35	0.625	5
5	35	0.25	0		16	35	0.625	10
6	35	0.167	0		17	35	0.625	20
7	35	0.125	0		18	35	0.625	30
8	35	0.083	0		19	35	0.625	40
9	35	0	5		20	35	0.625	50
10	35	0	10		21	35	0.625	60
11	35	0	20					

The viscosity of the samples were tested by TA Instruments AR-G2 rheometer. As seen in Figure 3-7, in this rheology test a circular plate, known as geometry, sits on the fluid cement paste and applies shear strain on the specimen by rotation.

The instrument was set up to apply shear rate from 100 (s^{-1}) to 0.01 (s^{-1}) over 10 minutes at room temperature of 25 Celsius degrees. Testing of the specimen from high to low shear rates was due to hardening of the cement pastes. Due to water-cement hydration, hardening of the cement paste advances during the rheology test, which solidifies the specimen. If the test had been conducted with ascending shear rate, after some time from the beginning of the test the progressed hardening of the cement paste would have caused slippage of the plate over the specimen at high shear rate.

The results of the rheology test are significantly affected by selected geometry. In these tests, parallel plate geometry was selected, which is recommended by Ferraris for rheology test on cement pastes. This geometry has a flat rough surface.

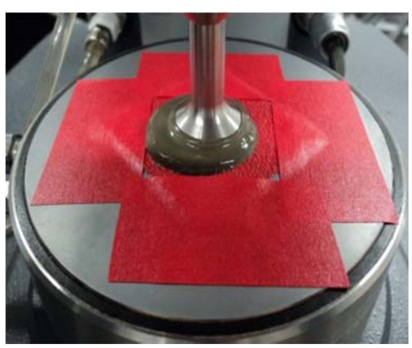


Figure 3-9 Test setup with rheometer instrument

The mixing method also affects the rheological response of the specimens Ferraris [27].

The control sample was made of 15 grams of cement with 5.25 g water giving 0.35 w/c ratio. Cement and water were mixed by vibration in a beaker for 60 seconds at 1000 rpm on vortex mixer. A steel stirrer was also used to stir the solution for a short time.

The mixed specimen was then put on the platform of the rheology instrument with about 4 mm thickness. At the beginning of the test, the geometry of the rheology instrument was set to apply 3.2 stresses on the specimen for keeping the stress the same for all of the samples. Then the test was started from 100 (s^{-1}) to 0.01 (s^{-1}) shear rate.

For the specimens with CNF and sucrose, first the chemicals and the water were vibration mixed for 120 seconds at 1000 rpm for enough time to yield a homogeneous solution. Then 15 grams of cement was added to the mixture and the solution was vibration mixed on vortex mixer for 60 seconds at 1000 rpm. The test type was set on steady state flow step.

The geometry used for rheology test was parallel plate with rough surface with 20 mm diameter, in which the plate of the instrument is flat and in parallel with the surface of the specimen. Parallel plate was used because previous studies found it to be the best geometry for this test. As the viscosity of the sample increases smaller diameter should be chosen for geometry, hence the 20 mm diameter in the conducted tests. A sand paper was put under the specimen on the platform to avoid rigid rotation of the specimen under the torque exerted by the geometry.

Oscillation procedure with steady state flow step was selected for the conducted rheology tests. The shear rate varied from 100 1/s to 0.01 1/s over 10 minutes.

It is known that the rheology of concrete is affected by the cement paste content, by contributing to the gap between the aggregates [12]. A parallel plate fluid rheometer was used to simulate the gap between the aggregates. The shear history of a cement paste with the same shear rate as it will experience in concrete [12]. The shear rate of cement paste in concrete during mixing and placement was established by Helmuth at 70 1/s [12]. Also the shear rate applied to the cement paste in concrete during a rheological test is from 3 to 24 1/s, as determined when the calculation are based on the shear rate applied to concrete in BTRHEOM rheometer [12].

The mixing method affects the rheological response of the cement paste, therefore a controlled speed mixer is the best method to ensure that the cement paste is always mixed in the same way [57] [59]. In our test the test speed was held constant for all experiments.

3-3. Mini-Slump Test

The mini-slump test was originally developed by Kantro (1980) and later modified by Zhro and Bremner (1998), which measures the consistency of cement paste [60].

The flow table test is intended for use in measuring the flow behavior of cement paste. The test is standardized in ASTM C230 [58]. Figure 3-10 shows the details of apparatus used.

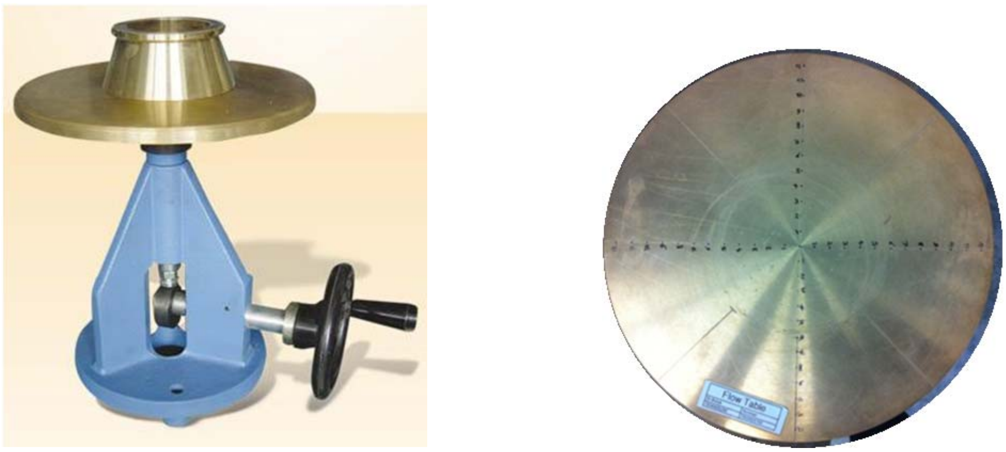


Figure 3-10 Flow Table with Brass plate and Mould

3-3-1. Mechanical Mixing of Hydraulic Cement Pastes

Kitchen aid mixer was used for the entire samples. For preparing the sample with cement and water a steel paddle was chosen for all tests. The mixer apparatus shall consist of a frame Figure 3-11. As the following steps, procedure will be explained according to ASTM C305 [61]

- Place the dry paddle and the dry bowl in the mixing position in the mixer
- Place all the mixing water in the bowl
- Add all the cement into water and allows to absorb the water for 30s
- Start mixing the cement and water with slow speed (140 ± 5 r/min) for 30s
- Then stop the mixer for 15s for cleaning the collected cement paste on the sides of the bowl

- Finally start mixing the cement and water for 60s at the medium speed (285 ± 5 r/min)

The product obtained after mixing cement with water is called cement paste. In this research cement paste has been used for mini-slump test.

Figure 3-11 Kitchen aid mixer and Paddle

3-3-2. Vicat Needle Test Procedure

The setting time of cement paste can be measured as an indication of workability [27]. Vicat needle test is one of the most common tests for testing cement paste (ASTM C192). This test method determines the time of setting of hydraulic cement by means of the Vicat needle. The Vicat apparatus shall consist of a frame Figure 3-12. The moveable rob is 300g with 1 mm diameter needle at the end. As the following steps, procedure will be explained the procedure of the test according to ASTM C191 [59].

- Quickly the fresh cement paste prepared quickly places into a ball with gloved hands.
- Then throw from one hand to the other hand for six times.

- Squeeze the ball and waiting for a few seconds then place it into the larger end of the ring and completely filling the ring with fresh cement paste.
- Use a trowel to remove and make the top surface of the mold clean.

After placing the fresh cement paste in a conical ring below the mold, test shall be started with setting up the needle on the top of the cement paste. Since the needle touches the top surface, we stop moving the bar because of calibration the instrument to zero procedure. The setup is ready to measure no less frequently than every 15 minutes. After 30 min the needle under its own weight is placed on the surface of fresh cement paste and allowed to settle inside the sample. The test is repeated each 15 minutes as mentioned before. The depth of penetration is recorded from the Vicat needle. Each penetration should take in different location on the cement paste because of making more accurate measurement.

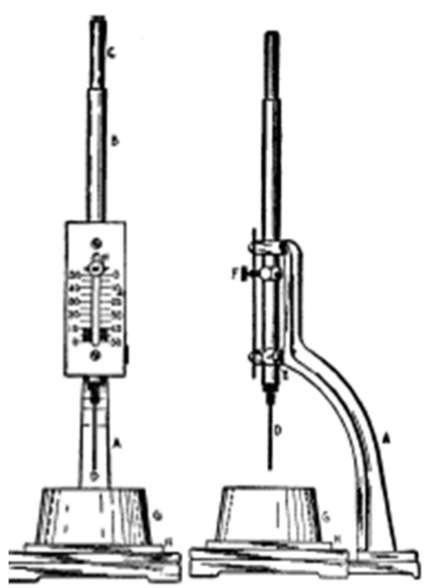


Figure 3-12 Vicat Apparatus [9]

3-3-3. Cement Paste

Cement paste can be used for different application such as masonry building, gluing tiles on the wall and etc. Cement paste always is known as ratio water over cement. W/c ratio can change the workability of cement paste with increasing or decreasing the amount of water or cement. To create a workable cement paste, Vicat Needle test has been used, which was explained before, to find the best W/c ratio for cement paste. In this research the W/c ratio is kept constant for all the rheology and flow tests. The reason that this ratio was chose is just because of knowing the effect of addictive in cement paste which causes to change the workability of the cement paste. The cement paste with constant ratio for this research is called control sample with 0.35 W/c. The molecular structure of cement before and after adding it with water is shown in Figure 3-13.

Figure 3-13 Cement Particle surface charge before and after mixing with water

3-3-4. Flow Table Setup and Test Procedures

The mini-slump test was carried out by using a cone with diameters of 7 cm at top and 10 cm at the bottom and 5 cm height Figure 3-14, according to ASTM C1437 [23].

First, the height and radius of each sample is recorded by camera during the slump test. Figure 3-14 is represented slump test at first drop with h_1, r_1 in the left figure and after n drops with h_n and r_n in the right figure.

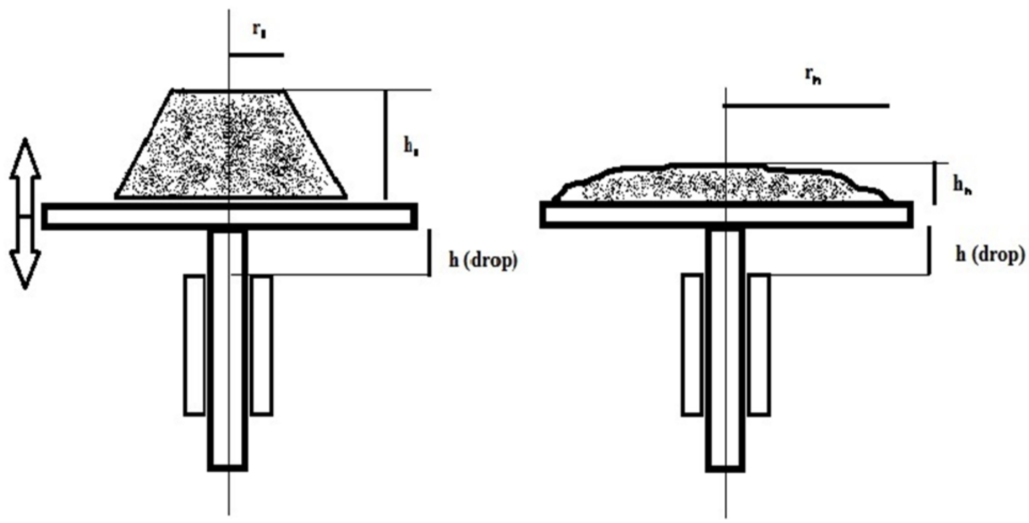


Figure 3-14 Mold for flow test

As the following steps, procedure will be explained the procedure of the test according to ASTM C1437 [23].

- Flow table should be clean and dry, and flow mold place at the center

- In two layers mortar should be placed in the mold about 25 mm (1 in.) in thickness and tamp 20 times with the tamper. Tamping shall be done after placing each layer of mortar.
- The tamping shall be just to ensure the uniformly filling of the mold.
- In this step the extra part of mortar should cut by the edge of the trowel with a sawing motion across the top of the mold.
- All the proses should take only one minute after completing the mixing operation until Lifting the mold away from the mortar.
- Immediately drop the table 25 times in 15 s, unless otherwise specified.
- After the mini-slump cone is lifted up from the cement paste sample, immediately drop the brass table 25 times in 15 seconds.

"The flow is the resulting increase in average base diameter of the mortar mass, expressed as a percentage of the original base diameter" ASTM C1437. Using a caliper, compute the flow in percent by dividing *"A"* by the original inside base diameter in millimeters and multiplying by 100. Where it is shown in the following equation [12]

A = average of four readings in millimeters, minus the original inside base diameter in millimeters divided by inside base diameter in millimeters. Report the flow to the nearest 1 %.

$$\text{Flow in Percent} = \frac{\text{A}-25}{25} \times 10 \qquad [12\ ]$$

The bigger the spread diameter, the lower viscosity of cement paste or the lower the yield stress of the cement paste [62].

In this research, two cameras were used to record the spread evolution over time and also height changing over time Figure 3-15. One of the cameras was setup at the top of the mini slump flow table to record all of cement paste movement over time. The other one was setup on the side of the table where it was recording the height changing over time. For all the mini slump tests, the cameras were fixed and the angle of cameras was constant when they were used for recording the movies.

Figure 3-15 Setup the cameras at top and side of the Mini flow table

The purpose of using two cameras was to record the spread evolution and height changing over time. These two parameters were then used to create the graphs.

Chapter 4. Results and Discussion

4-1. Description of the Case Study

A series of cement paste were prepared consisting of type general use (GU), tap water, cellulose nano fiber (CNF) and sucrose. Cement paste mixture design is shown in Figure 4-1. In this mixing set up only one water binder ratio has been applied which is water/cement ratio 0.35 (weight/weight) along with different dosages of CNF, sucrose and mixes of both.

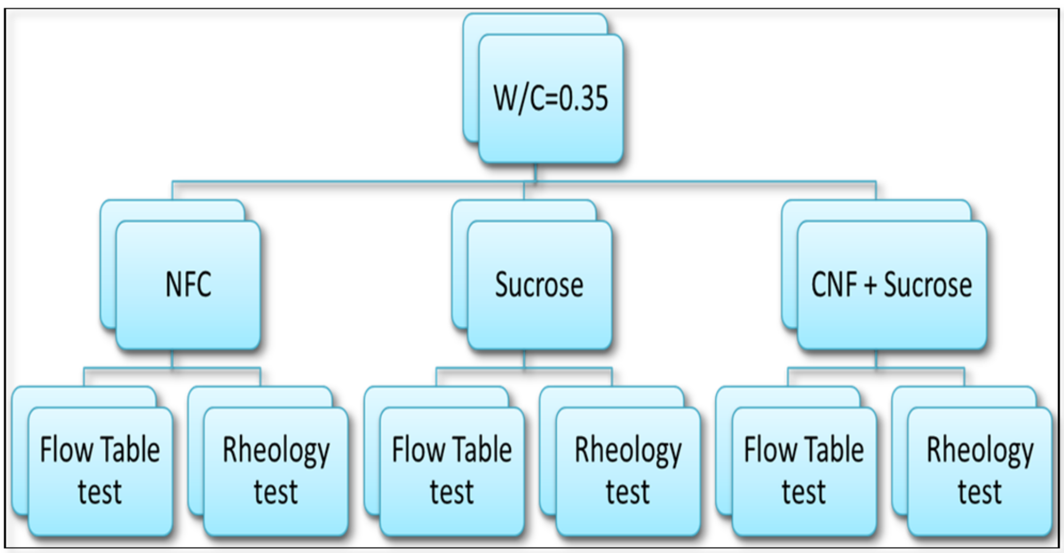


Figure 4-1 Mixture design

In this study, viscosity, spread versus drops and final spread were investigated through the rheology and mini slump test respectively. Simple test such as the slump-flow test was used to measure quality control of concrete or cement paste workability. Also, the rheometer AR-G2 was used for cement pastes in order to investigate and analyze the rheological parameters such as yield stress, τ_0 and viscosity, μ [63]. Obtaining knowledge about the rheological behavior of concrete or fresh cement paste is of significant interest and can be very helpful to optimize mixing of these materials. In addition, this information can be utilized to find appropriate application for concrete or fresh cement paste. Fresh concrete or cement paste is an unsolidified material which the way it can be processed is affected by its flow behavior [47]; therefore, the rheological parameters such as yield stress and viscosity are two significant factors in the creation of high quality concrete or cement paste [8], [64]. In this research, the correlation between the rheological behavior of fresh cement paste from the rheology tests and final spread from the mini flow table test has been investigated. In addition, rheology data is used along with the spread as a function of drops was camera recorded and used for modeling the rheological properties.

4-2. Vicat needle test results

Vicat needle test has been used to choose the amount of water required for normal consistency of hydraulic fresh cement paste [10], [29]. The test has been conducted in this research is a method including the manually operated standard Vicat apparatus. According to ASTM C187, 10 mm ± 1 mm is the minimum penetrations needle can go through the cement paste specimen so it is important to find the specific amount of water for mixing the cement with water to produce the fresh cement paste [10], [29]. Table 4-1 shows that four fresh cement pastes with different water/cement ratio have been prepared. The results indicate that test number 4 with 11 mm penetration is the only one that meets the ASTM C187 requirement.

Table 4-1 Vicat needle test on cement paste

Sample #	Cement (gr)	Water (gr)	ASTM Requirement	Result	water/cement
1	400	104	10 mm _+ 1 mm	8 mm	0.26
2	400	110	10 mm _+ 1 mm	14 mm	0.275
3	400	107	10 mm _+ 1 mm	12 mm	0.2675
4	400	105	10 mm _+ 1 mm	11 mm	0.2625

Therefore, the water cement ratio more than 0.2625 has to be used. It is important what property aspects are considered for cement paste. In this study, the rheological behaviors were considered to study, the minimum water/cement ratio was not chosen because of the changes in viscosity. For instance, with having some chemical additives into the fresh cement paste, it is a chance to reduce the viscosity of the sample, which, if it was chosen with the minimum w/c, it workability of the fresh cement paste would not reach to the ASTM C187 requirement [29]. Therefore, in this research, the water/cement ratio 0.35 was selected to investigate and control the rheological behavior of fresh cement paste. As a result, some chemical additives were used into the fresh cement paste for changing the viscosity of the sample. The results were showed that the workability of the sample is changed, however the sample was still workable and useful.

4-3. Rheology Test

4-3-1. Test Results and Discussion

The purpose of using the rheometer was to determine the rheological behavior of fresh cement paste then finding a relationship between the rheology parameters and mechanical behavior of fresh cement paste by using the mini-slump flow test. It was mentioned in material and methods chapter that rheological tests were conducted on 21 samples during this research. Chemical additives such as CNF and sucrose were added to the control sample. The results of the rheology tests indicate that the flow behavior of fresh cement paste is significantly affected by chemical additives. According to Table 4-2 the following tests were performed to determine the validity of methodology adopted:

Table 4-2 Test Modeling

Tests	Cement paste Water/cement (0.35)	Cement paste + CNF	Cement paste + sucrose	Cement Paste + CNF + sucrose
τ_y	✓	✓	✓	✓
μ	✓	✓	✓	✓

Rheology Data on cement paste with CNF

First of all, the flow test was performed on the cement paste (w/c of 0.35) with no admixtures as a control sample. Figure 4-2 shows viscosity versus shear rate for cement pastes with various amounts of CNF/cement weight ratio. The result for control sample in Figure 4-2 shows viscosity is decreasing from 1.E+06 (Pa.s) to

1.E+01 (Pa.s) whereas the shear rate is increasing from 0.01 (1/s) to 100 (1/s). Thus, as a non-Newtonian shear thinning material, viscosity decreases with an increase in shear rate. When the shear stress applied to the control sample is very low, the viscosity is in the highest amount; however, when the force increases viscosity begins to loss its stability so that it gradually drops. The physical meaning can be defined as followed: where the control sample is able to maintain its shape for a long time with no change it means the sample has high viscosity. Otherwise, it is called low viscosity sample because it cannot stay long enough in its stable shape. Therefore, as it is shown in Figure 4-2 viscosity is changing by the time for each different values of CNF.

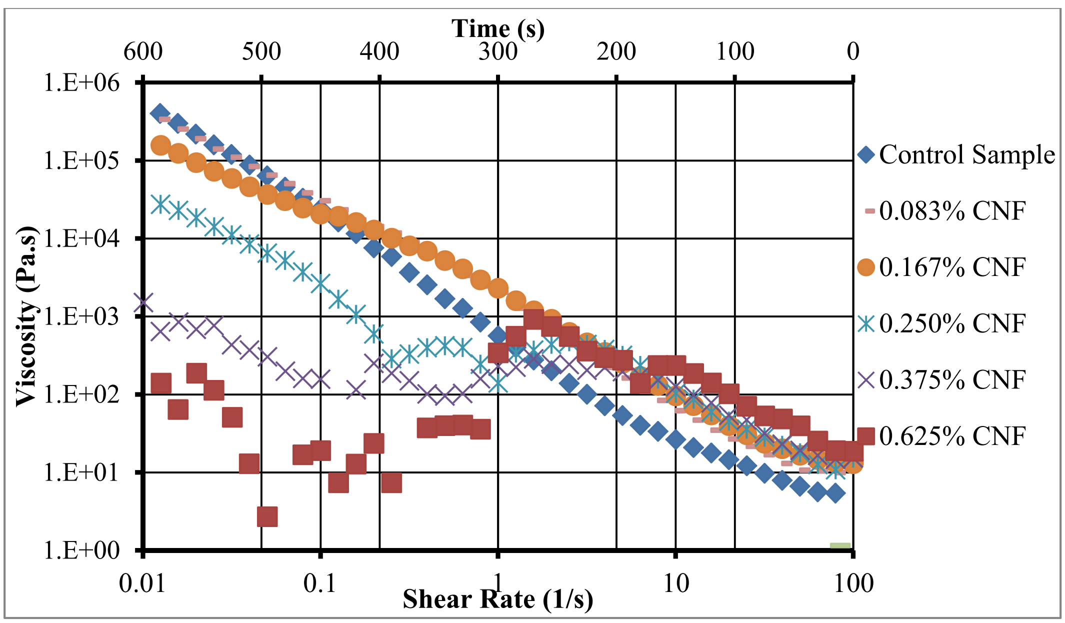


Figure 4-2 Viscosity vs. shear rate for cement pastes with CNF

As observed in Figure 4-2, the increase in CNF /cement ratio reduces the viscosity. This is well exhibited in Figure 4-3, which shows a power correlation between viscosity and CNF/cement weight ratio of the cement paste at 0.1 (s^{-1}) with convergence factor of 0.9756 % according to the following equation:

$$\mu = 51.23\left(\frac{\text{CNF}}{\text{c}}\right)^{-1.314} \qquad [13]$$

Where the υ is viscosity (Pa.s) and CNF/c is weight ratio of CNF to cement.

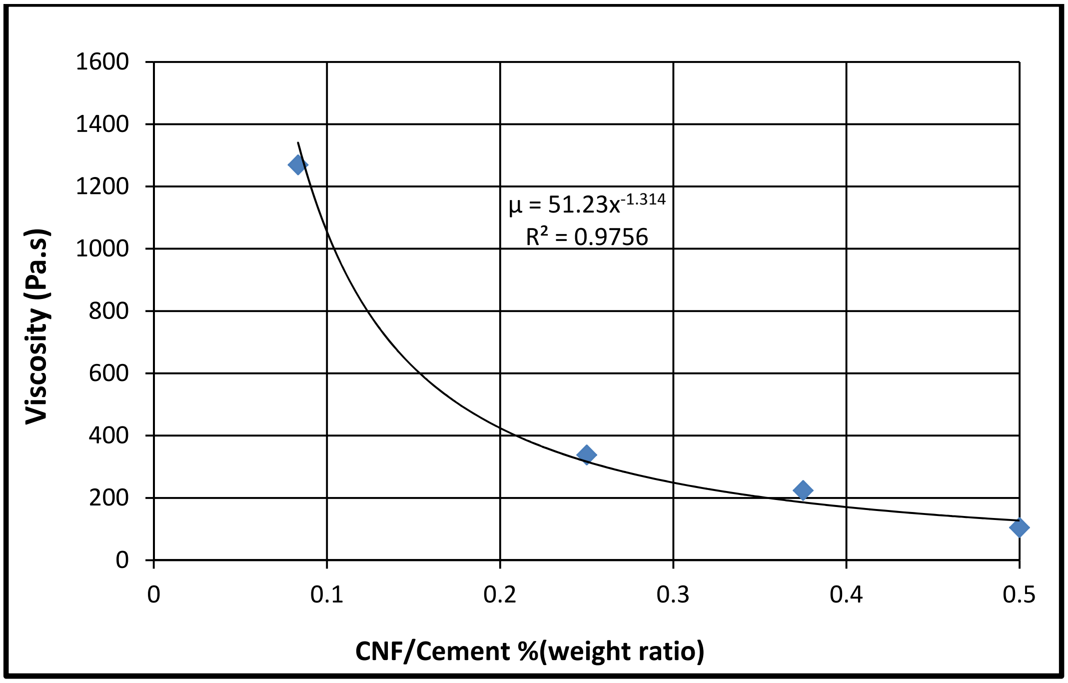


Figure 4-3 Viscosity CNF/c ratio of $\dot{\gamma} = 0.1\ s^{-1}$

Rheology data on cement paste with Sucrose

Figure 4-4 shows viscosity versus shear rate for cement pastes with sucrose. As seen in this figure, at later stages of the experiment, increase in sucrose/cement weight ratio decreases the viscosity of the cement paste. Figure 4-4 shows viscosity versus sucrose/cement weight ratio for range of between 0.01 (s-1) to 100 (s^{-1}) in shear rate. As seen in this figure, at lower shear rate, the viscosity of the cement paste with 10% sucrose/cement and more has an average value of 3.168 Pa.s at 10 (s^{-1}) shear rate, which is significantly lower than the value of 26.37 Pa.s for the control sample. However, as shear rate advances, the increase in sucrose/cement weight ratio further decreases the viscosity, which is exhibited by divergence of the curves for different sucrose/cement ratios.

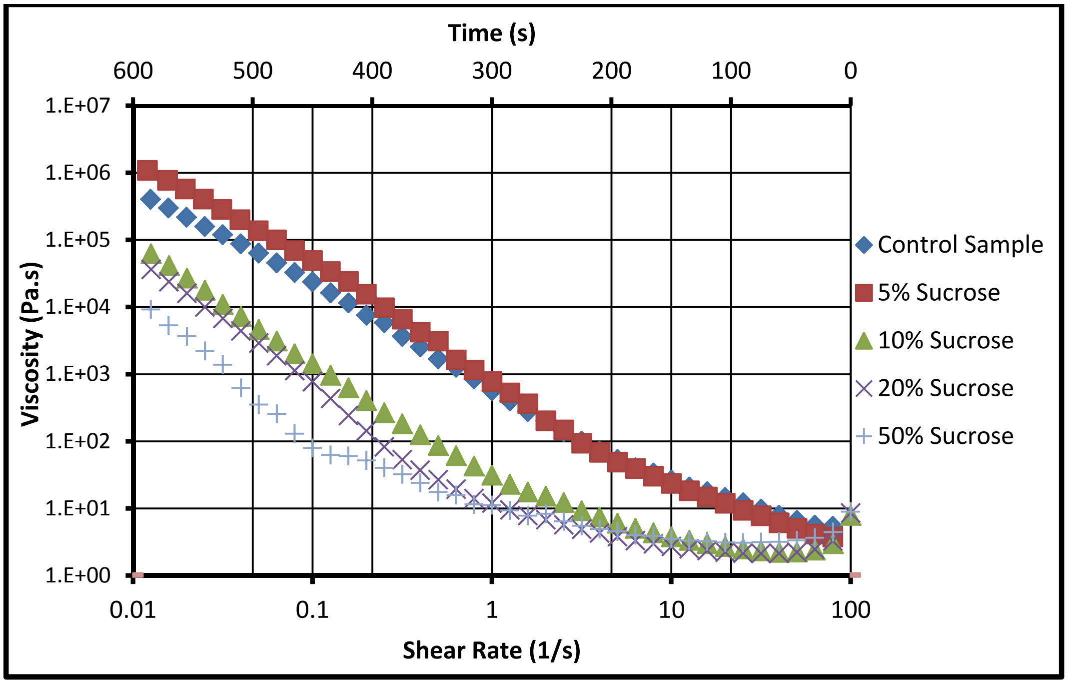


Figure 4-4 Viscosity vs. shear rate for cement pastes with sucrose

Rheology data on cement paste with CNF and Sucrose

Figure 4-5 shows viscosity versus shear rate of the cement pastes with 0.625% of CNF/cement ratio and varying sucrose/cement ratios along with control sample. As seen in this figure, if sucrose/cement ratio is more than 40%, the viscosity of the specimen is greater than that of the control cement paste before 0.1 sec^{-1} from the beginning of the test. However, bellow 0.1 sec^{-1} shear rate, the viscosity of the specimen with 40% sucrose/cement slightly decreases due to hardening of the cement paste caused by CNF.

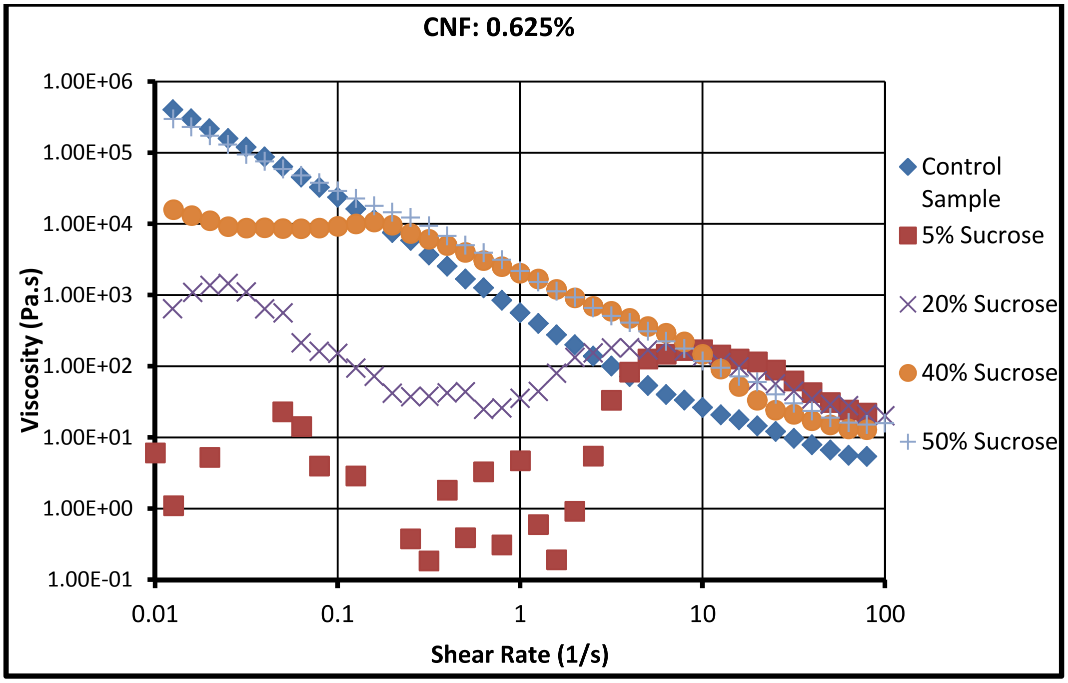


Figure 4-5 Viscosity vs. shear rate for cements pastes with sucrose and CNF

The yield stress and viscosity from the rheology software

One of the main purposes of using rheometer was to find out two important flow parameters of cement mixture: yield stress and shear dependent. The rheology TA advantage data analysis software has been used to analyses the data. According to Casson equation, the yield stresses were found from the software for each sample. Figure 4-6 shows the shear rates range have been chosen between 0.01 and 1 (1/s) because yield stress always happens at the low shear rate. For instance, the rheology test has been done on control sample and the yield stress were found equals 3052 Pa. According to graph, viscosity decreases with increasing strain rates, because cement paste is shear thinning [8], [9].

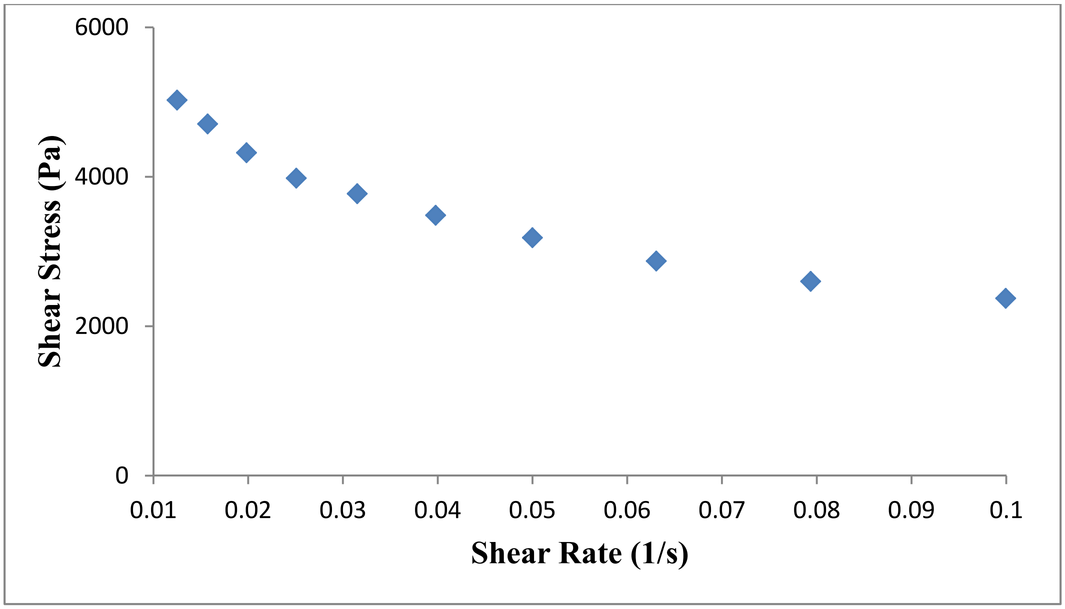


Figure 4-6 Shear stress vs. Shear rate for control sample

Four more tests have also been conducted on cement paste samples with 5% Sucrose/c, 0.375% CNF/c with 50% sucrose/c, 0.125% CNF/c and 0.167% CNF/c and then the yield stresses were found the same as control sample for all the above samples where their data is listed in Table 4-3 as following.

Table 4-3 Yield stress and viscosity, values caculated from Casson Plot

Sample	**W/c**	**CNF/c**	**Sucrose/c**	**Yield stress**	**Viscosity**
#	%	%	%	Pa	Pa.s
1	0.35	0.00E+00	0	2406	2.38E+04
2	0.35	0.625	0	2996	2.71E+04
3	0.35	0.5	0	45.02	2.20E+02
4	0.35	0.375	0	14.71	1.57E+02
5	0.35	0.25	0	298.5	2.65E+03
6	0.35	0.167	0	6785	4.94E+04
7	0.35	0.125	0	4249	4.13E+04
8	0.35	0.0833	0	3610	4.30E+04
9	0.35	0	0.05	3051	1.38E+04
10	0.35	0	0.1	5416	4.94E+04

11	0.35	0	0.2	203.6	1.42E+03
12	0.35	0	0.3	37.84	1.55E+02
13	0.35	0	0.4	22.08	8.18E+01
14	0.35	0	0.5	42.08	7.95E+01
15	0.35	0.375	0.05	3.03	8.00E-01
16	0.35	0.375	0.1	44.45	2.68E+02
17	0.35	0.375	0.2	19.38	1.51E+02
18	0.35	0.375	0.3	12.65	5.07E+00
19	0.35	0.375	0.4	390	9.27E+03
20	0.35	0.375	0.5	3597	3.42E+04
21	0.35	0.375	0.6	3992	4.16E+04

According to the Table 4-3, viscosities were also found for each sample from the rheology data for the low shear rates. As it is obvious in Table 4-3 the lowest yield stress happens for the sample with low viscosity. The viscosity is increasing since the yield is increasing too. The data from the Table 4-3 for yield stress and viscosity is plotted in Figure 4-7. Figure 4-7 shows a polynomial correlation between viscosity and yield stress with convergence factor of 0.994 % according to the following equation:

$$\mu = 8.6285\ \tau_0^2 + 793.22 \qquad [14]$$

Where the μ is viscosity (Pa.s) and τ_0 is yield stress.

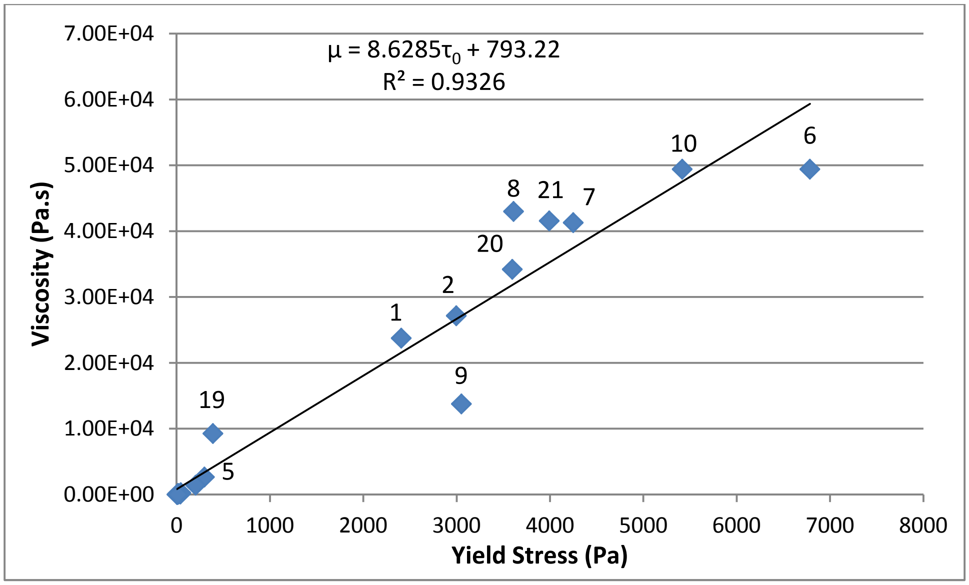


Figure 4-7 Viscosity vs. yield stress

According to the figure, viscosity is increasing from 22000 (Pa.s) to 50000 (Pa.s) whereas the shear rate is increasing from 2400 (1/s) to 6800 (1/s).

Yield stress and viscosity are two independent parameters supposed to be used to describe the flow behavior of cement paste. However, we have seen in special case; when yield stress and viscosity are not independent parameters but they are relate it. It was because of the limited amount of components we used in our formulations.

4-4. Mini Flow Slump Test

The slump test was prepared with the mini metal cone, open at both ends and sitting on a horizontal plate, is filled with cement paste, and lifted quickly and dropped 25 times in 15 seconds ($\frac{0.6\ sec}{1\ drop}$) [23]. The slump of the cement paste with/without additives is measured as shown in Figure 4-8.

Figure 4-8 Mini flow slump test

The flow of mini slump test is measure as a final spread of cement paste on flat table. The spread of fresh cement paste depends on two parameters such as viscosity, μ and yield stress, τ. The fresh cement paste will move only if the applied stress exceeds the yield stress and will stop, when the applied stress is below the yield stress. Therefore, the mini flow slump test is related to the yield stress. Moreover, a spread diameter of fresh cement paste depends on the viscosity of the sample but the viscosity is not calculable easily from the test. ASTM C1437 has been written based on the test and is used in other countries [23].

In this research, the mini slump test procedure was modified, where it was recoding while it drops 25 times in 15 seconds to allow the approximate calculation of both yield stress and the viscosity.

4-4-1. Results and Discussion

The mini flow table test was conducted 15 times for different mixtures. Three tests were chosen to excrement the relationship between spread and time. One of the tests is on cement paste with no additive material, the second one is mixed with 0.375% CNF/c and 50% sucrose and the last one is mixed only with 0.375% CNF/c. Figure 4 9 shows spread of fresh cement paste versus time for each three types of samples. According to the Figure 4 9, the diameter is increasing by the time. The polynomial plot was used to find the equation between the final spread and time. For example, Figure 4 9 shows a polynomial correlation between spread and time of the control sample with convergence factor of 0.987 % according to the following equation:

$$D=11.928+1.0262t-0.026t^2 \quad [15]$$

Where the D is the diameter of the fresh cement past and X is the time.

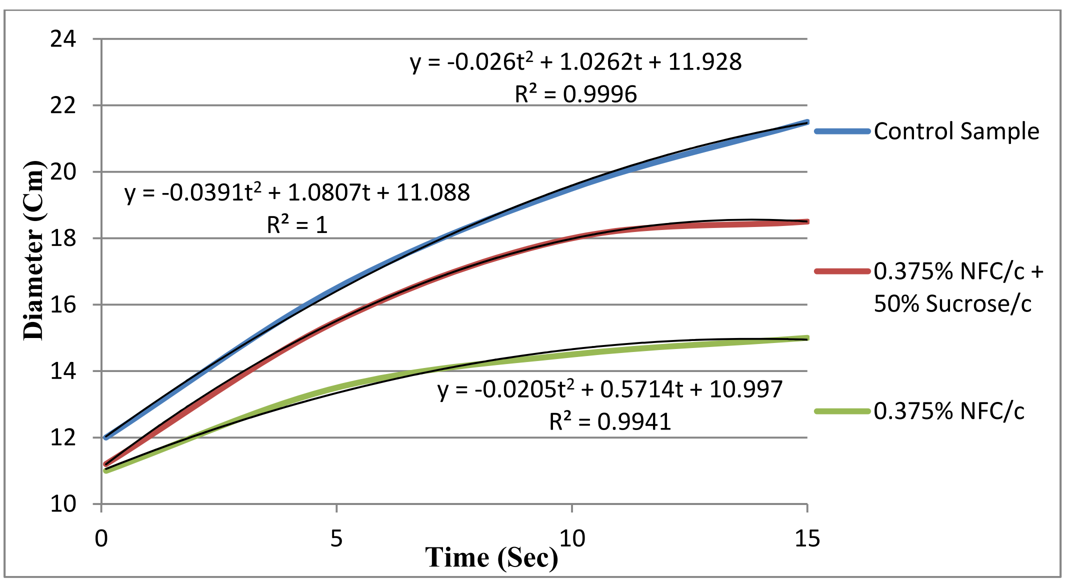


Figure 4-9 Spread vs. Time for different samples

Figure 4-10 shows spread of fresh cement paste versus drops for each three types of samples. According to the Figure 4-10, the diameter is increasing by number of drops. The polynomial plot was used to find the equation between the diameter and drops. For example, Figure 4-10 shows a polynomial correlation between spread and drops of the control sample with convergence factor of 0.999 % according to the following equation:

$$D \quad = 11.357 + 0.7228n - 0.0128n^2 \qquad [16]$$

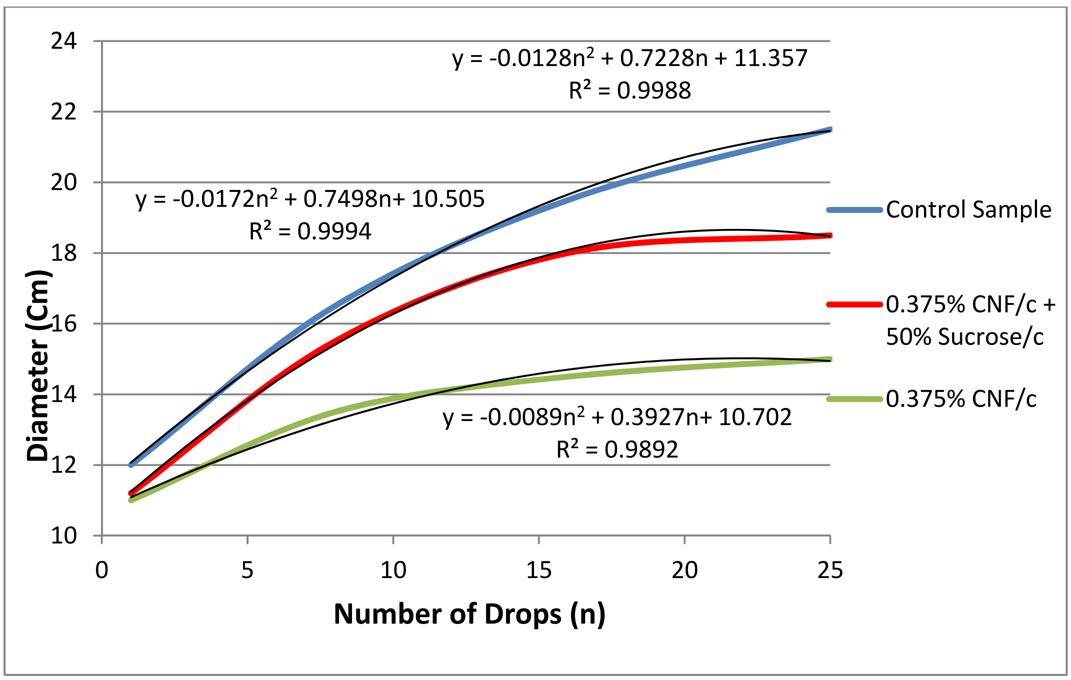


Figure 4-10 Diameter vs. Number of drops for different samples

Several tests have been conducted for the rheological behavior of cement paste. From the rheometer, the yield stress and viscosity have found for all of the specimens. Yield stress is found from the rheology software by using the Casson plot which was more fitted into our data to compare with the other plots such as

Bingham, power law, Herschel-Bulkley and Carreau. The Casson plot is driving from the equation as a following:

$$\tau^{\frac{1}{2}} = \tau_0{}^{\frac{1}{2}} + \mu^{\frac{1}{2}}\dot{\gamma}^{\frac{1}{2}} \qquad [17]$$

Viscosity is founds from the software for the shear rate in the low range less than 0.1 (1/s). Also from the camera recordings of mini slump tests, a spread evolution over time was obtained. The fitted data are listed in Table 4-4. To simplify the results, 5 different tests are chosen to find the correlation between rheological properties and spreading parameters, it is shown in Table 4-4.

Table 4-4 Yield stress, Viscosity and final spread values

Sample	**W/c**	**CNF/c**	**Sucrose/c**	**Yield stress**	**Viscosity**	**Final Spread**
#	%	%	%	Pa	Pa.s	cm
1	0.35	0	0	2406	23750	21.5
2	0.35	0	5	3052	3.04E+04	19
3	0.35	0.375	50	3597	3.42E+04	18.5
4	0.35	0.125	0	4249	4.13E+04	17.5
5	0.35	0.167	0	6785	4.94E+04	15

According to Table 4-4, five different samples have chosen to find the correlation between rheological behavior and time to final spread of fresh cement past. As it is shown in Table 4-4, the yield stress is increasing from control sample (sample 1) to control sample with 0.167% CNF/c (sample 5) and also viscosity is increasing with the same order as well as yield stress. However, it is different for time to final spread. Since, yield stress and viscosity are increasing the time to final spread is decreasing. For example, the control sample has the lowest yield stress; however its final spread is larger than the other samples.

The correlation between yield stress and final spread and also viscosity and final spread is plotted in Figure 4-11 and Figure 4-12.

4-5. Correlation between rheology test and flow test

4-5-1. Yield stress and final spread

Figure 4-11 shows yield stress versus spread of fresh cement paste for each five samples. According to the Figure 4-11, the yield stress is decreasing when final spread is increasing. The power and polynomial plot was used to find the equation between the final spread and drops. The polynomial plot was fitted better into our experimental data so that it has been chosen to describe the data. For example, Figure 4-11 shows a polynomial correlation between yield stress and final spread of the samples with convergence factor of 0.997 % according to the following equation:

$$\tau_y = 45590-3916.7D+88.71D^2 \quad [18]$$

Where the τ_y is the yield stress of the samples and D is final spread.

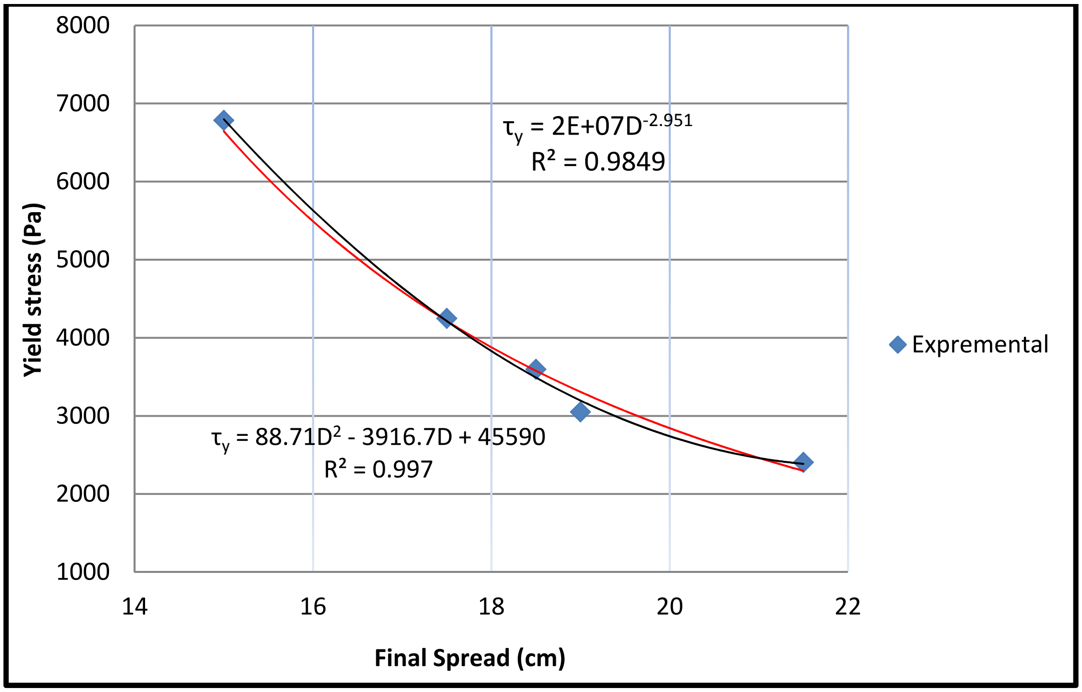


Figure 4-11 Yield Stress vs. final spread for different samples

4-5-2. Viscosity and final spread

Figure 4-12 shows viscosity versus spread of fresh cement paste for each five samples. The linear plot was used to find the equation between the viscosity and final spread. For example, Figure 4-12 shows a linear correlation between viscosity and final spread of the samples with convergence factor of 0.9671 % according to the following equation:

$$\mu=111244 - 4121.6D \qquad [19]$$

Where the μ is the viscosity of the samples and D is final spread.

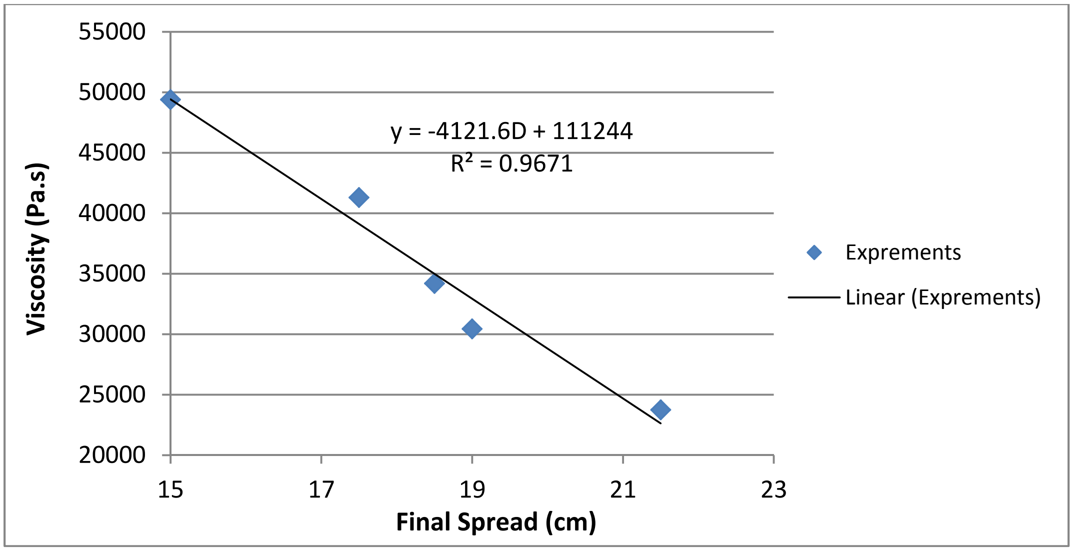


Figure 4-12 Viscosity vs. final Spread for different samples

4-5-3. Correlation between viscosity and drops (control sample)

Control sample has been chosen from all the samples as an example to find a relationship between the viscosity and drops.

First of all, from the camera (top view) the spread of control sample by drops has been recorded and it is shown in Figure 4-13.

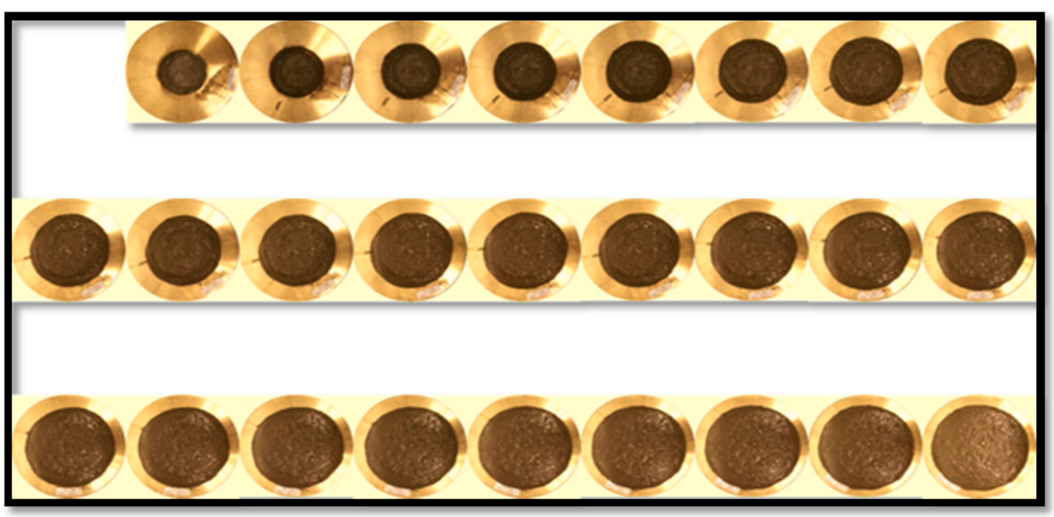


Figure 4-13 Spread time for cement paste

Figure 4-13 shows the spread of control paste where it is started from top left to top right and then from the first row to second and third. As it is shown, the diameter is getting bigger and bigger when drops happen. Therefore, each drop has its own unique spread diameter which is shown in Table 4-5. Then, using the equation [19] ($\mu = -4121.6D + 111244$) to calculate the viscosity from the final spread in each drops. Inputting the spread for each drop to the equation [6] and output is giving the viscosity for each time the sample drops, where the μ is viscosity and D is final spread. The results are shown in Table 4-5.

Table 4-5 Drops, Final Spread and viscosity for control sample

Drops	Spread	Viscosity	Drops	Spread	Viscosity
#	cm	Pa.s	#	cm	Pa.s
1	11	65906.4	**14**	19.4	31284.96
2	12.3	60548.32	**15**	19.6	30460.64
3	13.6	55190.24	**16**	20	28812
4	14.4	51892.96	**17**	20.1	28399.84
5	15.7	46534.88	**18**	20.3	27575.52
6	16.2	44474.08	**19**	20.6	26339.04
7	16.9	41588.96	**20**	20.7	25926.88
8	17.4	39528.16	**21**	20.9	25102.56
9	17.7	38291.68	**22**	21.1	24278.24
10	18	37055.2	**23**	21.2	23866.08
11	18.3	35818.72	**24**	21.3	23453.92
12	18.5	34994.4	**25**	21.5	22629.6
13	19	32933.6			

As it is shown, after each drop, the spread of fresh cement paste is getting bigger; however the viscosity is decreasing.

And finally, Thee last figure is plotted after inputting the final spread in to equation and extracting the viscosity.

Figure 4-14 shows a power and polynomial plots correlation between viscosity and number of drops for the control sample with convergence factor of 0.9954 % for polynomial plot and convergence factor of 0.9544 % for power plot. The polynomial plot is fitted to the experimental data so that it is been chosen according to the following equation:

$$\mu = 65423 - 3577.1n + 77.806n^2 \qquad [20]$$

Where the μ is viscosity and n is the number of drops for fresh cement paste.

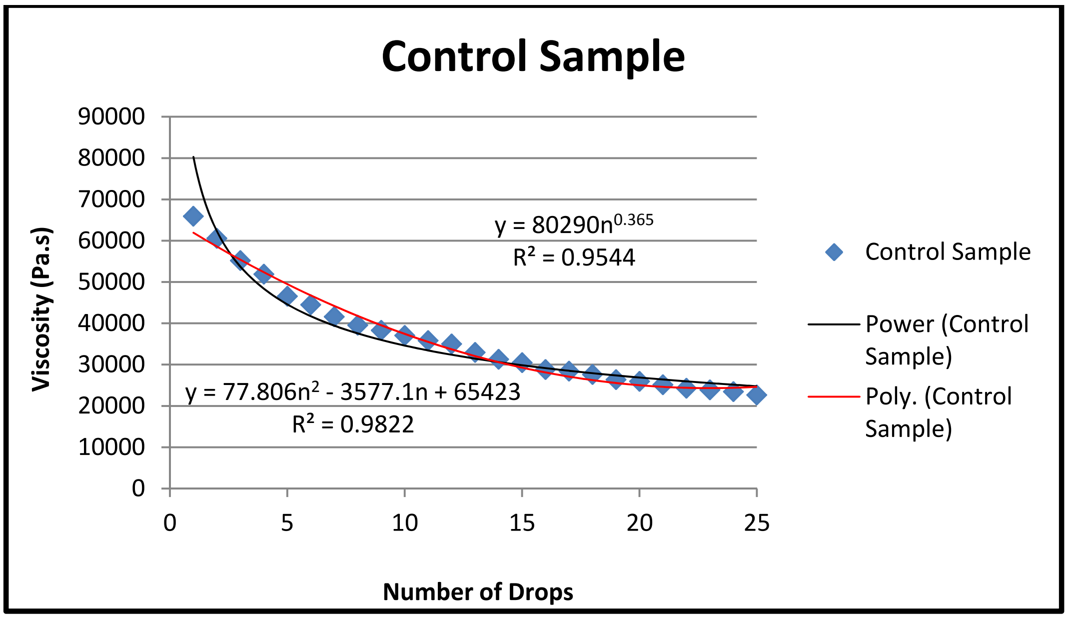


Figure 4-14 Viscosity vs. Number of drops for Control sample (special case)

The reason why the control sample is chosen to find the relationship between viscosity and drops is because drops are independent value for each viscosity. For example, two different samples with having two different viscosities cannot have the same final diameters after one drop. The reason is because the weight of samples and height of samples are the same; however they have a different viscosity and yield stress ranges. One drop can flow the sample different for the other one.

4-5-4. **Modeling rheology data and mini-slump flow test with the formulas for control sample (Specific case)**

From the slump flow table test some parameters are already known like mass of the specimen equals 0.7 kg, Δt equals $\frac{15\ (sec)}{25\ (drop)} = 0.6$ second and drop distance which equals 0.01 meter and shows in Table 4-6.

Table 4-6 Known parameters for control sample

Parameters	Unites	Amount
m	kg	0.7
Δt	sec	0.6
Drop Height	meter	0.01
R_f	cm	5.5
R_i	cm	4.5
R_{final}	cm	10.75
Δz	cm	5
h_t	cm	11.6

Acceleration of gravitation (g) is chosen 9.8 (m/s^2) and also pi (π) is chosen equals 3.14. Rf, Ri and R final are the radius of control sample relatively before first drop, after first drop and after 25 drops.

Some parameters such as ht and Δz calculated according to the cone geometry.

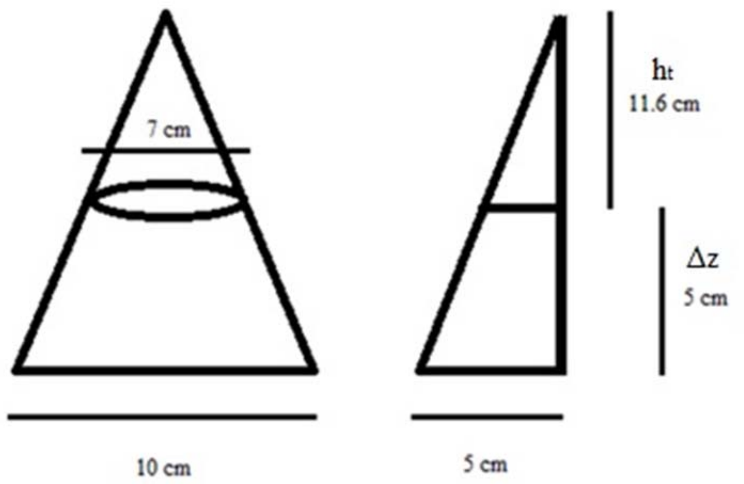


Figure 4-15 Cone Geometry

Figure 4-15 shows the cone geometry and it has used to calculate the volume of the cone. The volume of the cone is calculated using the equation [21]

$$V = \frac{\pi \times h}{12}\,(d^2 + db + b^2) \qquad [21]$$

Where, V is the volume of the cone, h is the height of cone, d is the top diameter of cone and b is the bottom diameter of the cone.

Then the density of the control sample is calculated using the equation [22]

$$\rho = \frac{wieght}{volume} \qquad [22]$$

Table 4-7 Calculated parameters

Parameters	**Unites**	**Amount**
Volume	(cm^3)	286.56
ρ	(kg/m^3)	0.2.443

The shear stress, shear rate, yield stress and viscosity can be calculated by using the known parameters such as volume, density and etc.

Equation [23] is used to calculate the shear stress for control sample.

$$\tau = \left(\frac{\rho \times g}{6}\right) \left[(z + h_t) - \frac{ht^3}{(z+ht)^2}\right] \qquad [23]$$

Equation [24] is used to calculate the yield stress for control sample.

$$\tau_0 = \frac{225 \times \rho \times g \times v^2}{128 \times \pi^2 \times R^5} \qquad [24]$$

Equation [25] is used to calculate the shear rate for control sample.

$$\dot{\gamma} = \frac{r_f - r_i}{\Delta z \times \Delta t} \qquad [25]$$

Is based on radius change, where r_f is the radius of the sample before first drop and r_i is radius of the sample after the first drop.

And then the viscosity of the control sample is calculated from the equation [26]

$$\mu = \frac{shear\ stress - yield\ stress}{shear\ rate} \qquad [26]$$

The results of shear stress, shear rate, yield stress and viscosity of control sample is calculated and is shown in table Table 4-8.

Table 4-8 Rheology parameters for cement paste

Test	Shear stress (Pa)	Shear rate (1/s)	Yield stress (Pa)	Viscosity (Pa.s)
Analytical	43.676	0.416	2.44	~100
Rheology	6566	0.416	2406	10000

According to the Table 4-8, the shear stress , shear rate, yield stress and viscosity for control sample have calculated from the analytical data, however the rheology data for these parameters have found from the rheology data using the AR-G2 Instrument. The data from the rheology have been selected with picking up at the same shear rate for control sample. As it is shown in Table 4-8, shear rate from the analytical data is equals to rheology data, but the shear stress, shear rate and viscosity are extensively high for rheology data.

According to the data, the model that has chosen to compare between the analytical and rheology methods is not good, because we did not predict the shear stress. Therefore, the model needs revision for further studies. In addition, the rheology procedure can be changed to get different data from the rheology. For example, the shear rate can be choose from low to high, where in this research the shear rate from high to low has been used.

Formulas which have viscosity and yield stress change independently are needed. For instance, new formulas can be found when sand or other materials are added into cement paste.

Chapter 5. Summary and Conclusion

5-1. Correlation between rheological properties and spreading parameters

In this part 21 samples tasted out on a dry horizontal plate while recorded by a video camera. Simultaneously, a sample of the cement paste was taken to the AR-G2 rheometer to determine the rheological properties.

In the rheology some parameters like rheology gap, temperature and applied force have not changed during the tests because to obtain reliable measures of cement paste rheology. Also, the mixing protocol for rheology tests and mini slump tests were with no changes for each tests. Also, for cement paste rheology, the measurements should be made in a variable geometry rheometer which parallel plate has been used in this research.

Finally, for the flow table test, the test was performed using the same procedure that is in ASTM C1437.

5-1-1. Yield stress and final spread

The yield stress is the most commonly measured value in fresh concrete or cement paste because it is related to the mini slump test. The yield stress has been found from different tests, however in this research the yield stress was found using the mini slump test where it has never been used before. The summary of the test result is coming in bellow:

$$\tau_y = 45590\text{-}3916.7D\text{+}88.71D^2$$

Where τ_0 is the yield stress of sample and D is the final spread of the sample. It means, using this equation to find the yield stress by having the final spread from the mini slump test.

5-1-2. Viscosity and final spread

Viscosity is the other value to measure in fresh concrete and cement paste from the mini slump test; however it has never been measure in mini slump test. In this research, the correlation between the viscosity and final spread was conducted and summary of result is brought in bellow:

$$\mu=111244 - 4121.6D$$

Where μ is the viscosity of sample and D is the final spread of the sample. It means, using this equation to find the viscosity by having the final spread from the mini slump test.

5-1-3. Viscosity and drops (control sample)

In this research, the correlation between the viscosity and drops for control sample were found using the rheology data and mini flow test data. In this research, to correlate these to parameters, first the equation from viscosity verses spread were used to find the viscosity for each spread and then it was correlated to drops. Finally, the viscosity of the sample during the 25 drops were found and plotted. The summary of result is shown in bellow:

$$\mu = 65423 - 3577.1n + 77.806n^2$$

μ is the viscosity of the sample and N is the number of drops only for control sample. It means, using this equation to find the viscosity by inputting the number of drops from the mini slump test.

These formulas are based on specific case, where we only have cement paste. For future research study on rheology behavior of cement with adding different kind of additives, these formulas cannot be used because viscosity and yield stress are independent parameters and they can be changed based on adding different additives into cement paste.

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