University of Alberta

INVESTIGATION OF THE FATE OF WASTEWATER PHOSPHORUS WITHIN THE PROCESSED KIMBERLITE CONTAINMENT AREA AT BHP'S EKATI™ DIAMOND MINE

by

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A thesis submitted to the Faculty of Graduate Studies and Research in partial fulfillment of the requirements for the degree of Master of Science.

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research for acceptance, a thesis entitled Investigation of the Fate of Wastewater Phosphorus within the Processed Kimberlite Containment Area at BHP's Ekati™ Mine submitted by Karen Patricia Graham in partial fulfillment of the requirements for the degree of Masters of Science in Environmental Engineering.

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ABSTRACT

The BHP Ekati™ Diamond mine discharges treated wastewater effluent to the processed kimberlite containment area. The goal of this research was to determine the adsorption of phosphorus on to the kimberlite portion of mine tailings going to the processed kimberlite containment area by conducting controlled laboratory isotherm investigations.

The results indicate that adding the wastewater effluent to the kimberlite mine tailings may be an effective method to adsorb the phosphorus found in the wastewater effluent. Using the Freundlich model, approximately 0.5 kg of dried kimberlite per litre of wastewater effluent is needed at a temperature of 18°C to reduce the concentration of phosphorus from 10 mg/L to 0.1 mg/L. The addition of coagulant and flocculant had no affect on the adsorption of phosphorus. Changes in pH and temperature did not cause any desorption of phosphorus.

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1 INTRODUCTION

The BHP Ekati™ Diamond mine is located 300 km northeast of Yellowknife. The mine is the first operating diamond mine in Canada. The permanent camp has a population of approximately 400 people. In the summer, when construction is occurring, the population may reach higher numbers. The wastewater from the camp and neighbouring worksites is collected and treated. In the past, the treated wastewater effluent was discharged into a nearby lake. The nutrients in the effluent were observed to increase algae growth and decrease dissolved oxygen levels in the lake as early as 1997 (Rescan, 2000). There was concern that the increased nutrient levels, particularly phosphorus, may affect downstream lakes, in the form of eutrophication and decreased winter dissolved oxygen concentrations (Rescan, 2000). To minimize the impact of increased nutrients, the discharge of the treated effluent was relocated to the processed kimberlite containment system.

Virtually nothing is known about the interaction between the clays and minerals that make up the processed kimberlite mixture and the wastewater effluent components, such as phosphorus. A preliminary bench scale study conducted by BHP found that the total suspended solids (TSS), phosphate (PO_4^{3-} and total phosphorus) and turbidity were reduced, beyond a straight mass calculated value, in the supernatant from a mixture of 1.9 parts wastewater to 100 parts processed kimberlite washwater (BHP, 1999). The measured values of 5-day, 20°C biochemical oxygen demand (BOD_5), ammonia (NH_3), nitrate and nitrite (NO_2^- and NO_3^-), total

kjeldal nitrogen (TKN), total organic carbon (TOC) and pH remained within 10% of the calculated values (BHP, 1999).

The objective of this research is to understand the advantages and possible future impacts of the addition of treated sewage to the processed kimberlite containment system. In particular, the adsorption of phosphorus on to the kimberlite component of the mine tailings was investigated. This study accomplished this objective by conducting controlled laboratory isotherm investigations. These investigations were performed at two different temperature conditions to represent the temperature extremes that the lakes in the area undergo throughout the year.

To accomplish the goals of this research, first equilibrium time experiments were completed at two different temperatures. These experiments would allow the determination of the contact time required for the phosphorus concentration to reach equilibrium. Second, adsorption isotherms were developed for the examination of the adsorption of phosphorus by processed kimberlite. The phosphorus adsorption isotherms were used to evaluate the fate of phosphorus in the processed kimberlite area. Third, the effects of parameters such as temperature, pH, and coagulant and flocculant addition were also determined. Finally, experiments were also conducted to examine the potential for the release of the phosphorus back into the water column of the processed kimberlite containment area. These experiments consisted of conducting desorption tests.

1.1 Diamond Mine Information

Mines remove ore-bearing rock from the ground. This rock has to be processed to remove the precious ore. The crushed rock remaining after ore removal is called tailings.

The majority of natural diamonds have been derived from deposits of kimberlite (BHP and DIA MET, 1995). For mineral processing involving diamonds there are three main operation units that are used in the reduction and diamond recovery process to separate the diamonds from the surrounding kimberlite. The kimberlite ore is first crushed and scrubbed, then concentrated by heavy medium separation and finally concentrated by X-ray sorting (BHP and DIAMET, 1995). The concentration of diamonds in the kimberlite ore is only one to two parts per 10,000,000 parts at the Northwest Territories deposit (BHP and DIA MET, 1995). This results in large amounts of material that must be processed to recover the diamonds.

Crushing, scrubbing and screening are done to reduce the size of the rock to be processed and to remove soft fines and clay minerals from the crushed ore. The specific gravity of diamond is 3.52 while that of the waste rock is only 2.7 and the kimberlite is 2.2 to 2.4 (BHP and DIA MET, 1995). This density difference allows the use of density separation processes. The heavy medium separation (HMS) step separates high density particles (including the diamonds) from low density particles through gravity separation. In this step, ground ferrosilicon and water is added to the ore to form a slurry in the HMS unit. The ferrosilicon is a suspension medium of

high density that enhances the separation of high and low density particles (BHP and DIA MET, 1995). The majority of the ferrosilicon is recovered and reused (BHP and DIA MET, 1995).

The recovery process involves further concentration by X-ray sorting. Diamonds luminesce under X-rays, which allows separation of luminescent material (mostly diamond) from non-luminescent material (no diamonds).

1.1.1 Ekati™ Mine Tailings

Tailings are produced in each of the concentration steps described in Section 1.1. Fine tailings and coarse tailings are treated separately at the BHP site (BHP and DIA MET, 1995).

The fine tailings are thickened to allow process water to be recycled and to reduce the volume of tailings to be disposed. This entire process is illustrated in Figure 1-1. A coagulant and flocculant are added to the thickener to encourage flocculation and settling. At the time samples were obtained, the flocculant was Magnafloc 156 and the coagulant was Magnafloc 368. They will be referred to as coagulant and flocculant throughout the rest of this work. The thickener overflow is recycled and used as process water. The underflow is sent to a storage tank prior to disposal. The wastewater effluent and other effluents are added to the storage tank prior to pumping to the containment area.

The fine tailings consist of kimberlite rock, coagulants and minor amounts of acid generating materials and heavy metals (BHP and DIA MET, 1995).

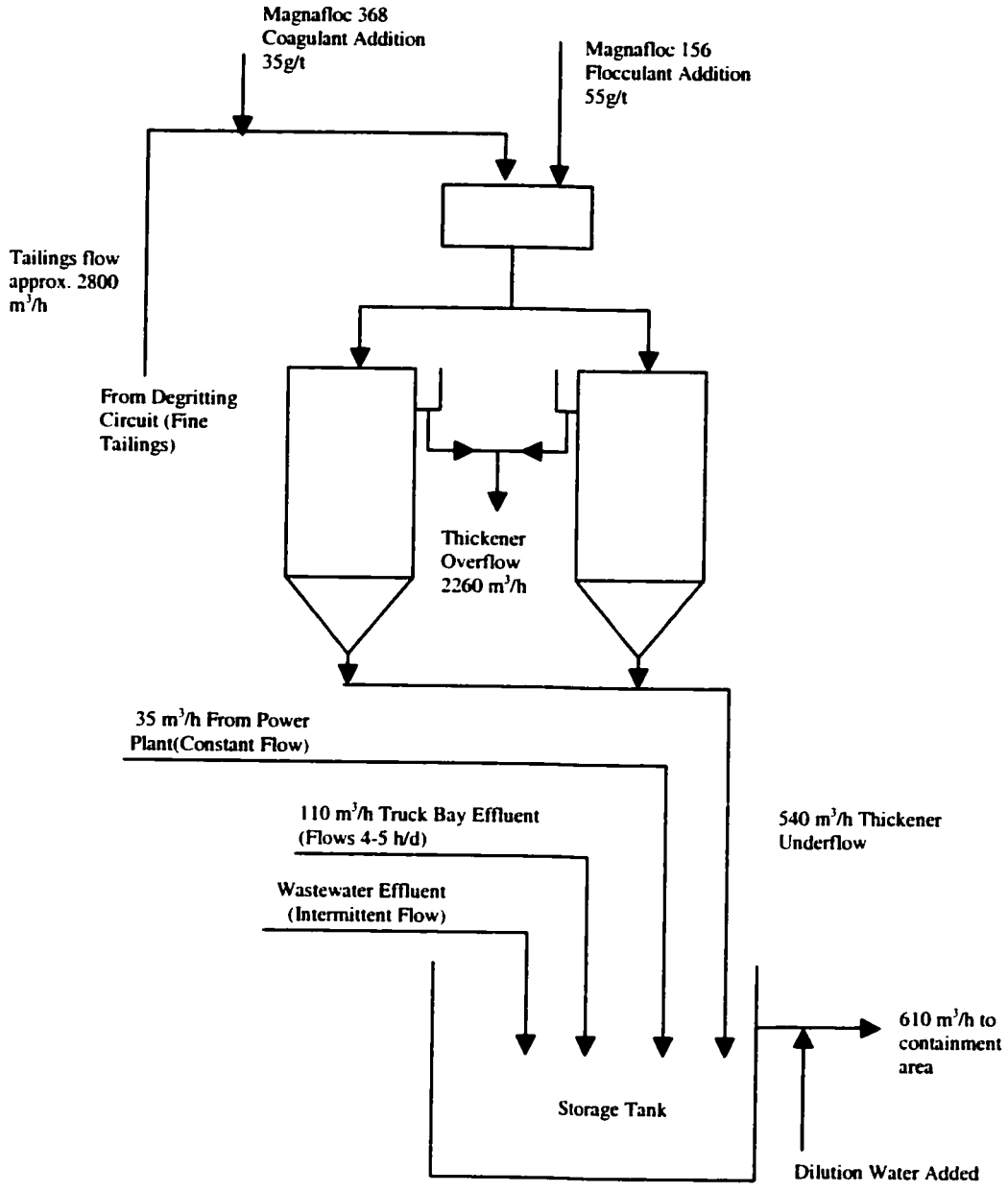

Figure 1-1 Mine Tailings Process Diagram

Approximately 60 percent of the total ore that is processed becomes fines. More than 75% of the fine tailings are generally sand size particles although it is anticipated that clay and silt contents may be in excess of 50% on occasion (BHP and DIA MET, 1995).

The fine tailings must be treated and/or disposed. The most common method used by mines is the use of tailings ponds (also called containment areas or impoundment ponds) to store the processed waste rock. If the tailings do not contain enough water, water must be added so that the tailings can be transported as slurry through pipes from the process plant to the tailings pond. Currently, BHP uses Long Lake as its fine tailings disposal area.

The purpose of tailings ponds is to remove the solids in the tailings by sedimentation. The ponds are used to contain the settled tailings and other contaminants. The water in the tailings pond is sometimes recycled to be used in the processing procedure as process water. The unsettled portion may be impounded indefinitely or until the water can meet effluent criteria. At the time this thesis was written, there were no restrictions on the phosphorus concentration of the effluent. The current criteria only consider suspended solids content.

The fine tailings are currently disposed of in Long Lake and will be for the first 20 years of operation at Ekati™ (BHP and DIA MET, 1995). Long Lake was divided into containment cells. The cells will be filled starting with the uppermost cells and moving to the lowermost cells as the previous cells are filled. The final cell will not receive tailings but will act as a final clarifier (BHP and DIA MET, 1995).

1.1.2 Fine Tailings Water Balance

It is very important to know all inputs that are going into the mine tailings storage tank prior to the mine tailings disposal in the containment area. It is known that the mine tailings, water from the power plant, water from the truck bay and wastewater effluent all flow into the storage tank (See Figure 1-1). The water from the truck bay and the wastewater effluent are intermittent flows whose flow rates are not currently measured. Both of these flows will contain phosphorus. It is important to understand how much phosphorus is going into the storage tank to understand its fate within the containment area.

The volume of water from the truck bay and its phosphorus concentration is unknown. It will be assumed that the volume is small in comparison to the volume of mine tailings. Its phosphorus contribution is therefore negligible.

The volume of wastewater will be estimated by assuming a certain wastewater production per capita. The phosphorus concentrations were determined from the Technical Reports provided by Alpha Laboratory Services Ltd (1999-2000).

1.1.3 Ekati™ Wastewater Treatment System

An extended aeration system is used to treat the wastewater generated at the Ekati™ site. This system was chosen due to its simple design and operation. There are three basic parts to the system: an aeration tank, a clarifier and a sludge storage tank. In the aeration tank, microorganisms use components of the wastewater as a food source. The solids formed tend to clump together to form sludge. Oxygen is added to this tank to supply the necessary oxygen for the microorganisms. The

sludge is separated by gravity in the clarifier and a portion of the sludge is recycled back into the aeration tank to speed up the treatment process. The excess sludge is held in an aerated sludge storage tank until land disposal.

1.2 Sorption

Sorption involves the assimilation of molecules of one substance by a material in a different phase. Adsorption (sorption on a surface) and absorption (sorption into bulk material) are two types of sorption phenomena. It is often difficult to determine which phenomenon is actually occurring.

Adsorption of a substance involves its accumulation at the interface between two phases, in this case between liquid and a solid. The molecule that accumulates, or adsorbs, at the interface is called an adsorbate, and the solid on which adsorption occurs is the adsorbent. Adsorbents of interest in water treatment include activated carbon, ion exchange resins, adsorbent resins, metal oxides, hydroxides, and carbonates, activated alumina, clays and other solids (AWWA, 1990). Adsorbents are held on the surface by various types of chemical forces such as hydrogen bonds, electrical interactions (cation/anion exchange, ligand exchange and di-polar bonds) and van der Waals forces (AWWA, 1990).

Absorption involves the penetration of molecules into the bulk of a solid or liquid, forming either a solution or compound.

1.2.1 Phosphorus Sorption by Clays

It is known that phosphorus in the form of phosphate is retained by soils through ligand exchange (He et al, 1997). This suggests that adsorption, not absorption is occurring. For this reason, the removal of phosphorus by kimberlite tailings is assumed to occur due to adsorption not absorption.

In studies conducted by Henmi and Huang (1995), the sorption of phosphorus by clays was examined. Phosphorus removal was determined to occur in the range of 2.25 to 10.1 mg P/g of clay depending on the type of clay used as the adsorbent (Henmi and Huang, 1995).

The removal of phosphorus by clays increases as the aluminum content of the clay increases (Henmi and Huang, 1995 and Clark and McBride, 1984). However, if the clay has a high organic content, its adsorption of phosphorus decreases no matter what aluminum content is present (Clark and McBride, 1984).

1.3 Kimberlite

Kimberlite is a volcanic rock. The kimberlite formation process results in a carrot-shaped pipe called a diatreme (BHP and DIA MET, 1995). Figure 1-2 shows an idealized view of a kimberlite pipe. The majority of natural diamonds have been derived from deposits of kimberlite (BHP and DIA MET, 1995). The diamonds have to be removed from the surrounding kimberlite rock. The diamond content varies

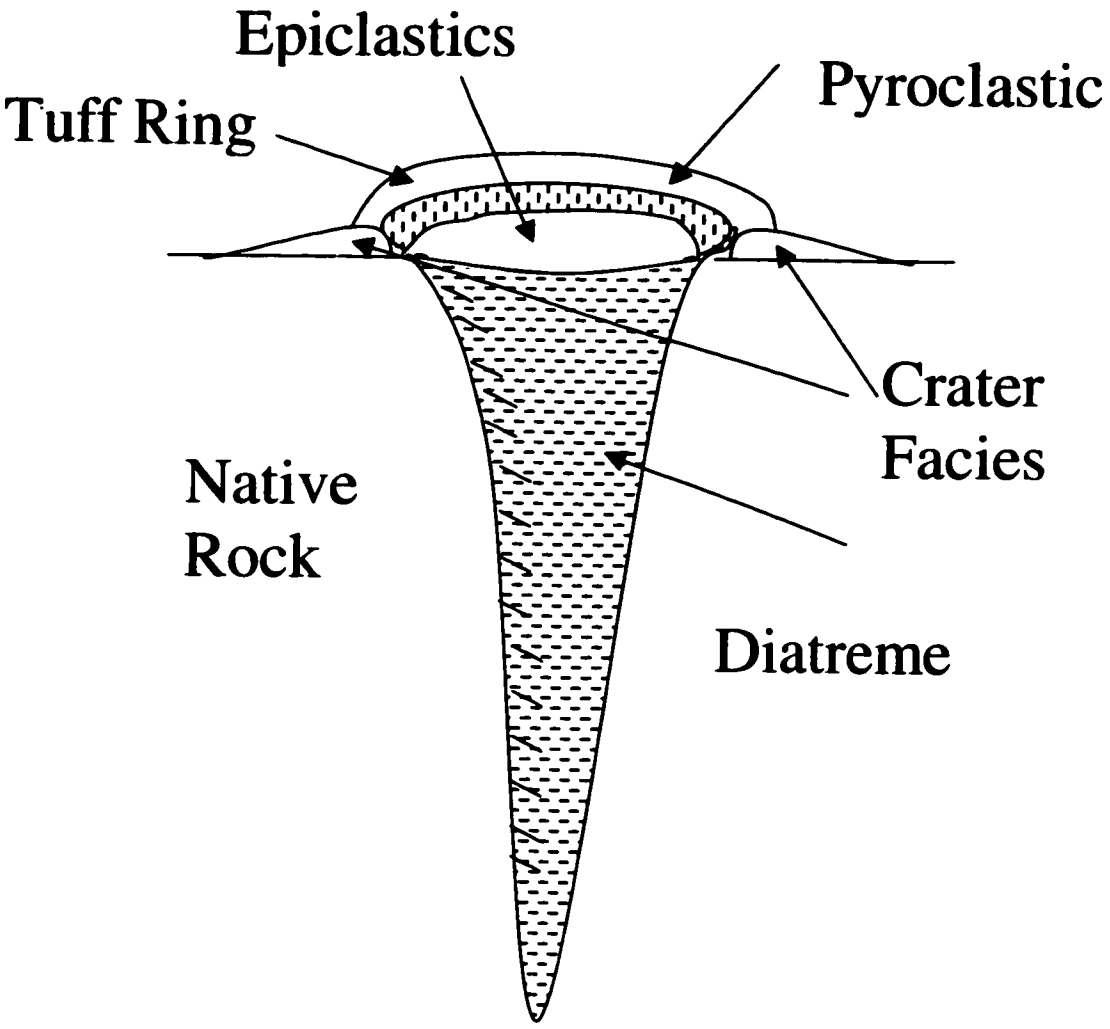

Figure 1-2 Idealized Kimberlite Pipe

throughout the kimberlite pipe. The diamond bearing kimberlites in the Northwest Territories were the first found in North America. The mineralogy of the kimberlite varies both vertically and horizontally throughout the pipe. The five kimberlite pipes that are currently approved for mining in the BHP project exhibit a wide range of characteristics (BHP and DIA MET, 1995).

The clay is an important component of the kimberlite for this study. As stated in Section 1.1.1, clay particles make up at least 25% of the kimberlite in the mine tailings. First, the clay is the source of the fine colloidal suspensions of solids that take a very long time to settle and secondly, and more importantly, the clay is a more powerful adsorbent than the silt and the sand.

1.3.1 Kimberlite Adsorption

The use of kimberlite tailings as an adsorbent was examined in two recent research articles. The first article by Sudhakar and Dikshit (1999) compared the endosulfan removal performance of several different materials, including kimberlite tailings, to activated charcoal. Endosulfan is a pesticide that is commonly used in agriculture to control common garden pests. The purpose of the study was to evaluate low cost adsorbing material for the removal of endosulfan from water. The results from batch experiments were used to develop adsorption kinetic profiles, to determine equilibrium time and to develop adsorption isotherms. The materials evaluated were wood charcoal, kimberlite tailings, silica and macro fungi *sojar caju*. All of the adsorbents were prepared using the same method. The procedure was as follows:

1. Clean raw material thoroughly with distilled water.
2. Pulverize the washed and dried material.
3. Sieve to obtain a mean size of 0.200 mm.
4. Wash with distilled water to remove fine materials.
5. Dry at 110°C for 10 hours.

6. Cool in a dessicator to room temperature.
7. Re-sieve the material to required size.
8. Store material in airtight bottles.

To develop adsorption kinetic profiles, endosulfan spiked water of a specified concentration was placed in polyethylene bottles with an adsorbent dose of 20 g/L. The bottles were shaken on a mechanical shaker at 150 rpm. Samples were withdrawn at certain time intervals over a 24 hour period to determine the resulting endosulfan concentrations. The kimberlite tailings were found to remove 86.5% of the endosulfan. The non-homogeneous texture of the kimberlite tailings was reported as the cause of its relatively poor performance in comparison to the activated charcoal whose removal was 94.5% (Sudhakar and Dikshit, 1999). It was determined that the major part of the adsorption occurred within the first 2 hours of contact (Sudhakar and Dikshit, 1999). A contact time of 6 hours was deemed to be adequate to achieve equilibrium concentrations for all adsorbents, including the kimberlite tailings (Sudhakar and Dikshit, 1999).

To develop the adsorption isotherms, varying concentrations of endosulfan were dosed with 20 g/L of adsorbent. The sample bottles were shaken for a period of 6 hours prior to the measurement of equilibrium endosulfan concentration. In Sudhakar and Dikshit's study, the adsorption of endosulfan onto the kimberlite tailings was modeled using the BET (Brunauer, Emmet and Teller) model. The maximum adsorptive capacity of the kimberlite mine tailings was 0.8821 mg/g

(Sudhakar and Dikshit, 1999). This adsorption capacity was higher than silica but much lower than wood charcoal and *sojar caju*.

A second article by Dikshit et al. (2000) investigated the adsorption of arsenic onto kimberlite tailings. Batch and column studies were conducted to determine the feasibility of using kimberlite tailings in an adsorption column to remove arsenic from groundwater. The effects of pH, adsorbent dose and adsorbent particle size on the arsenic removal performance of the kimberlite tailings were studied. Batch desorption studies were also conducted by saturating kimberlite with arsenic and placing the arsenic containing kimberlite in distilled water. The chemical composition of the kimberlite tailings was determined by Inductively Coupled Plasma (ICP) spectroscopy.

The Freundlich isotherm was used to determine the maximum adsorptive capacity. For arsenic, the adsorption capacity of the kimberlite tailings was 0.25 mg/g for the batch study and 0.27 mg/g for the fixed bed study (Dikshit *et al.*, 2000). The adsorption of arsenic was found to be pH dependent, with higher adsorption capacities near the neutral pH range (Dikshit *et al.*, 2000). The performance of the kimberlite tailings was compared with activated alumina. The adsorption capacity of kimberlite for arsenic was found to be one quarter of the adsorption capacity of activated alumina in column studies (Dikshit *et al.*, 2000). The kimberlite tailings were prepared by the same procedure used in Sudhakar and Dikshit (1999).

It was found that 50% of the adsorption of arsenic was completed in 2 hours of contact time (Dikshit *et al.*, 2000). After 8 hours, there was 91% removal of the

arsenic by the kimberlite tailings (Dikshit *et al.*, 2000). A 10 to12 hour contact period was used to ensure equilibrium conditions in the rest of the experiments (Dikshit *et al.*, 2000).

Arsenic removal was found to increase rapidly with adsorbent dose up to a dose of 20 g/L of kimberlite. Beyond a 20 g/L dosage, an increase in the removal of arsenic was found to be marginal with increasing dose (Dikshit *et al.*, 2000).

The removal efficiency of arsenic was found to increase as the absorbent particle size was decreased (Dikshit *et al.*, 2000). However, the study only used two different mean particle sizes (0.212 and 0.387 mm).

Both studies (Sudhakar and Dikshit, 1999; Dikshit et al., 2000) show that kimberlite has the ability to adsorb endosulfan and arsenic but not to the same extent as commercially available adsorbents such as activated charcoal and activated alumina. Since kimberlite was found to adsorb both endosulfan and arsenic, it is probable that kimberlite will also adsorb phosphorus.

1.4 Clays

Since clay is a very important constituent of kimberlite, the kimberlite will have to be analyzed for its clay content. This section discusses clays and the available techniques for characterizing clays. There are two particle categories in soils: granular and colloidal. The granular particles are gravel, sand and silt while the colloidal particles are clay-sized particles.

There are different methods to characterize both of these types of particles in soil. The methods for characterizing clay size particles include: X-ray diffraction and fluorescence, differential thermal analysis, optical (microscope) study of aggregates, electron microscopy, chemical methods, surface area determination and infrared spectroscopy. No one method is satisfactory for the identification of a variety of minerals in soils due to the interaction and interference of the minerals during measurements (Yong and Warkentin, 1975). Several methods are usually used to identify the minerals that are present (Yong and Warkentin, 1975).

The properties of clays vary spatially in clay deposits (van Olphen and Fripiat, 1979). The variation in properties may be due to differences in overburden pressure, extent of weathering and other factors (van Olphen and Fripiat, 1979).

The following sections outline techniques that are currently being used to characterize clays.

1.4.1 X-Ray Diffraction

This method is the most commonly used method of identification of clay minerals (Yong and Warkentin, 1975). The diffraction lines can identify the minerals that are present in the clay. Clays consist of crystals. Different clays have crystals of different dimensions. Which therefore allows different minerals to be identified by X-ray diffraction. The most common method to accomplish this is to use a Light Element Energy Dispersive X-ray Analysis (EDX) attachment on a scanning electron microscope (SEM).

1.4.2 Differential Thermal Analysis

Differential thermal analysis (DTA) determines the temperature at which changes occur in a mineral when it is heated continuously to a high temperature (Yong and Warkentin, 1975). The intensity of the change can be related to the amount of mineral that is present. DTA is based on the fact that clays lose water or go through phase changes that either require or give off heat. The temperatures at which these reactions occur are indicative of the mineral that is involved and can be used to identify the minerals.

1.4.3 Infrared Spectroscopy

Clay minerals have adsorption bands in the infrared region of the energy spectrum because the molecular bond vibration frequencies are in the infrared region. The absorption frequencies can be used to identify the molecular bonds, which allow the type of mineral to be determined (Yong and Warkentin, 1975). Many of the bonds are not specific to one type of clay mineral and other methods of characterization of clay would be necessary to identify the clay (Yong and Warkentin, 1975).

A variation of infrared spectroscopy is inductively coupled plasma (ICP) spectroscopy. The sample to be analyzed, if solid, is normally first dissolved and then mixed with water before being fed into the plasma. Atoms in the plasma emit light (photons) with characteristic wavelengths for each element. This light is recorded by optical spectrometers and then calibrated against standards. The technique provides a quantitative analysis of the original sample.

1.4.4 Electron Microscopy

Transmission and scanning electron microscopy can be used to study the shape and arrangement of clay particles (Yong and Warkentin, 1975). This method involves visually comparing the shape and arrangement of clay particles to photographed standards. This method is time consuming and requires knowledge of standard clay shapes and arrangements.

1.4.5 Surface Area

The surface area of clay particles varies with the different type of clay minerals. The activity of clay, such as its adsorption of water, increases with increasing surface area. The surface area is determined by measuring the amount of a liquid or a gas that is required to cover the surface of the clay (Yong and Warkentin, 1975). Water vapour, nitrogen and organic liquids have been used. The majority of the differences in properties between clay minerals such as water retention or plasticity can be explained by the differing surface areas (Yong and Warkentin, 1975). Surface area is not a commonly used measurement because it is very time consuming. The liquid limit is closely related to surface area and is often used to estimate the surface area because it is much simpler to measure (Yong and Warkentin, 1975).

1.4.6 Cation Exchange Capacity

The cation exchange capacity of clay is generally understood to be equivalent to the layer charge (van Olphen and Fripiat, 1979). Exchangeable cations are positively

charged ions that are attracted to the surface of the clay particles to balance the negative charge of the clay. They are called exchangeable because another cation or cations can easily replace one cation or cations. The quantity of exchangeable cations held by the clay is the cation exchange capacity and equals the negative charge of the clay.

The capacity is measured by determining the total amount of cations in exchange positions on the clay surface. Most methods replace the various cations that are present on the natural clay surface with a single cation. The total amount of this cation is determined by measuring the total amount of the single cation species after washing the clay. Two cations that are commonly used are ammonium and barium.

1.5 Performance Evaluation of Adsorbents

The performance evaluation of adsorbents is conducted by several different methods depending on the properties of the substance to be removed by the adsorbent. Different substances are used in the tests to mimic the behaviour of the actual substance being adsorbed. The following sections describe two different methods currently being used to evaluate adsorbents. After investigating both of these tests, it was determined that neither was relevant to the adsorption of phosphorus by the kimberlite because both of these tests mimic the ability of an adsorbent to adsorb substances with high molecular weights. Section 1.5.3 will discuss adsorption isotherms.

1.5.1 Tannin Test

The tannin test is a measure of an adsorbents ability to remove high molecular weight impurities (large organics). It is a jar test procedure that is described in AWWA B600-78 Standard. The tannin value is defined as the dosage of adsorbent required to reduce a standard tannic acid concentration from 20 mg/L to 2.0 mg/L.

1.5.2 Methylene Blue Adsorption

Methylene blue was initially used to evaluate the ability of an adsorbent to remove colour from solution. It is now used to determine the ability of an adsorbent to remove larger molecules. The methylene blue number is the mass of methylene blue adsorbed by 1.0 g of adsorbent at a residual concentration of 1.0 mg/L. Methylene blue has a molecular weight of 319.9 g/mol and its structure is $C_{16}H_{18}N_3ClS$ as compared to phosphorus, which is most commonly found as PO_4^{-3}, HPO_4^{-2}, and $H_2PO_4^{-}$ in wastewater effluent.

1.5.3 Adsorption Isotherms

An adsorption isotherm describes the distribution of the solute between the liquid phase (solution) and the solid phase (in this research, the kimberlite or processed kimberlite mixture) at a constant temperature. This isotherm represents the relationship between the amount of a substance adsorbed to the absorbent and the amount of that same substance in solution at equilibrium. This relationship can allow the determination of the amount of solute that is adsorbed per mass of adsorbent under a given set of conditions.

There are several models that can be used to describe adsorption. The two most common models are the Freundlich and Langmuir equations. The Freundlich model is the more commonly used model for environmental applications. The Langmuir model is used to describe single layer adsorption while the Freundlich equation has the ability to describe multi-layer adsorption.

The Freundlich Equation is outlined in Table 1-1. Taking the logarithm of both sides of the Freundlich equation linearizes the equation, allowing the model parameters to be easily determined (Table 1-1). In the Freundlich model, the constants K and $1/n$ are designated as the Freundlich parameters. These parameters can be estimated by performing a linear regression. K has been used as an indicator of the adsorption capacity of the adsorbent. As the value of K increases the adsorbent capacity of the adsorbent increases. $1/n$ has been used as an indicator of the strength of adsorption. As the value of $1/n$ decreases the strength of the adsorbent bonds increases.

The Langmuir equation is outlined in Table 1-2. Rearranging the Langmuir equation allows the model parameters to be easily determined (Table 1-2). The constant q_{max} represents the maximum value of q_e that can be achieved as C_e is increased (i.e. when the monolayer is saturated). The constant b is related to the energy of adsorption. As the strength of the adsorption bond increases the value of b increases.

Table 1-1 Freundlich Equation

$$q_e = KC_e^{1/n}$$

And: $q_e = \frac{C_o - C_e}{M}$

Where: C_o: initial solute concentration (mg solute/L)

C_e: equilibrium solute concentration (mg solute/L)

M: adsorbent dosage (g adsorbent/L)

q_e: adsorbent loading (mg solute/g adsorbent)

K: constant, Freundlich parameter (L/g adsorbent)

$1/n$: constant, Freundlich parameter (unitless)

In linearized form:

$$\log q_e = \log K + \frac{1}{n}\log C_e$$

Where: log K= ordinate intercept at Ce = 1.0

$1/n$ = slope

Table 1-2 Langmuir Equation

$$q_e = \frac{q_{max} b C_e}{1 + b C_e}$$

And: $q_e = \frac{C_o - C_e}{M}$

Where: C_o: initial solute concentration (mg solute/L)

C_e: equilibrium solute concentration (mg solute/L)

M: adsorbent dosage (g adsorbent/L)

q_e: adsorbent loading (mg solute/g adsorbent)

q_{max}: constant, saturation coefficient (mg solute/g adsorbent)

b: constant (L/mg)

In linearized form:

$$\frac{C_e}{q_e} = \frac{1}{q_{max} b} + \frac{C_e}{q_{max}}$$

Where: q_{max}= 1/slope

b= $1/(q_{max} * y_{intercept})$

2 MATERIALS AND METHODS

2.1 Kimberlite Characterization

The Scanning Electron Microscope (SEM) in the Earth and Atmospheric Sciences Department at the University of Alberta is equipped with Light Element Energy Dispersive X-ray Analysis (EDX) capability. This feature allows an elemental analysis of a sample. This method allows a range of particle sizes to be analyzed and is not limited to only clay particles. This method was chosen in part due to equipment availability. For these reasons, the EDX method was chosen to characterize the kimberlite instead of the other clay characterization methods discussed in Section 1.4.

The EDX capabilities of the SEM were used to conduct an elemental analysis of kimberlite tailings samples. ICP Spectroscopy was also used to validate the EDX results. The following sections will discuss the methods used to prepare and analyze the kimberlite tailings. The results of these analyses will be reported in Section 3.1.

2.1.1 Kimberlite Tailings Samples

Samples of the fine tailings were obtained from three different locations along the tailings process (Figure 2-1). Location 1 was chosen to obtain a kimberlite tailings sample free of any coagulant and flocculant. Location 2 was chosen to obtain a kimberlite tailings sample containing coagulant and flocculant, prior to the addition of truck bay effluent, wastewater effluent and power plant effluent. Location 3 was chosen to obtain a sample that represents the final tailings effluent.

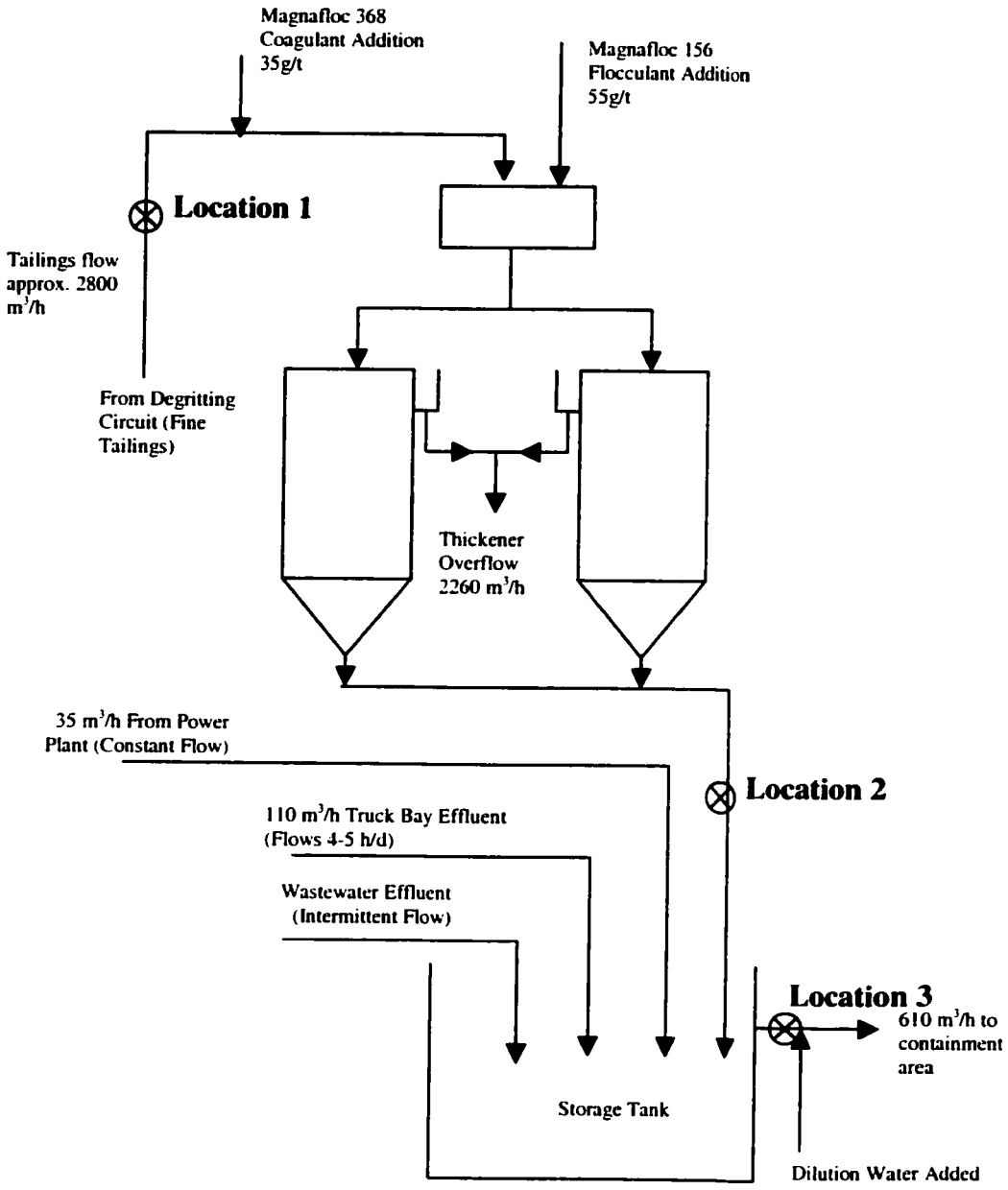

Figure 2-1 Sampling Locations

An initial sample was obtained from Location 3 in April 2000. This sample will be referred to as April Final Effluent (AFE). A sample from Location 1 was taken in June 2000 will be referred to as June Thickener Feed (JTF). A sample from Location 3 was taken in June 2000 will be referred to as June Final Effluent (JFE). Approximately 50 L of sample was collected at each location. The 50 L samples were sealed in 100 L drums and shipped to the Environmental Engineering Building at the University of Alberta. The samples were stored in a temperature controlled room at 5°C.

2.1.2 Obtaining a Representative Kimberlite Tailings Subsample

During shipping and storage, settlement of the larger particles in the kimberlite tailings occurred in the sample drums. The solids therefore needed to be resuspended by mixing before any subsamples could be removed. The mixing was accomplished by stirring the drum with a big stir stick to dislodge as much of the solids from the bottom of the drum as possible. Once the majority of solids were in suspension, a hand held electric rotor was used to keep the solids suspended long enough to ensure a homogenous solution was created. When the solution appeared to be homogenous (after about 10 minutes), approximately 2 L of kimberlite tailings was removed. This procedure was followed whenever a subsample of kimberlite tailings was required.

2.1.3 Light Element Energy Dispersive X-ray Analysis (EDX)

George Braybrook, the SEM technician, suggested that the kimberlite tailings sample be sieved to separate it into four different size fractions. This separation was done to ensure that the smaller particles of soil would not be blocked out by the larger particles when the X-rays were directed at the sample. The four different size fractions were: greater than 355 μm, 150 to 355 μm, 45 to 150 μm and less than 45 μm. These size ranges were chosen according to the available sieve sizes.

The kimberlite tailings were separated into the four different size fractions by wet sieving. Three sieves were stacked on top of one another with the largest sieve size on top and the smallest sieve size on the bottom. A portion of tailings was added to the top sieve. The tailings were wet sieved by rinsing the sieves with de-ionized water until soil particles were no longer passing through the sieves. The particles passing through the bottom sieve were collected as the smallest size fraction (<45 μm). The kimberlite tailings particles were removed from the sieve by flipping the sieve upside down and rinsing with de-ionized water. The resulting kimberlite tailings and water mixture was placed in a drying oven to evaporate the water. A grab sample of each size fraction was mounted on a 12 mm diameter stub that was covered with carbon tape. This stub could be placed into the SEM for analysis after being coated with gold.

An elemental analysis was completed on three different samples. The three samples analyzed were i) the AFE, ii) the JTF and iii) the JFE. These three different samples were chosen to attempt to determine whether there was a significant

difference in the elemental composition of the fine tailings at different times and locations within the processing plant.

The results of the elemental analysis are more accurate when a smaller area on the stub is analyzed. For this reason, four different areas of each stub were analyzed to obtain a more representative elemental composition. For each sample and size fraction, a duplicate stub was also analyzed. The reported results are an average of the eight areas that were analyzed. The results of the elemental analysis are reported as an atomic percentage. The results are reported in Section 3.1.1. After a size fraction analysis was completed, the results from each size fraction were weighted accordingly to obtain an overall elemental composition for each sample.

2.1.4 ICP Spectroscopy

ICP Spectroscopy was used to determine the chemical composition of the kimberlite tailings. A subsample of the Thickener Feed sample received from BHP in June 2000 was obtained according to the procedure discussed in Section 2.1.2. Norwest Labs in Edmonton analyzed this subsample using ICP Spectroscopy. The results are reported in Section 3.1.2.

2.2 Kimberlite Preparation for Adsorption Experiments

2.2.1 Preliminary Experiments

The kimberlite tailings preparation techniques discussed in Section 1.3.1 were used as a starting point for the preparation techniques of this study. However, the

kimberlite tailings in that research by Sudhakar and Dikshit (1999) and Dikshit et al. (2000) were sieved to obtain a consistent particle size. The kimberlite tailings from the Ekati™ mine consist of a wide range of particle sizes. The wastewater effluent comes into contact with this range of particle sizes in the kimberlite tailings. This range of particle sizes should be maintained throughout the experiments to represent the conditions that actually occur at the mine site.

Preliminary studies were conducted in order to become familiar with the equipment and methods involved with determining phosphorus concentrations. The procedure for preparing the kimberlite originally included drying a subsample of kimberlite in an oven at 110°C. During this drying, there is a possibility that substances in the water may precipitate onto the adsorption sites of the soil particles in the kimberlite. The substances of concern were any substances that are routinely found in water, such as calcium, magnesium, sodium, potassium, chloride and sulphate. This precipitation could directly affect the ability of the kimberlite to adsorb phosphorus.

One possible solution to this problem was to centrifuge the samples to separate the soil particles from the water. The next step was to determine how many rinse cycles were required to remove the substances in the water that could precipitate onto the kimberlite. As a first attempt, a subsample was centrifuged at 6500 rpm for 20 minutes and the supernatant was decanted and sent to Norwest Labs for an analysis of the cations and anions present in the supernatant. The results are reported in Section 3.1.3. From these anions and cations, an indicator will be chosen to determine the

number of rinses required to clean the kimberlite tailings. The indicator will be chosen from the anions and cations according to its ease of measurement and its high initial concentration.

Kimberlite samples were therefore centrifuged, the supernatant was decanted and de-ionized water was added to rinse the solids. Samples were then centrifuged again. This process of rinsing and centrifuging was repeated 4 times. The indicator concentration was monitored in duplicate after each cycle. The results are reported in Section 3.1.3. The results will be used to determine the number of rinse cycles required to clean the kimberlite tailings.

2.2.2 Kimberlite Tailings Preparation Procedure

Based on preliminary experiments; the following kimberlite tailings preparation procedure was adopted:

1. Dislodge solids from bottom of sample drum by manually stirring with a yardstick.
2. Use a hand held electric rotor to keep solids in suspension, forming a homogeneous mixture.
3. Remove a subsample of kimberlite tailings.
4. Centrifuge the kimberlite tailings at 6500 rpm for 20 minutes
5. Decant the supernatant
6. Add de-ionized water to rinse the kimberlite tailings.
7. Repeat Steps 4 through 6 for the number of rinse cycles determined in Section 3.1.3. In the last rinse cycle, no de-ionized water is added.

8. Dry the remaining solids at 65°C (This temperature was chosen because the centrifuge bottles are plastic and cannot withstand higher temperatures)
9. Cool to room temperature in a dessicator.
10. Grind the dried material with a mortar and pestle to break the dried kimberlite tailings down into smaller sizes.
11. Store material in airtight bottles until used.

2.3 Adsorption Studies

The following sections outline the methodology followed to complete the adsorption studies. These studies consisted of batch experiments to determine equilibrium times and adsorption isotherms.

2.3.1 General Procedures

All chemicals used were of analytical reagent grade. All glassware was soaked in a solution of 10% nitric acid for three hours and rinsed twice with de-ionized water prior to use to ensure no contamination by phosphorus. All solutions were prepared with de-ionized water. A stock solution of phosphate was prepared using potassium phosphate in de-ionized water. Orthophosphate was used since it was anticipated that the phosphorus in the treated wastewater effluent at Ekati™ is, for the most part, in the form of orthophosphate. This assumption is based on the fact that literature suggests that other forms of phosphate such as polyphosphates and metaphosphates are converted to orthophosphate when they come into contact with microorganisms that are found in raw wastewater (Metcalf and Eddy, 1991). This breakdown of other

forms of phosphate to orthophosphate by the microorganisms occurs in a relatively short period of time (Metcalf and Eddy, 1991) and was therefore assumed to occur in the extended aeration tank at the Ekati™ site.

2.3.2 Kimberlite Tailings Preparation

The procedure described in Section 2.2.2 was followed throughout the equilibrium time studies.

2.3.1 Equilibrium Time Studies

The equilibrium time studies were completed to determine the contact time required for the adsorption of phosphorus by the kimberlite to reach equilibrium. The studies were conducted at two different temperatures, 18°C and 5°C, to represent the two extremes of lake temperatures near the mine site. The tumblers were placed in temperature controlled rooms, set at 18 $\pm$ 0.5°C for the warmer temperature boundary and 5 $\pm$ 0.5°C for the lower temperature boundary. The equilibrium time determined from these experiments was used as the contact time for the adsorption isotherm studies.

At the start of the experiments, the pH was measured and found to be 7 $\pm$ 0.5 and the dosage of kimberlite was 2.5 g/L. This set of conditions was chosen for simplicity. The initial phosphate concentration was 10 mg P/L of orthophosphate.

A starting concentration of 10 mg/L was chosen as an estimate of the maximum phosphorus concentration of the sewage effluent. The largest observed

total phosphorus concentration of the sewage effluent was 11 mg/L (Alpha Laboratory Services Ltd. Feb 1999 to January 2000).

The results of the equilibrium time studies are reported in Section 3.2.2. The equilibrium time determined from this procedure will be used as the contact time in the following experiments detailed in Sections 2.3.2 through 2.3.4.

The equilibrium time experiments were carried out using the procedure outlined below:

1. 190 mL of de-ionized water was placed in a 250 mL bottle
2. Three different samples served as controls:

   (i) De-ionized water

   (ii) De-ionized water with kimberlite

   (iii) De-ionized water with phosphorus
3. For most samples, prepared kimberlite was added to the bottle. For the control samples (i) and (iii), no kimberlite was added.
4. The bottle was capped and sealed with Teflon tape.
5. Several bottles were loaded into one of two custom-built rotary tumblers (used to keep the samples well mixed). The tumbler was either located in a 5°C or a 18°C temperature control room.
6. The tumblers were rotated at 10 revolutions per minute (rpm) for 24 hours.
7. 10 mL of 200 mg/L phosphate stock solution was placed in each 250 mL bottle (bringing the initial phosphorus concentration of the entire solution to 10 mg/L). The phosphorus was added at this point because it was observed

in preliminary experiments that additional mixing was required to allow the water to break up the dried kimberlite into finer particles.

8. The bottles were reloaded into the tumblers and rotated at 10 revolutions per minute.
9. Three bottles were removed at each time interval. The bottles were removed after contact times ranging from 10 minutes to 14 days.
10. The solutions from the bottles were filtered through a pretreated 2 mm glass fiber pre-filter and then filtered through a pretreated 0.45 μm membrane filter to remove the kimberlite. Pretreatment of filters involved pre-soaking prior to use following the procedure outlined in *Standard Methods* (APHA, 1992). Pre-soaking involved soaking the filters for one hour in de-ionized water, changing the water and soaking for another three hours.
11. The filtrate from Step 10 was collected and analyzed in duplicate for phosphate using the Vanadomolybdophosphoric Acid Method described in *Standard Methods* (APHA, 1992). The Vanadomolybdophosphoric Acid Colourimetric method was chosen due to its simplicity and its applicable concentration range. To ensure that the total phosphorus concentration was being measured, the Persulfate Digestion Method (APHA, 1992) was used to release all the phosphorus as orthophosphate, which is the form of phosphorus that is measured by the Vanadomolybdophosphoric Acid Colourimetric method. A Novaspec 3000 UV-visible spectrophotometer was

used for the measurement of absorbance. The techniques used to determine the phosphorus concentrations are discussed further in Section 2.3.5.

2.3.2 Kimberlite Tailings Adsorption Isotherm Procedures

The adsorption isotherm experiments were carried out using a procedure similar to that outlined in Section 2.3.1. Steps 1 through 8 were completed. In Step 3, kimberlite dosages ranged from 2.5 g/L to 150 g/L. Each dosage was applied to three separate bottles. Dosages higher than 150 g/L were not used due to long filtration times exceeding 18 hours. After Step 8, the bottles were tumbled for a contact time of 48 hours (determined from equilibrium time studies). The bottles were then removed from the tumblers and Steps 10 and 11 were completed.

2.3.3 Adsorption Isotherm Procedures for Altered Absorbent

During processing of the kimberlite tailings at the Ekati™ mine, coagulant and flocculant are added to help settle the tailings. The coagulant used in this research was Magnafloc 368 and the flocculant used was Magnafloc 156. To prevent confusion they will simply be referred to as coagulant and flocculant. The addition of coagulant and flocculant may affect the ability of the kimberlite to adsorb phosphorus. In addition, some of the additives may result in the removal of phosphorus from solution. For this reason, the adsorption isotherm experiments were repeated, with the addition of various chemicals and with different experimental conditions.

Six different batch experiments were completed. The six batches used the following as the adsorbent:

1. Phosphorus adsorption by coagulant and kimberlite
2. Phosphorus adsorption by flocculant and kimberlite
3. Phosphorus adsorption by coagulant, flocculant and kimberlite
4. Phosphorus adsorption by coagulant
5. Phosphorus adsorption by flocculant
6. Phosphorus adsorption by coagulant and flocculant

The kimberlite, coagulant and flocculant adsorption isotherm experiments were carried out using a procedure similar to that outlined in Section 2.3.1. Steps 1 through 8 were completed. In Step 2, the second control was de-ionized water with the adsorbent. In this case, the adsorbent was the kimberlite tailings with coagulant and flocculant added. In Step 3, the adsorbent was added. For batches 1 through 3, the kimberlite dosage ranged from 2.5 g/L to 150 g/L. In these batches, the flocculant and coagulant were added in the same proportion to each other as they are found in the actual kimberlite tailings at the Ekati™ mine site. The dosages are approximately 35 g of coagulant and 55 g of flocculant per tonne of processed kimberlite. Each dosage was applied to three separate bottles. In batches 4 through 6, the coagulant and flocculant dosages were higher than the dosages used in batches 1 through 3. This increased dosage was used to determine if dosage rate had any affect on the adsorption of phosphorus. After Step 8, the bottles were tumbled for a contact time of 48 hours (determined from equilibrium time studies). The bottles were then

removed from the tumblers and Steps 10 and 11 from Section 2.3.1 were completed. These experiments were only conducted at a temperature of 18°C. The results are reported in Section 3.2.4. If the results indicated that the addition of coagulant and flocculant had a significant impact on phosphorus adsorption, the experiments would have been repeated at a temperature of 5°C.

2.3.4 Desorption Isotherm Procedures

Batch laboratory experiments were completed to determine if a change in pH or a change in temperature would cause the phosphorus to desorb from the kimberlite. A kimberlite dosage of 40 g/L was used throughout the desorption isotherm experiments. This dosage was chosen due to its ease of filtration (higher dosages required much longer to filter) and the phosphorus equilibrium concentration was on average around 3 mg/L. This starting concentration allowed for any subsequent changes in concentration to be measured easily.

The desorption isotherm experiments were carried out using a procedure similar to that outlined in Section 2.3.1. Steps 1 through 8 were completed. In Step 2, an additional control was added. This control contained kimberlite and phosphorus. After Step 8, the samples were mixed in the tumblers for 48 hours. After 48 hours, the samples were removed and either the temperature conditions or the pH conditions were changed. For temperature condition changes, the samples were either moved from the 5°C tumbler to the 18°C tumbler or vice versa. For pH changes, a fixed amount of acid or base was added to the sample before being

reloaded in the tumbler. The acid and base dosages used are reported in . All pH changes were completed at 18°C.

Acetic acid is the main component of organic acid that is produced during anaerobic digestion of sludge (Metcalf and Eddy, 1991). Anaerobic digestion may be occurring in the sediment layer at the bottom of the kimberlite containment area. This digestion would cause the pH to lower and the main reason for the pH change would be an increase in acetic acid. For this reason, acetic acid was used to decrease the pH. Sodium hydroxide was used to increase the pH.

Sodium hydroxide was chosen because it is one of the most commonly used bases for pH adjustment. The sodium hydroxide also has a low tendency to precipitate out of solution no matter what the pH (Metcalf and Eddy, 1991).

The dosages of acid and base ranged from 0.1 mg/L to 10 mg/L. Each dosage was applied to three bottles. These dosages were chosen to cover a pH range of approximately 3 to 10. After the acid or base was added, the samples were reloaded into the 18°C tumbler. After the temperature or pH change, the samples were tumbled for 48 hours prior to completing Steps 10 and 11 from Section 2.3.1. The pH level of the samples was measured prior to phosphorus determination. The results are presented in Sections 3.2.5 and 3.2.6.

2.3.5 Phosphorus Determination Procedures

There are two major steps involved in phosphorus determination. The first step is digestion and the second step is the phosphorus determination. As previously mentioned in Section 2.3.1, the phosphorus concentration was determined using the

Persulfate Digestion and the Vanadomolybdophosphoric Acid methods. The following paragraphs provide more detail on these methods.

The first step, digestion, is used to convert the different forms of phosphorus such as polyphosphates, metaphosphates, organic phosphates and organic phosphorus to dissolved orthophosphate. Dissolved orthophosphate can be measured in the second step using a colourimetric technique. The conversion is completed by acid hydrolysis at boiling-water temperatures. The dissolved orthophosphate concentration can be determined through one of three different colourimetric methods.

In the Persulfate Digestion Method, a 50 mL portion of sample is brought to acidic conditions with sulfuric acid before the addition of persulfate. The sample can then be either boiled on a hot plate for 30 minutes or placed in an autoclave for 30 minutes. The autoclave was used initially because it requires no supervision while the sample is being heated. However, contamination problems occurred when the samples were autoclaved. A description of the contamination problems and of the solution to this problem that was found are discussed in Appendix A. For the reasons discussed in Appendix A, samples were digested by boiling on a hot plate until 10 mL of sample was remaining. After the sample was cooled, it was neutralized with sodium hydroxide and made up to a volume of 100 mL with de-ionized water. The digested sample was then analyzed by one of the colourimetric methods for phosphorus determination.

The colourimetric method was chosen according to the expected concentration of the sample. The sample can always be diluted to ensure that it falls within the acceptable concentration range for the chosen colourimetric method. The Vanadomolybdophosphoric Acid Colourimetric method was chosen due to its simplicity and its applicable concentration range. For all of the colourimetric methods, the absorbance of a certain wavelength of light is used to determine the concentration of phosphate. In the Vanadomolybdophosphoric Acid Colorimetric method, a yellow vanadomolybdophosphoric acid is formed. The intensity of the yellow colour is proportional to the phosphate concentration.

There are three different wavelengths of light that can be used to determine the relative intensity of the yellow colour that is formed. The wavelength is chosen according to the expected concentration of the sample. Table 2-1 shows the wavelengths that can be used and their corresponding phosphate concentration range. This concentration range refers to the concentration of phosphate in the solution whose absorbance is measured by the spectrophotometer and not the concentration of phosphate in the original sample.

Table 2-1 Phosphorus Concentration Ranges by Wavelength

Wavelength (nm)	Phosphorus Concentration Range (mg P/L)
400	1 to 5
420	2 to 10
470	4 to 18

To carry out the Vanadomolybdophosphoric Acid Colourimetric method, 35 mL or less of the digested sample was added to a 50 mL volumetric flask. The volume of digested sample that was transferred to the volumetric flask was chosen so that the concentration in the flask would fall within the range of 1 to 18 mg P/L once the volume was brought up to 50 mL. 10 mL of vanadate-molybdate reagent was added and the volume was made up to 50 mL. The reagent was prepared according to the directions in *Standard Methods* (APHA, 1992). The colour is given at least ten minutes to develop and then the absorbance is measured at the desired wavelength. The original sample is diluted during the digestion step and during the colourimetric step. These dilutions are taken into account when the final concentration of the sample is determined. A sample calculation is provided in Appendix B.

From preliminary experiments, it was determined that all of the samples should fall into the concentration range covered by a wavelength of 420 nm. The absorbance of all samples was measured at a wavelength of 420 nm. If necessary, the absorbance of the samples was measured at one of the other two wavelengths. A calibration curve was created for each wavelength. The standards used to create the calibration curve were carried through the same digestion procedure as the samples.

3 RESULTS AND DISCUSSION

3.1 Kimberlite Characterization

3.1.1 Light Element Energy Dispersive X-ray Analysis (EDX)

The Light Element Energy Dispersive X-ray Analysis (EDX) capabilities of the Scanning Electron Microscope (SEM) were used to conduct an elemental analysis of kimberlite samples. The procedure set out in Section 2.1.3 was followed. The results of the elemental analysis are reported as an atomic percentage for each size fraction in Table 3-1.

The overall elemental composition of the mine tailings did not vary substantially over the different size fractions or sample types (Figure 3-1). The similarities in the elemental composition of the different samples are more evident when looking at a single size fraction (Figure 3-2). The elemental composition of the fine tailings does not appear to change substantially over time (from April to June 2000) or throughout the fine tailings concentration process (Table 3-2). For this reason, for the rest of this research it was assumed that the tailings composition would not change significantly over time.

3.1.2 ICP Spectroscopy

ICP Spectroscopy was used to determine the chemical composition of the kimberlite tailings. The JTF sample received from BHP in June 2000 was analyzed by this method. Table 3-3 provides the results of this analysis.

Table 3-1 Elemental Analyses of Kimberlite Tailings

	Atomic Percentage by Size Fraction											
	June Final Effluent (JFE)				June Thickener Feed (JTF)				April Final Effluent (AFE)			
	>355 µm	150 to 355 µm	45 to 150 µm	<45 µm	>355 µm	150 to 355 µm	45 to 150 µm	<45 µm	>355 µm	150 to 355 µm	45 to 150 µm	<45 µm
Si	53.47	46.32	55.51	52.65	49.10	48.67	51.88	53.13	53.57	53.39	57.11	56.22
Mg	25.34	24.94	22.44	21.50	31.78	28.16	26.15	22.17	22.30	22.13	18.54	17.59
Fe	8.49	11.84	9.23	10.89	8.37	10.45	9.04	8.94	8.82	9.08	8.79	7.85
Al	7.27	9.56	7.46	8.25	6.39	7.08	7.43	8.90	9.06	8.80	8.88	10.75
Ca	4.01	1.98	4.13	5.50	3.07	3.09	3.51	4.93	4.56	4.29	4.76	5.34
K	0.65	2.49	0.81	1.09	0.24	0.82	0.65	0.81	1.11	1.18	1.23	1.37
Na	0.48	2.10	0.30	0.08	0.76	1.01	1.10	0.76	0.45	0.43	0.30	0.35
Other	0.29	0.77	0.13	0.05	0.29	0.71	0.25	0.36	0.13	0.71	0.39	0.52
Total	99.71	99.23	99.87	99.95	99.71	99.29	99.76	99.64	99.87	99.29	99.61	99.48

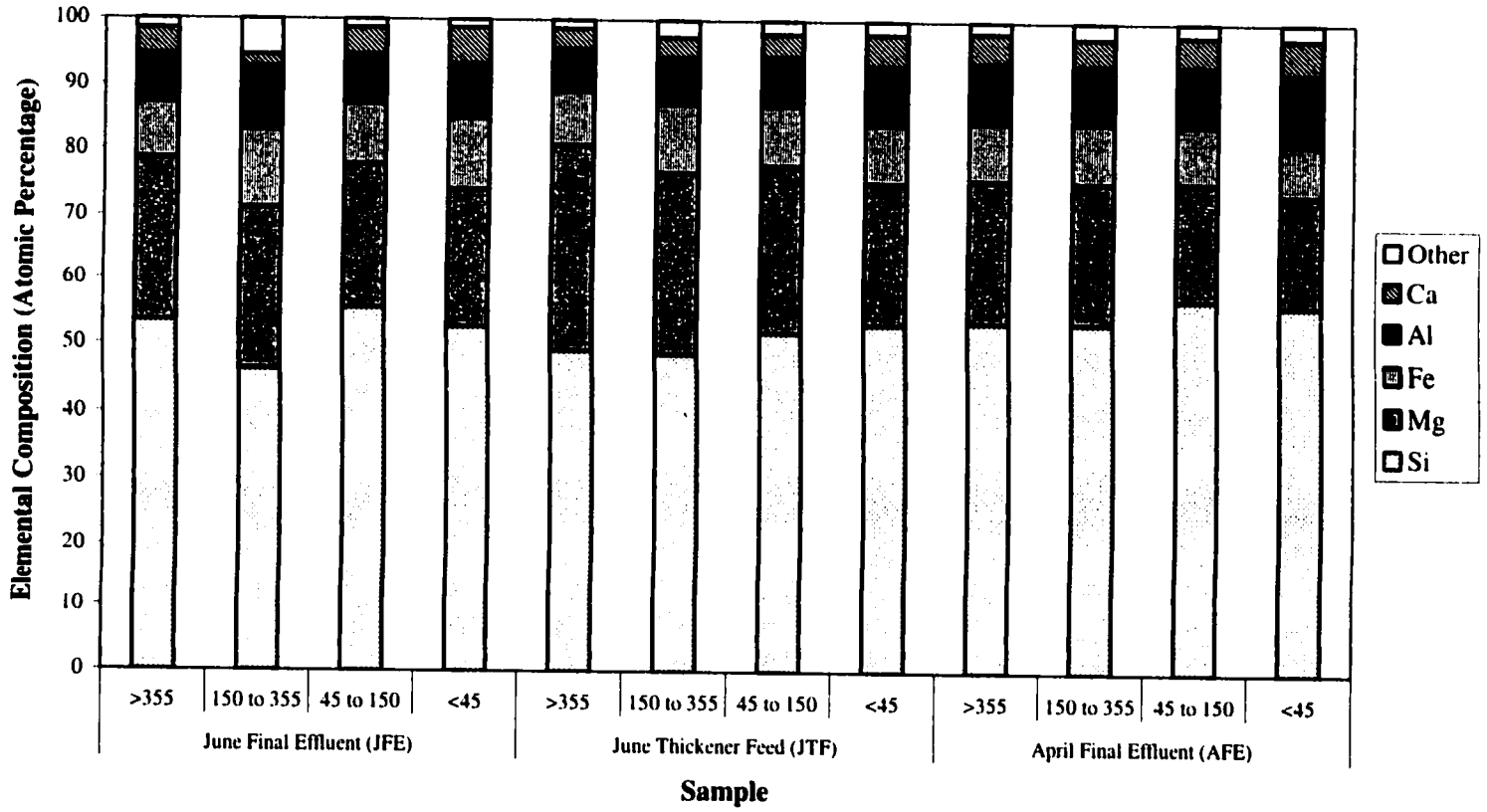

Figure 3-1 Elemental Compositions of Samples by Size Fraction (in μm)

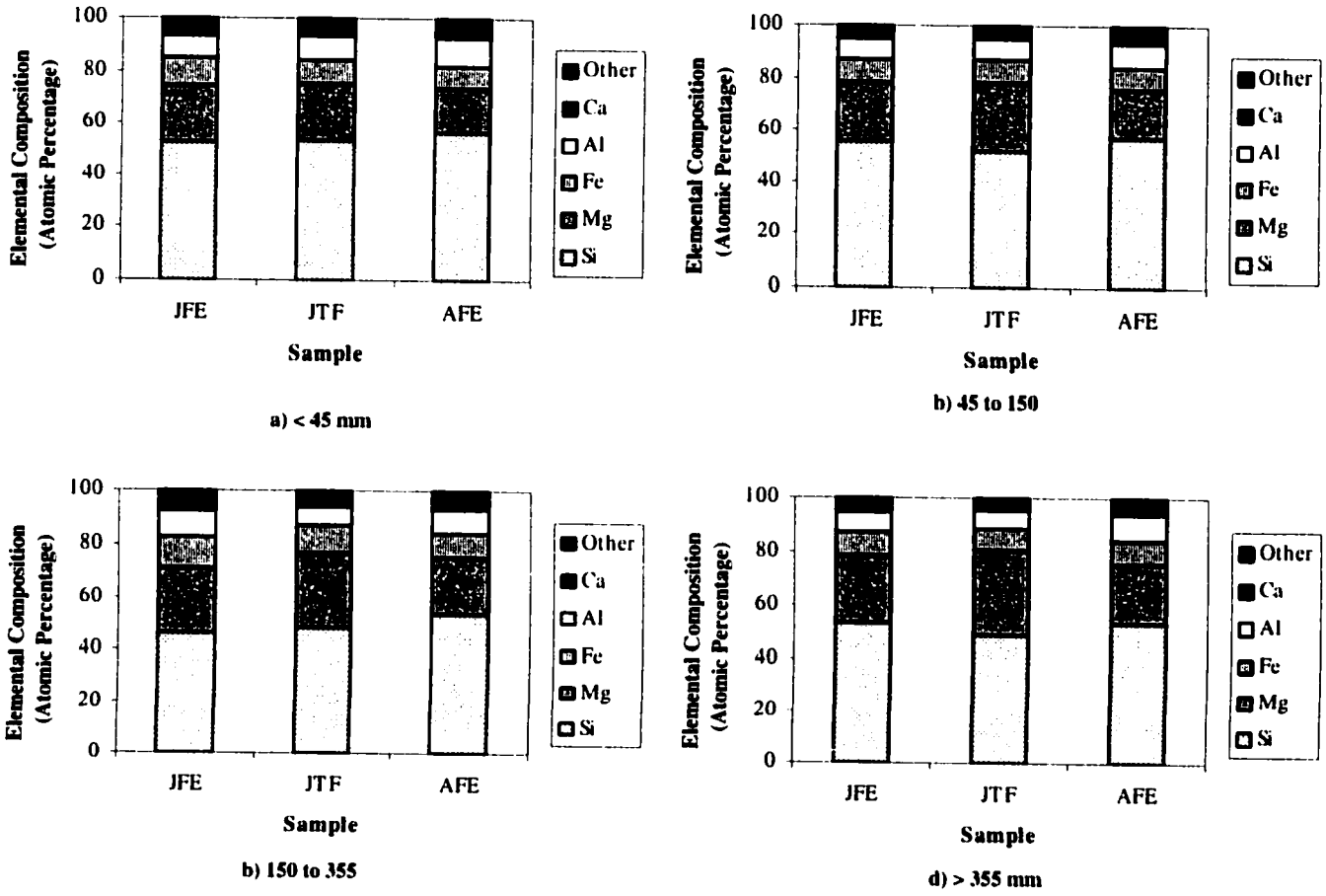

Figure 3-2 Elemental Compositions of Samples (June Final Effluent (JFE), June Thickener Feed (JTF) and April Final Effluent (AFE)) by Size Fraction

Table 3-2 Average Percent Difference in Elemental Composition of Kimberlite Over Time and Throughout Tailings Process

	Average Percent Difference	
	JTF and JFE (Through Tailings Process)	JFE and AFE (Over Time)
Si	3	6
Mg	12	17
Fe	10	18
Al	10	13
Ca	6	10
Other	11	8
Average	9	12

Table 3-3 Chemical Composition of June 2000 Thickener Feed (as determined by ICP Spectroscopy)

Analyte		Result	Unit
Silicon	SiO_2	47.8	% (dry weight)
Magnesium	MgO	23.8	% (dry weight)
Iron	Fe_2O_3	6.32	% (dry weight)
Aluminum	$Al2O_3$	5.38	% (dry weight)
Calcium	CaO	2.61	% (dry weight)
Potassium	K_2O	1.23	% (dry weight)
Titanium	TiO_2	0.353	% (dry weight)
Sodium	Na_2O	0.228	% (dry weight)
Phosphorus	P_2O_5	0.19	% (dry weight)
Barium	BaO	0.156	% (dry weight)
Manganese	MnO	0.0902	% (dry weight)
Strontium	SrO	0.04	% (dry weight)
Zirconium	ZrO_2	0.0038	% (dry weight)
Total Oxides		101	% (dry weight)
Loss on Ignition		12.9	% (dry weight)
Chromium		602	µg/g dry
Zinc		94.3	µg/g dry
Vanadium		77	µg/g dry

The ICP Spectroscopy and mean EDX results are comparable (Table 3-4). The difference between the methods is less than 5 percent for all of the elements analyzed.

Table 3-4 Elemental Percentage Comparisons of ICP and EDX Results

	Elemental %		
	ICP	EDX	Difference
Si	47.8	52.6	4.8
Mg	23.8	23.6	0.2
Fe	6.3	9.3	3.0
Al	5.4	8.3	2.9
Ca	2.6	4.1	1.5
K	1.2	1.0	0.2
Na	0.2	0.7	0.4
Other	0.5	0.4	0.1

3.1.3 Kimberlite Preparation for Adsorption Experiments

A subsample of kimberlite tailings was centrifuged at 6500 rpm for 20 minutes and the supernatant was decanted and sent to Norwest Labs for an analysis of the cations and anions present in the supernatant. The results are shown in Table 3-5. As discussed in Section 2.2.1, an indicator of the number of rinses required to clean the kimberlite tailings was chosen from the list of cations and anions shown in Table 3-5. Hardness was chosen as the indicator because the initial hardness of the indicator was well above detection limits and a simple titration procedure is required to measure hardness.

Table 3-5 Dissolved Metals, Anions and Cations Found in Supernatant

Dissolved Metals	Results (mg/L)
Aluminum	0.028
Antimony	<0.005
Arsenic	<0.01
Barium	0.0512
Berylium	<0.0005
Bismuth	0.008
Boron	0.032
Cadmium	<0.0005
Chromium	<0.0008
Cobalt	0.0008
Copper	<0.001
Lead	<0.002
Lithium	0.003
Molybdium	0.004
Nickel	0.013
Phosphorus	<0.03
Selenium	<0.004
Silicon	1.02
Silver	<0.001
Strontium	0.448
Sulphur	79.5
Thalium	<0.004
Tin	<0.003
Titanium	0.0009
Vanadium	0.004
Zinc	0.0065
Routine Water	
Calcium	30.7
Magnesium	47.7
Sodium	12.2
Potassium	27.1
Iron	0.016
Manganese	0.0184
Chloride	10.6
Nitrate - N	0.571
Nitrite - N	0.042
Sulphate - S	79.5
Phosphate	<0.05

The procedure of rinsing described in Section 2.2.1 was followed. The hardness indicator was monitored after each rinse cycle. After one rinse, the hardness of the supernatant was significantly lower. Subsequent rinses did not notably alter the hardness concentration (Figure 3-3). The results of these tests indicate that all subsamples of kimberlite should be centrifuged twice (1 rinse) prior to being used in any experiments.

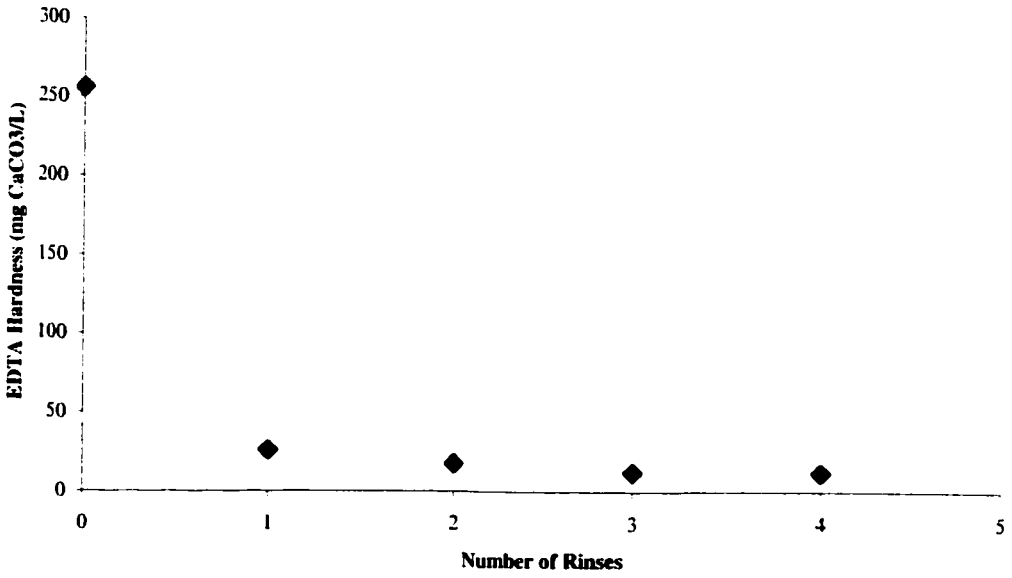

Figure 3-3 Hardness of Supernatant vs. Number of Rinses

3.2 Adsorption Studies

3.2.1 Calibration Curve

A calibration curve was prepared for each wavelength with at least six phosphate standards measured in duplicate for each wavelength. The calibration

curve for the 420 nm wavelength is shown in Figure 3-4. The calibration curve provides a relationship between absorbance and phosphate concentration. Once the absorbance of each digested sample was measured the concentration was determined using the calibration curve as illustrated in Appendix C. The data used to prepare the calibration curves and the calibration curves for wavelengths of 400 and 470 μm can be found in Appendix C.

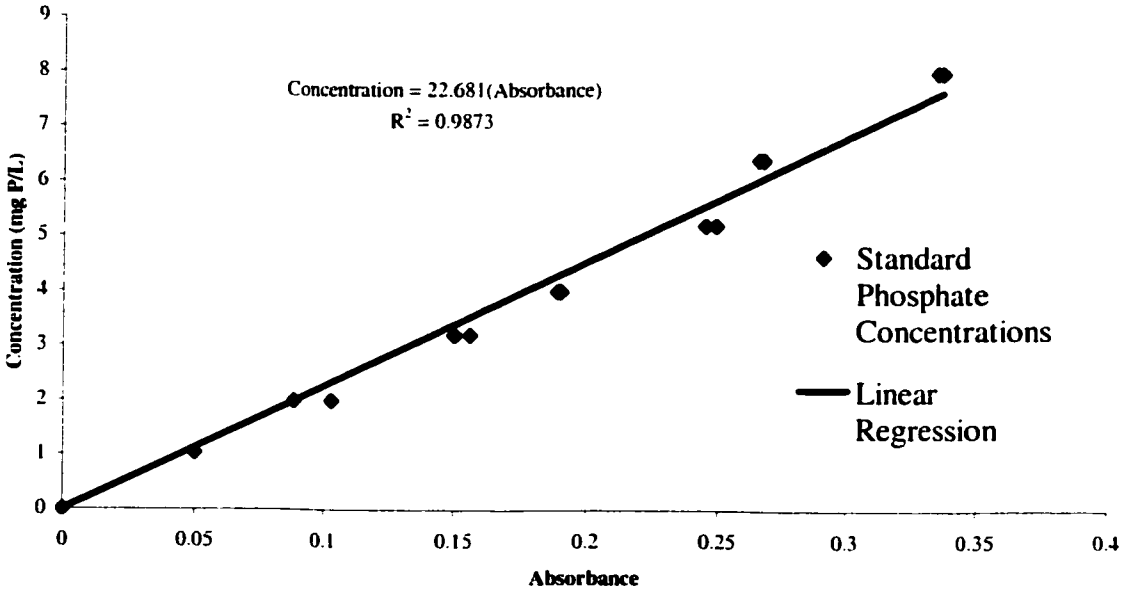

Figure 3-4 Phosphate Calibration Curve for 420 nm Wavelength

3.2.2 Equilibrium Time Studies

The results of the equilibrium time studies are presented in Figure 3-5. The data presented in Figure 3-5 represents the average of two separate runs for each

temperature. The phosphorus concentration at both temperatures decreased rapidly during the first six hours of contact time with the kimberlite (Figure 3-5). After the first six hours, the rate of adsorption of phosphorus decreased. The phosphorus concentration remained constant after approximately one full day of contact. It was assumed that at this point equilibrium had been reached. This assumption was reinforced when the concentration of samples removed after a contact time of 14 days were found to have an average concentration of 8.8 mg/L. This concentration was approximately the same as the concentration after 1 day of contact time.

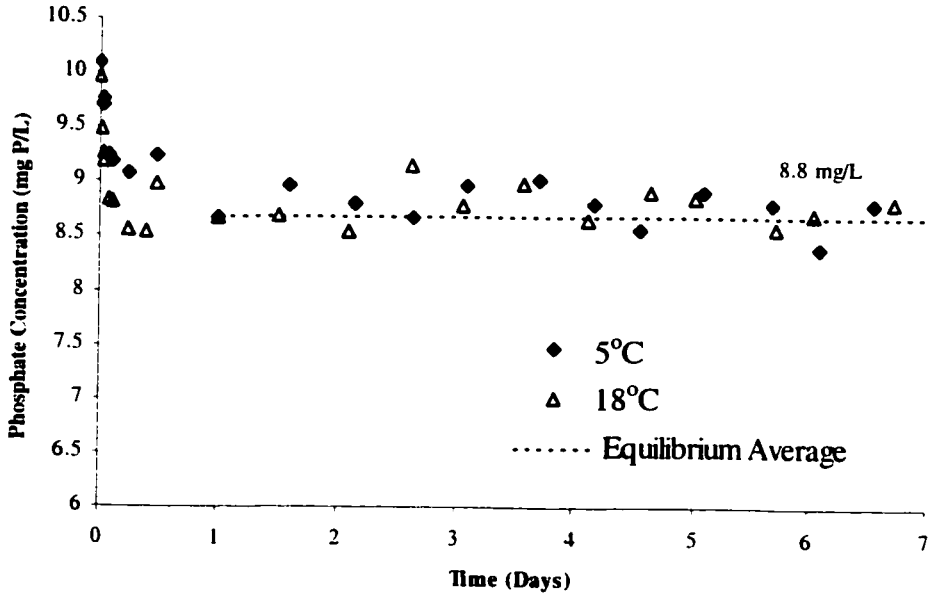

Figure 3-5 Equilibrium Time Studies

As can be observed from Figure 3-5, the time to reach equilibrium and the equilibrium concentration was found to be the same for both temperatures. The

equilibrium concentration was found to be 8.8 mg P/L for both temperature conditions. This translates to a removal of 0.5 mg of phosphorus per gram of kimberlite. The removal of arsenic was found to be 0.25 mg per g of kimberlite (Dikshit et al., 2000) and the removal of endosulfan was found to be 0.88 mg per g of kimberlite (Sudhakar and Dikshit, 1999). Phosphorus removal by kimberlite is greater than arsenic removal and less than endosulfan removal. Phosphorus removal was determined to occur in the range of 2.25 to 10.1 mg P/L depending on the type of clay used as the adsorbent (Henmi and Huang, 1995). On average, the clay content of the fine tailings is 25%. According to this percentage, the removal of phosphorus by kimberlite tailings should fall in the range of 0.56 to 2.5 mg per g of kimberlite. The results fall slightly below this range. This may be due to the interaction of the different types of clay present in the kimberlite tailings.

3.2.3 Kimberlite Adsorption Isotherms

The adsorption capacity of kimberlite tailings was modeled using the Langmuir and the Freundlich isotherms given in Table 1-1 and Table 1-2. The experimental equilibrium data for 18°C were fitted with the Langmuir and Freundlich isotherms and are presented in Figure 3-6 and Figure 3-7. From these figures, it is clear that the Freundlich isotherm models the adsorption capacity of kimberlite tailings much better than the Langmuir model. The Freundlich model will be used throughout the rest of this research.

When the Freundlich isotherm is plotted on a linear scale, it can be seen that the equilibrium concentration (C_e) approaches the initial concentration of 10 mg/L as

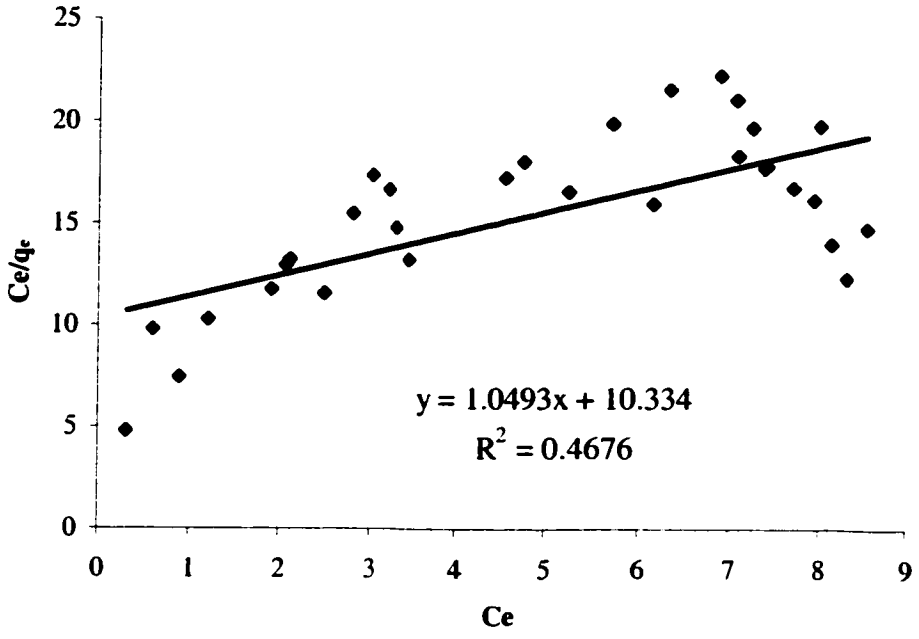

Figure 3-6 18°C Adsorption Data modeled with Langmuir Isotherm

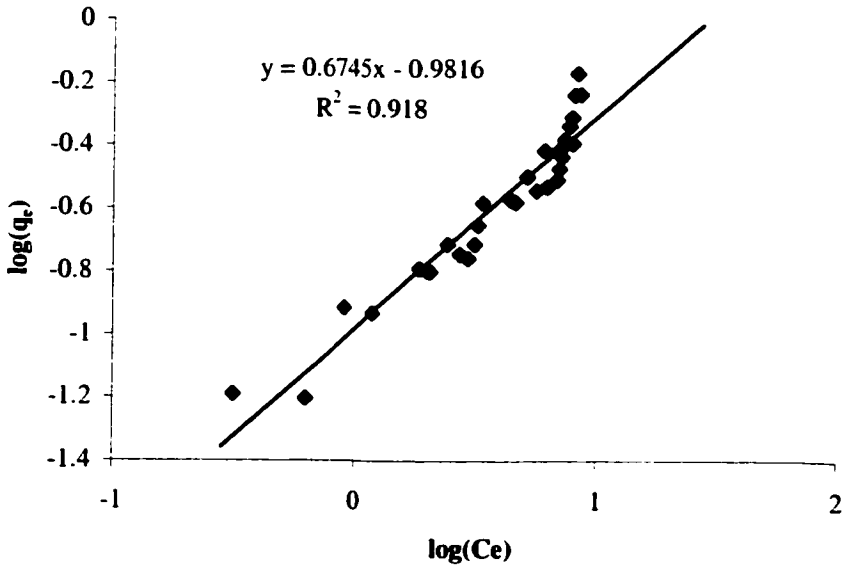

Figure 3-7 18°C Adsorption Data modeled with Freundlich Isotherm

the adsorbent loading increases (Figure 3-8). This is visible in the Freundlich isotherm plotted on a log scale as the tail that is visible above the linear regression line on the right hand side of the graph (Figure 3-7).

The experimental equilibrium data for both temperatures were fitted with the Freundlich isotherm equation and these are presented in Figure 3-7 and Figure 3-9. The Freundlich isotherm constants for temperatures of 18°C and 5°C are summarized in Table 3-6. From these results, it can be seen that there is little difference between the adsorption of phosphorus at 5°C and 18°C.

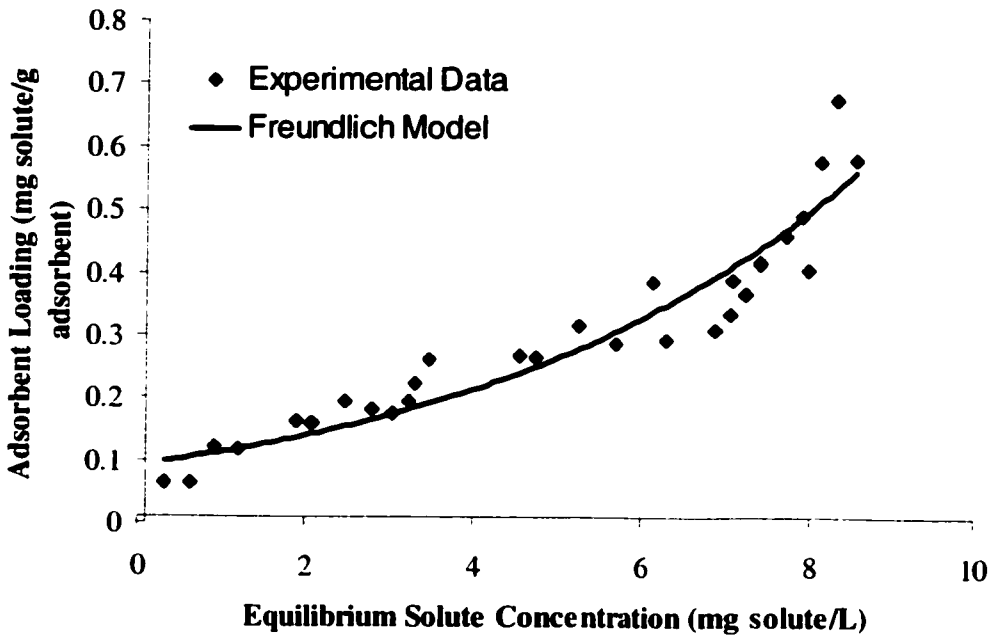

Figure 3-8 18°C Adsorption Data modeled with Freundlich Isotherm on a Linear Scale

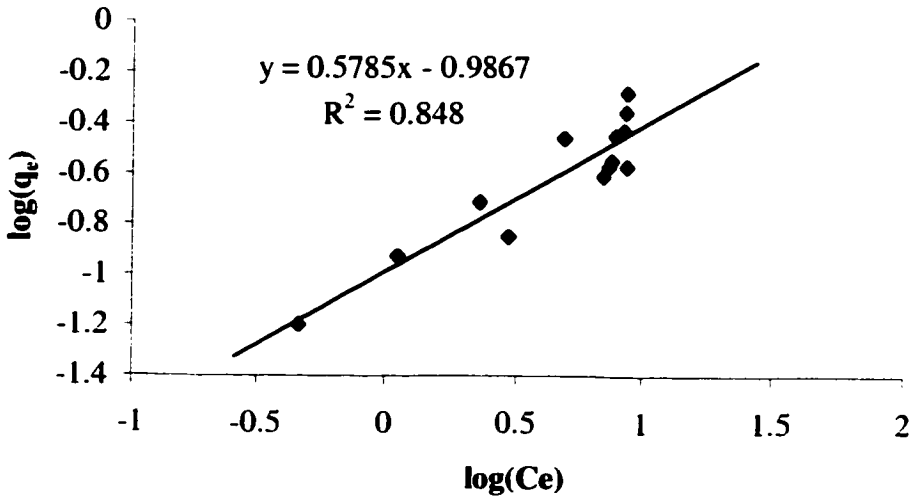

Figure 3-9 5°C Kimberlite Adsorption Isotherm

Table 3-6 Freundlich Isotherm Constants for Phosphorus Adsorption by Kimberlite Tailings at 5°C and 18°C

Temperature Condition	1/n	95% Confidence Interval of 1/n	log(Kf)	95% Confidence Interval of log(Kf)	Number of Data Points	R^2
18°C	0.67	0.57 to 0.78	0.98	0.93 to 1.0	31	0.92
5°C	0.58	0.42 to 0.75	0.99	0.86 to 1.1	13	0.85

The adsorption of phosphorus may be affected when there is other substances found in the water. These substances may include TOC, sulfate, nitrate, nitrite and other compounds. They effect of competitive adsorption may need to be studied if phosphorus is found in the water column of the kimberlite containment area.

3.2.4 Kimberlite, Coagulant and Flocculant Adsorption Isotherms

The adsorption capacity of the kimberlite tailings with the addition of coagulant and/or flocculant was also modeled using the linearized form of the Freundlich isotherm (as given in Table 1-1).

Since the results presented in Section 3.2.3 indicate that there was little difference in phosphorus adsorption at 5°C and 18°C, the experimental data for this section were only collected at 18°C. The experimental equilibrium data for the first three adsorbent conditions were fitted with the Freundlich isotherm equation and are presented in Figure 3-10 through Figure 3-12. The Freundlich isotherm constants for the three different adsorbent conditions are reported in Table 3-7. These constants are similar to the constants determined in Section 3.2.3 (Table 3-6). The Freundlich constants for coagulant and flocculant addition fall within the 95% confidence interval of the constants found for kimberlite alone (Table 3-7). Since the Freundlich constants fall within this confidence interval, it can be concluded that the addition of coagulant and flocculant have little or no effect on the adsorption of phosphorus by kimberlite tailings.

Since the coagulant and flocculant dosages in the first three batches of experiments were low, additional experiments were completed with higher concentrations of coagulant and flocculant to determine if these have any phosphorus adsorption capabilities. These experiments were completed without any kimberlite being present in the samples.

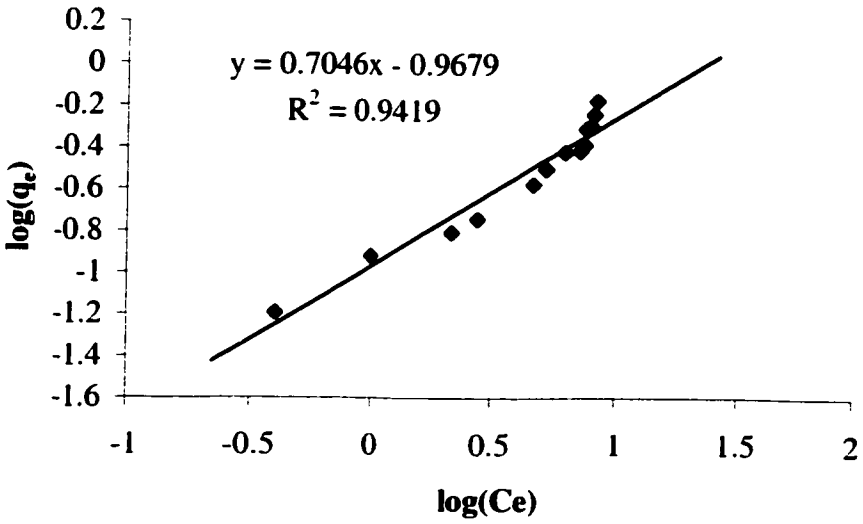

Figure 3-10 Kimberlite and Coagulant Adsorption Isotherm

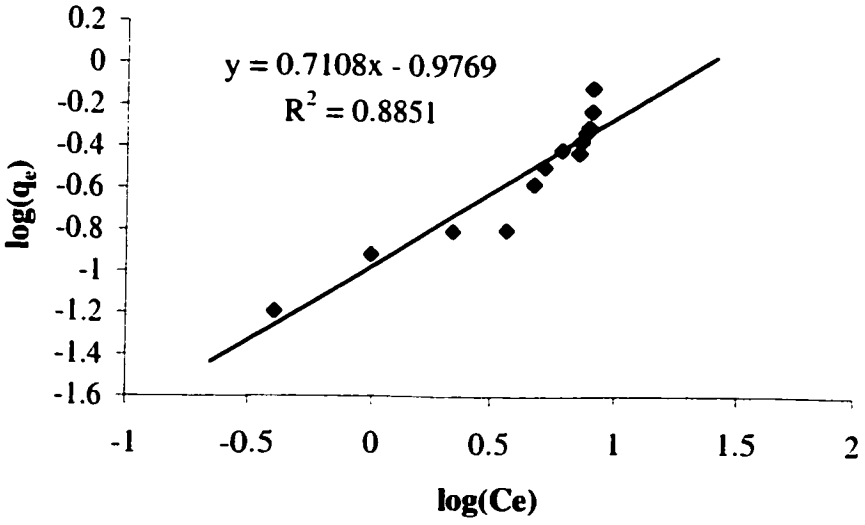

Figure 3-11 Kimberlite and Flocculant Adsorption Isotherm

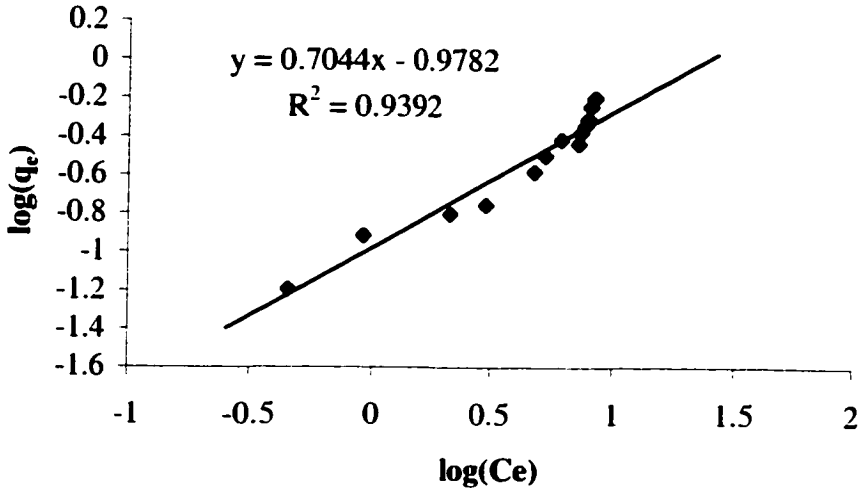

Figure 3-12 Kimberlite, Coagulant and Flocculant Adsorption Isotherm

Table 3-7 Freundlich Isotherm Constants for Different Absorbent Conditions

Adsorbent	1/n	95% Confidence range of 1/n	Log(K_f)	95% Confidence range of log (K_f)
Kimberlite	0.68	0.60 to 0.75	0.98	0.93 to 1.0
Kimberlite and Coagulant	0.70	0.59 to 0.82	0.97	0.88 to 1.0
Kimberlite and Flocculant	0.71	0.54 to 0.88	0.98	0.85 to 1.1
Kimberlite, Coagulant and Flocculant	0.70	0.59 to 0.82	0.98	0.89 to 1.1

The dosages of coagulant and flocculant are summarized in Table 3-8. These dosages were chosen to keep the coagulant and flocculant in the same proportion to each other as they are found in the actual mine tailings at the Ekati™ mine site. The resulting phosphorus concentrations after coagulant addition are reported in Table 3-9. The average phosphorus concentration of three control samples that had no flocculant or coagulant added was 9.8 mg P/L (Table 3-9). There was no trend in removal of phosphorus as the coagulant and flocculant dose was increased (Figure 3-13). The 95% confidence limits on the average phosphorus concentration of the control shows that the differences in phosphorus concentrations after the addition of coagulant and flocculant may be due to random error alone. These results indicate that the addition of coagulant and flocculant have no effect on phosphorus concentrations in solution at 18°C. It is therefore assumed that the affect of the addition of coagulant and flocculant will also be negligible at 5°C. For this reason, the experiments were not repeated at 5°C.

Table 3-8 Coagulant and Flocculant Dosages

	Dose (g/L)	
Dose Number	Coagulant	Flocculant
1	0.000075	0.000125
2	0.00025	0.000375
3	0.00075	0.001125
4	0.005	0.0075
5	0.0125	0.01875
6	0.025	0.0375
7	2.5	3.75

Table 3-9 Phosphorus Concentrations after Flocculant and Coagulant Addition

Dose Number	Coagulant	Flocculant	Coagulant and Flocculant
1	9.8	9.9	9.8
2	9.9	9.9	9.9
3	9.9	9.8	9.7
4	9.7	9.7	9.9
5	9.9	9.7	9.7
6	9.9	9.8	9.7
7	9.7	9.7	9.7
Control Average			9.8

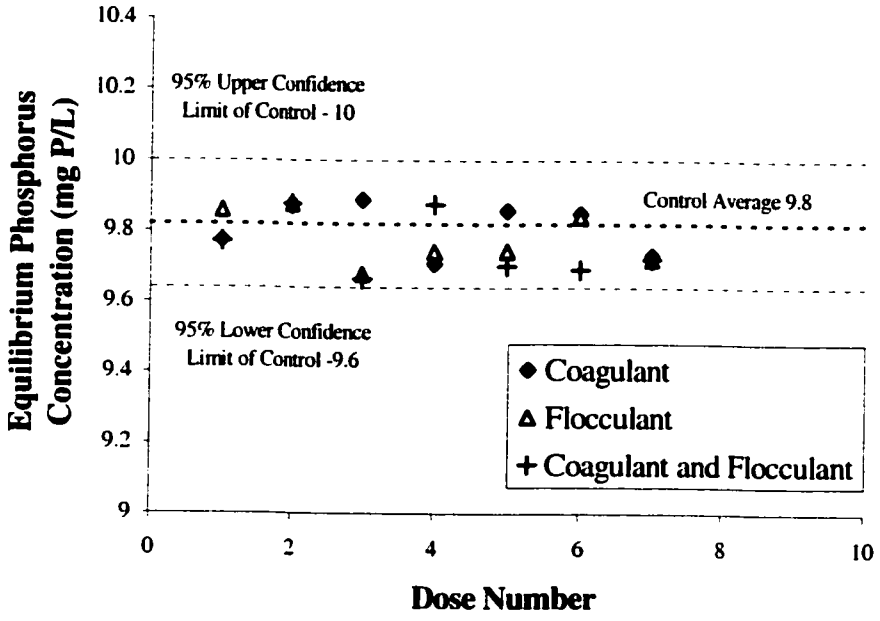

Figure 3-13 Phosphorus Concentrations After Coagulant and Flocculant Addition

3.2.5 Effect of Temperature

As explained in Section 2.3.4, once equilibrium between kimberlite and phosphorus had been reached, samples were moved from either 18°C to 5°C or from 5°C to 18°C and allowed to reach a new equilibrium. The resulting concentrations are summarized in Table 3-10. From the 95% confidence interval of the controls that underwent no temperature change it can be seen that the change in temperature had no effect on phosphorus concentration (Table 3-10).

Table 3-10 Equilibrium Phosphorus Concentration after Temperature Change

	Equilibrium Phosphorus Concentration (mg P/L)		
Sample #	Control	5°C to 18°C	18°C to 5°C
1	2.9	3.0	2.8
2	2.9	2.9	2.8
3	2.9	2.9	2.9
Average	2.8	2.9	2.8
95% Confidence Interval of Average	2.8 to 3.0	2.8 to 3.0	2.8 to 2.9

The phosphorus concentration was determined two days after the temperature change was completed. The desorption process may take longer than two days to occur. If phosphorus is found in the water column of the kimberlite containment area, the desorption experiments may need to be repeated with a longer desorption time.

3.2.6 Effect of pH

Increasing amounts of acetic acid and sodium hydroxide were added to select samples as described in Section 2.3.4. The resulting equilibrium phosphorus concentrations are reported in Table 3-11. Table 3-11 also displays the resulting pH. There is no observable trend in equilibrium phosphorus concentration as the pH increases or decreases (Figure 3-14). From the 95% confidence interval of the controls that underwent no pH change it can be seen that the addition of acetic acid and the addition of sodium hydroxide had no effect on phosphorus concentration (Figure 3-14). From these results, it appears that pH change has no desorption effects.

As mentioned above, the addition of acetic acid and sodium hydroxide have little effect on the phosphorus concentration in solution. It is therefore assumed that the effect of pH changes due to the addition of other acids and bases will also be negligible.

The phosphorus concentration was determined two days after the addition of acetic acid or sodium hydroxide. The desorption process may take longer than two days to occur, however this is unlikely at the given conditions. If phosphorus is found in the water column of the kimberlite containment area, the desorption experiments may need to be repeated with a longer desorption time, or other mechanisms resulting in release should be examined.

Table 3-11 Effect of Acetic Acid Addition on Phosphorus Concentration

Acetic Acid Dose (mg/L)	0.1	0.5	1	5	10
Resulting pH	6.8	6.7	6.5	5.4	4.5
Average Equilibrium Concentration (mg P/L)	2.8	2.9	2.9	2.8	2.9
95% Confidence Interval of Average	2.6 to 3.0	2.9 to 3.0	2.8 to 3.1	2.7 to 3.0	2.8 to 3.0
Sodium Hydroxide Dose (mg/L)	0.1	0.5	1	5	10
Resulting pH	7.1	7.3	7.5	8.2	9.1
Average Equilibrium Concentration (mg P/L)	3.0	2.8	3.0	2.9	2.8
95% Confidence Interval of Average	2.9 to 3.1	2.7 to 3.0	3.0 to 3.0	2.6 to 3.2	2.6 to 3.0

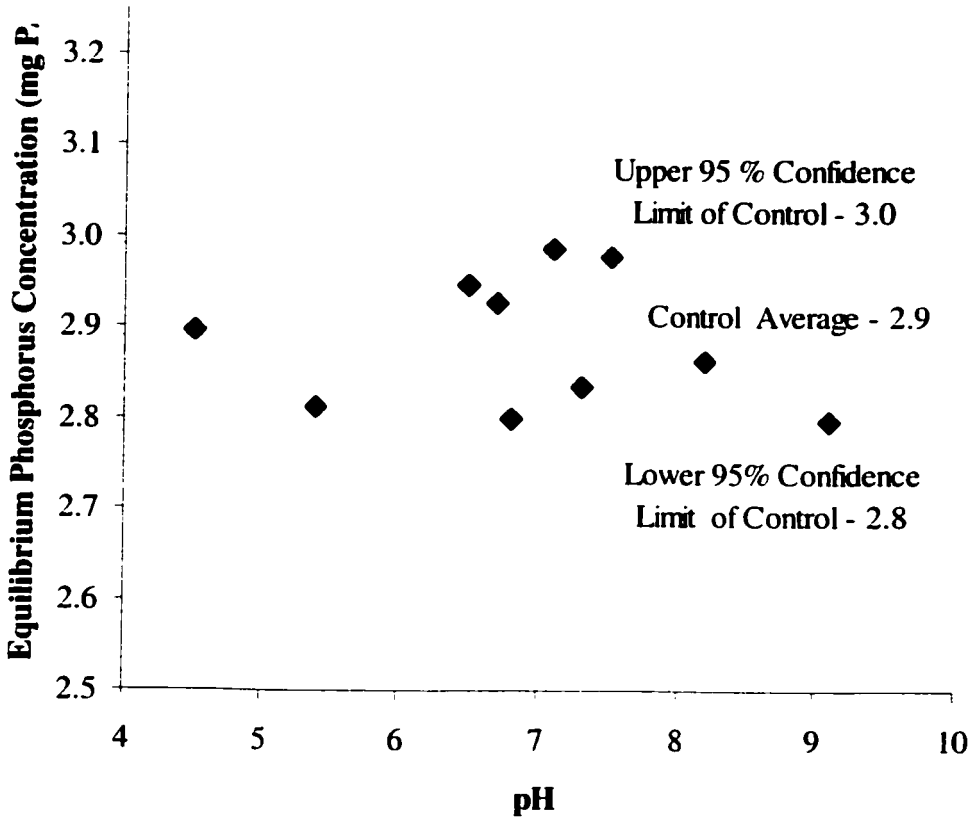

Figure 3-14 Effect of pH Change on Phosphorus Concentration

The interaction of temperature and pH change was not determined (i.e. changing the temperature and the pH at the same time). If further experiments were completed, the interaction of these two parameters should be determined.

3.3 Significance of Results

The Ekati™ mine site currently has no regulations on the amount or concentration of phosphorus that can be released into the environment. From the isotherms, however, a rough calculation can be used to determine how much dried

kimberlite would be needed to adsorb a certain amount of phosphorus. Based on the results presented here, changes in temperature, changes in pH and the addition of coagulant and flocculant have no observable effect on the adsorption of phosphorus by kimberlite. Therefore the isotherms presented in Section 3.2.3 can be used.

According to the results presented in Table 3-6 and at the particle sizes used in this study, to reduce the concentration of phosphorus from 10 mg/L to 0.1 mg/L approximately 0.5 kg of dried kimberlite per litre of wastewater would be needed at a temperature of 18°C and approximately 0.4 kg of dried kimberlite per litre of wastewater at a temperature of 5°C.

An estimated maximum of 320,000 L of wastewater could be produced in a day at the mine site based on a camp population of 800 with a per capita production rate of 400 L of wastewater per person per day. If the treated wastewater has a phosphorus concentration of 10 mg/L and at a temperature of 18°C, it would require approximately 160 tonnes of dried kimberlite of the size used in this study per day to reduce the phosphorus concentration to 0.1 mg/L.

Just after start-up, the mine site was processing 9,000 tonnes of ore per day and is currently in the process of increasing the amount to 18,000 tonnes per day. When the ore is processed, approximately 60 percent of the ore becomes fines. This percentage translates into the current disposal of fines being roughly 5,400 to 10,800 tonnes of dried kimberlite per day. This amount is well above the 160 tonnes needed to reduce the phosphorus concentration of 320,000 L of wastewater from 10 mg/L to 0.1 mg/L, based on the results presented here. Even though the kimberlite adsorption

of phosphorus is low according to the results presented here, with proper mixing of the wastewater effluent with the fine tailings, there is more than an adequate amount of kimberlite available to remove the phosphorus when the pH is 7.0 and the temperature is 18°C.

All laboratory experiments were conducted while keeping the samples well mixed. Once the tailings are sent to the kimberlite containment area they will be subjected to an entirely different set of conditions. There will be a dynamic component including settling and resuspension and temperature changes that cannot be modeled adequately in a laboratory. These dynamic changes may cause desorption. It will be necessary to monitor the phosphorus concentration levels in the containment area to determine if desorption is occurring under these conditions. The phosphorus concentration levels will have to be monitored to determine if water from the containment area can be released into the environment. There are no regulations on effluent phosphorus concentrations presently, however, in the future a minimum phosphorus concentration level may be set by government regulations.

If an unacceptable level of phosphorus is found in the containment area effluent, the laboratory experiments conducted here may have to be repeated with slight changes to determine the cause of the high phosphorus concentration levels.

The adsorption of phosphorus may be affected when there are other substances found in the water. These substances may include TOC, sulfate, nitrate, nitrite and other compounds. The adsorption of wastewater constituents was briefly studied in a preliminary study conducted by BHP. The adsorption of phosphorus still

occurred when other wastewater compounds were present (BHP, 1999). There may be the potential of other substances competing with phosphorus for adsorption on the kimberlite tailings. Theses substances may include the truck wash bay detergent or any other compounds or chemicals that are added to the kimberlite tailings.

During the desorption studies the phosphorus concentrations were measured two days after the temperature or pH change. The desorption process may take longer than two days, however this is unlikely to occur under the given conditions. The desorption experiments may need to be repeated allowing more time for desorption to occur.

The removal of phosphorus from the wastewater effluent prior to discharge may be the only way to prevent phosphorus from being released into the downstream water system. This is true whether the discharge is released into a nearby lake or into the mine tailings containment area. Phosphorus is not readily used up in the aquatic environment. It is recycled and reused within the aquatic environment. Unlike nitrogen that can be removed into the air, once phosphorus is present in a water system, it is difficult to remove. For this reason, if there is concern about increased aquatic plant growth, it is suggested that phosphorus removal from the wastewater prior to discharge be investigated. There are two basic options for phosphorus removal in wastewater treatment plants – chemical precipitation and biological removal. Due to lower operation and maintenance requirements, chemical precipitation may be the preferred method for removal of phosphorus. Alum, ferric chloride and ferric sulfate are the most common methods to accomplish phosphate

precipitation. If this method were used, the sludge that is generated would have to be removed to an acceptable disposal location. The disposal of the sludge would have to ensure that no phosphorus would be released to the local environment. Either building a storage pond or pit on site that would prevent the release of phosphorus to the environment or shipping the sludge to an existing disposal site capable of containing the sludge would be required.

Only the fate of the phosphorus in kimberlite tailings was studied in this work. The fate of the other nutrients, such as nitrogen and microorganisms such as fecal coliform, should be studied to determine whether the addition of wastewater effluent to the kimberlite containment area is an adequate disposal method for these materials.

4 CONCLUSIONS

From the results, it appears that adding the wastewater effluent to the kimberlite mine tailings may be an effective method to adsorb the phosphorus found in the wastewater effluent.

In controlled batch laboratory experiments, the adsorption of phosphorus by kimberlite at a pH of 7.0 reached equilibrium after approximately one day of contact time at temperatures of 18°C and 5°C. The equilibrium concentration was 8.8 mg P/L when the initial concentration was 10 mg/L and 2.5 g dried kimberlite/ L was added. This adsorption translates to a removal of 0.5 mg of phosphorus per gram of kimberlite.

Using the Freundlich model and the parameters determined herein, approximately 0.5 kg of dried kimberlite per litre of wastewater was needed at a temperature of 18°C to reduce the concentration of phosphorus from 10 mg/L to 0.1 mg/L. For a 95% confidence in this performance, 0.7 kg of dried kimberlite per litre of wastewater was needed. At 5°C, only 0.4 kg of dried kimberlite per litre of wastewater is needed to reduce the concentration of phosphorus from 10 mg/L to 0.1 mg/L. For a 95% confidence in this performance, 0.8 kg of dried kimberlite per litre of wastewater was needed.

Based on the results presented herein, even though the adsorption of phosphorus per gram of dried kimberlite is low, there is more than an adequate amount of kimberlite available to remove the phosphorus loading due to the addition

of treated wastewater to the kimberlite tailings, when the pH is 7.0 and the temperature is 18°C.

At the mine site coagulant and flocculant are added to the kimberlite tailings prior to the addition of the treated wastewater. In laboratory experiments however, the coagulant and flocculant did not appear to have an affect on the adsorption of phosphorus by the kimberlite.

Changes in temperature and pH did not appear to cause any desorption in experiments conducted in this work. However, under different environmental conditions desorption may result.

All laboratory experiments were conducted while keeping the samples well mixed. Once the tailings are sent to the kimberlite containment area, they will be subjected to an entirely different set of local conditions. The phosphorus concentration levels will have to be monitored to determine if water from the containment area can be released into the environment. There are no regulations on effluent phosphorus concentrations presently. In the future a minimum phosphorus concentration level of the effluent from the containment area may be set by government regulations.

5 RECOMMENDATIONS

During the desorption studies the phosphorus concentrations were measured two days after the temperature or pH change. The desorption process may take longer than two days, however, this is unlikely to occur at the given conditions. The desorption experiments may need to be repeated allowing more time for desorption to occur.

The adsorption of phosphorus may be affected when there are other substances found in the water. These substances may include TOC, sulfate, nitrate, nitrite and other compounds. There may be the potential of other substances competing with phosphorus for adsorption on the kimberlite tailings. These substances may include the truck wash bay detergent or any other compounds or chemicals that are added to the kimberlite tailings. Some experiments may need to be completed to determine the effects of competitive adsorption

Based on the unknowns of the environmental conditions in the tailing pond, the removal of phosphorus from the wastewater effluent prior to discharge may be the only way to positively insure that the no phosphorus will be released into the downstream water system. For this reason, it is suggested that the downstream impact of phosphorus be investigated carefully. If the downstream impacts are deemed to be of an unacceptable magnitude then phosphorus removal may need to be completed.

There are two basic options for phosphorus removal in wastewater treatment plants – chemical precipitation and biological removal. Due to lower operation and

maintenance requirements, ease of operation, and the ability to adjust performance quickly, chemical precipitation is recommended to remove the phosphorus. Alum, ferric chloride and ferric sulfate are the most common methods to accomplish phosphate precipitation. If this method were used, the sludge that is generated would have to be disposed of in an environmental compatible manner. The disposal of the sludge would have to ensure that no phosphorus would be released to the local environment. For a 95% confidence in this performance, 0.7 kg of dried kimberlite per litre of wastewater was needed.

Only the fate of the phosphorus in the kimberlite fine tailings was studied in this work. The fate of the other nutrients, such as nitrogen and microorganisms such as fecal coliform, should be studied to determine whether the addition of wastewater effluent to the kimberlite containment area is an adequate disposal method for these materials.

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A Discussion of Contamination Problems

Problems associated with phosphorus concentration determination were encountered during the course of this project. The problems and the methods used to address these problems will be discussed in this appendix.

A.1 Preliminary Phosphorus Adsorption Studies

A preliminary set of adsorption experiments were conducted prior to the beginning of experiments for this study. The results of this study indicated that the equilibrium time for phosphate adsorption would be longer than twenty-four hours. For this reason another equilibrium study had to be conducted with the time frame extending past twenty-four hours to determine when equilibrium was actually reached. During the preliminary studies, the Persulfate Digestion method was used to digest the samples and the Ascorbic Acid Colorimetric method was used to determine the phosphate concentrations. The equilibrium time studies after the preliminary phosphate adsorption experiment used the Vanadomolybdophosphoric Acid Colorimetric method to determine the phosphate concentrations and the digestion was still completed according to the Persulfate Digestion method. The next section will describe briefly the methods used in this study. For a complete description of these procedures, see *Standard Methods* (APHA, 1992).

A.2 Phosphorus Determination

This section will describe the methods used to prepare the samples for phosphate concentration measurements as well as describing in more detail the digestion and colorimetric methods used in this study. This description is provided to

give a better understanding of the problems encountered while measuring the phosphate concentrations of the prepared samples.

The kimberlite for both the preliminary studies and the equilibrium studies was prepared as described in Section 2.3.2. The preparation of the samples followed the procedure listed in Section 2.3.1

The rest of this section provides a more detailed description of Step 11 of Section 2.3.1. Two separate 50 mL portions of each sample were analyzed for phosphate to minimize the possibility of an incorrect phosphate measurement being recorded.

The digestion step is used to convert the different forms of phosphorus such as polyphosphates, metaphosphates, organic phosphates and organic phosphorus to dissolved orthophosphate. Dissolved orthophosphate can be measured using a colourimetric technique. The conversion is completed by acid hydrolysis at boiling-water temperature. The dissolved orthophosphate concentration can be determined through one of three different colourimetric methods.

In the Persulfate Digestion Method a 50 mL portion of sample is brought to acidic conditions with sulfuric acid before persulfate is added. The sample can then be either boiled on a hot plate for 30 minutes or placed in an autoclave for 30 minutes. The autoclave was initially used because it requires no supervision while the sample is being heated. After the sample is removed from the autoclave it is neutralized with sodium hydroxide and made up to a volume of 100 mL. The digested sample is then analyzed by one of the colourimetric methods of phosphorus determination.

The colourimetric method is either chosen by the expected concentration of the sample or by the preference of the analyzer. The sample can always be diluted to ensure that it falls within the acceptable concentration range for the chosen colourimetric method. However, as the dilution factor is increased, the error in the phosphorus concentration is also increased. The Vanadomolybdophosphoric Acid Colourimetric method was chosen due to its simplicity and its concentration range. For all of the colourimetric methods the absorbance of a certain wavelength of light is used to determine the concentration of phosphate. In the Vanadomolybdophosphoric Acid Colorimetric method, a yellow vanadomolybdophosphoric acid is formed. The intensity of the yellow colour is proportional to the phosphate concentration. There are three different wavelengths of light that can be used to determine the relative intensity of the yellow colour that is formed. The wavelength is chosen according to the expected concentration of the sample. Table 1 shows the wavelengths that can be used and their corresponding phosphate concentration range. A calibration curve must be created for each wavelength. The standards used to create the calibration curve must be carried through the same digestion procedure as the samples.

Table A-1 Phosphate Concentration Ranges by Wavelength

Wavelength (nm)	Phosphate Concentration Range (mg P/L)
400	1 to 5
420	2 to 10
470	4 to 18

To complete the Vanadomolybdophosphoric Acid Colourimetric method, 35 mL or less of the digested sample is added to a 20 mL volumetric flask. The volume of digested sample that is transferred to the volumetric flask is chosen so that the concentration in the flask will fall within the range of 1 to 18 mg P/L once the volume is brought up to 50 mL. 10 mL of vanadate-molybdate reagent is added and the volume is made up to 50 mL. The colour is given at least ten minutes to develop and then the absorbance is measured at the desired wavelength.

A.3 First Attempt at Equilibrium Study

As in the preliminary set of experiments the temperature of the first equilibrium time study was carried out at a temperature of 18°C ± 0.5. The pH was neutral and the dosage of kimberlite was 2.5 g/L. This set of conditions was chosen for simplicity. The initial phosphate concentration was 10 mg P/L of orthophosphate. Only orthophosphate has been used since it is anticipated that the phosphorus in the water from the wastewater treatment plant at Ekati™ is, for the most part, in the form of orthophosphate. This assumption is based on the fact that literature suggests that other forms of phosphate are changed to orthophosphate when they come into contact with microorganisms that are found in raw sewage. (Metcalf and Eddy, 1991) This breakdown of other forms of phosphate to orthophosphate by the microorganisms occurs in a relatively short period of time. (Metcalf and Eddy, 1991) This breakdown would occur in the extended aeration tank at the Ekati™ site. If necessary other forms of phosphate will be used at a later date. (For example, the forms of

phosphate in the truck bay wash detergent may need to be studied.) The samples are still put through the digestion step to ensure that if any other forms of phosphorus that may be added with the kimberlite are measured.

A calibration curve was prepared for each wavelength with at least six phosphate standards measured in duplicate for each wavelength. The resulting calibration curve provided a relationship between absorbance and phosphate concentration. The calibration curves and the data used to create them can be found in Appendix C. Once the absorbance of each digested sample is measured the concentration may be determined using the calibration curve as illustrated in Appendix B.

The first attempt at completing the equilibrium study showed a wide variability in measured phosphate concentrations (Figure A-1). The points plotted in this figure are an average of four measurements: two samples, each measured in duplicate. Figure A-1 shows that, in some cases the concentration of phosphorus in the sample was higher than the original amount of phosphate (10 mg/L). These results indicated that there was a problem in the methodology.

Further review of the raw data revealed that there was often a major difference in absorption measurements taken from the same sample (Table A-2). The average difference in absorbance readings is 0.033. This difference translates to a difference in concentration of over 3 mg/L in the original sample (taking into account dilution). The results were deemed unacceptable and implied that the methodology needed to be reviewed.

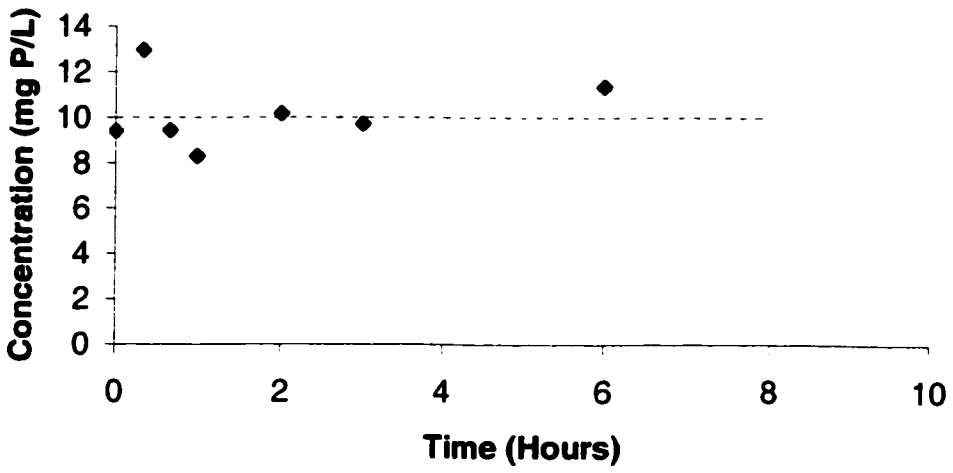

Figure A-1 First Attempt at Equilibrium Time

Table A-2 Raw Data from First Attempt

Time (hours)	Sample 1 Absorbance at 420		Difference	Sample 2 Absorbance at 420		Difference
0	0.222	0.189	0.033	0.136	0.136	0
0.33	0.225	0.18	0.045	0.237	0.187	0.05
0.67	0.178	0.169	0.009	0.175	0.163	0.012
1	0.166	0.176	0.01	0.137	0.136	0.001
2	0.187	0.149	0.038	0.188	0.191	0.003
3	0.176	0.185	0.009	0.159	0.176	0.017
6	0.228	0.186	0.042	0.185	0.164	0.021
12	0.328	0.472	0.144	0.299	0.264	0.035
18	0.221	0.179	0.042	0.217	0.166	0.051
24	0.305	0.23	0.075	0.227	0.278	0.051
36	0.227	0.172	0.055	0.205	0.228	0.023
48	0.273	0.229	0.044	0.169	0.21	0.041
84	0.196	0.16	0.036	0.171	0.168	0.003
DI	0.108	0.077	0.031	0.063	0.052	0.011
Average Difference				0.033		

A.4 Potential Sources of Contamination

At this point, it was suggested that some contamination of the samples was occurring to cause the elevated phosphate concentrations (i.e. higher than the initial values). At least one standard and one blank (de-ionized water with no phosphate added) were analyzed with each batch of samples. Since the standards and blanks appeared to be unaffected by the contamination at first, it was concluded that the samples were being contaminated. Possible sources of contamination of the samples were identified. These sources included the filters used to separate the kimberlite, the water used to prepare the samples, the kimberlite itself, and improper acid rinsing of glassware to remove residual phosphate. Each of these will be discussed below.

A.4.1 Filters

Initially the filters were prepared following the procedure set out in *Standard Methods* (APHA, 1992). The method initially chosen for this study was to soak 25 filters for one hour in 1 L of de-ionized water, change the water and soak for another three hours. Since preliminary work indicated that the filtration through the 0.45 μm membrane filter would take then 20 minutes, a glass fiber pre-filter was used prior to the membrane filter. The glass fiber pre-filters were prepared using the same presoaking method described for the membrane filters.

To remove possible contamination from the filters, the filters were soaked according to the procedure described above and then five 100 mL portions of de-

ionized water was rinsed through the filters. This treatment should remove any contaminants that were attached to the filters.

A.4.2 Water

For preparing all samples, blanks and standards, de-ionized water was used. The same water source was used to prepare the blanks, standards and samples. On occasion water was acquired at different times throughout the day and sometimes on completely different days. To ensure that the source water was not the problem, an ELGA water treatment system was used to treat the de-ionized water to ensure high-quality water. The ELGA treated water was used in all experiments following the first attempt at equilibrium time experiment.

A.4.3 Kimberlite

From the ICP Spectroscopy of the kimberlite solids after centrifuging, it was found that 0.19% of the solid was made up of phosphorus. (Norwest Laboratory Services Ltd., 2000) For this reason it is unlikely that the kimberlite was the source of additional phosphorus. To eliminate the kimberlite as a source of phosphorus, another set of blanks containing de-ionized water and a kimberlite dosage of 2.5 g/L was run through the same procedure as the other prepared samples. From the difference between the 10 mg/L standard and the kimberlite blanks, the phosphorus contribution of the kimberlite can be determined.

A.4.4 Glassware

On the advice of one of the environmental engineering lab technicians, all glassware was soaked in an acid bath of ten percent acetic acid. The glassware was removed and rinsed twice with de-ionized water. To remove the possible contamination from improper acid washing, the glassware was acid washed prior to the analysis of each batch of samples. After a few batches of samples were analyzed, the acid bath was switched to a ten percent nitric acid solution. *Standard Methods* recommends rinsing the glassware with hot, dilute hydrochloric acid (APHA, 1992). The ten percent nitric acid solution was found to be effective in phosphorus studies conducted in the summer of 2000 by others in our department. It is also not necessary to acid wash the glassware prior to every use of the glassware as long as it is rinsed thoroughly after each use.

A.4.5 Results After Changes

After all of these changes were implemented, another set of experiments was completed. Three samples, instead of two, were removed from the tumbler at each time interval. The results from the second attempt at determining the equilibrium time can be seen in Figure A-2. The samples taken over the first day have reasonable phosphate concentrations (i.e. less than the original concentration of 10 mg/L). After the first day, however, the majority of the sample concentrations are either around the initial concentration of 10 mg/L or higher.

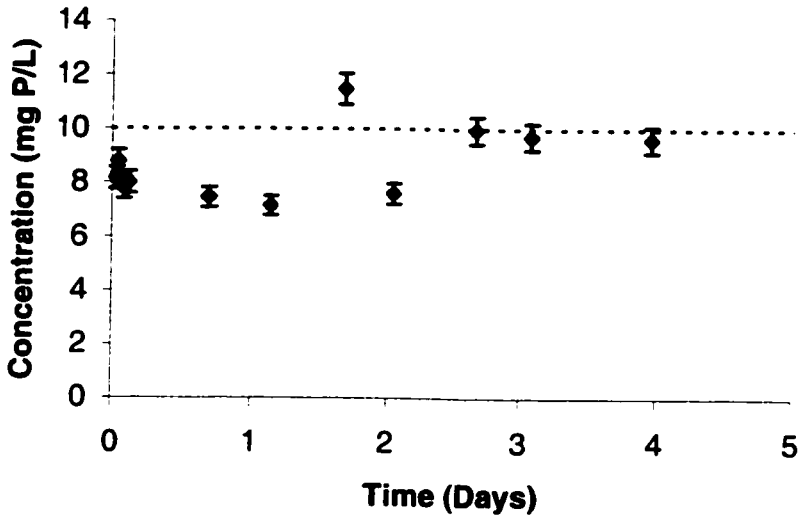

Figure A-2 Second Attempt at Determining Equilibrium Time

When a batch of samples were analyzed at least one 0 mg P/L standard and at least one other standard with a concentration in the range of 1 to 20 mg p/L were put through the digestion and colourimetric methods with the samples. During the second attempt at equilibrium time, it was observed that the same sort of contamination also affected the standards. Table A-3 shows a portion of the standard concentrations collected during the second attempt. From Table A-3 it can be seen that some batches had all of the standards having measured concentrations close to the expected concentrations (Batch 2). Other batches had the entire set of standards having measured concentrations that did not match the actual concentrations (Batch 1). Other batches had the blanks not measuring as zero and the other standards measuring close to the actual concentration (Batch 5). During the second attempt

the standards and the prepared samples showed evidence of some sort of contamination.

Table A-3 Measured Standard Concentrations

	Concentration in mg P/L				Concentration in mg P/L		
Batch	Standard	Measured	Difference	Batch	Standard	Measured	Difference
1	0	8.8	8.8	4	14	15.9	1.9
	14	25.5	11.5		14	15.1	1.1
	14	28.3	14.3		0	7.9	7.9
	4	9.9	5.9		0	3.7	3.7
	4	12.7	8.7		12	13.4	1.4
	8	22.2	14.2		12	14.1	2.1
2	0	0.6	0.6		10	12.2	2.2
	0	0.6	0.6		10	11.6	1.6
	0	0.5	0.5		8	11.7	3.7
	0	0.8	0.8		8	10.8	2.8
	0	0.3	0.3		6	10.6	4.6
	0	0.1	0.1		6	8.4	2.4
	4	4.4	0.4	5	0	9.0	9.0
	4	4.2	0.2		0	4.0	4.0
3	4	3.6	0.4		8	8.9	0.9
	4	4.3	0.3		8	8.6	0.6
	1	1.1	0.1		2	1.9	0.1
	1	1.4	0.4		2	1.9	0.1
	0	2.1	2.1	6	0	8.0	8.0
	0	1.4	1.4		0	5.0	5.0
	2	1.7	0.3		2	1.9	0.1
	2	1.9	0.1		2	1.7	0.3

A.5 Other Potential Sources of Contamination or Error

After additional experiments, it appeared that some form of contamination was appearing in both the standards and the prepared samples. Other potential sources of contamination or error were investigated. It was believed that the contamination

might be occurring in either the digestion step or the colourimetric step. Other potential sources of contamination or error that were identified included the spectrophotometer, the water used to prepare standards and samples, the digestion process using the autoclave, and the chemicals used throughout the procedure. These potential sources of error are discussed below.

A.5.1 Spectrophotometer

A batch of samples and standards were analyzed using two different spectrophotometers. The absorbance readings of the two spectrophotometers differed but one spectrophotometer absorbance readings were consistently higher then the other (Figure A-3). The difference between the two absorbance readings increased as the absorbance increased (Figure A-3). The difference between the spectrophotometers in both cases was very small. The difference between the two spectrophotometers ranged from 1 to 33 units with an average difference of 10. These results suggest that the spectrophotometer is not the source of error.

A.5.2 Water

The ELGA water treatment system treats the Environmental Engineering Building's de-ionized water. While the experiments were being run, the ELGA system had a new filter installed. If the building's de-ionized water was contaminated, the water used to create the standards and samples may be the source of contamination even when the water was run through the ELGA water treatment system prior to analysis. Another batch of standards using three different sources of

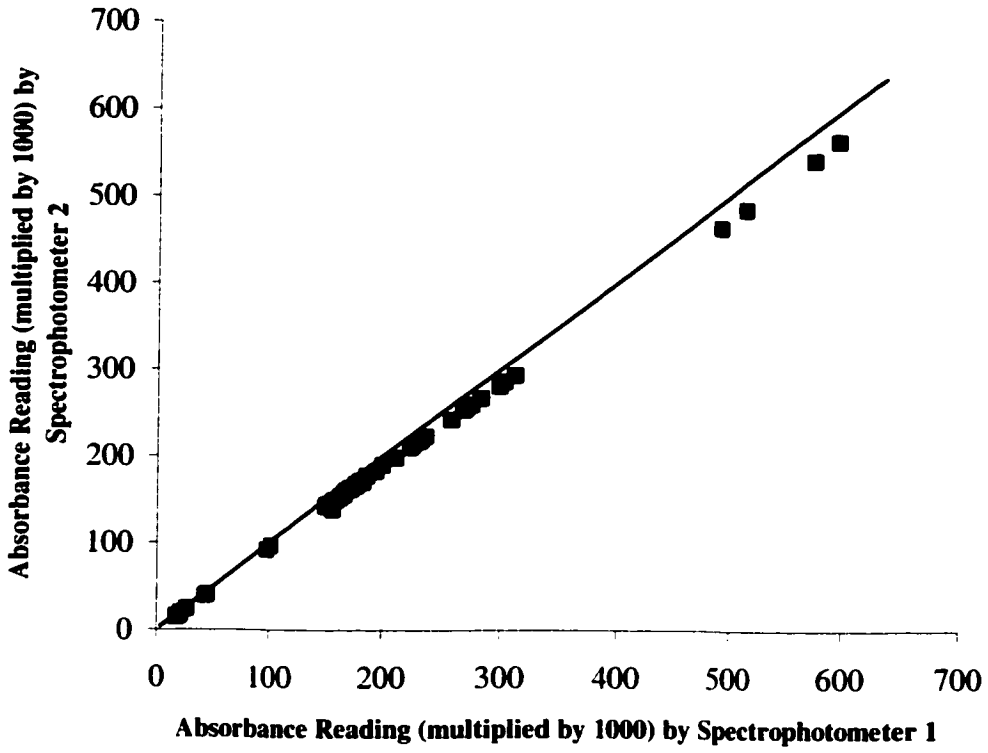

Figure A-3 Comparison of Spectrophotometer Absorbance Readings

water was analyzed. The three sources of water were de-ionized water from the Environmental Engineering Building, de-ionized water from the Environmental Engineering Building run through the ELGA unit in the Environmental Engineering Building and de-ionized water from the university wide system run through a different ELGA unit. Eight blanks (no phosphate added) from each source of water were analyzed through the digestion and colourimetric steps. The average of the eight samples is displayed in Table A-4. All blanks yielded some value of phosphate, despite the fact that different water sources were used. Since the probability of all

three sources of water being contaminated is low, it was concluded that the source of the water was not the source of contamination.

Table A-4 Testing Different Water Sources

Water Source	Average Phosphate Concentration (mg P/L)
DI	7.18
DI+ELGA	8.61
University DI +ELGA	9.96

A.5.3 Digestion Process

The digestion process is used to convert all forms of phosphate to orthophosphate. This is necessary because the colourimetric methods can only determine the concentration of phosphate in the form of orthophosphate. Since the original concentration of the sample is 10 mg P/L of orthophosphate, the samples can also be analyzed without the digestion step. By analyzing the same sample using an undigested portion and a digested portion it can be determined whether the digestion is contributing to the contamination. After removing two 50 mL portions of sample for digesting there is still enough sample to determine the orthophosphate concentration of the remaining undigested sample. The undigested samples from the first hour of one of the equilibrium time experiments were analyzed for phosphate. The measured phosphate concentrations of the undigested samples appear to be

reasonable. The concentrations slowly decrease over the first hour (Figure A-4). The same samples were carried through the digestion step prior to phosphate concentration determination. The digested samples showed evidence of contamination and the measured concentrations did not agree with the concentrations measured without digestion (Figure A-4). The samples were prepared with a starting orthophosphate concentration of 10 mg P/L. No other phosphorus is in the samples. Therefore the digestion step should not cause higher concentrations in the samples. These results indicate that the contamination is occurring in the digestion step.

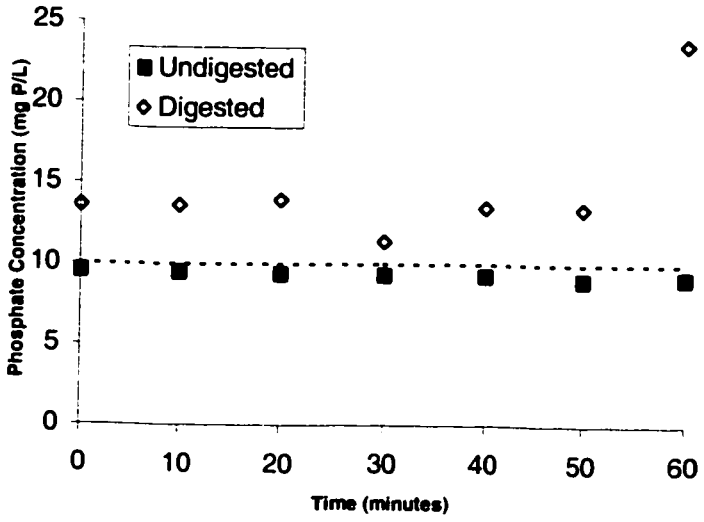

Figure A-4 Comparisons of Digested and Undigested Samples

To try and eliminate the autoclave as the source of contamination during the digestion process, a batch of blanks were digested in a different autoclave. The blanks included eight samples of de-ionized water from the Environmental

Engineering Building and eight samples of the same de-ionized water run through an ELGA unit. The results indicated that contamination occurred again (Table A-5).

Table A-5 Samples Digested in a Different Autoclave

Water Source	Expected Phosphate Concentration (mg P/L)	Average Phosphate Concentration (mg P/L)
DI	0	6.22
ELGA	0	9.92

Another batch of standards was analyzed using chemicals from a different laboratory. These chemicals were received at different times then the chemicals used throughout the rest of the experiments. This batch also showed evidence of contamination.

A.6 Other Digestion and Colourimetric Methods

There are different digestion and colourimetric methods that can be used during phosphate determination. As stated previously, the Vanadomolybdophosphoric Acid Colorimetric method was chosen for two reasons (1) for its simplicity and (2) for the fact that the sample concentrations fall within the range of concentration for this method without dilution (only a very small dilution is required during the digestion step). If this method cannot provide consistent results then another method must be used. Alternatively, if the Persulfate Digestion method is the cause of contamination, then a different digestion method can be used. Figure

A-5 displays the combinations of digestion and colourimetric methods that were used to determine which methods would provide consistent results.

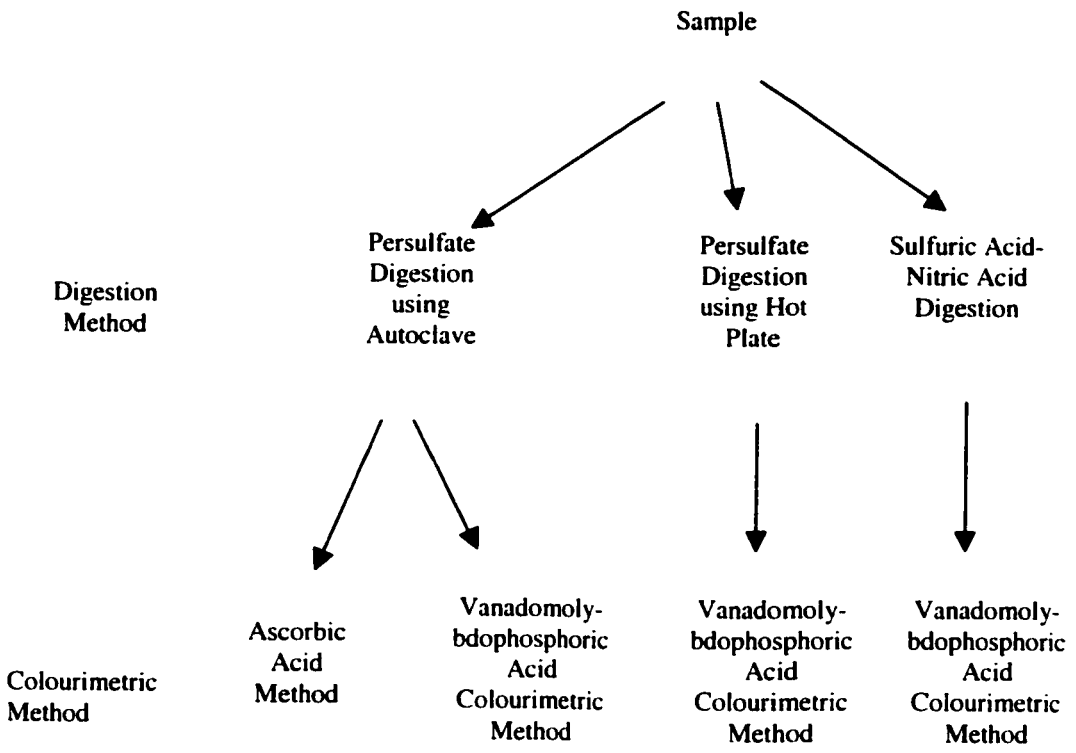

Figure A-5 Digestion and Colourimetric Combinations

A batch of standards was digested using the Persulfate Digestion method with the autoclave. Both the Vanadomolybdophosphoric Acid Colorimetric method and the Ascorbic Acid Method were used to determine the phosphate concentration of each standard. See *Standard Methods* for the procedures involved (APHA, 1992). The standards were diluted when measuring their concentration with the Ascorbic Acid method. The measured concentrations using the Ascorbic Acid Method agreed

with the expected concentrations while the concentrations determined by the Vanadomolybdophosphoric Acid Colorimetric method showed higher then expected concentrations (Table A-6). These results indicate that the contamination is not phosphorus. They also show an average contamination of 1.54 mg P/L (Table A-6). It is possible that the elevated phosphorus concentrations may be caused by ions or substances that cause positive interference with the Vanadomolybdophosphoric Acid Colorimetric method. The three substances that can positive interference are iron at a concentration greater than 100 mg/L, and silica and arsenate if heated (*APHA, 1992*). The combination of Persulfate Digestion and the Ascorbic Acid methods can be used to measure phosphate concentrations.

Table A-6 Ascorbic Acid and Vanadomolybdophosphoric Acid Methods

	Concentration (mg P/L)		
	Ascorbic	Vanado.	Difference
Blank	0	2.01	2.01
1 mg/L Std	0.77	1.45	0.68
2 mg/L Std	2.01	3.75	1.74
8 mg/L Std	8.23	9.7	1.47
16 mg/L Std	16.45	18.27	1.82
		Average	1.54

The Vanadomolybdophosphoric Acid method is a simpler procedure and would not involve diluting the sample. For this reason, two other types of digestion were attempted. The Persulfate Digestion method was attempted again except the samples were boiled on a hot plate instead of being heated in the autoclave. The

Sulfuric Acid-Nitric Acid Digestion method was also used. Combining either of these two digestion methods with the Vanadomolybdophosphoric Acid Method results in measured concentrations of standards being within 5% of the expected concentrations (Table A-7 and Table A-8).

Table A-7 Persulfate Digestion with Hot Plate with Vanadomolybdophosphoric Acid

Sample	Average Concentration	Percent Difference
DI	0.03	-
ELGA	0.04	-
2 mg/L Std	2.04	1.9
4 mg/L Std	4.19	4.8
6 mg/L Std	6.28	4.7
7 mg/L Std	7.31	4.4
10 mg/L Std	9.95	0.5
11 mg/L Std	11.02	0.2
16 mg/L Std	16.11	0.7
17 mg/L Std	17.26	1.6

Table A-8 Sulfuric Acid-Nitric Acid Digestion with Vanadomolybdophosphoric Acid

Sample	Average Concentration	Percent Difference
DI	0.05	-
ELGA	-0.03	-
3.5 mg/L	3.66	4.55
7.5 mg/L	7.47	0.35

The three combinations of digestion and colourimetric methods discussed in this section provided consistent results. One of these combinations must be chosen to

determine the phosphate concentrations of samples for the remainder of the experiments. The Vanadomolybdophosphoric Acid method is preferred because it does not involve the same degree of dilution as the Ascorbic Acid method. The higher the dilution of the samples prior to phosphate determination will increase the error. The Vanadomolybdophosphoric Acid method is also simpler and involves more stable reagents then the Ascorbic Acid method (APHA, 1992). The Vanadomolybdophosphoric Acid method will be used throughout the rest of the experiments.

Using the Vanadomolybdophosphoric Acid method limits the digestion to either the Persulfate Digestion method using the hot plate or the Sulfuric Acid-Nitric Acid Digestion method. The Sulfuric Acid-Nitric Acid Digestion method involves placing 100 mL of sample in a digestion tube and adding concentrated acid. The digestion tubes are heated to between 200°C and 250°C. The boiling that occurs is very violent. If the solutions are not well mixed, the boiling often causes bubbles that will boil over, out of the digestion tube, causing sample to be lost. The boiling can also cause cross-contamination if sample from one tube spills over to neighbouring tubes because the tubes are placed closely together with nothing sealing their tops. In an attempt to stop the boiling, different types of boiling beads and boiling rods were tried. The boiling could not be stopped consistently by any of the beads or rods tested. The Persulfate Digestion method using hot plates to heat the samples is the preferred digestion method when using the Vanadomolybdophosphoric Acid method.

To confirm that the combination of Persulfate Digestion method and Vanadomolybdophosphoric Acid method will work for samples that are carried through the entire kimberlite addition and filtration steps, a quick experiment was conducted. The tumbler was loaded with four different types of samples: de-ionized water (DI), de-ionized water with kimberlite added (DI w/K), 10 mg P/L and samples with an initial concentration of 10 mg P/L with a 2.5 g/L dosage of kimberlite (1,2 and 3). Three of each sample was loaded on the tumbler. Samples 1, 2 and 3 were removed at three different times. All samples were treated as discussed in Section A.2. The concentrations of all of the samples are reported in Table 9. Table 9 also shows that the differences in absorption readings of the same sample are very small (unlike the readings in Table A-2).

Table A-9 Sample Absorbance Readings and Concentrations using Persulfate Digestion with Hot Plate and Vanadomolybdophosphoric Acid Method

	Absorbance at 420							
	A		B		C			Sample Phosphate Concentration (mg P/L)
Sample	1	2	1	2	1	2	Average	
DI	0.001	0.003	-0.002	0.001	-0.001	0.003	0.001	0.047
DI w/K	0.005	0.002	0.004	0	0.002	0.001	0.002	0.132
10 mg/L	0.169	0.165	0.175	0.173	0.17	0.172	0.171	9.684
1	0.159	0.164	0.167	0.162	0.166	0.165	0.164	9.296
2	0.154	0.155	0.155	0.156	0.153	0.152	0.154	8.747
3	0.149	0.148	0.15	0.152	0.154	0.153	0.151	8.568

The combination of Persulfate Digestion using a hot plate and the Vanadomolybdophosphoric Acid method provides reasonable results. This

combination was used to determine phosphate concentrations in the rest of the experiments during this research.

B SAMPLE CALCULATIONS

B.1 Percent Difference

$$PercentDifference = \frac{X_0 - X_1}{X_o} * 100$$

Where: X_o: initial value of interest

X_1: new value of interest

For X_o=25.4 and X_1=22.4

$$PercentDifference = \frac{25.4 - 22.4}{25.4} * 100$$

$$= 11.8\%$$

B.2 Hardness

The procedure for hardness involves a titration. The volume of titrant (EDTA) used is recorded.

Total Hardness (as mg/L $CaCO_3$) = (A * B * 1000) / V_s

Where: A: mL of titrant (EDTA)

B: mg CaCO3 equivalent to 1.00 ml EDTA titrant

V_s: Volume of sample (mL)

For A=12.8 and B=1 and V_s=50

Total Hardness = (12.8*1*1000)/(50)

= 256 mg $CaCO_3$/L

B.3 Phosphate Concentrations from Adsorbance Readings

$$C_{Sample} = C_{cuvette} * \frac{V_D}{V_{50}} * \frac{V_U}{V_{100}}$$

Where: C_{Sample}: Phosphate Concentration of Sample

$C_{cuvette}$: Phosphate Concentration in Cuvette (mg P/L)

$$C_{cuvette} = 23.258 * Absorbance_{420}$$

(23.258 found from calibration curves shown in Appendix C)

$Absorbance_{420}$ = Absorbance at 420 wavelength

(Substitute different standard curve relationship for different wavelength)

V_D = Volume of Digested sample added to 50 mL volumetric flask (mL)

V_{50} = 50 (Since 50 mL volumetric flask is used)

V_U = Volume of sample that is digested (50 mL, unless stated otherwise)

V_{100} = 100 (Representing the 100 mL flask that is used in the digestion procedure)

For $Absorbance_{420}$ = 0.222

$$C_{cuvette} = 23.258 * Absorbance_{420}$$

= 23.258*(0.222)

=5.16 mg P/L

$$C_{Sample} = C_{cuvette} * \frac{V_{50}}{V_D} * \frac{V_{100}}{V_U}$$

$$C_{Sample} = 5.16 * \frac{50}{35} * \frac{100}{50}$$

C_{Sample} = 14.8 mg P/L

C Calibration Curves

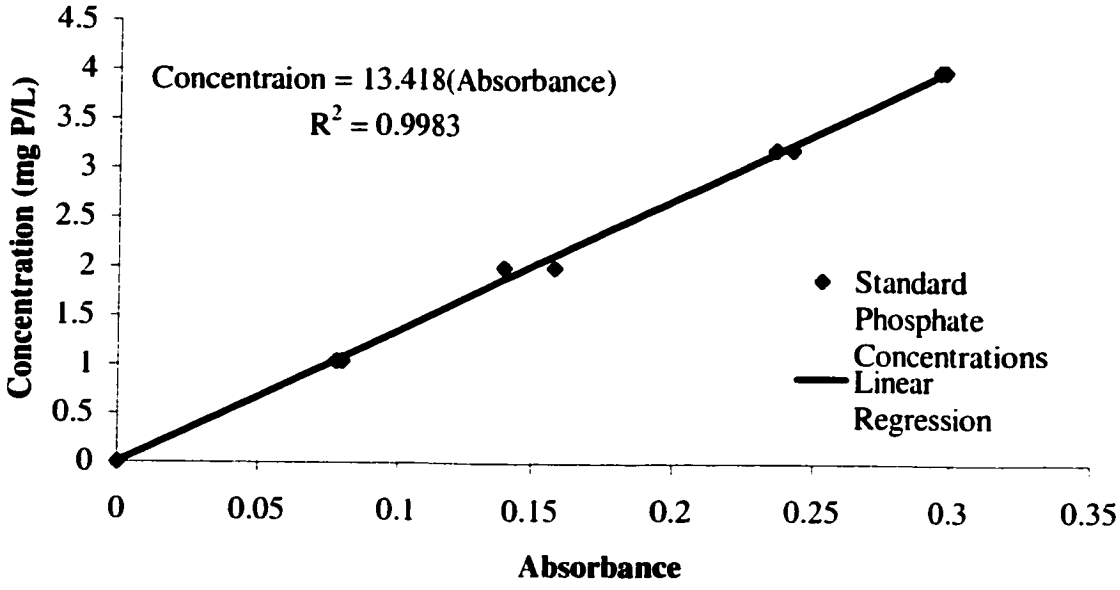

Figure C-1 Phosphorus Calibration Curve for 400 nm Wavelength

Table C-1 Phosphorus Calibration Curve Data for 400 nm, Wavelength

Standard Concentration (mg/L)	Absorbance at 400 nm
0	0
0	-0.003
1.04	0.078
1.04	0.08
2	0.14
2	0.158
3.2	0.242
3.2	0.236
4	0.298
4	0.296

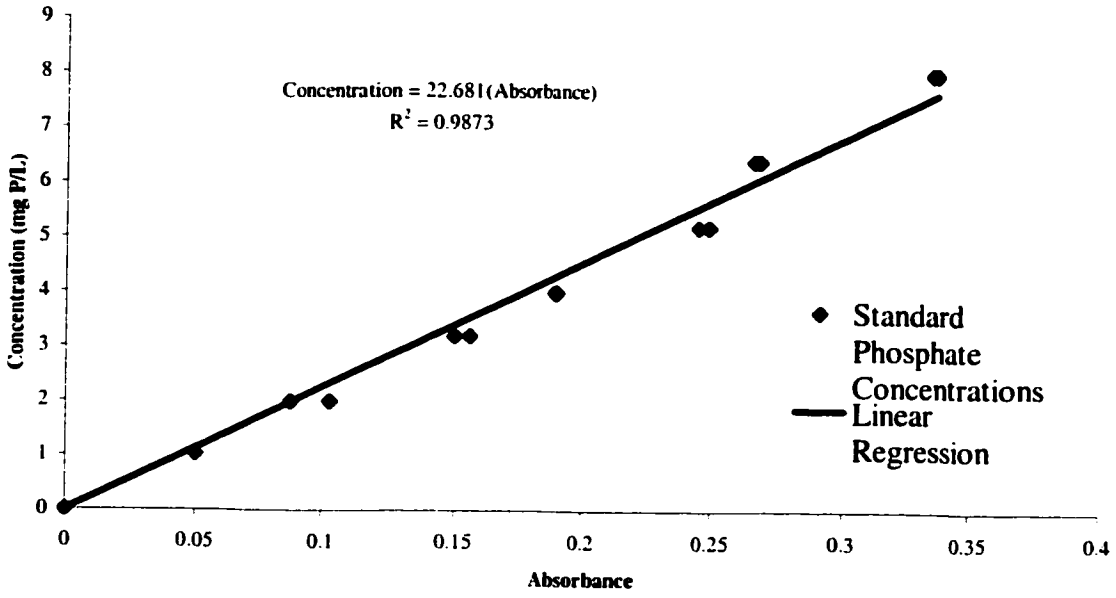

Figure C-2 Phosphorus Calibration Curve for 420 nm Wavelength

Table C-2 Phosphorus Calibration Curve Data for 420 nm Wavelength

Standard Concentration (mg/L)	Absorbance at 420 nm
0	0
0	0
2	0.088
2	0.103
3.2	0.156
3.2	0.15
4	0.19
4	0.189
5.2	0.245
5.2	0.249
6.4	0.266
6.4	0.268
8	0.337
8	0.335
10.4	0.422
10.4	0.421

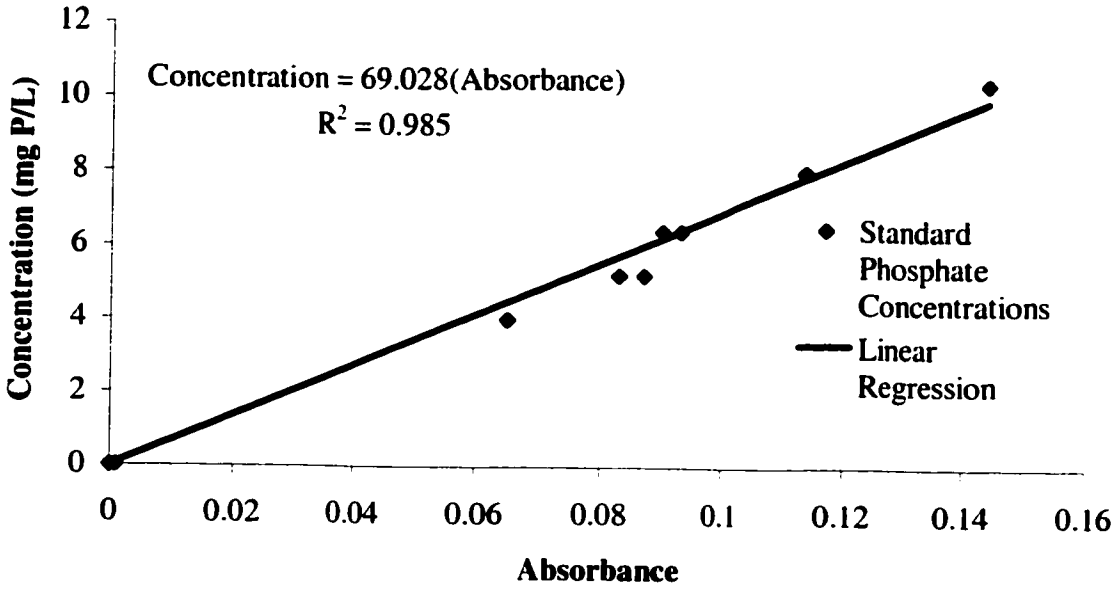

Figure C-3 Phosphorus Calibration Curve for 470 nm Wavelength

Table C-3 Phosphorus Calibration Curve Data for 470 nm Wavelength

Standard Concentration (mg/L)	Absorbance at 470 nm
0	0
0	0.001
4	0.065
4	0.065
5.2	0.083
5.2	0.087
6.4	0.09
6.4	0.093
8	0.114
8	0.114
10.4	0.144
10.4	0.144

D Raw Data

D.1 EDX Data

Table D-1 EDX Raw Data

	Atomic %					
	1	2	3	4	Stub Average	Overall Average
Stub 1						JTF <45
Mg	21.51	22.22	22.83	20.70	21.82	22.17
Al	9.43	9.04	8.62	9.63	9.18	8.90
Si	52.33	52.25	52.54	52.38	52.38	53.13
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	4.72	4.58	5.81	4.66	4.94	4.93
K	1.13	0.76	0.94	1.21	1.01	0.81
Fe	8.96	9.78	7.96	8.84	8.89	8.94
Cr	0.00	0.00	0.00	0.53	0.13	0.07
Na	1.92	1.36	0.80	1.43	1.38	0.76
Ti	0.00	0.00	0.48	0.59	0.27	0.29
Stub 2						
Mg	20.95	23.12	22.64	23.37	22.52	
Al	9.11	8.86	8.57	7.94	8.62	
Si	55.58	51.00	54.31	54.66	53.89	
S	0.00	0.00	0.00	0.00	0.00	
Ca	3.47	7.92	4.23	4.03	4.91	
K	0.83	0.40	0.52	0.69	0.61	
Fe	10.03	7.28	9.43	9.22	8.99	
Cr	0.00	0.00	0.00	0.00	0.00	
Na	0.00	0.59	0.00	0.00	0.15	
Ti	0.00	0.85	0.31	0.08	0.31	

	1	2	3	4	Stub Average	Overall Average
Stub 1						JTF 45-150
Mg	29.25	24.34	31.03	26.15	27.69	26.15
Al	6.62	8.49	6.14	6.65	6.98	7.43
Si	49.48	51.37	47.74	52.93	50.38	51.88
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	3.04	3.69	2.74	2.88	3.09	3.51
K	0.63	0.49	0.68	0.68	0.62	0.65
Fe	9.95	10.09	10.15	9.16	9.84	9.04
Cr	0.00	0.00	0.00	0.00	0.00	0.00
Na	1.03	1.09	1.38	1.53	1.26	1.10
Ti	0.00	0.42	0.12	0.01	0.14	0.24

Atomic %						
Stub 2						
Mg	23.19	26.00	25.61	23.61	24.60	
Al	6.73	7.63	9.47	7.71	7.89	
Si	54.75	52.56	52.18	54.05	53.39	
S	0.00	0.00	0.00	0.00	0.00	
Ca	4.07	4.08	2.91	4.64	3.93	
K	0.89	0.59	0.73	0.51	0.68	
Fe	9.50	7.69	7.68	8.08	8.24	
Cr	0.01	0.00	0.00	0.00	0.00	
Na	0.53	1.22	0.95	1.08	0.95	
Ti	0.33	0.23	0.47	0.32	0.34	
	1	2	3	4	Stub Average	Overall Average
Stub 1						JTF 150-355
Mg	26.45	28.73	29.23	28.14	28.14	28.16
Al	7.93	7.86	6.55	6.34	7.17	7.08
Si	48.22	43.79	48.11	51.22	47.84	48.67
S	0.00	0.66	0.00	0.00	0.17	0.08
Ca	2.80	3.33	4.18	3.23	3.39	3.09
K	1.00	0.41	0.87	0.51	0.70	0.82
Fe	11.95	14.20	9.66	9.13	11.24	10.45
Cr	0.00	0.00	0.00	0.00	0.00	0.34
Na	1.14	0.84	1.25	1.20	1.11	1.01
Ti	0.52	0.19	0.15	0.23	0.27	0.29
Stub 2						
Mg	28.74	29.90	27.65	26.44	28.18	
Al	7.09	4.94	7.04	8.91	7.00	
Si	49.82	49.04	50.70	48.49	49.51	
S	0.00	0.00	0.00	0.00	0.00	
Ca	1.58	3.17	4.45	2.01	2.80	
K	1.04	0.60	0.71	1.44	0.95	
Fe	10.40	9.93	8.15	10.18	9.67	
Cr	0.56	1.43	0.00	0.72	0.68	
Na	0.68	0.53	1.03	1.41	0.91	
Ti	0.10	0.45	0.26	0.39	0.30	

	Atomic Percentage					
	1	2	3	4	Stub Average	Overall Average
Stub 1						JTF >355
Mg	30.42	33.40	37.31	32.38	33.38	31.78
Al	6.60	6.58	4.41	6.17	5.94	6.39
Si	50.09	47.63	46.38	49.92	48.51	49.10
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	2.45	2.73	1.39	1.79	2.09	3.07
K	0.04	0.19	0.00	0.28	0.13	0.24
Fe	8.71	8.58	9.44	7.96	8.67	8.37
Cr	0.00	0.00	0.24	0.00	0.06	0.04
Na	1.68	0.88	0.61	1.31	1.12	0.76
Ti	0.00	0.00	0.23	0.20	0.11	0.26
Stub 2						
Mg	34.72	31.10	28.84	26.05	30.18	
Al	4.51	7.15	7.51	8.16	6.83	
Si	47.94	50.12	49.05	51.69	49.70	
S	0.00	0.00	0.00	0.00	0.00	
Ca	3.39	3.20	5.17	4.47	4.06	
K	0.19	0.30	0.44	0.50	0.36	
Fe	8.81	7.43	8.37	7.63	8.06	
Cr	0.00	0.04	0.00	0.00	0.01	
Na	0.00	0.55	0.17	0.87	0.40	
Ti	0.44	0.11	0.46	0.61	0.41	

	1	2	3	4	Stub Average	Overall Average
Stub 1						AFE <45
Mg	18.92	17.13	17.11	18.27	17.86	17.59
Al	10.18	10.62	10.70	11.16	10.67	10.75
Si	56.75	56.36	57.69	55.77	56.64	56.22
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	4.33	5.64	6.52	5.91	5.60	5.34
K	1.37	1.39	1.12	1.56	1.36	1.37
Fe	7.41	8.68	6.81	7.04	7.49	7.85
Cr	0.00	0.00	0.00	0.00	0.00	0.15
Na	0.00	0.00	0.00	0.00	0.00	0.35
Ti	1.02	0.18	0.04	0.30	0.39	0.38

Atomic Percentage						
Stub 2						
Mg	18.06	17.23	17.81	16.21	17.33	
Al	9.59	11.64	11.12	10.99	10.84	
Si	55.62	54.89	57.11	55.60	55.81	
S	0.00	0.00	0.00	0.00	0.00	
Ca	5.28	4.99	4.40	5.62	5.07	
K	1.44	1.25	1.24	1.58	1.38	
Fe	8.45	7.90	7.59	8.92	8.22	
Cr	0.55	0.00	0.64	0.00	0.30	
Na	0.49	1.25	0.10	0.99	0.71	
Ti	0.50	0.88	0.00	0.09	0.37	

	1	2	3	4	Stub Average	Overall Average
Stub 1						AFE 45-150
Mg	18.97	17.82	18.22	18.95	18.49	18.54
Al	10.19	8.92	10.21	8.64	9.49	8.88
Si	54.58	58.54	55.20	55.75	56.02	57.11
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	5.41	4.07	4.90	5.70	5.02	4.76
K	1.25	1.60	1.36	1.39	1.40	1.23
Fe	9.19	8.73	9.56	9.56	9.26	8.79
Cr	0.00	0.00	0.00	0.00	0.00	0.10
Na	0.00	0.00	0.00	0.00	0.00	0.30
Ti	0.42	0.33	0.54	0.00	0.32	0.29
Stub 2						
Mg	18.73	18.72	15.65	21.29	18.60	
Al	9.01	9.17	7.15	7.76	8.27	
Si	56.23	55.91	65.82	54.86	58.21	
S	0.00	0.00	0.00	0.00	0.00	
Ca	5.06	5.26	2.91	4.73	4.49	
K	1.44	1.32	0.70	0.81	1.07	
Fe	8.26	8.86	6.94	9.18	8.31	
Cr	0.41	0.39	0.00	0.00	0.20	
Na	0.64	0.37	0.24	1.13	0.60	
Ti	0.21	0.00	0.59	0.24	0.26	

	Atomic Percentage					
	1	2	3	4	Stub Average	Overall Average
Stub 1						AFE 150-355
Mg	21.36	19.34	22.35	21.71	21.19	22.13
Al	8.41	8.44	8.76	8.55	8.54	8.80
Si	54.84	55.30	53.32	54.57	54.51	53.39
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	4.28	5.54	5.35	4.03	4.80	4.29
K	1.18	0.95	0.99	1.01	1.03	1.18
Fe	9.45	9.53	8.11	9.43	9.13	9.08
Cr	0.00	0.00	0.00	0.00	0.00	0.11
Na	0.00	0.00	0.00	0.00	0.00	0.43
Ti	0.47	0.90	1.15	0.68	0.80	0.60
Stub 2						
Mg	23.74	23.09	21.77	23.65	23.06	
Al	9.98	8.08	9.58	8.63	9.07	
Si	50.92	53.04	53.71	51.39	52.27	
S	0.00	0.00	0.00	0.00	0.00	
Ca	3.63	3.89	4.00	3.57	3.77	
K	1.57	0.98	1.26	1.49	1.33	
Fe	9.07	9.55	8.64	8.87	9.03	
Cr	0.00	0.30	0.10	0.50	0.23	
Na	0.73	0.83	0.66	1.18	0.85	
Ti	0.37	0.22	0.30	0.71	0.40	

	1	2	3	4	Stub Average	Overall Average
Stub 1						AFE >355
Mg	22.99	23.04	19.80	26.06	22.97	22.30
Al	7.72	10.09	10.35	6.98	8.79	9.06
Si	57.09	53.87	56.30	52.96	55.06	53.57
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	4.02	3.72	4.24	4.99	4.24	4.56
K	0.77	1.33	1.28	0.80	1.05	1.11
Fe	7.42	7.99	7.81	7.80	7.76	8.82
Cr	0.00	0.00	0.00	0.00	0.00	0.00
Na	0.00	0.00	0.00	0.00	0.00	0.45
Ti	0.00	0.00	0.20	0.40	0.15	0.13

Atomic Percentage						
Stub 2						
Mg	20.55	22.06	21.89	22.02	21.63	
Al	10.52	9.57	9.20	8.06	9.34	
Si	50.78	55.28	50.46	51.81	52.08	
S	0.00	0.00	0.00	0.00	0.00	
Ca	5.68	3.04	5.38	5.41	4.88	
K	2.09	0.79	0.94	0.89	1.18	
Fe	8.93	8.83	11.19	10.55	9.88	
Cr	0.00	0.00	0.02	0.00	0.01	
Na	1.28	0.45	0.71	1.19	0.91	
Ti	0.15	0.00	0.20	0.08	0.11	

	1	2	3	4	Stub Average	Overall Average
Stub 1						JFE <45
Mg	20.06	21.44	22.74	22.34	21.65	21.50
Al	7.79	10.07	7.35	7.59	8.20	8.25
Si	54.12	48.92	54.02	53.30	52.59	52.65
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	4.91	5.23	5.36	5.55	5.26	5.50
K	1.11	1.30	1.04	1.07	1.13	1.09
Fe	12.01	13.06	9.49	10.17	11.18	10.89
Cr	0.00	0.00	0.00	0.00	0.00	0.01
Na	0.00	0.00	0.00	0.00	0.00	0.08
Ti	0.00	0.00	0.00	0.00	0.00	0.05
Stub 2						
Mg	21.06	21.33			21.20	
Al	7.43	9.26			8.35	
Si	51.89	53.67			52.78	
S	0.00	0.00			0.00	
Ca	6.15	5.79			5.97	
K	1.30	0.72			1.01	
Fe	12.10	8.52			10.31	
Cr	0.08	0.00			0.04	
Na	0.00	0.45			0.23	
Ti	0.00	0.28			0.14	

	Atomic Percentage					
	1	2	3	4	Stub Average	Overall Average
Stub 1						JFE 45-150
Mg	22.02	26.04	20.66	19.50	22.06	22.44
Al	7.48	3.41	9.95	9.33	7.54	7.46
Si	53.31	63.13	56.93	53.56	56.73	55.51
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	4.02	0.64	2.94	8.97	4.14	4.13
K	0.46	0.62	1.34	0.80	0.81	0.81
Fe	12.70	6.16	8.18	7.83	8.72	9.23
Cr	0.00	0.00	0.00	0.00	0.00	0.03
Na	0.00	0.00	0.00	0.00	0.00	0.30
Ti	0.00	0.00	0.00	0.00	0.00	0.10
Stub 2						
Mg	23.42	22.99			23.21	
Al	7.04	7.56			7.30	
Si	52.46	53.67			53.07	
S	0.00	0.00			0.00	
Ca	4.31	3.88			4.10	
K	0.81	0.83			0.82	
Fe	10.44	10.04			10.24	
Cr	0.09	0.07			0.08	
Na	0.99	0.79			0.89	
Ti	0.44	0.17			0.31	

	1	2	3	4	Stub Average	Overall Average
Stub 1						JFE 150-355
Mg	25.73	23.12	24.92	25.37	24.79	24.94
Al	8.97	10.23	10.07	11.17	10.11	9.56
Si	47.21	46.41	46.47	45.12	46.30	46.32
S	0.00	0.00	0.00	0.00	0.00	0.10
Ca	1.44	1.44	2.10	1.97	1.74	1.98
K	2.21	2.93	2.76	2.53	2.61	2.49
Fe	12.18	13.63	10.40	11.35	11.89	11.84
Cr	0.00	0.00	0.00	0.00	0.00	0.04
Na	1.06	1.94	2.16	1.89	1.76	2.10
Ti	0.00	0.00	0.00	0.00	0.00	0.23

Atomic Percentage						
Stub 2						
Mg	25.98	25.16	26.19	23.06	25.10	
Al	9.03	9.26	7.53	10.20	9.01	
Si	42.48	48.91	48.20	45.74	46.33	
S	0.79	0.00	0.00	0.00	0.20	
Ca	2.18	1.94	1.65	3.09	2.22	
K	2.76	2.16	1.73	2.83	2.37	
Fe	11.39	11.40	12.59	11.81	11.80	
Cr	0.19	0.00	0.02	0.11	0.08	
Na	4.57	1.07	1.70	2.44	2.45	
Ti	0.63	0.10	0.41	0.70	0.46	
	1	2	3	4	Stub Average	Overall Average
Stub 1						JFE >355
Mg	27.42	24.20	24.67	28.74	26.26	25.34
Al	7.14	6.97	6.08	5.69	6.47	7.27
Si	55.51	52.71	56.99	52.68	54.47	53.47
S	0.00	0.00	0.00	0.00	0.00	0.00
Ca	2.90	6.64	3.52	3.26	4.08	4.01
K	0.43	0.93	0.62	0.53	0.63	0.65
Fe	6.59	8.50	7.37	8.57	7.76	8.49
Cr	0.00	0.00	0.00	0.00	0.00	0.04
Na	0.00	0.00	0.00	0.00	0.00	0.48
Ti	0.00	0.04	0.74	0.52	0.33	0.25
Stub 2						
Mg	24.54	24.65	28.06	26.04	25.82	
Al	6.59	7.60	8.24	9.11	7.89	
Si	54.52	53.42	49.62	51.88	52.36	
S	0.00	0.00	0.00	0.00	0.00	
Ca	3.89	4.36	3.20	2.54	3.50	
K	0.64	0.40	0.42	0.96	0.61	
Fe	9.30	9.47	9.48	8.90	9.29	
Cr	0.03	0.00	0.00	0.00	0.01	
Na	0.44	0.11	0.47	0.54	0.39	
Ti	0.06	0.00	0.49	0.03	0.15	

D.2 Hardness Data

Table D-2 Hardness Raw Data

	Buret Volume			Buret Volume					
Sample	Start	Stop	Volume Used	Start	Stop	Volume Used	Average Difference	Hardness	Number of Rinses
0-1	1.34	14.33	12.99	33.99	46.84	12.85	12.80	255.9	0
0-2	21.30	33.90	12.60	5.60	18.34	12.74			
1-1	15.15	16.05	0.90	18.34	19.29	0.95	1.31	26.2	1
1-2	19.58	21.30	1.72	19.29	20.96	1.67			
2-1	17.45	18.20	0.75	4.79	5.60	0.81	0.88	17.6	2
2-2	18.34	19.41	1.07	20.96	21.85	0.89			
3-1	16.05	16.60	0.55	3.40	4.12	0.72	0.63	12.6	3
3-2	16.80	17.38	0.58	4.12	4.79	0.67			
4-1	17.38	17.95	0.57	18.61	19.14	0.53	0.60	11.9	4
4-2	17.95	18.61	0.66	19.14	19.76	0.62			

D.3 Calibration Curve Data

See Appendix C.

D.4 Equilibrium Time Data

Table D-3 5°C Equilibrium Time Absorbance Readings

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 1	0	0			0
Batch 1	0	0			1
Batch 2	0	0			0
Batch 2	0	0			1
Batch 3	0	0			0
Batch 3	0	0			-4
Batch 4	0	0			0
Batch 4	0	0			0
Batch 5	0	0			0
Batch 5	0	0			5

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 3	50	2			83
Batch 3	100	2			78
Batch 3	100	2			78
Batch 4	50	3			110
Batch 4	100	3			114
Batch 4	100	3			114
Batch 5	50	4			151
Batch 5	100	4			164
Batch 5	100	4			162
Batch 1	50	8			319
Batch 1	100	8			339
Batch 1	100	8			338
Batch 2	50	10			545
Batch 2	100	10			550
Batch 2	100	10			553
Batch 4	11		10		177
Batch 4	11		10		178
Batch 4	12		10		175
Batch 4	12		10		176
Batch 5	8		DI		28
Batch 5	8		DI		11
Batch 5	9		DI		8
Batch 5	9		DI		0
Batch 5	10		DI		1
Batch 5	10		DI		1
Batch 5	11		DI w/k		3
Batch 5	11		DI w/k		3
Batch 5	12		DI w/k		4
Batch 5	12		DI w/k		3
Batch 5	13		DI w/k		10
Batch 5	13		DI w/k		4
Batch 1	7			0.01	171
Batch 1	7			0.01	169
Batch 1	8			0.01	162
Batch 1	8			0.01	162
Batch 1	9			0.01	171
Batch 1	9			0.01	170
Batch 2	11			0.03	158
Batch 2	11			0.03	158
Batch 2	12			0.03	162
Batch 2	12			0.03	159
Batch 2	13			0.03	162
Batch 2	13			0.03	161
Batch 1	4			0.04	175
Batch 1	4			0.04	163

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 1	5			0.04	167
Batch 1	5			0.04	171
Batch 1	6			0.04	238
Batch 1	6			0.04	214
Batch 1	13			0.08	163
Batch 1	13			0.08	163
Batch 1	14			0.08	161
Batch 1	14			0.08	160
Batch 2	1			0.08	154
Batch 2	1			0.08	154
Batch 1	10			0.13	163
Batch 1	10			0.13	163
Batch 1	11			0.13	172
Batch 1	11			0.13	162
Batch 1	12			0.13	169
Batch 1	12			0.13	169
Batch 2	8			0.25	160
Batch 2	8			0.25	160
Batch 2	9			0.25	241
Batch 2	9			0.25	160
Batch 2	10			0.25	152
Batch 2	10			0.25	156
Batch 4	4			0.25	153
Batch 1	1			0.50	156
Batch 1	1			0.50	161
Batch 1	2			0.50	162
Batch 1	2			0.50	163
Batch 1	3			0.50	169
Batch 1	3			0.50	159
Batch 2	5			1.00	151
Batch 2	5			1.00	151
Batch 2	6			1.00	151
Batch 2	6			1.00	150
Batch 2	7			1.00	153
Batch 2	7			1.00	152
Batch 2	2			1.58	156
Batch 2	2			1.58	154
Batch 2	3			1.58	158
Batch 2	3			1.58	156
Batch 2	4			1.58	157
Batch 2	4			1.58	157
Batch 3	6			2.13	154
Batch 3	6			2.13	148
Batch 3	7			2.13	149
Batch 3	7			2.13	155

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 3	8			2.13	148
Batch 3	8			2.13	154
Batch 3	3			2.63	167
Batch 3	3			2.63	318
Batch 3	4			2.63	173
Batch 3	4			2.63	152
Batch 3	5			2.63	153
Batch 3	5			2.63	169
Batch 4	2			2.63	147
Batch 4	3			2.63	147
Batch 4	3			2.63	149
Batch 2	14			3.08	158
Batch 2	14			3.08	153
Batch 3	1			3.08	278
Batch 3	1			3.08	149
Batch 3	2			3.08	147
Batch 3	2			3.08	155
Batch 4	2			3.08	144
Batch 3	12			3.71	157
Batch 3	12			3.71	309
Batch 3	13			3.71	159
Batch 3	13			3.71	154
Batch 3	14			3.71	150
Batch 3	14			3.71	165
Batch 4	1			3.71	186
Batch 3	9			4.17	151
Batch 3	9			4.17	149
Batch 3	10			4.17	64
Batch 3	10			4.17	152
Batch 3	11			4.17	155
Batch 3	11			4.17	152
Batch 4	1			4.17	147
Batch 4	8			4.54	147
Batch 4	8			4.54	146
Batch 4	9			4.54	149
Batch 4	9			4.54	149
Batch 4	10			4.54	150
Batch 4	10			4.54	151
Batch 4	5			5.08	144
Batch 4	5			5.08	146
Batch 4	6			5.08	187
Batch 4	6			5.08	167
Batch 4	7			5.08	157
Batch 4	7			5.08	143
Batch 5	1			5.08	150

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 5	1			5.08	145
Batch 5	3			5.67	153
Batch 5	3			5.67	155
Batch 5	4			5.67	149
Batch 5	4			5.67	148
Batch 5	5			5.67	150
Batch 5	5			5.67	150
Batch 4	13			6.08	147
Batch 4	13			6.08	146
Batch 4	14			6.08	142
Batch 4	14			6.08	148
Batch 5	2			6.08	146
Batch 5	2			6.08	146
Batch 5	6			6.54	159
Batch 5	6			6.54	163
Batch 5	7			6.54	155
Batch 5	7			6.54	562
Batch 5	14			6.54	150
Batch 5	14			6.54	152

Table D-4 18°C Equilibrium Time Absorbance Readings

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 1	0	0			0
Batch 1	0	0			2
Batch 2	0	0			0
Batch 2	0	0			2
Batch 3	0	0			0
Batch 3	0	0			0
Batch 4	0	0			0
Batch 4	0	0			2
Batch 5	0	0			0
Batch 5	0	0			0
Batch 3	50	2			83
Batch 3	100	2			77
Batch 3	100	2			78
Batch 1	50	3			119
Batch 1	100	3			119
Batch 1	100	3			119
Batch 4	50	5			213
Batch 4	100	5			224
Batch 4	100	5			224

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 5	50	6			228
Batch 5	100	6			241
Batch 5	100	6			240
Batch 2	50	7			309
Batch 2	100	7			318
Batch 2	100	7			307
Batch 5	7		10		161
Batch 5	7		10		161
Batch 5	8		10		164
Batch 5	8		10		161
Batch 4	1		DI		6
Batch 4	1		DI		2
Batch 4	2		DI		0
Batch 4	2		DI		1
Batch 4	3		DI		1
Batch 4	3		DI		3
Batch 3	12		DI w/k		3
Batch 3	12		DI w/k		15
Batch 3	13		DI w/k		2
Batch 3	13		DI w/k		2
Batch 3	14		DI w/k		1
Batch 3	14		DI w/k		2
Batch 4	10			0	176
Batch 4	10			0	175
Batch 4	11			0	175
Batch 4	11			0	177
Batch 4	12			0	176
Batch 4	12			0	175
Batch 1	7			0.01	159
Batch 1	7			0.01	167
Batch 1	8			0.01	164
Batch 1	8			0.01	164
Batch 1	9			0.01	151
Batch 1	9			0.01	161
Batch 2	11			0.02	167
Batch 2	11			0.02	177
Batch 2	12			0.02	162
Batch 2	12			0.02	165
Batch 2	13			0.02	164
Batch 2	13			0.02	164
Batch 2	8			0.02	164
Batch 2	8			0.02	162
Batch 2	9			0.02	165
Batch 2	9			0.02	160
Batch 2	10			0.02	175

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 2	10			0.02	160
Batch 2	5			0.03	169
Batch 2	5			0.03	169
Batch 2	6			0.03	170
Batch 2	6			0.03	169
Batch 2	7			0.03	160
Batch 2	7			0.03	157
Batch 1	4			0.04	159
Batch 1	4			0.04	169
Batch 1	5			0.04	165
Batch 1	5			0.04	162
Batch 1	6			0.04	159
Batch 1	6			0.04	162
Batch 1	1			0.06	156
Batch 1	1			0.06	148
Batch 1	2			0.06	166
Batch 1	2			0.06	160
Batch 1	3			0.06	157
Batch 1	3			0.06	160
Batch 1	13			0.08	158
Batch 1	13			0.08	157
Batch 1	14			0.08	160
Batch 1	14			0.08	157
Batch 2	1			0.08	162
Batch 2	1			0.08	159
Batch 1	10			0.13	162
Batch 1	10			0.13	179
Batch 1	11			0.13	158
Batch 1	11			0.13	158
Batch 1	12			0.13	156
Batch 1	12			0.13	165
Batch 2	2			0.25	169
Batch 2	2			0.25	159
Batch 2	3			0.25	156
Batch 2	3			0.25	161
Batch 2	4			0.25	156
Batch 2	4			0.25	158
Batch 3	9			0.54	201
Batch 3	9			0.54	178
Batch 3	10			0.54	163
Batch 3	10			0.54	165
Batch 3	11			0.54	158
Batch 3	11			0.54	159
Batch 2	14			1.08	160
Batch 2	14			1.08	162

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 3	1			1.08	149
Batch 3	1			1.08	151
Batch 3	2			1.08	156
Batch 3	2			1.08	157
Batch 3	6			1.48	159
Batch 3	6			1.48	290
Batch 3	7			1.48	159
Batch 3	7			1.48	160
Batch 3	8			1.48	160
Batch 3	8			1.48	155
Batch 3	3			2.00	146
Batch 3	3			2.00	158
Batch 3	4			2.00	149
Batch 3	4			2.00	159
Batch 3	5			2.00	160
Batch 3	5			2.00	158
Batch 4	7			2.58	157
Batch 4	7			2.58	156
Batch 4	8			2.58	155
Batch 4	8			2.58	155
Batch 4	9			2.58	153
Batch 4	9			2.58	153
Batch 4	4			3.13	147
Batch 4	4			3.13	156
Batch 4	5			3.13	149
Batch 4	5			3.13	150
Batch 4	6			3.13	152
Batch 4	6			3.13	183
Batch 5	4			3.58	156
Batch 5	4			3.58	157
Batch 5	5			3.58	152
Batch 5	5			3.58	160
Batch 5	6			3.58	164
Batch 5	6			3.58	158
Batch 5	1			4.08	154
Batch 5	1			4.08	156
Batch 5	2			4.08	150
Batch 5	2			4.08	152
Batch 5	3			4.08	153
Batch 5	3			4.08	153
Batch 5	12			4.50	162
Batch 5	12			4.50	160
Batch 5	13			4.50	282
Batch 5	13			4.50	328
Batch 5	14			4.50	163

Batch	Sample	Standard	Control	Time (Days)	Absorbance at 420
Batch 5	14			4.50	158
Batch 5	9			5.06	148
Batch 5	9			5.06	148
Batch 5	10			5.06	151
Batch 5	10			5.06	181
Batch 5	11			5.06	152
Batch 5	11			5.06	177
Batch 5	4			6.71	155
Batch 5	4			6.71	155
Batch 5	5			6.71	159
Batch 5	5			6.71	156

D.5 Adsorption Isotherm Data

Table D-5 Adsorption Isotherm Absorbance Readings

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Run 1				
Batch 1	0	0		0
Batch 1	0	0		0
Batch 2	0	0		0
Batch 2	0	0		0
Batch 1	50	1		97
Batch 1	100	1		926
Batch 1	100	1		952
Batch 2	50	1		741
Batch 2	100	1		397
Batch 2	100	1		405
Batch 1	7		0.5	143
Batch 1	7		0.5	146
Batch 1	8		0.5	147
Batch 1	8		0.5	147
Batch 1	9		0.5	148
Batch 1	9		0.5	148
Batch 1	14		1.25	129
Batch 1	14		1.25	132
Batch 1	13		1.5	127
Batch 1	13		1.5	123
Batch 1	12		1.75	126
Batch 1	12		1.75	123

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Batch 2	13		2	126
Batch 2	13		2	130
Batch 2	14		2	128
Batch 2	14		2	125
Batch 2	11		2.5	109
Batch 2	11		2.5	111
Batch 2	12		2.5	114
Batch 2	12		2.5	112
Batch 2	8		3	96
Batch 2	8		3	102
Batch 2	9		3	105
Batch 2	9		3	106
Batch 2	10		3	98
Batch 2	10		3	96
Batch 2	6		5	64
Batch 2	6		5	65
Batch 2	7		5	57
Batch 2	7		5	58
Batch 2	4		6	118
Batch 2	4		6	114
Batch 2	5		6	59
Batch 2	5		6	58
Batch 1	11		7	98
Batch 2	2		7	108
Batch 2	3		7	50
Batch 2	3		7	50
Batch 1	4		8	54
Batch 1	4		8	55
Batch 1	5		8	56
Batch 1	5		8	54
Batch 1	6		8	51
Batch 1	6		8	50
Batch 1	1		10	47
Batch 1	1		10	43
Batch 1	2		10	37
Batch 1	2		10	36
Batch 1	3		10	42
Batch 1	3		10	42
Batch 1	10		10	66
Batch 1	10		10	76
Batch 2	1		10	41
Batch 2	1		10	33
Run 2				
Batch 1	0	0		0
Batch 1	0	0		0

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Batch 1	50	4		131
Batch 1	100	4		157
Batch 1	100	4		158
Batch 1	6		0.5	152
Batch 1	6		0.5	154
Batch 1	12		0.5	148
Batch 1	12		0.5	149
Batch 1	3		1	144
Batch 1	3		1	139
Batch 1	4		1	138
Batch 1	4		1	137
Batch 1	5		1	141
Batch 1	5		1	145
Batch 1	1		2	121
Batch 1	1		2	122
Batch 1	2		2	127
Batch 1	2		2	126
Batch 1	11		2	116
Batch 1	11		2	118
Batch 1	9		4	80
Batch 1	9		4	82
Batch 1	10		4	82
Batch 1	10		4	83
Batch 1	14		8	40
Batch 1	14		8	39
Batch 1	7		15	25
Batch 1	7		15	21
Batch 1	8		15	20
Batch 1	8		15	20
Batch 1	13		15	25
Batch 1	13		15	17

D.6 Kimberlite, Coagulant and Flocculant Adsorption Isotherm Data

Table D-6 Kimberlite with Coagulant Adortption Isotherm Data

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Batch 1	0	0		0
Batch 1	0	0		0
Batch 2	0	0		0

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Batch 2	0	0		0
Batch 1	50	1		97
Batch 1	100	2		120
Batch 1	100	2		122
Batch 2	50	3		140
Batch 2	100	4		160
Batch 2	100	4		161
Batch 1	7		0.5	142
Batch 1	7		0.5	145
Batch 1	8		0.5	146
Batch 1	8		0.5	148
Batch 1	9		0.5	147
Batch 1	9		0.5	149
Batch 1	14		1.25	128
Batch 1	14		1.25	131
Batch 1	13		1.5	124
Batch 1	13		1.5	120
Batch 1	12		1.75	125
Batch 1	12		1.75	120
Batch 2	13		2	124
Batch 2	13		2	126
Batch 2	14		2	128
Batch 2	14		2	124
Batch 2	11		2.5	105
Batch 2	11		2.5	111
Batch 2	12		2.5	116
Batch 2	12		2.5	110
Batch 2	8		3	100
Batch 2	8		3	95
Batch 2	9		3	96
Batch 2	9		3	99
Batch 2	10		3	97
Batch 2	10		3	95
Batch 2	6		5	64
Batch 2	6		5	61
Batch 2	7		5	55
Batch 2	7		5	59
Batch 2	4		6	120
Batch 2	4		6	116
Batch 2	5		6	57
Batch 2	5		6	60
Batch 1	11		7	97
Batch 2	2		7	110
Batch 2	3		7	54
Batch 2	3		7	56

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Batch 1	4		8	57
Batch 1	4		8	51
Batch 1	5		8	54
Batch 1	5		8	58
Batch 1	6		8	50
Batch 1	6		8	49
Batch 1	1		10	46
Batch 1	1		10	42
Batch 1	2		10	37
Batch 1	2		10	31
Batch 1	3		10	41
Batch 1	3		10	42
Batch 1	10		10	64
Batch 1	10		10	69
Batch 2	1		10	40
Batch 2	1		10	35

Table D-7 Kimberlite and Flocculant Adsorption Isotherm Data

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Batch 1	0	0		0
Batch 1	0	0		0
Batch 2	0	0		0
Batch 2	0	0		0
Batch 1	50	5		155
Batch 1	100	6		169
Batch 1	100	6		170
Batch 2	50	7		185
Batch 2	100	8		206
Batch 2	100	8		204
Batch 1	7		0.5	139
Batch 1	7		0.5	142
Batch 1	8		0.5	143
Batch 1	8		0.5	145
Batch 1	9		0.5	144
Batch 1	9		0.5	146
Batch 1	14		1.25	125
Batch 1	14		1.25	128
Batch 1	13		1.5	121
Batch 1	13		1.5	117
Batch 1	12		1.75	122
Batch 1	12		1.75	117

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Batch 2	13		2	121
Batch 2	13		2	123
Batch 2	14		2	125
Batch 2	14		2	121
Batch 2	11		2.5	102
Batch 2	11		2.5	108
Batch 2	12		2.5	113
Batch 2	12		2.5	107
Batch 2	8		3	97
Batch 2	8		3	92
Batch 2	9		3	93
Batch 2	9		3	96
Batch 2	10		3	94
Batch 2	10		3	92
Batch 2	6		5	61
Batch 2	6		5	58
Batch 2	7		5	52
Batch 2	7		5	56
Batch 2	4		6	117
Batch 2	4		6	113
Batch 2	5		6	54
Batch 2	5		6	57
Batch 1	11		7	94
Batch 2	2		7	107
Batch 2	3		7	51
Batch 2	3		7	53
Batch 1	4		8	54
Batch 1	4		8	48
Batch 1	5		8	51
Batch 1	5		8	55
Batch 1	6		8	47
Batch 1	6		8	46
Batch 1	1		10	43
Batch 1	1		10	39
Batch 1	2		10	34
Batch 1	2		10	28
Batch 1	3		10	38
Batch 1	3		10	39
Batch 1	10		10	61
Batch 1	10		10	66
Batch 2	1		10	37
Batch 2	1		10	32

Table D-8 Kimberlite, Coagulant and Flocculant Data

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Batch 1	0	0		0
Batch 1	0	0		0
Batch 2	0	0		0
Batch 2	0	0		0
Batch 1	50	5		155
Batch 1	100	6		169
Batch 1	100	6		170
Batch 2	50	7		185
Batch 2	100	8		206
Batch 2	100	8		204
Batch 1	7		0.5	148
Batch 1	7		0.5	151
Batch 1	8		0.5	152
Batch 1	8		0.5	154
Batch 1	9		0.5	153
Batch 1	9		0.5	155
Batch 1	14		1.25	134
Batch 1	14		1.25	137
Batch 1	13		1.5	130
Batch 1	13		1.5	126
Batch 1	12		1.75	131
Batch 1	12		1.75	126
Batch 2	13		2	130
Batch 2	13		2	132
Batch 2	14		2	134
Batch 2	14		2	130
Batch 2	11		2.5	111
Batch 2	11		2.5	117
Batch 2	12		2.5	122
Batch 2	12		2.5	116
Batch 2	8		3	106
Batch 2	8		3	101
Batch 2	9		3	102
Batch 2	9		3	105
Batch 2	10		3	103
Batch 2	10		3	101
Batch 2	6		5	70
Batch 2	6		5	67
Batch 2	7		5	61
Batch 2	7		5	65
Batch 2	4		6	126

Batch	Sample	Standard	Kimberlite Dose	Absorbance at 420
Batch 2	4		6	122
Batch 2	5		6	63
Batch 2	5		6	66
Batch 1	11		7	103
Batch 2	2		7	116
Batch 2	3		7	60
Batch 2	3		7	62
Batch 1	4		8	63
Batch 1	4		8	57
Batch 1	5		8	60
Batch 1	5		8	64
Batch 1	6		8	56
Batch 1	6		8	55
Batch 1	1		10	52
Batch 1	1		10	48
Batch 1	2		10	43
Batch 1	2		10	37
Batch 1	3		10	47
Batch 1	3		10	48
Batch 1	10		10	70
Batch 1	10		10	75
Batch 2	1		10	46
Batch 2	1		10	41

Table D-9 Coagulant Adsorption Data

Batch	Sample	Coagulant Dose	Absorbance at 420
Batch 1	7	1	176
Batch 1	7	1	174
Batch 1	8	1	172
Batch 1	8	1	171
Batch 1	9	1	169
Batch 1	9	1	168
Batch 1	14	2	169
Batch 1	14	2	170
Batch 1	13	2	172
Batch 1	13	2	173
Batch 1	12	2	174
Batch 1	12	2	172
Batch 2	13	3	177
Batch 2	13	3	175
Batch 2	14	3	173
Batch 2	14	3	172
Batch 2	11	3	170
Batch 2	11	3	169
Batch 2	12	4	170
Batch 2	12	4	171
Batch 2	8	4	173
Batch 2	8	4	174
Batch 2	9	4	175
Batch 2	9	4	173
Batch 2	10	5	174
Batch 2	10	5	172
Batch 2	6	5	170
Batch 2	6	5	169
Batch 2	7	5	167
Batch 2	7	5	166
Batch 2	4	6	167
Batch 2	4	6	168
Batch 2	5	6	170
Batch 2	5	6	171
Batch 1	11	6	172
Batch 2	2	6	170
Batch 2	3	7	171
Batch 2	3	7	170
Batch 1	4	7	168
Batch 1	4	7	167
Batch 1	5	7	168
Batch 1	5	7	169

Table D-10 Flocculant Adsorption Data

Batch	Sample	Coagulant Dose	Absorbance at 420
Batch 1	7	1	174
Batch 1	7	1	172
Batch 1	8	1	170
Batch 1	8	1	169
Batch 1	9	1	167
Batch 1	9	1	166
Batch 1	14	2	167
Batch 1	14	2	168
Batch 1	13	2	170
Batch 1	13	2	171
Batch 1	12	2	172
Batch 1	12	2	170
Batch 2	13	3	175
Batch 2	13	3	173
Batch 2	14	3	171
Batch 2	14	3	170
Batch 2	11	3	168
Batch 2	11	3	167
Batch 2	12	4	168
Batch 2	12	4	169
Batch 2	8	4	171
Batch 2	8	4	172
Batch 2	9	4	173
Batch 2	9	4	171
Batch 2	10	5	172
Batch 2	10	5	170
Batch 2	6	5	168
Batch 2	6	5	167
Batch 2	7	5	165
Batch 2	7	5	164
Batch 2	4	6	165
Batch 2	4	6	166
Batch 2	5	6	168
Batch 2	5	6	169
Batch 1	11	6	170
Batch 2	2	6	168
Batch 2	3	7	169
Batch 2	3	7	168
Batch 1	4	7	166
Batch 1	4	7	165
Batch 1	5	7	166
Batch 1	5	7	167

Table D-11 Coagulant and Flocculant Adsorption Data

Batch	Sample	Coagulant Dose	Absorbance at 420
Batch 1	7	1	177
Batch 1	7	1	175
Batch 1	8	1	173
Batch 1	8	1	172
Batch 1	9	1	170
Batch 1	9	1	169
Batch 1	14	2	170
Batch 1	14	2	171
Batch 1	13	2	173
Batch 1	13	2	174
Batch 1	12	2	175
Batch 1	12	2	173
Batch 2	13	3	178
Batch 2	13	3	176
Batch 2	14	3	174
Batch 2	14	3	173
Batch 2	11	3	171
Batch 2	11	3	170
Batch 2	12	4	171
Batch 2	12	4	172
Batch 2	8	4	174
Batch 2	8	4	175
Batch 2	9	4	176
Batch 2	9	4	174
Batch 2	10	5	175
Batch 2	10	5	173
Batch 2	6	5	171
Batch 2	6	5	170
Batch 2	7	5	168
Batch 2	7	5	167
Batch 2	4	6	168
Batch 2	4	6	169
Batch 2	5	6	171
Batch 2	5	6	172
Batch 1	11	6	173
Batch 2	2	6	171
Batch 2	3	7	172
Batch 2	3	7	171
Batch 1	4	7	169
Batch 1	4	7	168
Batch 1	5	7	169
Batch 1	5	7	170

D.7 Temperature Change Desorption Data

Table D-12 Temperature Change Desorption Data

Batch	Sample	Absorbance at 420
Batch 1	Ctl	174
Batch 1	Ctl	172
Batch 1	Ctl	170
Batch 1	Ctl	169
Batch 1	Ctl	167
Batch 1	Ctl	166
Batch 1	WtoC	167
Batch 1	WtoC	168
Batch 1	WtoC	170
Batch 1	WtoC	171
Batch 1	WtoC	172
Batch 1	WtoC	170
Batch 2	CtoW	175
Batch 2	CtoW	173
Batch 2	CtoW	171
Batch 2	CtoW	170
Batch 2	CtoW	168
Batch 2	CtoW	167
Batch 2	CtoW	168

D.8 pH Change Desorption Data

Table D-13 Acetic Acid Desorption Data

Batch	Sample	Acid Dose	Absorbance at 420
Batch 1	7	0.1	178
Batch 1	7	0.1	176
Batch 1	8	0.1	174
Batch 1	8	0.1	173
Batch 1	9	0.1	171
Batch 1	9	0.1	170
Batch 1	14	0.5	171
Batch 1	14	0.5	172
Batch 1	13	0.5	174
Batch 1	13	0.5	175
Batch 1	12	0.5	176
Batch 1	12	0.5	174
Batch 2	13	1	179
Batch 2	13	1	177
Batch 2	14	1	175
Batch 2	14	1	174
Batch 2	11	1	172
Batch 2	11	1	171
Batch 2	12	5	172
Batch 2	12	5	173
Batch 2	8	5	175
Batch 2	8	5	176
Batch 2	9	5	177
Batch 2	9	5	175
Batch 2	10	10	176
Batch 2	10	10	174
Batch 2	6	10	172
Batch 2	6	10	171
Batch 2	7	10	169
Batch 2	7	10	168

Table D-14 Sodium Hydroxide Desorption Data

Batch	Sample	Base Dose	Absorbance at 420
Batch 1	7	0.1	176
Batch 1	7	0.1	174
Batch 1	8	0.1	172
Batch 1	8	0.1	171
Batch 1	9	0.1	169
Batch 1	9	0.1	168
Batch 1	14	0.5	169
Batch 1	14	0.5	170
Batch 1	13	0.5	172
Batch 1	13	0.5	173
Batch 1	12	0.5	174
Batch 1	12	0.5	172
Batch 2	13	1	177
Batch 2	13	1	175
Batch 2	14	1	173
Batch 2	14	1	172
Batch 2	11	1	170
Batch 2	11	1	169
Batch 2	12	5	170
Batch 2	12	5	171
Batch 2	8	5	173
Batch 2	8	5	174
Batch 2	9	5	175
Batch 2	9	5	173
Batch 2	10	10	174
Batch 2	10	10	172
Batch 2	6	10	170
Batch 2	6	10	169
Batch 2	7	10	167
Batch 2	7	10	166