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Kinetic Analysis of the Ion Exchange of Sodium from Subbituminous Coal

by
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This thesis submitted by Jason Allen Chesley in partial fulfillment of the requirements for the Degree of Master of Science from the University of North Dakota is hereby approved by the Faculty Advisory Committee under whom the work has been done.

Dorab N. Barcá

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Tom Baseway

This thesis meets the standards for appearance and conforms to the style and format requirements of the Graduate School of the University of North Dakota, and is hereby approved.

Title Kinetic Analysis of the Ion Exchange of Sodium from Subbittuminous Coal

Department Chemical Engineering

Degree Master of Science

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Signature Jason Allen Chesley

Date 9/26/83

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ABSTRACT

The removal of Na from subbituminous coal by ion exchange was investigated on a batch basis with H-ion donated by dilute aqueous H_2SO_4. The exchange of K, Mg, Ca, and Al from the coal was also observed. The four parameters studied were initial H_2SO_4 concentration, coal particle size, residence time, and liquid to solid mass ratio.

Results from this investigation indicate that at equilibrium 95 % of the initial Na content of the subbituminous coal can be removed by ion exchange with dilute aqueous H_2SO_4. It was also determined that Na removal is directly proportional to the residence time, and inversly proportional to particle size. The concentration of H-ion, as controlled by the liquid to solid mass ratio, and initial H_2SO_4 concentration have no significant effect on the rate of Na removal, provided there is sufficient H-ion present to complete the exchange.

Experimental results indicated that particle diffusion was the rate determining step. The following Vermuelen's approximation was used to predict the kinetic response for the removal of Na by ion exchange with good results.

$$\% \text{ of initial Na removed} = \left[1 - \exp\left(- \frac{\bar{D}t\pi^2}{r_o^2}\right)\right]^{1/2} *X*(100\%)$$

where D is the self-diffusion coefficient for H-Na ion exchange within the particle, mm^2/min; t is the time of the exchange, min; r_o is the radius of the exchange particle, mm; and X^o is the fraction of Na removal at equilibrium.

The H-ion:Na-ion self-diffusion coefficient was found to be nearly constant over the range of H_2SO_4 solution concentration used, at a value of 0.0018 mm^2/min (3.0×10^{-7} cm^2/sec).

Chapter I

INTRODUCTION

Even in the bleakness of the recent recession, coal is one of the few bright spots on the U.S. economic scene. Coal consumption reached an all time peak of 838 million tons in 1981 and was only 3 % below this in 1982, due to a weakened economy (1).[1] Predictions for 1983 suggest a repeat of the 1982 consumption level with a chance for increased production tied with the expected economic recovery (2). Helping to sustain the boost in coal demand is its prime customer, the electric utilities. Coal's share of electric power generation in the U.S. reached 53 % in 1982 and was off by only 1 % in 1983 (1,2). Continued growth in coal-fired steam electric power plants is certain with the commitment of 150 new units (79,670 MW) through 1997, and a possible additional 30 power plants (15,113 MW) proposed for construction by the year 2000 (3).

A problem encountered in the use of coal-fired power plants is ash fouling on the fire-side of the heat exchanger tubes. Ash fouling obstructs gas flow in boiler passes, re-

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[1] Numbers in parenthesis that are underlined refer to references cited at the end of this report.

duces heat transfer, lowers efficiency, and causes hot spots (4). Traditionally, there have been two basic methods used to overcome ash fouling. One method is to modify the boiler design. Unfortunately, such modifications represent a substantial capital expenditure (5). The other method, which may be economically preferable, is using either a fuel additive or some method of fuel treatment. The use of fuel additives has been studied with varied results, while fuel treatment by the use of ion exchange has been shown to be effective (4,6,7,8,9,10). The use of fuel additives or treatment has the added advantage of allowing high-fouling low rank coals to be burnt in existing facilities.

Two of the major parameters linked with ash fouling of western U.S. low rank coals are Na and ash contents. As shown by many pilot plant and utitlity boiler tests, any combination of high values of Na (above 5 % Na_2O in the ash) and ash (above 8 % in dry coal) can lead to high fouling potential (9).

The practice today is to burn only low Na coals or mixtures of low and high Na coals (9). With the supply of clean burning, low-fouling run-of-mine coals being depleted, some method of treatment to allow the burning of lower grade, higher fouling coals is needed and carries many advantages over boiler modifications.

Coal, for practical purposes, is only a generic title for a solid which is very heterogeneous in nature. Na content in the coal can vary from mine to mine, as well as in different locations in the same seam in the same mine.

Most of the Na and K, and some of the Ca and Mg found in low rank coals, are present in the organic structure of the coal as salts of humic acid (11). Since low rank coals such as lignite and subbituminous coal are salts of a weak acid, these ions can be exchanged with H-ions donated from an acid in an aqueous medium, such as a H_2SO_4 solution.

The present work investigates the ion exchange of Na from a Montana subbituminous coal with H-ion, donated by dilute H_2SO_4, as determined in a stirred beaker on a batch basis. Four parameters were studied. These were initial H_2SO_4 concentration, coal particle size, residence time, and liquid to solid mass ratio.

Chapter II

PREVIOUS EXPERIMENTAL WORK

The removal of Na from lignite via ion exchange has been studied using batch (7,12,13,14) and continuous countercurrent (13,15,16,17) systems. Some additional work has been done on removal of Na from subbituminous coal (9,18).

Work done on the batch basis used water, CaO, $CaCl_2$, HCl, and H_2SO_4 solutions as exchange media. Variables studied included residence time, particle size, initial solution concentration, liquid to solid mass ratio, agitation, and moisture content of the coal. The major results from past batch work were: a) Na is a cation which was readily exchangeable from low rank coal; b) ionic solutions were more effective in removing Na by ion exchange than distilled water, indicating Na in the coal was not present as water soluble salts; c) particle size was a critical parameter in the exchange of Na; and d) agitation had no effect on ion exchange as long as it was greater than a minimum value.

Studies completed on the continuous countercurrent system used solutions of H_2SO_4, or CaO and other calcium salt solutions as exchange media. Parameters investigated include solid residence time, initial solution concentration, liquid to solid mass ratio, particle size, and mositure content of the coal. Two major results from the past continuous work were: a) among exchange media used, H_2SO_4 and $CaCl_2$ solutions were found quite effective due to the fact H and Ca are highly ionic in these solutions; and b) increasing the liquid to solid mass ratio above 2/1 does not yield a significant increase in Na removal. The results from the continuous countercurrent system corroborate the work done on the batch basis.

Studies have shown that Na in the ash contributes to both ash deposit strength and quantity for low rank coals (12). Ion exchange as a means of coal preparation has been found to be effective in the reduction of Na in the coal and thus effective in the reduction of ash fouling problems (17).

Work done with lignite, both batch and continuous basis, have used integration of Ficks law to predict the response of the removal of Na by ion exchange (13,15,16). One investigation used Vermuelen's approximation, the theoretical integrated equation used in this report, to predict the kinetic response of the removal of Na from lignite on a continuous basis with good correlation (16).

Chapter III

THEORY OF ION EXCHANGE

3.1 INTRODUCTION

The process of ion exchange is a redistribution of ions from one environment to another. With very few exceptions, this exchange is reversible. Additionally, ion exchange is a stoichiometric process, with any ion removed from the exchange medium replaced by an equivalent ion of the same sign from the ion exchanger, thereby maintaining electroneutrality.

Ion exchangers are defined as insoluble solid materials which carry exchangeable cations and/or anions. If the ion exchanger contains ion exchangeable cations, it is called a cation exchanger; and conversly if the ion exchanger contains exchangeable anions, it is called an anion exchanger.

The ion exchanger consists of a framework held together by chemical bonds or lattice energy. This framework possesses either a surplus positive or negative charge, compensated by mobile ions with an opposite charge, the so called A counter-ions. Able to move freely within the framework of the ion exchanger, the A counter-ions may be replaced by other counter-ions of equivalent charge from solution. Y co-ions exist in the solution in order to preserve electroneutrality of the solution.

Figure 1 shows a diagram of the ion exchange system and the process of ion exchange is illustrated by the following example. An ion exchanger can be compared to a sponge, with the A counter-ions moving freely within the pores. The sponge is immersed in an electrolyte solution, made up of mobile Y co-ions, which have the same sign as the fixed ionic groups in the framework of the ion exchanger, and B counter-ions. The A counter-ions in the sponge can leave through the pores, but since electroneutrality of the sponge must be maintained, a stoichiometrically equivalent number of B counter-ions from the solution must enter the

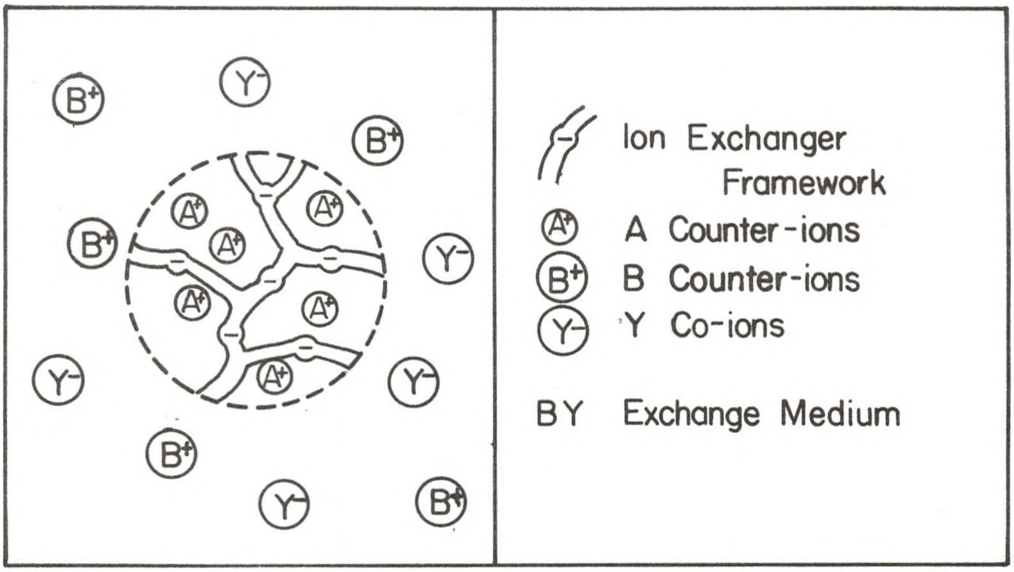

FIGURE 1 - Illustration of Ion Exchange System

pores to compensate. Thus, a counter-ion can only leave the sponge upon simultaneous entry of an equivalent ion.

According to this simple model, the amount of counter-ion in the ion exchanger is a constant, governed only by the magnitude of the charge of the framework of the ion exchanger, and independent of the nature of the counter-ion. In reality, this situation is complicated by the possibility of electrolyte sorption into the ion exchanger.

Normally, the pores of the ion exchanger may contain not only counter-ions, but also solvent and solutes, which entered the pores upon contact with a solution. Sorption is usually defined as the uptake of solute, and presence of the solvent may cause swelling of the ion exchanger.

The sorption of the electrolyte solution increases the number of B counter-ions and accompaning Y co-ions in the ion exchanger. Thus, sorption means that the counter-ion content of an ion exchanger depends not only upon the magnitude of the framework charge, but also on the Y co-ion content.

Ion exchange is a diffusion process, dependent upon the mobility of the counter-ions. This diffusion, generally does not follow the simple, well known rate laws; rather electric forces affect the fluxes of ions, causing deviations.

As a rule, the ion exchanger prefers one counter-ion to another. This implies that redistribution is not strictly statistical. Some of the most important causes for preferance are: a) the size and valence of the counter-ion influence the electrostatic interactions between the charged framework and the counter-ions, b) other interactions between ions and their environment beyond electrostatic forces are effective, and c) the narrow pores may prevent large counter-ions from entering the ion exchanger.

3.2 DONNAN POTENTIAL

Equilibria of ion exchangers using electrolyte solutions are complex, and the observed effect of different concentrations of exchange medium on removal of A counter-ions is explained by the Donnan potential.

Consider a low rank coal as an ion exchanger, placed in a dilute solution of a strong electrolyte, such as H_2SO_4. If no electrolyte is assumed to be sorped in the ion exchanger, a large concentration difference exists between the two phases; high A counter-ion concentration in the ion exchanger and high Y co-ion concentration in the electrolyte. If there was no charge associated with the ions, the concentration gradients would be levelled to equilibrium values by diffusion. Since the ions are charged, diffusion would disturb electroneutrality. Movement of both the A counter-ion into solution and Y co-ion into the ion exchanger causes the build up of charges, a positive charge in the solution and a negative charge in the ion exchanger. The first few ions which diffuse build up an electric potential difference, called the Donnan potential, between the ion exchanger and electrolyte solution. This potential pulls the positive A counter-ions back into the ion exchanger and negative Y co-ions back into solution. An equilibrium is thus established where the gradient set up by the concentration differences is balanced by the induced electric field. This effect is analogous in the anion exchanger with opposite signs for the various species.

The Donnan potential repels Y co-ions from entering the ion exchanger and thus maintains the internal Y co-ion concentration at an equlilbrium level. Since Y co-ion uptake and electrolyte sorption are equivalent because of electroneutrality, the electrolyte is at least partially excluded from entering the ion exchanger.

The presence of large amounts of Y co-ions in the ion exchanger will have a strong influence on the mobility of the counter-ions. Usually the amount of Y co-ion in the ion exchanger is kept low by the Donnan potential, but if the concentration of the exchange medium is strong, some Y co-ions may be forced into the ion exchanger, thus influencing counter-ion mobility. Consequently, changing exchange medium concentration could affect the rate of ion exchange by causing Y co-ions to be forced into the ion exchanger.

3.3 KINETICS

The ion exchange process can be separated into five steps. These steps are: a) diffusion of the counter-ions from the bulk of the solution across the film layer clinging to the ion exchanger particle surface, b) diffusion of the counter-ions from the surface of the ion exchanger through the pores to the exchange site, c) the chemical exchange of the counter-ions, d) diffusion of the exchanged counter-ions outwards from the exchange site to the surface through the pores of the ion exchanger, and e) diffusion of the exchanged counter-ions across the film layer to the bulk solution (19). Chemical exchange and the movement of species across boundaries are considered to be quite rapid.

By having the agitation at a high rate, the film clinging to the ion exchanger particle is assumed to be very thin, and migration through it is quite rapid. Consequently, particle diffusion within the ion exchanger particle is considered to be the rate controlling step in the present experimental investigation.

While it is true that Donnan potential keeps substantial amounts of Y co-ion from entering the ion exchanger during ion exchange, it does not hinder exchange of the counter-ions. Thus, Y co-ions do not participate in ion exchange and they have little effect on the rate of exchange.

3.3.1 Particle Diffusion

Since ion exchange is a diffusion process, rate laws can be derived using diffusion equations. Though appearing simple at first glance, the derivation is not as straight forward as it would seem. The problem can only be tackled by using certain boundary conditions. One of the simplest is the case of isotopic exchange, where the system is assumed to be in equilibrium except for an isotopic distribution (for example $^1H:^2H$). The following derivation is taken primarily from work done by Helfferich for a system where particle diffusion is rate controlling (19).

Ficks first law describes the flux of the isotope A in the ion exchanger.

$$J_A = -\overline{D} \text{ grad } \overline{C}_A \quad (1)$$

where J_A is the flux (in moles per unit time and cross section), C_A is the concentration of A, and D is the self-diffusion coefficient. (Quantities with bars refer to the interior of the ion exchanger)

Time dependence of the concentration is related to the flux by the following material balance, Ficks second law.

$$\frac{\partial \bar{C}_A}{\partial t} = -\mathrm{div}\ J_A \tag{2}$$

where t is time of ion exchange

Combining equations (1) and (2), including the restrictions of spherical geometry for the ion exchanger and constant diffusion coefficient, yields in spherical coordinates

$$\frac{\partial \bar{C}_A}{\partial t} = \bar{D}\left(\frac{\partial^2 \bar{C}_A}{\partial r^2} + \frac{2}{r}\frac{\partial \bar{C}_A}{\partial r}\right) \tag{3}$$

where r is the radial diffusion path in the ion exchanger sphere

Equation (3) may be solved using appropriate initial and boundary conditions. One simple initial condition is that the concentration of A counter-ions in the ion exchanger is uniform, with no A present in the exchange medium. This initial condition can be written as:

For $r > r_o$ at $t = 0$, $C_A(r) = 0$

For $0 \leqslant r \leqslant r_o$ at $t = 0$, $\bar{C}_A(r) = \bar{C}_{Ao}$ = constant (4)

where r_o is ion exchanger particle radius and C_{Ao} is the initial concentration of A counter-ions in the ion exchanger.

Two additional simplifying conditions may also be considered. The first, and the one used in this analysis, applies when the concentration of A counter-ion in solution remains neglible throughout the ion exchange process. This condition is met in batch experiments if the solution volume is so large that

$$\overline{C}\overline{V} << CV \tag{5}$$

> where $\underline{C}$ is the total concentration of counter-ions, $\overline{V}$ is the total volume of ion exchange material, and V is volume of solution.

This condition is called "infinite solution volume". If requirement (5) is not met, then a second boundary condition called "finite solution volume" must be applied. This second boundary condition is more general and will not be discussed here since infinitie solution volume holds true in the presesnt study.

Considering an infinite solution volume with particle diffusion rate controlling, the concentration of A counter-ions at the ion exchanger surface is the same as that in the bulk solution and this concentration will not change.

$$\text{For } r = r_o \text{ at } t > 0, \quad \Delta\overline{C}_A(t) = 0 \tag{6}$$

The solution of equation (3) under the conditions of Equations (4) and (6) yields a function $C_A(r,t)$. Integration of this function through the ion exchanger particles gives the following expression:

$$U(t) = \frac{\bar{Q}_{Ao} - \bar{Q}_A(t)}{\bar{Q}_{Ao} - \bar{Q}_{A\infty}} = 1 - \frac{6}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{n^2}\exp\left(-\frac{\bar{D}t\pi^2 n^2}{r_o^2}\right) \qquad (7)$$

where $Q_A(t)$ is the amount of A in the ion exchanger at time t, Q_{Ao} is the initial amount of A in the ion exchanger, $Q_{A\infty}$ is the amount of A at equilibrium in the ion exchanger, and U(t) is the fraction of attainment of equilibrium.

A summary of the conditions used for Equation 7 includes: a) a constant diffusion coefficient, b) no isotope (counter-ion) A initially in the exchange medium, c) spherical exchange particles, d) a well stirred electrolyte solution for a exchange medium, e) particle diffusion as rate controlling, and f) an isotopic distribution of counterions.

Less accurate than Equation (7), but more useful for practical work, is Vermeulen's approximation. It is the Vermeulen's approximation that will be used in this investigation.

$$U(t) = \left[1 - \exp\left(-\frac{\bar{D}t\pi^2}{r_o^2}\right)\right]^{1/2} \qquad (8)$$

Equation 8, Vermuelen's approximation, gives the fraction of equilibrium attained and not fraction ion removed. The fraction ion removed can be determined by multiplying the fraction of equilibrium attained by the equilibrium value for the fraction ion removed.

% Ion Removed = U(t)*X*(100%) (9)

where X is the equilibrium value for the fraction ion removed (In this report the symbol * shall be used as a multiplication sign.)

The half time, $t_{1/2}$, of ion exchange may be readily found by the use of equation (7). Substituting U(t) = 0.5 yields

$$t_{1/2} = 0.030 \frac{r_o^2}{\bar{D}} \tag{10}$$

It can be seen from the derived equations that the relative rate of ion exchange is proportional to the diffusion coefficient in the ion exchanger and inversly proportional to the square of the ion exchanger particle radius.

In Equations (7) and (8), there is no dependence on solution concentration for the exchanger rate. Solution concentration will have an effect on the self-diffusion coefficient, since the mobility of the B counter-ion from the exchange medium will be affected by a change in Y co-ion content of the ion exchanger. The Donnan potential will generally restrict the amount of Y co-ion entering the ion exchanger, but if the Y co-ion concentration becomes large enough, Y co-ions may be forced into the particle and affect the mobility of the counter-ions. While there is not a direct relationship between the exchange rate and solution concentration, the concentration will have an indirect effect by influencing the mobility of counter-ions and thus influencing the self-diffusion coefficient.

Equations (7) and (8) are derived under the assumption of isotopic exchange. Unfortunately, quantitative treatment of ion exchange is much more complicated. The fact that the fluxes of two different ionic species may couple together means that the system cannot be described by one constant diffusion coefficient. More complex derivations for the ion exchange rate equations can be made using the Nernst-Planck equations. Work done utilizing these more complex equations helped support the explanation concerning interactions of electrical fluxes and other complications which exist (13). In the present investigation, it was decided to use the less complicated Vermuelen's approximation for a theoretical integrated equation. Coal is far from being the ideal ion exchanger for which the rate equation derivations are made. For example, coal particles are not spherical nor are they homogeneous. Use of the more complex equations for such a non-ideal ion exchanger such as coal, does not seem appropriate.

Chapter IV

EXPERIMENTAL EQUIPMENT AND PROCEDURE

4.1 EXPERIMENTAL PROCEDURE

Utilizing a batch method of investigation, the ion exchange of subbituminous coal with a H_2SO_4 solution was studied. This was done in a 250 ml beaker, in which measured quantities of the coal particles and acid solution were combined. The resulting mixture was agitated by a 3-blade, propeller stirrer rotating at approximately 200 rpm. This procedure was developed in previous work (13).

The solid and liquid residue were separated by suction filtration after the designated time for the individual run, and stored in separate containers for further analyses. The as received coal was also analyzed. An average analysis for the Nerco, Montana subbituminous coal is given in Table 1, and a compilation of the various as received coal analysis is found in Table 5 in Appendix B.

TABLE 1

ANALYSIS OF TEST COAL

MODIFIED PROXIMATE ANALYSIS (AS RECEIVED BASIS)

CONSTITUENT	PERCENT
Moisture	23.4
Ash	2.8
Fixed Carbon and Volatile Matter	73.8
Total	100.0

SELECTED ASH CONSTITUENTS (AS RECEIVED BASIS)

CONSTITUENT	PERCENT IN ASH
Na_2O	9.6
K_2O	0.5
MgO	5.2
CaO	20.7
Al_2O_3	12.6

4.2 DESCRIPTION OF PARAMETERS

The four parameters investigated were initial H_2SO_4 concentration, coal particle size, residence time, and liquid to solid mass ratio. A summary of the various parameters used for the runs is found in Table 2.

The three initial H_2SO_4 concentrations used were 0.1070, 0.1565, and 0.1959 N. According to theory, a change in exchange medium concentration should have no effect on the rate of ion exchange if particle diffusion is rate determining. A directly proportional effect on the rate of ion exchange would indicate that film diffusion is prevalent. The different acid solutions were prepared by diluting concentrated H_2SO_4 with deionized distilled water and then titrating the solution with a standard NaOH solution to determine its normality.

Four different subbituminous coal particle sizes were used. Each size has a radius, r_o, relating to the radius of the next larger sized particle by the relationship $r^2 = 10/r^2$. This distribution of sizes was used since, according to theory, the ion exchange rate should be proportional to $1/r^2$ if particle diffusion is rate determining (<u>19</u>). The four particle sizes used correspond to the following Tyler mesh sizes: -4 to +6, -14 to +20, -35 to +48, and -80 to +100 mesh, and to a mean particle radii of: 2.013, 0.502,

TABLE 2

TEST CONDITIONS

Particle Radius, mm	H_2SO_4 Conc., N	Residence Time, minutes	Liquid/Solid Mass Ratio
2.013	0.1070	1440 (24 hour),180,150,120,80, 40,30,20,10,7,5	10/1
0.502	0.1070	1440 (24 hour),150,80,40,30,20, 10,5,2	10/1
0.178	0.1070	40,30,20,10,7,5	10/1
0.082	0.1070	40,30,20,10,5,2	10/1
2.013	0.1565	1440 (24 hour),150,120,80,40, 30,20,10,5,2	10/1
0.502	0.1565	240,120,80,60,40,30,20, 10,5,2	10/1
0.178	0.1565	40,30,20,10,5,2	10/1
2.013	0.1959	1440 (24 hour),180,40,30,20, 10,5,2	10/1
0.502	0.1959	240,80,40,30,20,10,5,2	10/1
0.178	0.1959	60,40,30,20,10,5,2	10/1
0.502	0.1070	1440 (24 hour),240,60,30,20, 10,5,2	5/1
0.502	0.1070	1440 (24 hour),60,30,20, 10,5,2	2/1
0.502	0.1959	1440 (24 hour),60,30,20, 10,5,2	5/1

0.178, and 0.082 mm, respectively. Separation into the different particle sizes was accomplished by the use of a Cenco Meizer Sieve Shaker.

Runs using differing liquid to solid mass ratios were accomplished by altering both the H_2SO_4 solution (liquid) amount as well as the subbitiuminous coal (solid) amount. The liquid to solid mass ratios of 10/1, 5/1, and 2/1 were made by the following ratios of amount of H_2SO_4 solution to amount of as received coal: 60 g/6 g, 50 g/10 g, and 40 g/20 g, respectively.

4.3 ANALYSIS OF SAMPLES

Subbitiumious coal and liquid residues were analyzed using ASTM procedures (20). After each run was completed, a sample of the processed subbituminous coal was weighed into a crucible and dried for 2 hours at 100°C (212°F), thus determining its moisture content. Ash content for the processed coal sample was determined by placing the dried coal sample in in a muffle furnace, gradually raising the temperature to 750°C (1382°F), and then holding at that temperature for 2 hours.

The metal ion concentration of the ash was determined for Na, the ion of interest, as well as for K, Mg, Ca, and Al. This was done by first making a pellet from the ash, by combining approximately 0.1 g of ash with 0.6 g of $LiBO_4$.

This mixture of ash and $LiBO_4$ was put into a graphite crucible and heated in a muffle furnace to 975°C (1787°F), forming the pellet upon cooling. The pellet was then dissolved in a mixture of 5 ml of concentrated HNO_3 diluted to 40 ml with distilled water. Having dissolved the pellet, the resulting solution was further diluted to 100 ml with distilled water. This final solution was analyzed for ion content by the use of an Atomic Absorption Spectrophotometer (AA), comparing the dissolved pellet solution to standard concentration solutions. A Perkin-Elmer model 303 Atomic Absorption Spectrophotometer equipped with a model DCR1 Concentration Readout was used.

The liquid residue was also analyzed for metal content by the use of the AA. However, in the analysis of the liquid residue, no preparation was required.

The results obtained from the AA are given in parts per million (ppm). The data for the liquid and solid residue was converted to percent ion removed for the subbituminous coal based on original as received coal analysis. After determining the closure for the run, the values were normalized for the percent ion removed. Sample calculations are shown in Appendix A, and the raw data for the runs is found in Appendix B. Graphs were drawn for the normalized percent ion removed data as a function of time.

Chapter V

RESULTS AND DISCUSSION

5.1 RESULTS

Using the analysis obtained from the liquid residue, solid residue, and coal as received, the removal of Na, the ion of interest, as well as K, Mg, Ca, and Al by ion exchange was determined by the use of a computer program, written for a TI-59 calculator in an earlier work (21). The complete program as well as an example of a print-out of results are shown in Appendix A. The results determined for all ions studied are tabulated in Tables 32-44, Appendix C, for the 13 sets of runs as a function of time for varying particle size, initial H_2SO_4 concentration, and liquid to solid mass ratio. The normalized percent ion removed for all ions investigated is plotted as a function of time in Figures 2-14, showing the order of removal.

Figures 2-5 and the data in the corresponding Tables 6-9, 19-22 (Appendix B), and 32-35 (Appendix C) were obtained for a series of runs as a function of time at a constant initial H_2SO_4 concentration of 0.1070 N and a liquid to solid mass ratio of 10/1 for 4 particle sizes. Likewise,

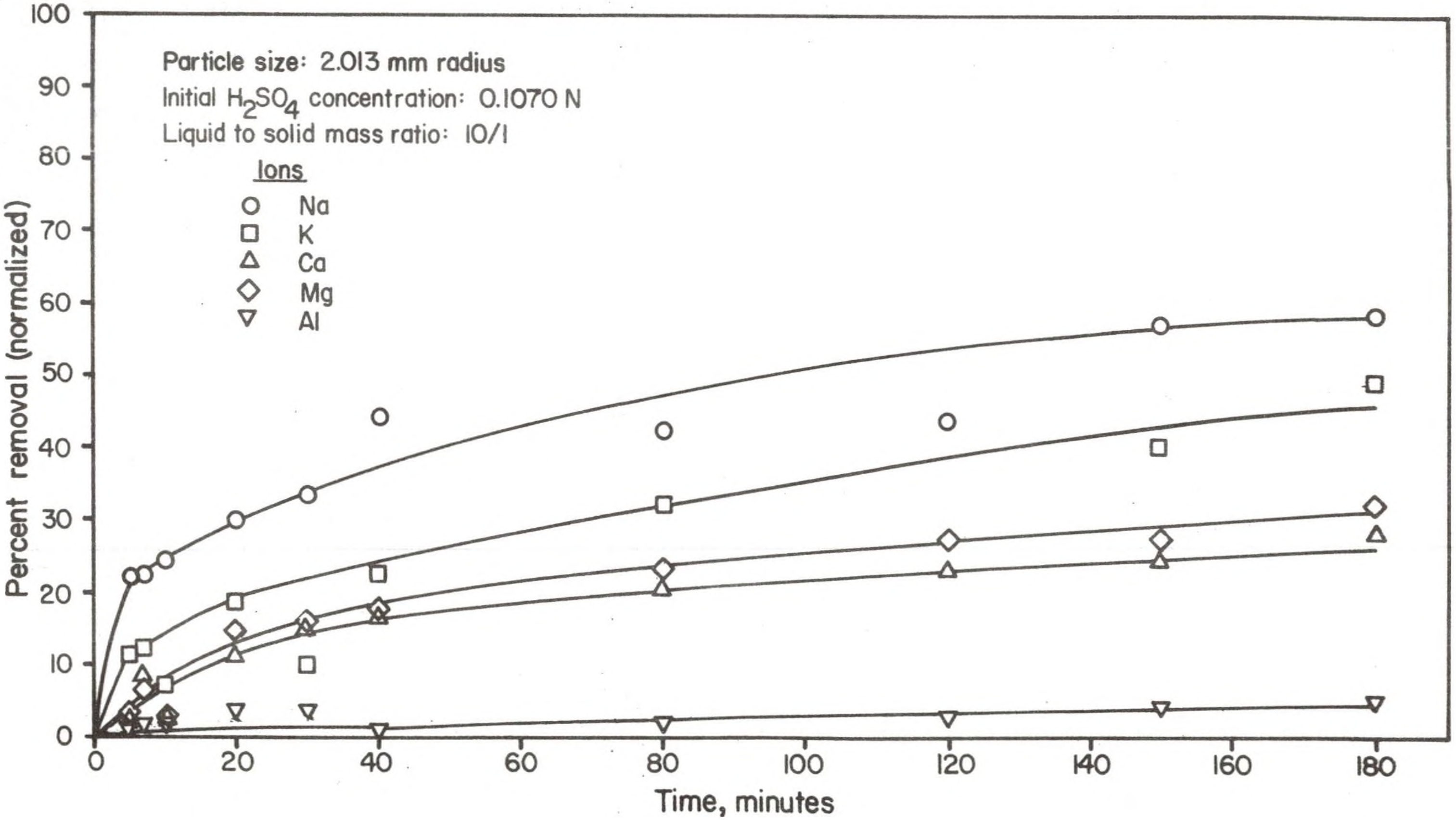

FIGURE 2 - Cation Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 2.013 mm Particle Radius (4-6 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

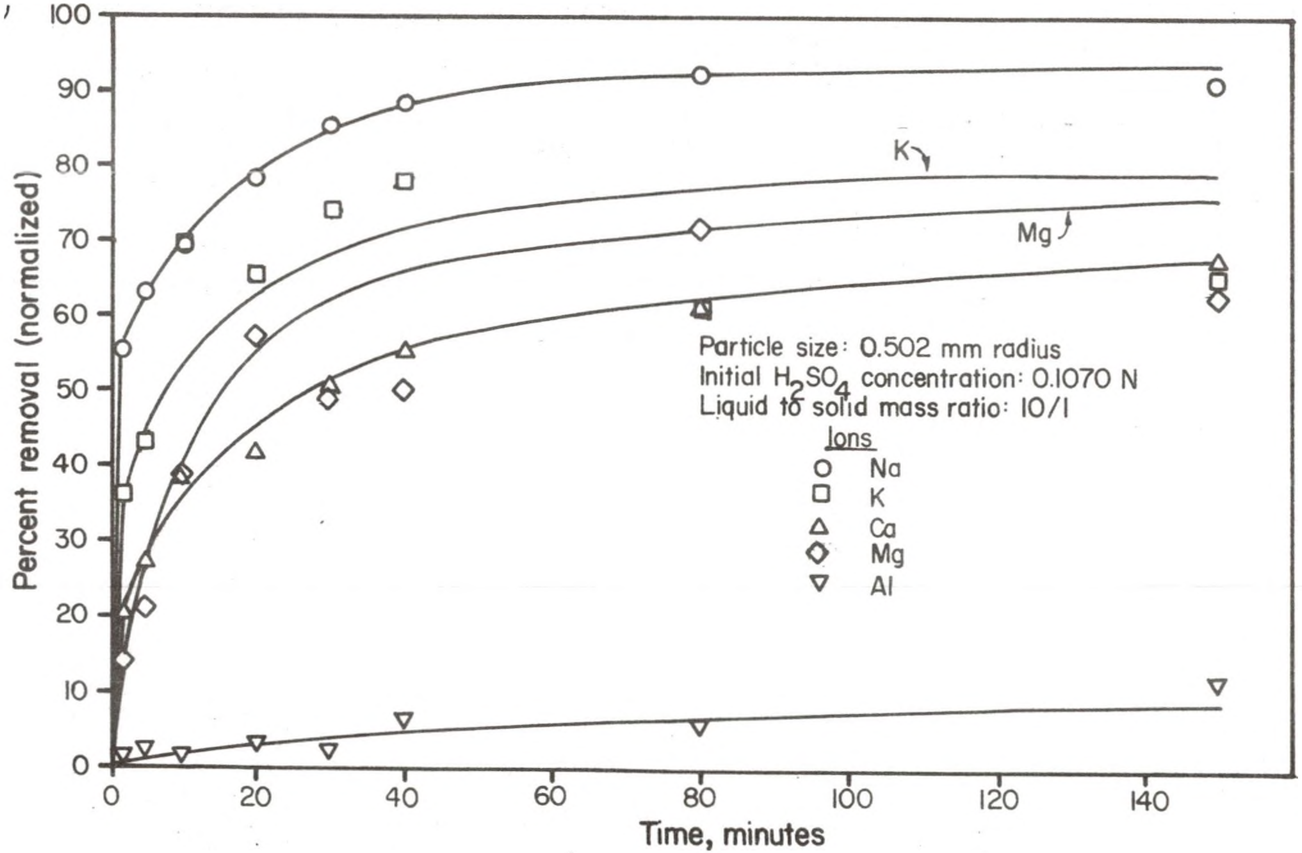

FIGURE 3 - Cation Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

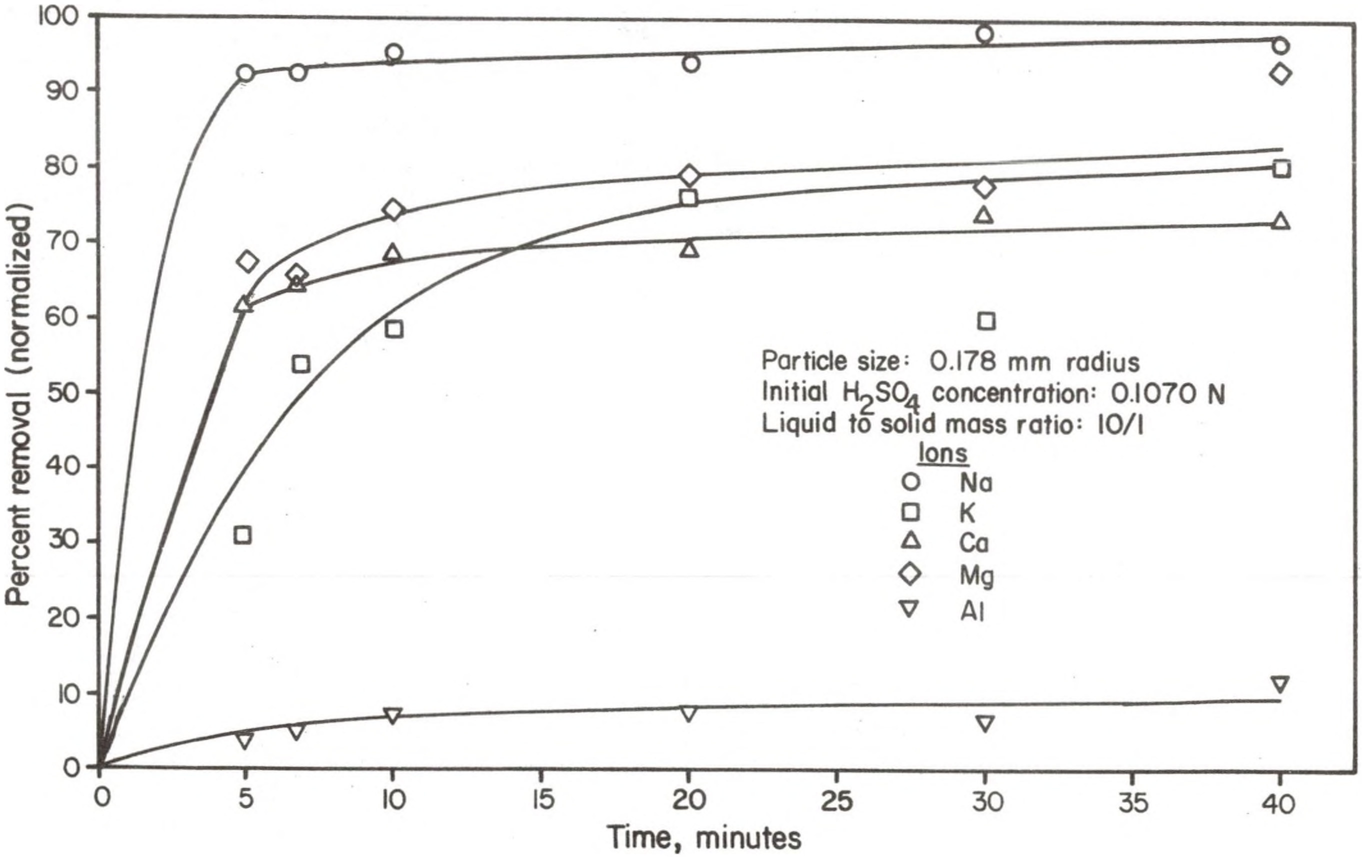

FIGURE 4 - Cation Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.178 mm Particle Radius (35-48 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

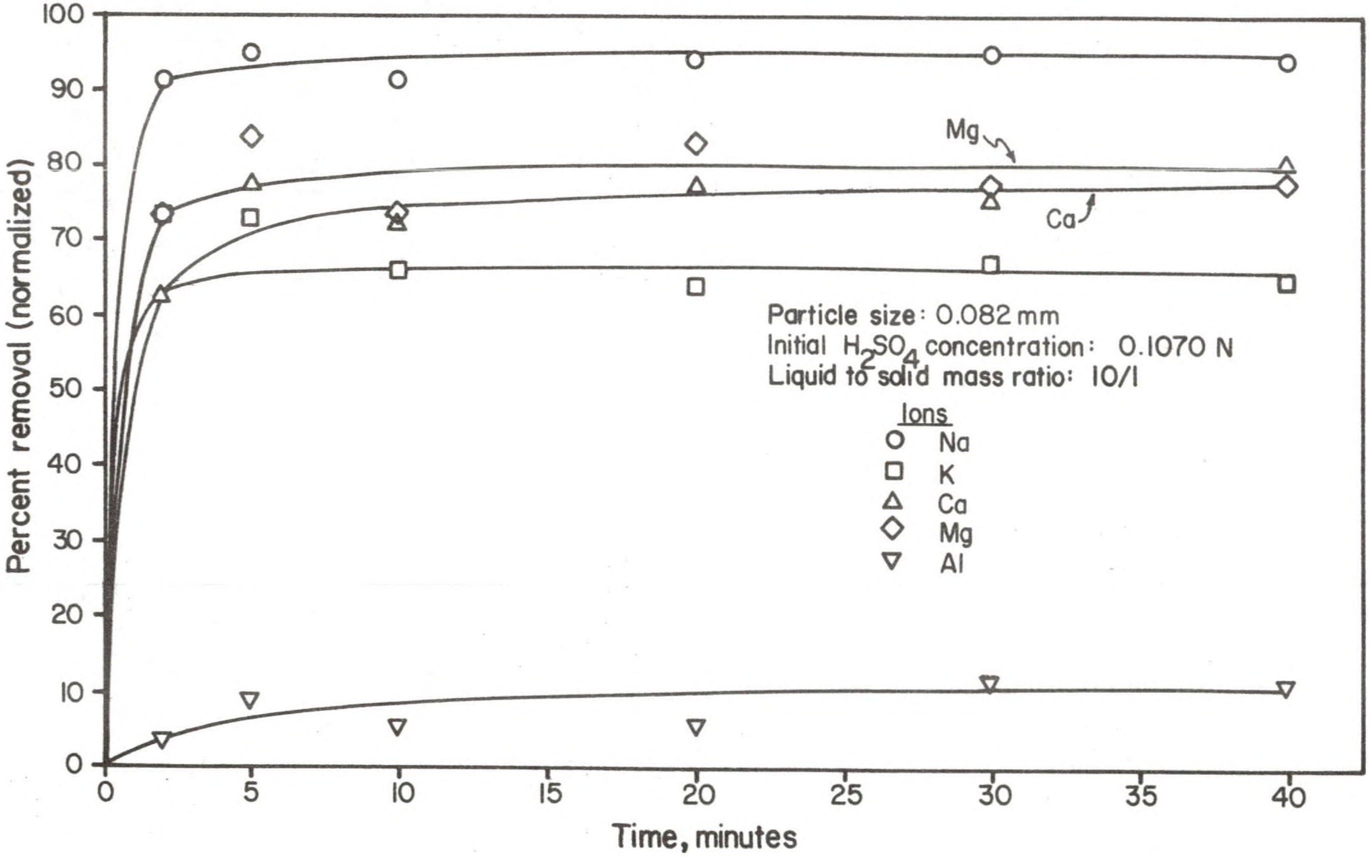

FIGURE 5 - Cation Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.082 mm Particle Radius (80-100 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

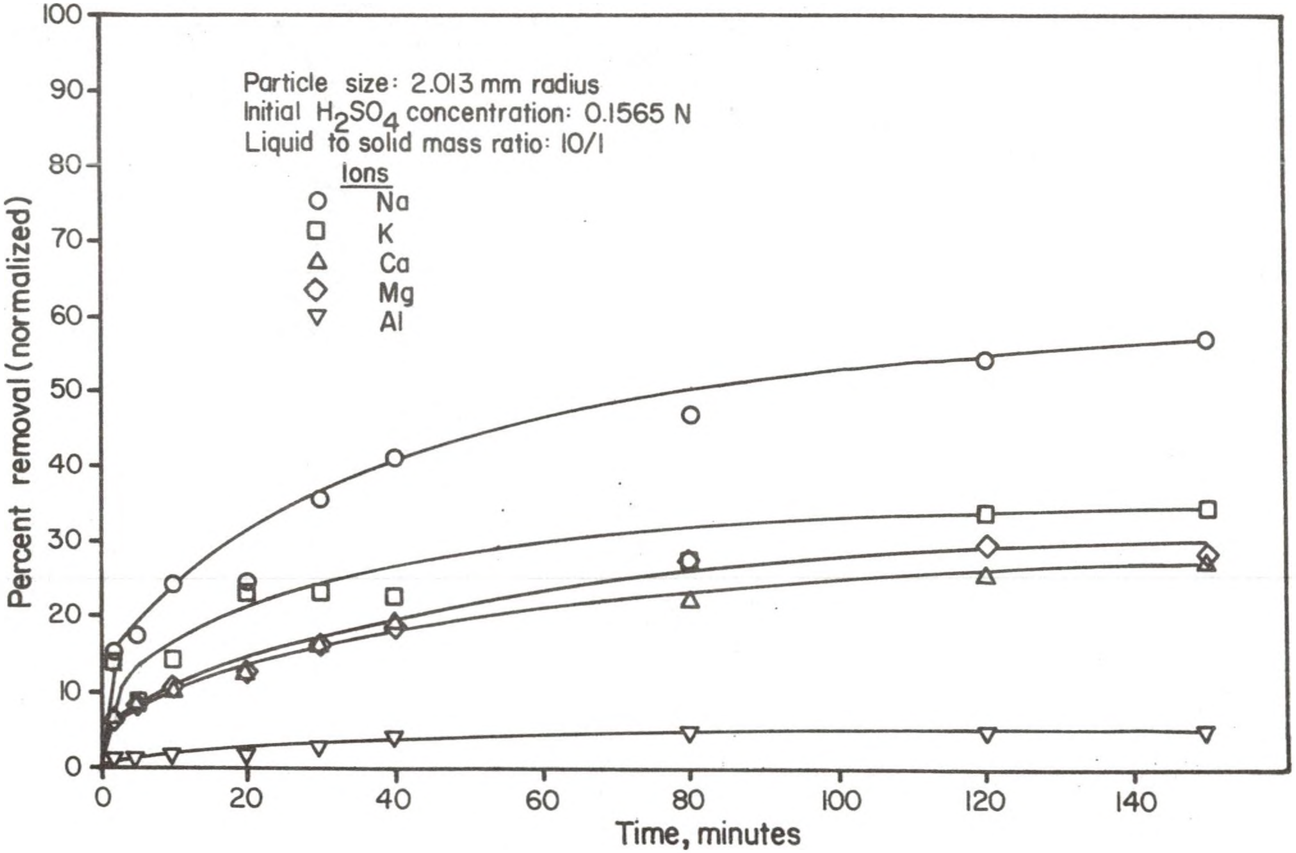

FIGURE 6 - Cation Removal as a Function of Time Using 0.1565 N H_2SO_4 Solution, 2.013 mm Particle Radius (4-6 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

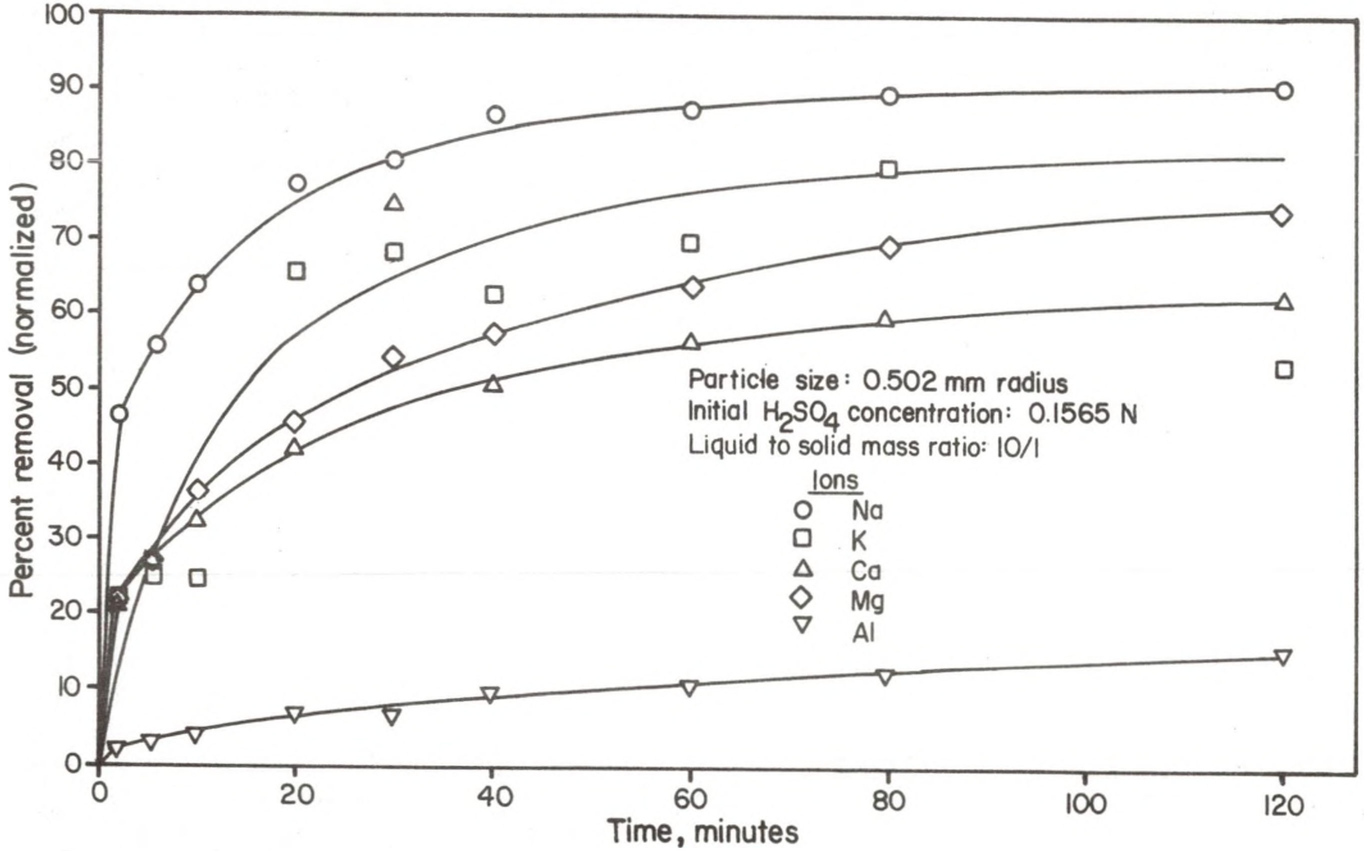

FIGURE 7 - Cation Removal as a Function of Time Using 0.1565 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

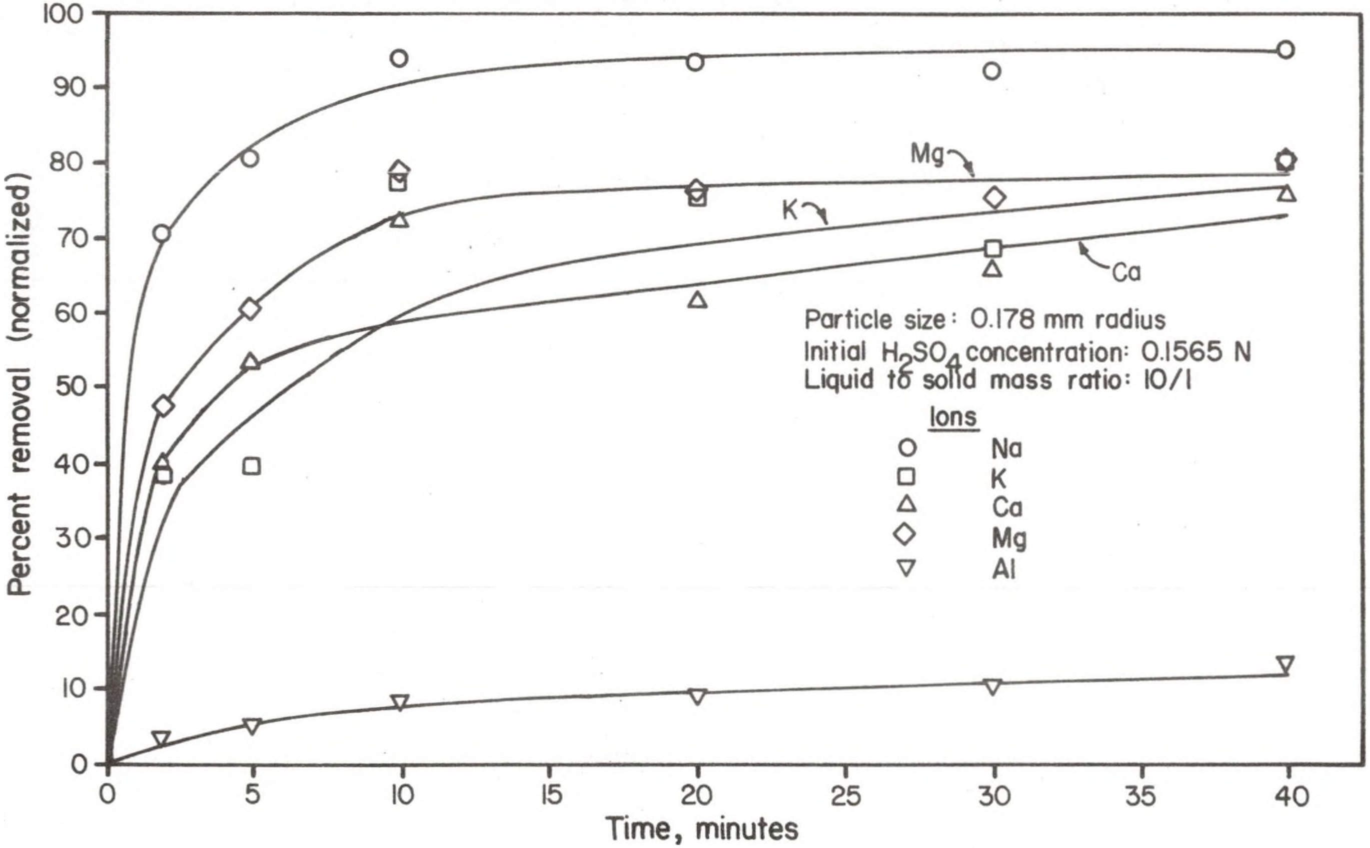

FIGURE 8 - Cation Removal as a Function of Time Using 0.1565 N H_2SO_4 Solution, 0.178 mm Particle Radius (35-48 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

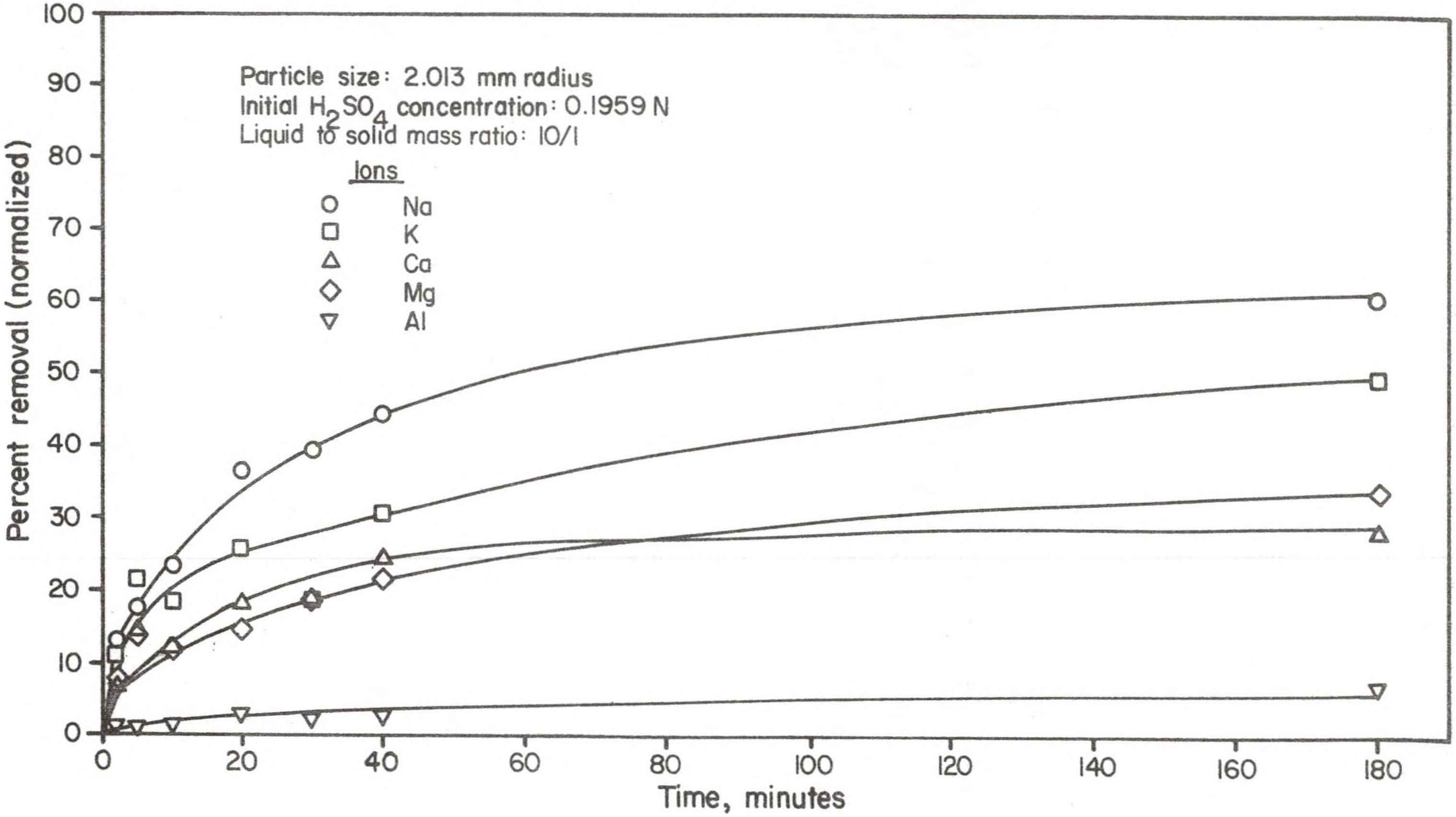

FIGURE 9 - Cation Removal as a Function of Time Using 0.1959 N H_2SO_4 Solution, 2.013 mm Particle Radius (4-6 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

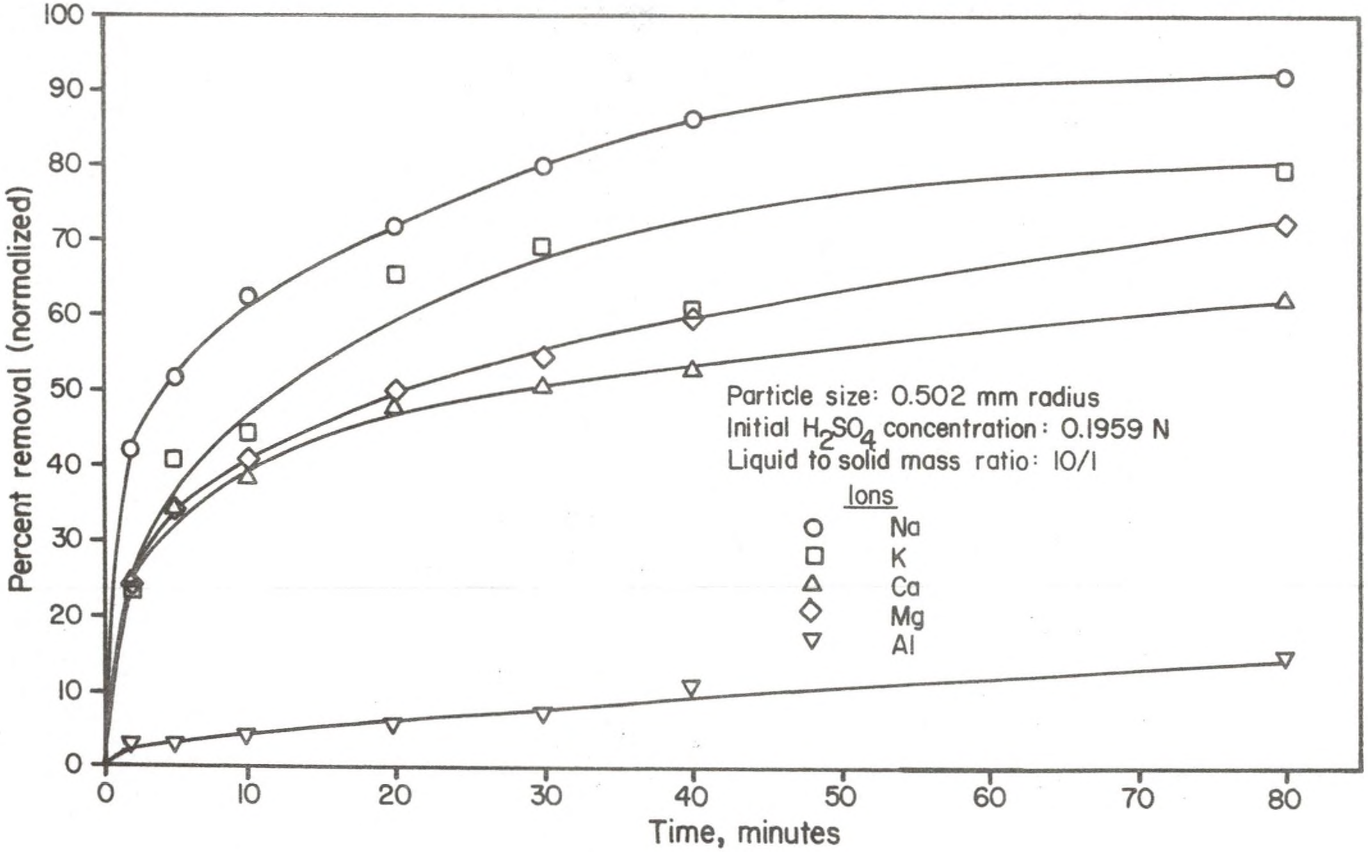

FIGURE 10 - Cation Removal as a Function of Time Using 0.1959 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

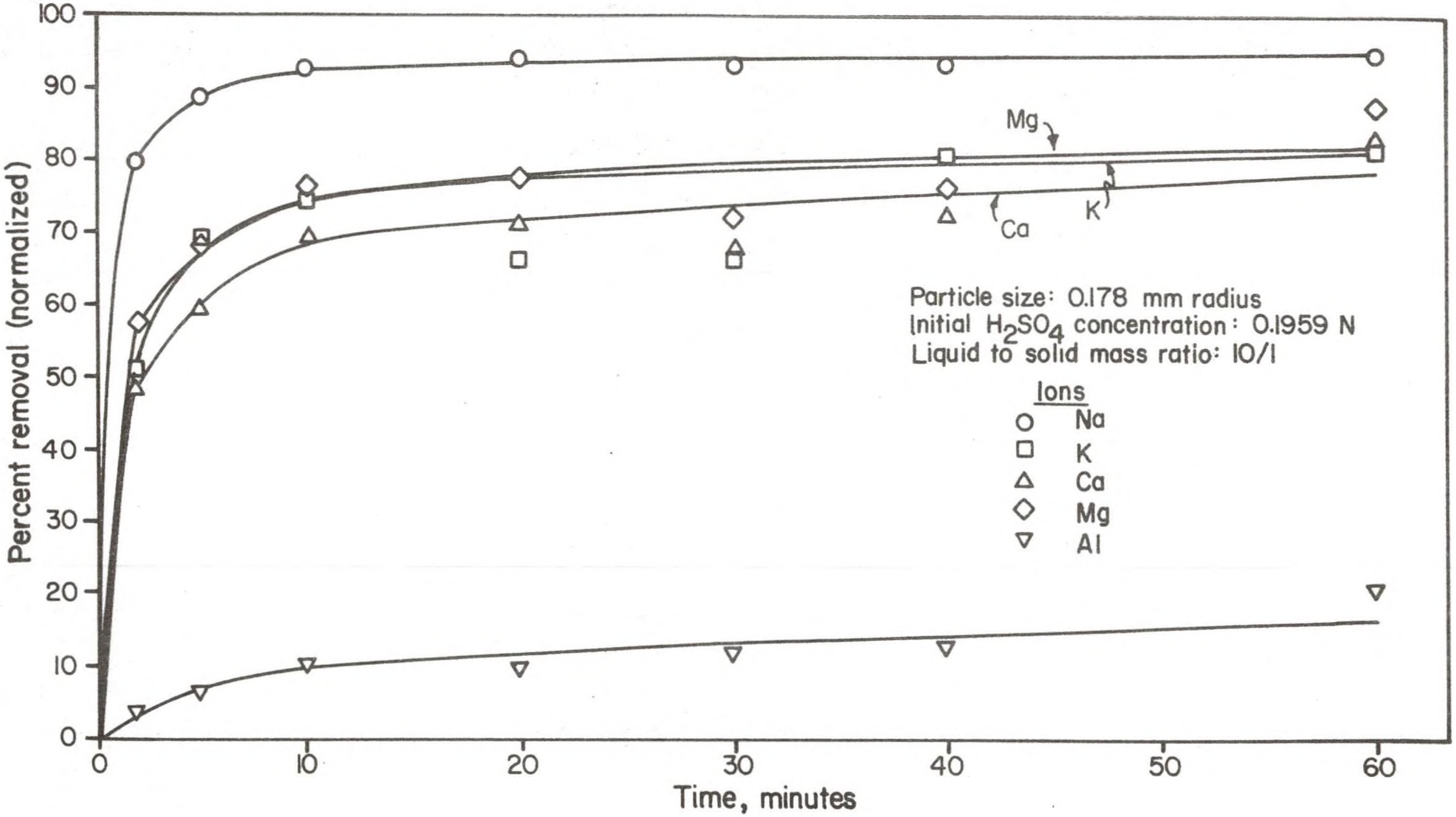

FIGURE 11 - Cation Removal as a Function of Time Using 0.1959 N H_2SO_4 Solution, 0.178 mm Particle Radius (35-48 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

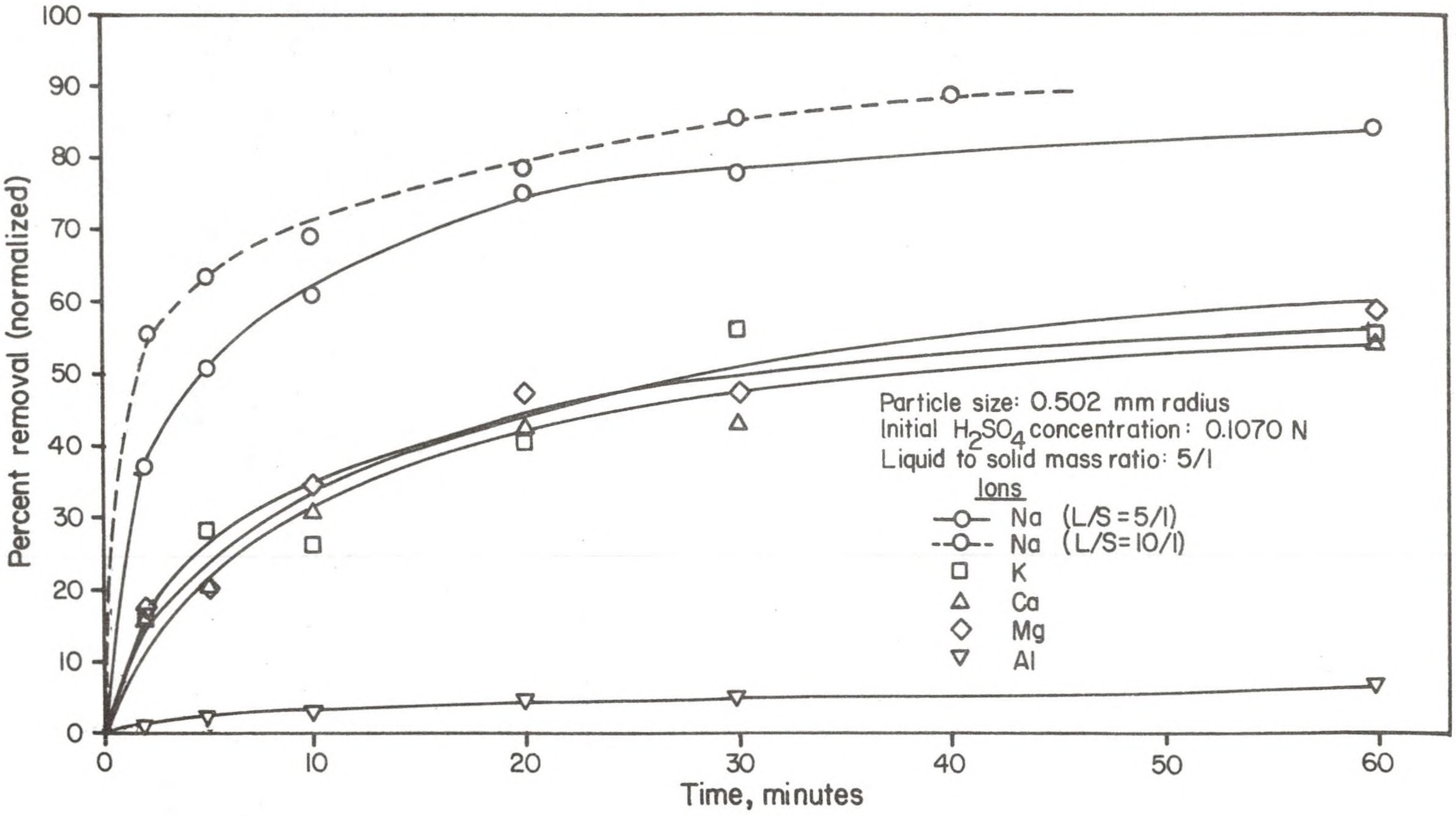

FIGURE 12 - Cation Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 5/1 Liquid to Solid Mass Ratio

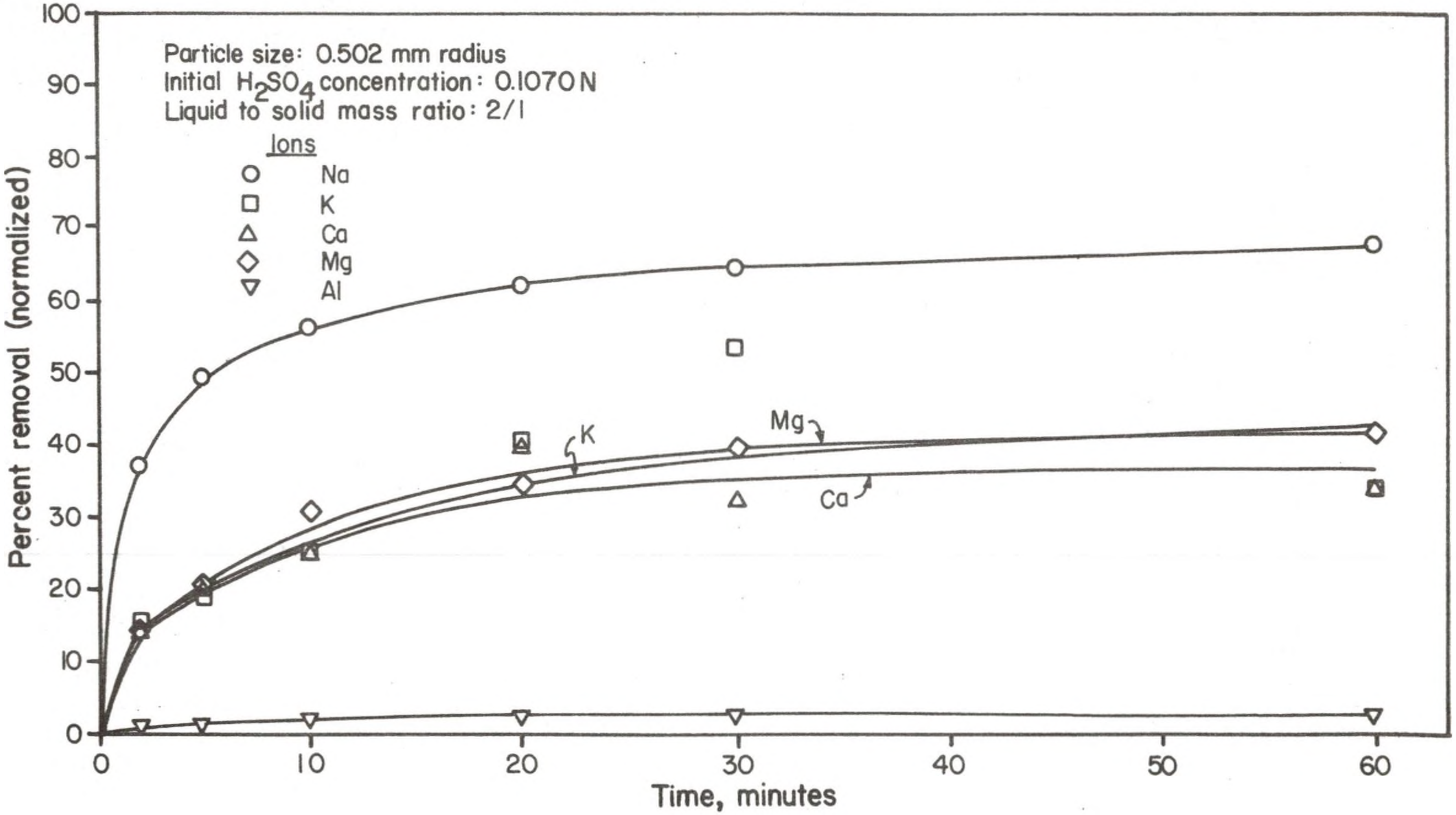

FIGURE 13 - Cation Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 2/1 Liquid to Solid Mass Ratio

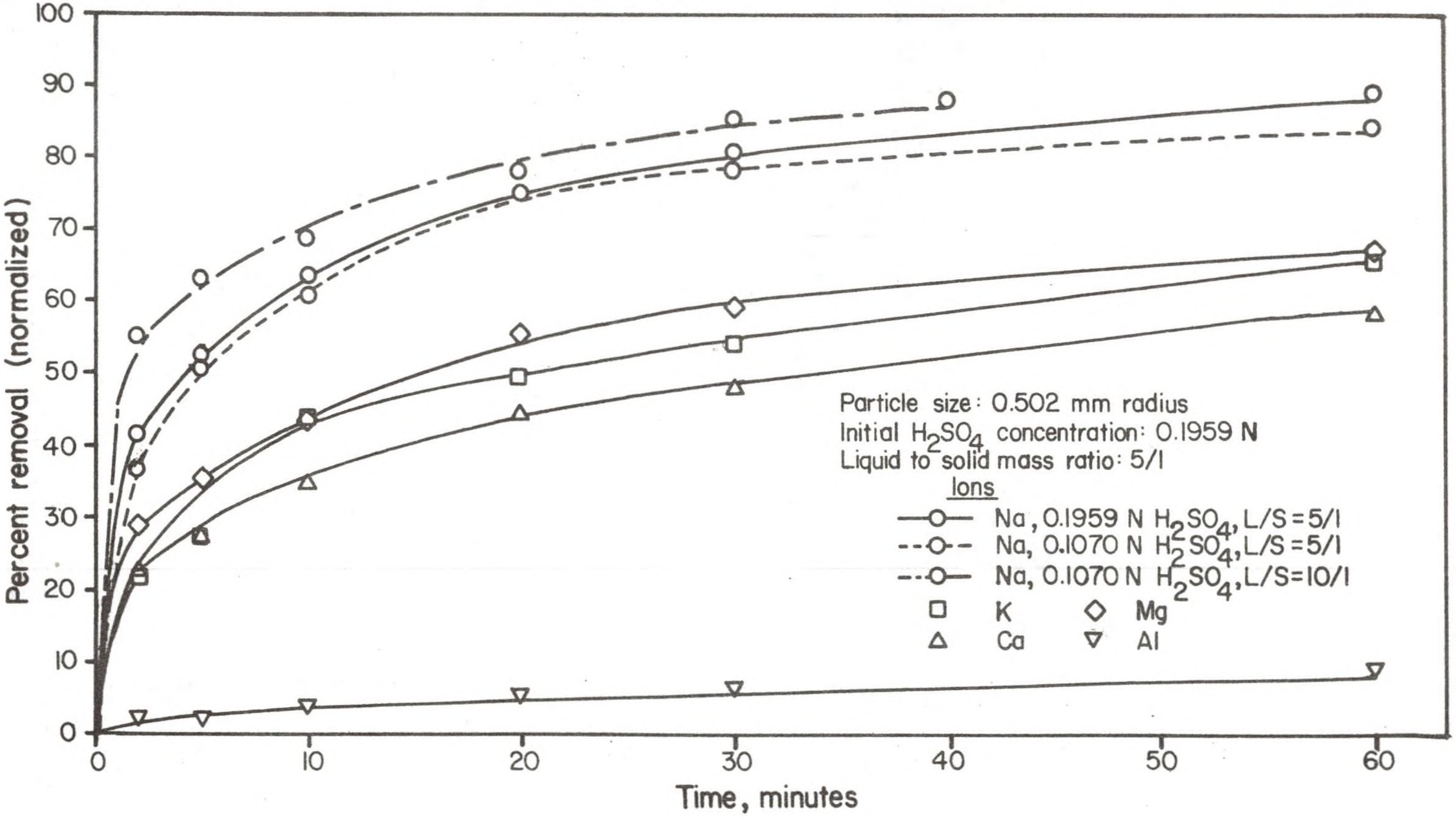

FIGURE 14 - Cation Removal as a Function of Time Using 0.1959 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 5/1 Liquid to Solid Mass Ratio

Figures 6-8 and Tables 10-12, 23-25 (Appendix B), and 36-38 (Appendix C) were prepared for a constant initial concentration of H_2SO_4 of 0.1565 N and a liquid to solid mass ratio of 10/1 for 3 particle sizes. In Figures 9-11, and Tables 13-15, 26-28 (Appendix B), and 39-41 (Appendix C), initial H_2SO_4 concentration was 0.1959 N and the liquid to solid mass ratio was 10/1 for 3 particle sizes. The liquid to solid mass ratio was altered in Figures 12-14 and Tables 16-18, 29-31 (Appendix B), and 42-44 (Appendix C), to 5/1, 2/1, and back to 5/1, respectively. A constant particle size of 0.502 mm was used and the initial H_2SO_4 concentration was 0.1070 N for Figures 12 and 13 and 0.1959 N for Figure 14.

The material balance closures for Na were quite good, ranging from about 95 % to 115 %. Of the remaining 4 ions, K was by far the most erratic. Most closures for Mg, Ca, and Al ranged from 85 % to 100 %, while K closures ranged from 50 % up to 120 %. The scatter in the closures for K analysis may be attributed to the trace amount of K present in the subbituminous coal. With the low ash coal used, remembering the heterogeneous character of coal, very minute fluctuations in K amount could throw the closure off by substantial amounts. Additionally, some blame must go also to inaccuracies of the atomic absorption (AA) analysis.

5.2 INFLUENCE OF PARTICLE SIZE, INITIAL SULFURIC ACID CONCENTRATION, AND RESIDENCE TIME

5.2.1 Particle Size

The effect of particle size on the removal of Na is clearly illustrated by Figure 15; a plot of Na removed as a function of time for the 4 particle sizes investigated, at a commmon initial concentration of H_2SO_4 of 0.1070 N and liquid to solid mass ratio of 10/1. To give the graph a common basis for convergence, the value for the amount of Na removed at equilibrium was assumed to be 95 % for all 4 particle sizes used. The value 95 % is an average value over all the runs for the amount of Na removed at equilibrium. Though the equilibrium value for Na removal should be a constant value for all particle sizes of the same coal, experimental error caused a scatter in this analysis. The equilibrium values for Na removal for all the runs, which can be found in Tables 32-44, ranged from 92 % to 97 %.

It is evident from Figure 15 that there is an inversly proportional relationship between particle size and Na removal, i.e., decreasing particle size increases the rate of Na removal. The observed relationship between particle size and Na removal was found to be significant by statistical analysis described in this section.

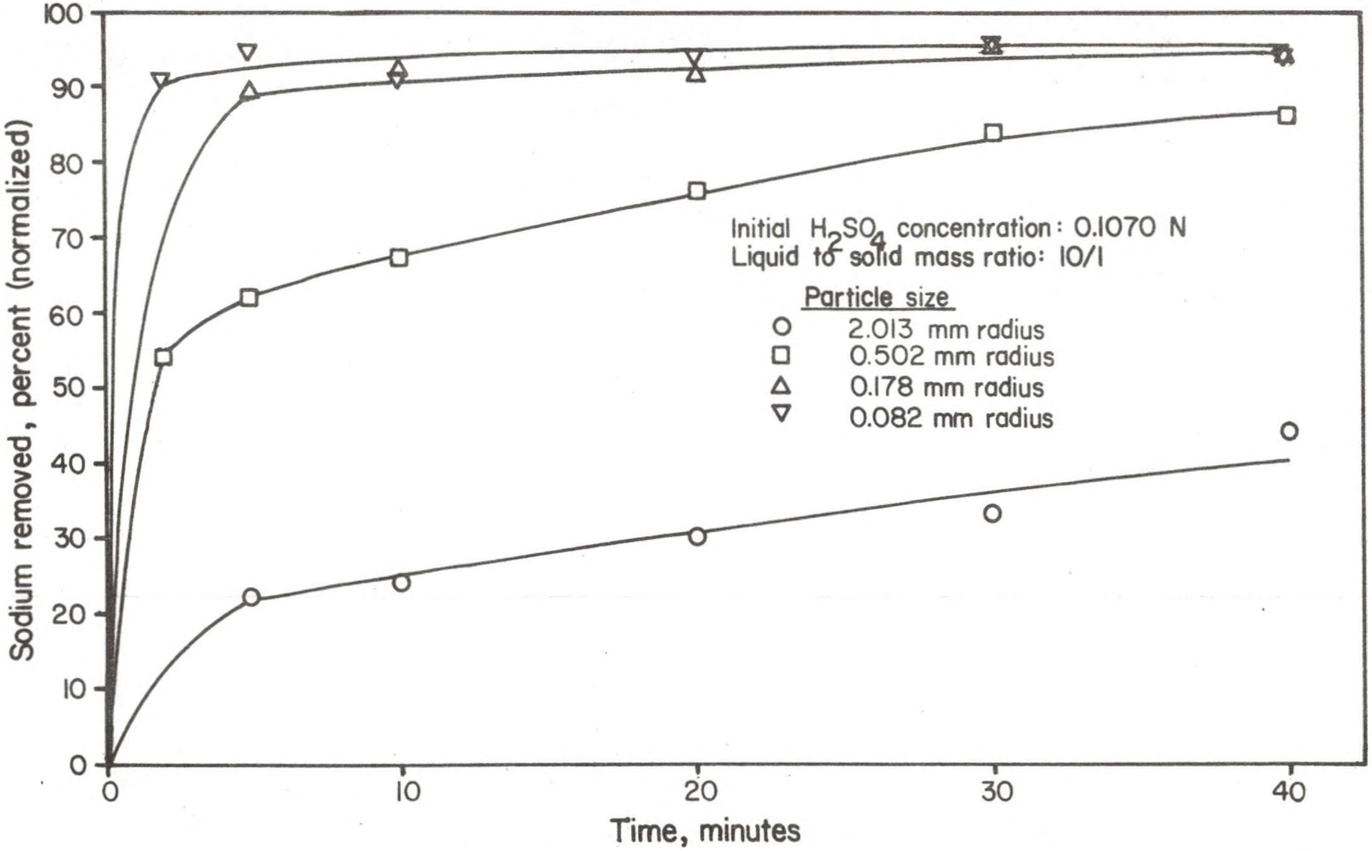

FIGURE 15 - Sodium Removal as a Function of Time Showing the Effect Particle Size Has on Sodium Removal Using 0.1070 N H_2SO_4 Solution and 10/1 Liquid to Solid Mass Ratio

5.2.2 Initial Sulfuric Acid Concentrtion

Figure 16, a plot of Na removed as a function of time for the 3 initial H_2SO_4 concentrations used, at a common particle size of 0.502 mm and liquid to solid mass ratio of 10/1, shows the effect initial H_2SO_4 concentration has upon Na removal. A value of 95 % Na removed at equilibrium was also used in this case. The graphs for the 3 varying initial H_2SO_4 solution concentrations show close agreement. As shown by statistical analysis later in the section, there is no significant difference between the curves for the 3 concentrations of 0.1070, 0.1565, and 0.1959 N H_2SO_4.

5.2.3 Residence Time

It is evident from Figures 2-14 that Na removal is a function of time. The removal of Na increases with time until an equilibrium value is reached.

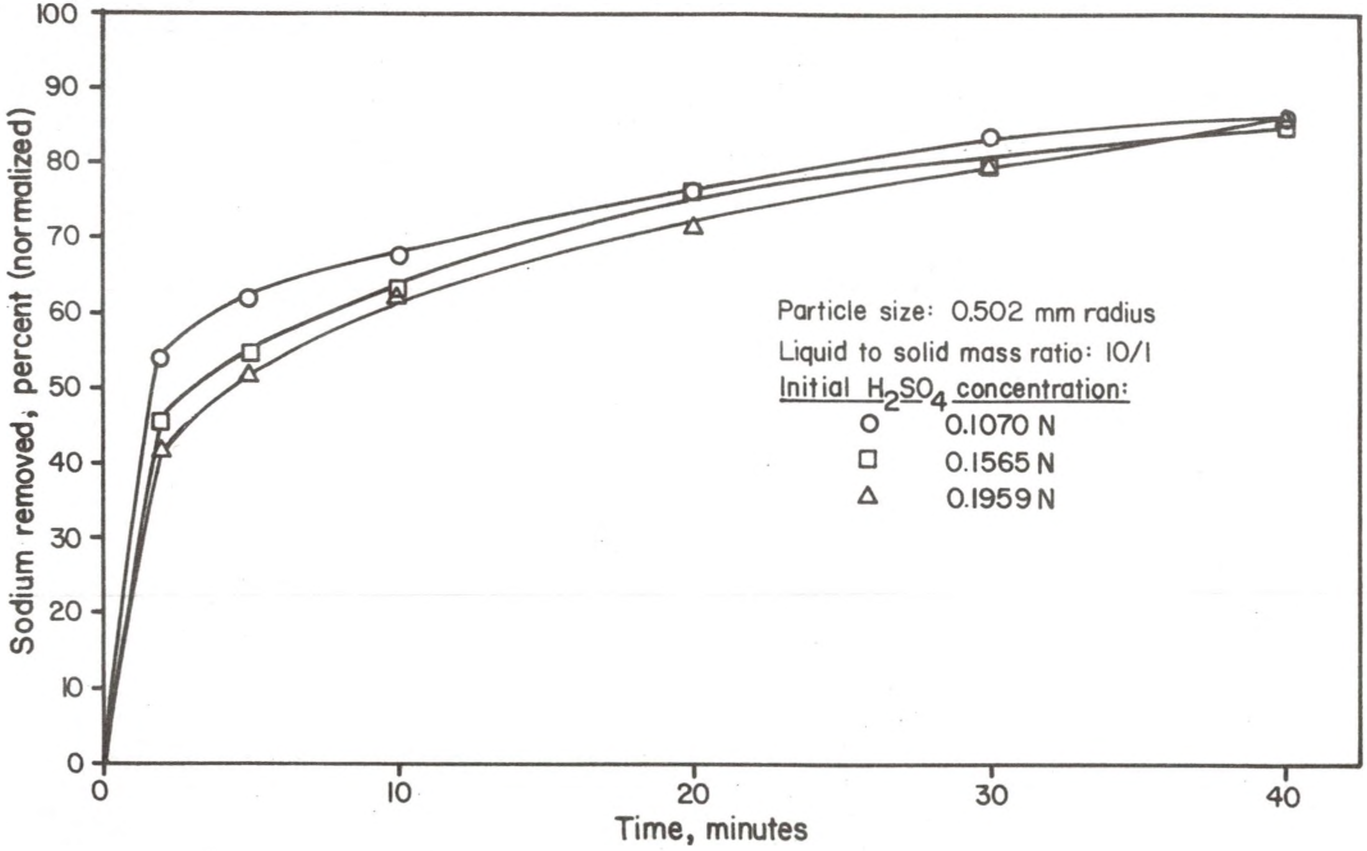

FIGURE 16 - Sodium Removal as a Function of Time Showing the Effect Initial H_2SO_4 Concentration Has on Sodium Removal Using 0.502 mm Particle Radius (14-20 Mesh Size) and 10/1 Liquid to Solid Mass Ratio

5.2.4 Statistical Analysis

A 3-way test of variance was completed to determine statistically the significance of the 3 parameters of particle size, initial concentration of H_2SO_4 solution, and residence time on Na removal. Statistical results from this analysis are found in an analysis of variance (ANOVA) table, Table 3, and the sample calculations for the ANOVA table are located in Appendix D.

In Table 3, the sum of squares and the mean square are simply the partitioning of the variablity of experimental data into the various sources of error. In this investigation, all 3 parameters were assumed to be fixed level (Model 1) errors. This implies that the statistical analysis is only concerned with the specific levels used for the 3 parameters. In using fixed level (Model 1) errors, the variablity of the experimental values partitioned to each parameter was separately compared with the variability attributed to experimental "slop" by the use of an F test, to determine if there was a significant difference between the variablity associated with the parameter compared to the variablity associated with experimental error (22).

In the working equation and both null and alternate hypothesis (H_o and H_a respectively) used in Table 3, the symbols A, B, and C represent the error associated with the parameters of time, particle size, and initial H_2SO_4

TABLE 3

ANOVA TABLE

Working Equation: $X_{ijk} = \mu + A_i + B_j + C_k + \varepsilon$

Null Hypothesis $H_o: A_i = B_j = C_k = 0$
Alternate Hypothesis $H_a: A_i \neq 0$ or $B_j \neq 0$ or $C_k \neq 0$

Source	D.F.	Sum of Squares	Mean Square	Estimated Mean Square	F_{calc}	F^1
Mean	1	193,874.3	-	-	-	-
Time	4	2409.2	602.3	$\sigma^2 + (9/4) * \Sigma A_i^2$	24.02	3.19
Particle Size	2	30,670.2	15,335.1	$\sigma^2 + (15/2) * \Sigma B_j^2$	611.70	4.11
H_2SO_4 Concentration	2	102.0	51.0	$\sigma^2 + (15/2) * \Sigma C_k^2$	2.03	4.11
Error	36	902.5	25.1	σ^2	-	-
Total	45	227,958.2	-	-	-	-

D.F. = Degrees of Freedom

[1] Reference (22)

concentration, respectively. The null hypothesis, H_o, states that there is no significant effect related with any of the parameters studied, while conversly the alternate hypothesis, H_a, states that there is at least one significant effect among the parameters. A value for the Type 1 error, probability of rejecting the null hypothesis when it is true, was chosen to be the commonly used value of 5 %. A F value greater than 3.19 for the variable (source) of time and 4.11 for the variables (sources) of particle size and initial H_2SO_4 concentration indicated that there was a significant influence for that parameter (22).

From the F-test found in the ANOVA table, it was determined that residence time and particle size have a significant effect on Na removal at a 95 % confidence level. Also, initial H_2SO_4 concentration was found to have no significant effect over the concentration range investigated. From the values for the mean square determined for the ANOVA table, particle size was found to have a greater influence on Na removal than residence time as particle size showed a greater variability in experimental values.

5.2.5 Rate Controlling Step

The assumption that particle diffusion was the rate controlling step was supported by all the above findings. The determination that particle size is inversly proportional to the removal of Na supports the assumption of particle diffusion as rate controlling since with a shorter diffusion path (smaller particle size), the rate of diffusion and thus Na removal should increase. Likewise, increased residence time allows a longer period for ion exchange to occur, increasing Na removal. The fact that initial H_2SO_4 concentration had no significant effect on Na removal is strong evidence that particle diffusion, and not film diffusion, was the rate controlling mechanism for the ion exchange. If film diffusion had been rate controlling, initial concentration of H_2SO_4 solution should have had a directly proportional relationship on the removal of Na (19). Since initial concentration of H_2SO_4 solution showed no significant effect, the rate controlling step must have been particle diffusion. By setting the agitation rate at a high rate such that the film layer clinging to the coal particle was very thin, particle diffusion as the rate controlling step was expected.

5.3 INFLUENCE OF LIQUID TO SOLID MASS RATIO

5.3.1 Liquid to Solid Mass Ratio

The effect of liquid to solid mass ratio upon removal of Na is shown in Figure 17, which is a plot of Na removed as a function of time for the 4 mass ratios used, at a common particle radius of 0.502 mm and H_2SO_4 concentrations of 0.1070 N and 0.1959 N. Once again, to achieve common convergence a value of 95 % Na removed at equilibrium was assumed. The curves for the 3 liquid to solid mass ratios (10/1, 5/1, and 2/1) show no significant difference between mass ratios as long as there was sufficient H-ion content to complete the exchange. This observation was supported by statistical means later in this section. The graphs for the 10/1 and 5/1 mass ratios are quite similiar, but the 2/1 mass ratio illustrates the effect of not having sufficient H-ion content present. Though the graph for the 2/1 mass ratio is initially similiar to the graphs for the 10/1 and 5/1 mass ratios, it falls far short of the 95 % Na removed at equilibrium, as the H-ion (B counter-ion) was used up before the usual equilibrium value was reached. An additional set of runs was made at a constant ratio of equivalents of Na to equivalents of H-ion in the exchange medium of 0.18, which is the same as the 10/1 mass ratio and 0.1070 N H_2SO_4 concentration. This data set was done at a 5/1 mass ratio and 0.1959 N H_2SO_4 concentration. The graphs for these two

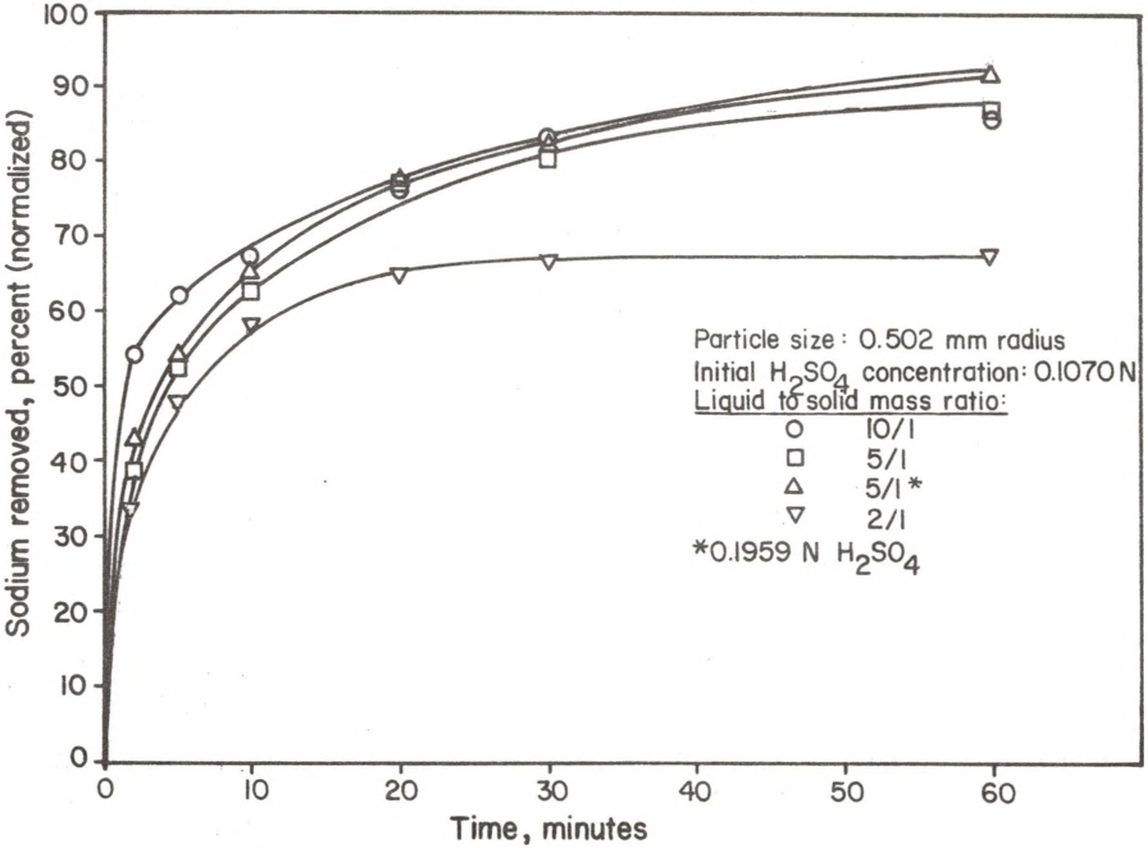

FIGURE 17 - Sodium Removal as a Function of Time Showing the Effect Liquid to Solid Mass Ratio Has on Sodium Removal Using 0.502 mm Particle Radius (14-20 Mesh Size) and 0.1070 N H_2SO_4 Solution

runs at constant equivalent ratio show no significant difference from the graphs of the other mass ratios in which there was sufficient H-ion present to complete the exchange, and this observation is verified by the following statistical analysis.

5.3.2 Statistical Analysis

A "student t-test" was done for the data obtained for the liquid to solid mass ratios (10/1, 5/1, and 5/1 with an equivalents ratio of 0.18) that had sufficient H-ions present to complete the exchange. The sample calculations are shown in Appendix D. It was determined that no significant difference existed, at a 95 % significance level, between the data obtained for the mass ratios of 10/1, 5/1, and 5/1 (with the similar ratio of equivalents of Na-ion to equivalents of initial H-ion as the 10/1 mass ratio and 0.1070 N H_2SO_4 concentration).

5.3.3 Rate Controlling Step

The results for varying liquid to solid mass ratio also confirm that particle diffusion was rate controlling. The mechanism for particle diffusion should be independent of H-ion content, and that is what was found in this investigation.

5.4 THE THEORETICAL INTEGRATED EQUATION

The Vermuelen's approximation, Equation 8, was used in conjuction with Equation 9 to predict the kinetic response of the ion exchange of Na from subbituminous coal. Figures 18-30 are plots of Na removal as a function of time for both the experimental work and predictions from Vermuelen's approximation. The 13 figures correspond to the 13 sets of timed runs in which the parameters of particle size, initial H_2SO_4 concentration, and liquid to solid mass ratio were varied. A computer program, listed in Appendix A, was used to determine the predicted values of Na removal from the Vermuelen's approximation. The most important parameter in Vermuelen's approximation is the H-ion:Na-ion self-diffusion coefficient in the exchange particle. Equation 10 was used, knowing the half time of the ion exchange, to determine the H-ion:Na-ion self-diffusion coefficient. Values for the H-ion:Na-ion self-diffusion coefficient are compiled in Table 4 for the various sets of runs. Equations 8, 9, and 10 again are:

$$U(t) = \left[1 - \exp\left(- \frac{\bar{D}t\pi^2}{r_o^2}\right)\right]^{1/2} \tag{8}$$

$$\%\ \text{Ion Removed} = U(t)*X*(100\%) \tag{9}$$

$$t_{1/2} = 0.030 \frac{r_o^2}{\bar{D}} \tag{10}$$

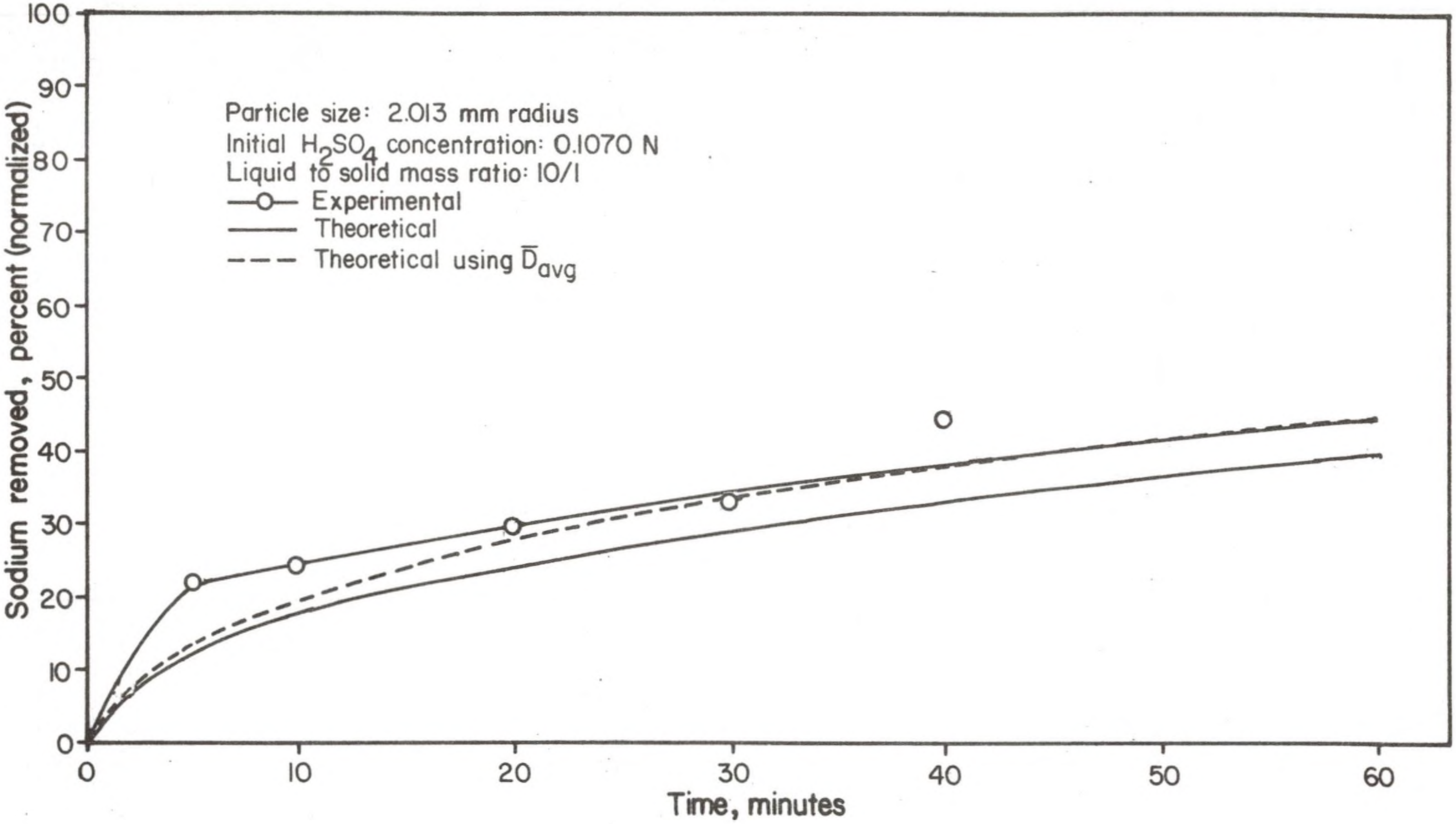

FIGURE 18 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 2.013 mm Particle Radius (4-6 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

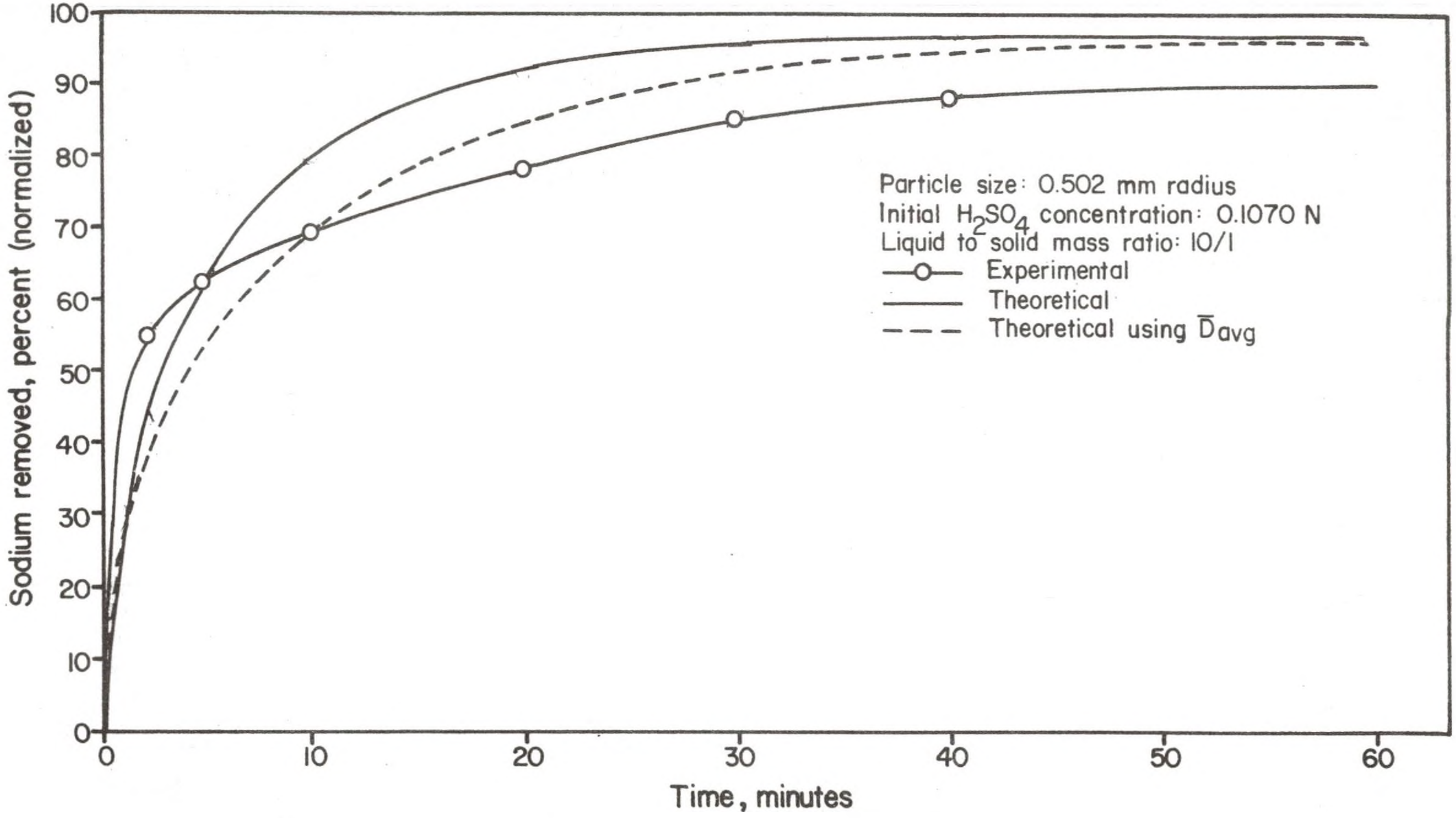

FIGURE 19 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

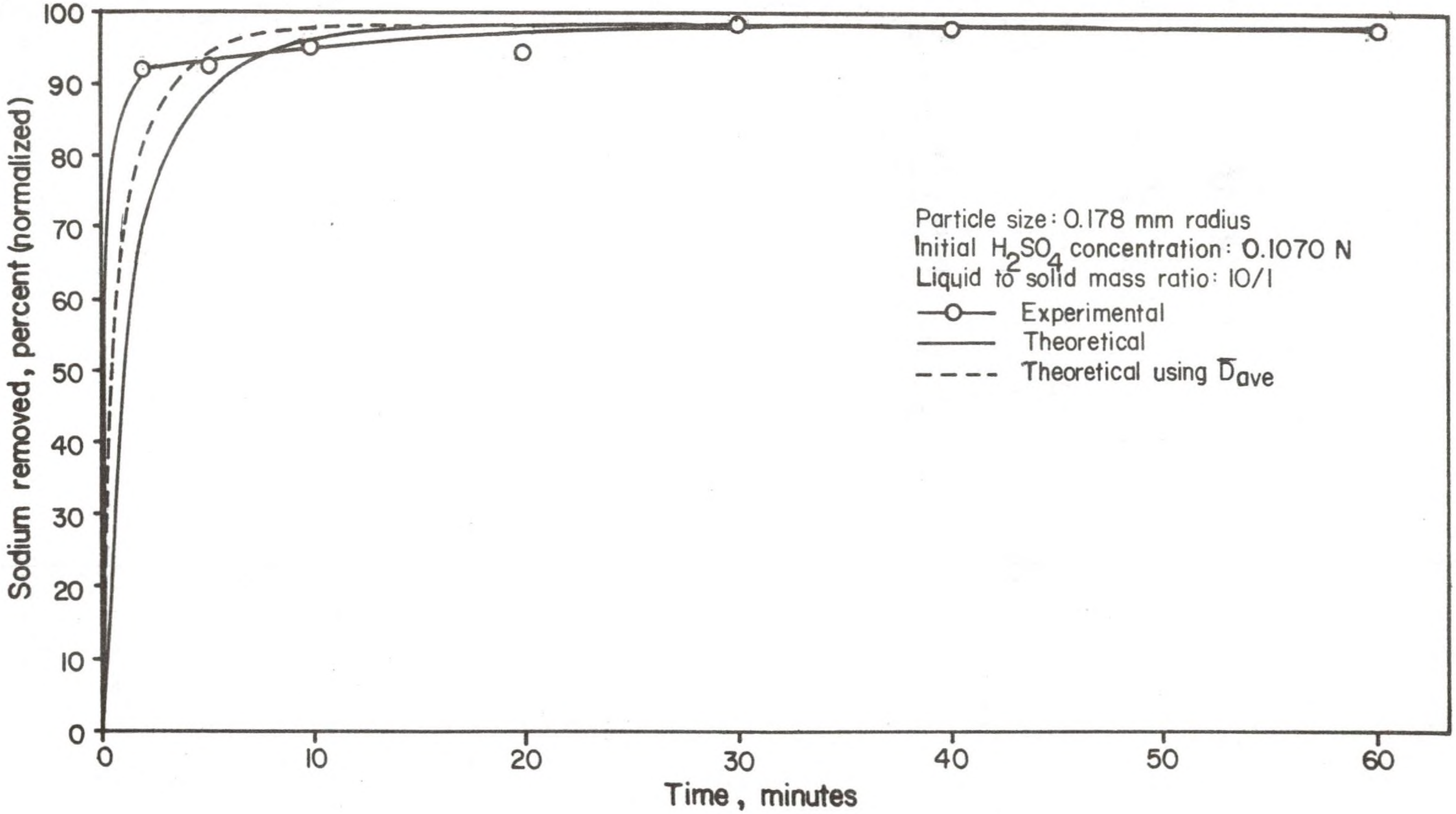

FIGURE 20 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.178 mm Particle Radius (35-48 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

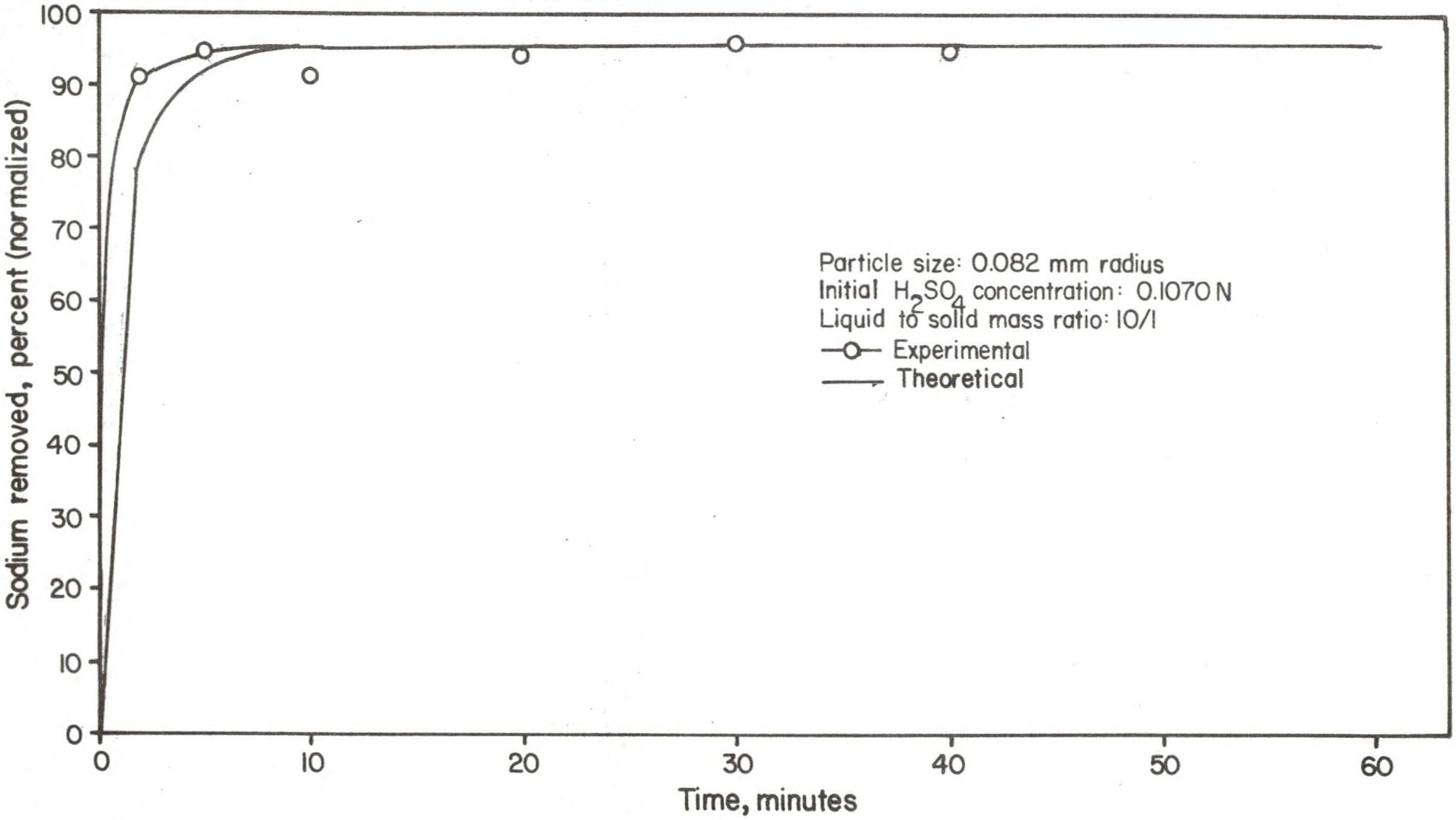

FIGURE 21 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.082 mm Particle Radius (80-100 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

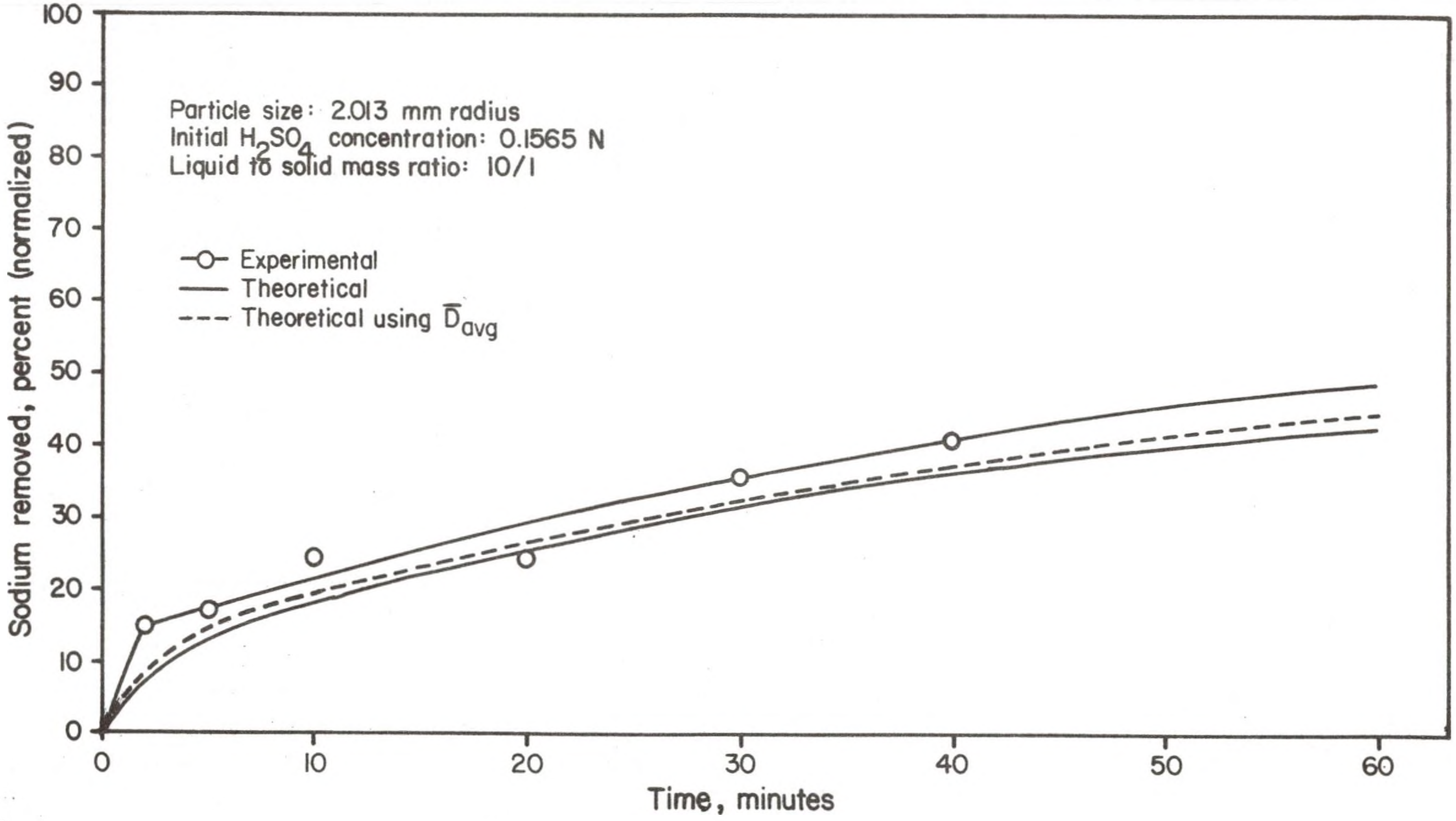

FIGURE 22 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1565 N H_2SO_4 Solution, 2.013 mm Particle Radius (4-6 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

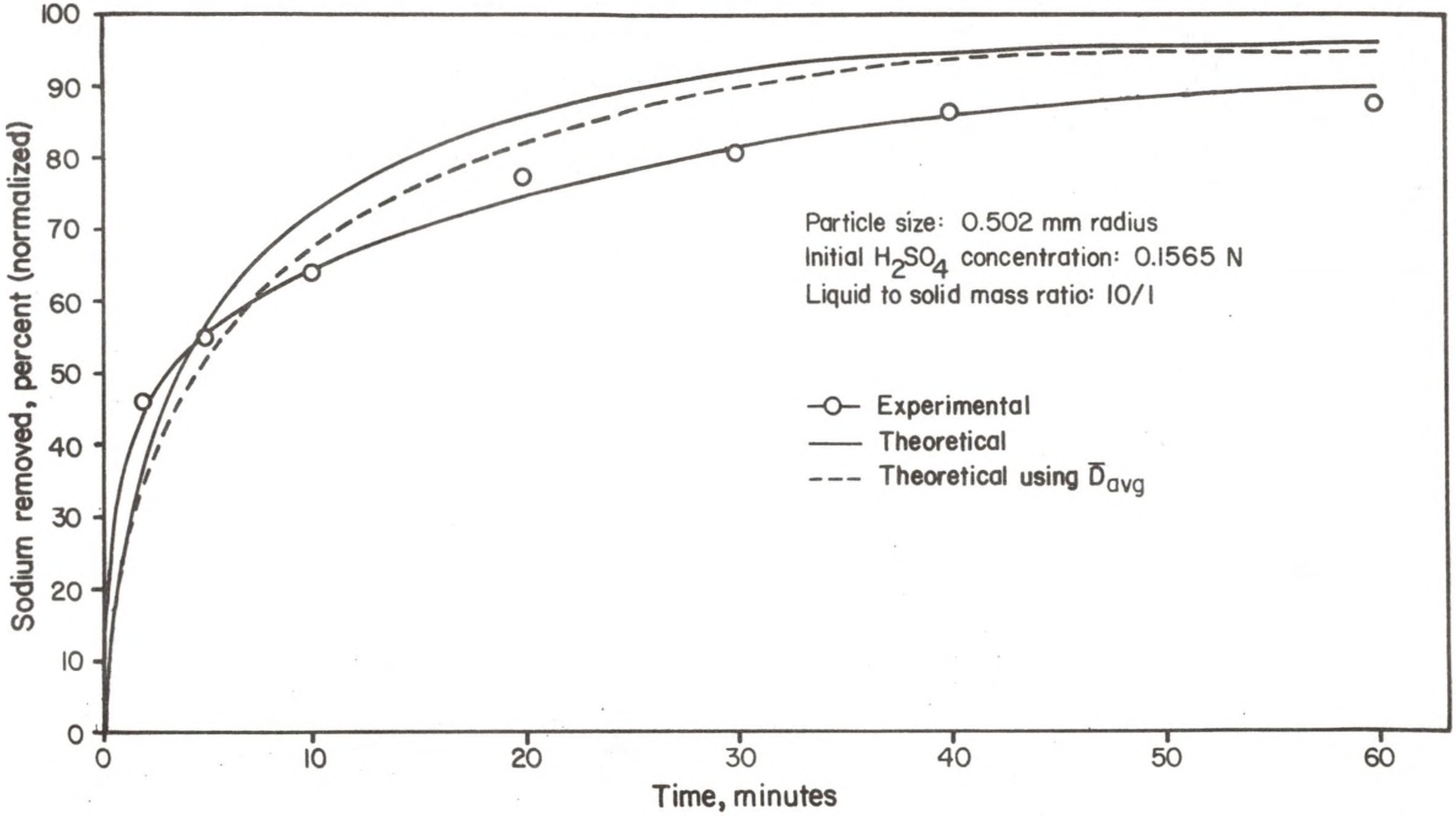

FIGURE 23 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1565 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

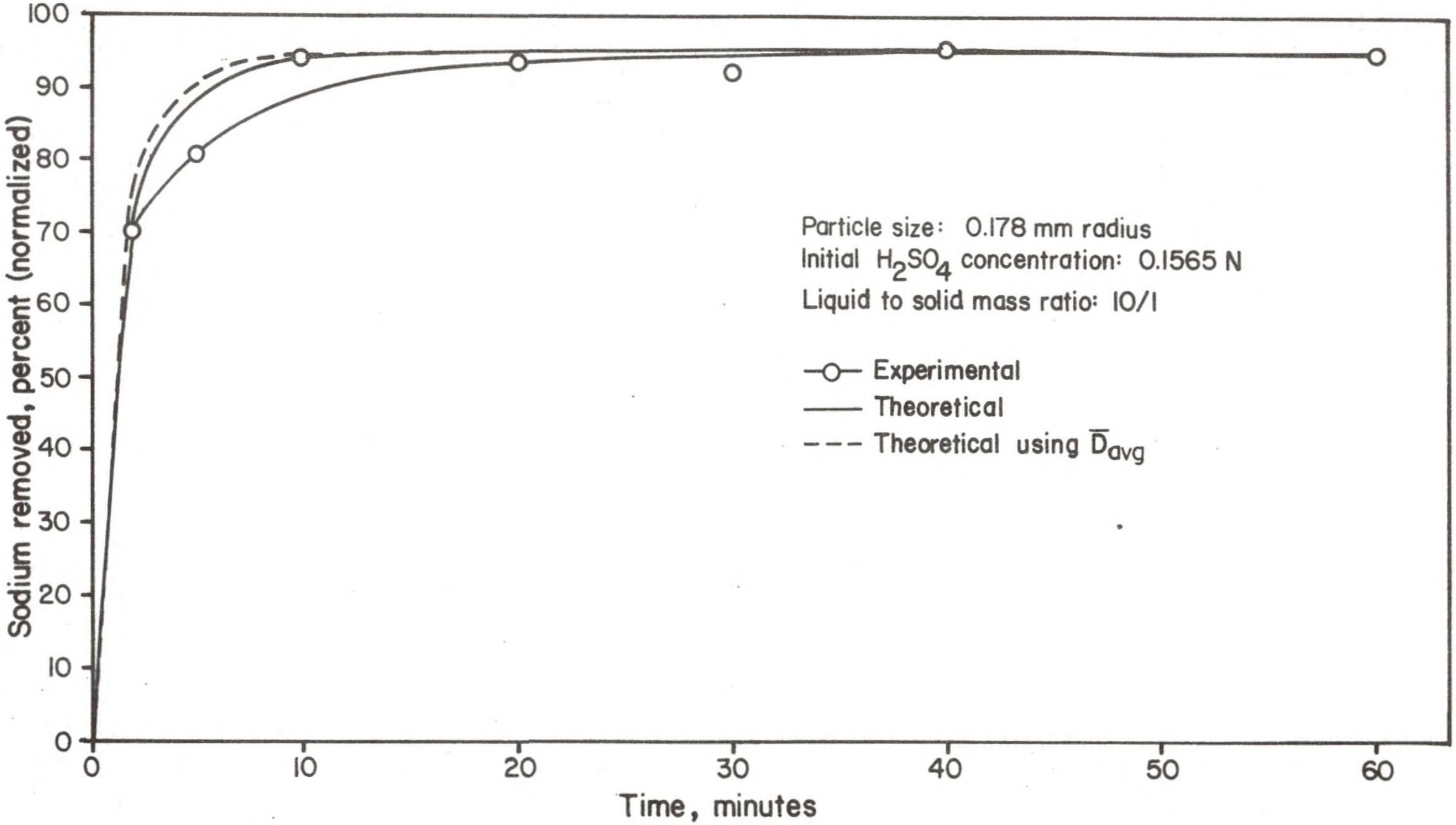

FIGURE 24 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1565 N H_2SO_4 Solution, 0.178 mm Particle Radius (35-48 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

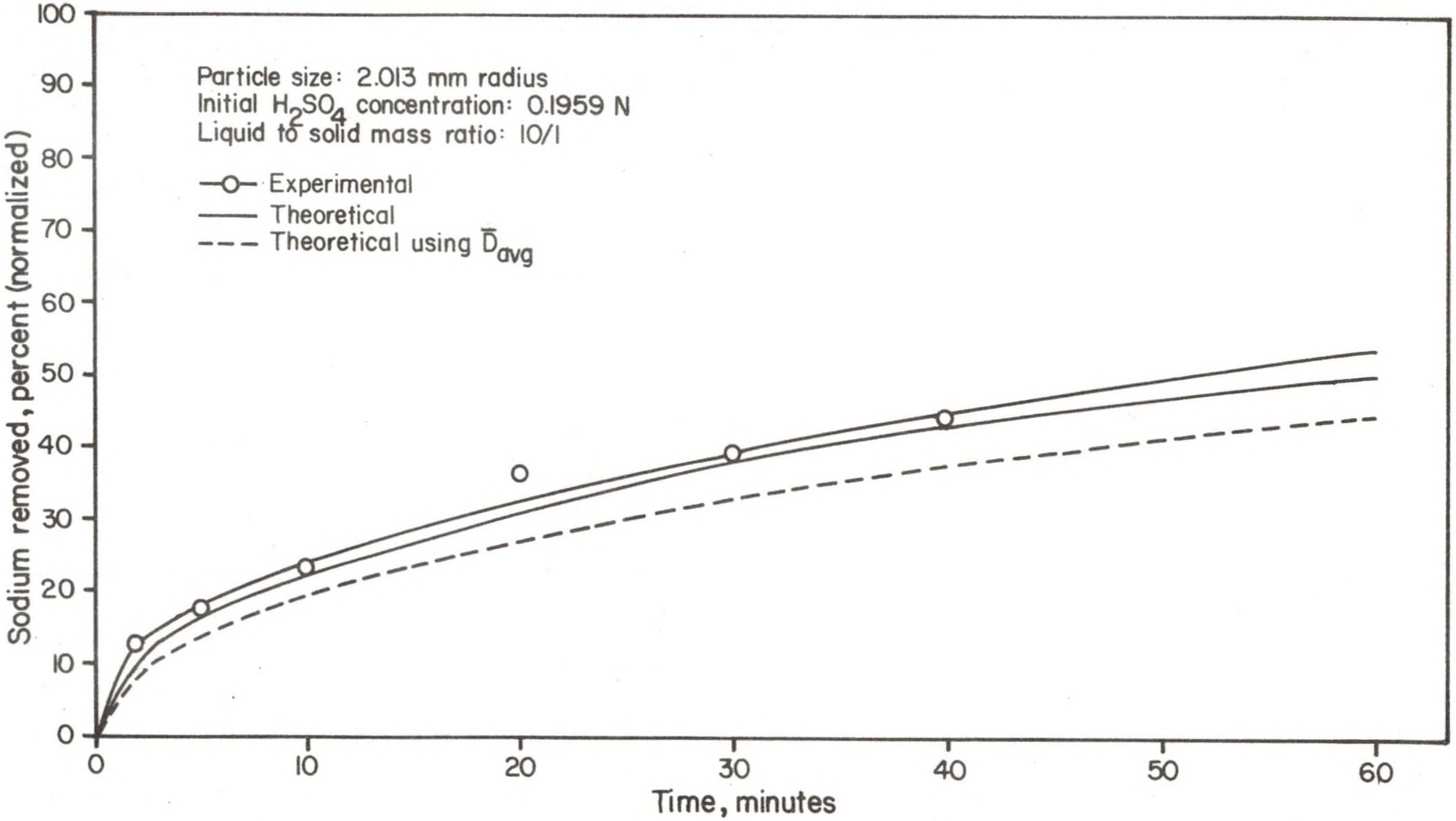

FIGURE 25 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1959 N H_2SO_4 Solution, 2.013 mm Particle Radius (4-6 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

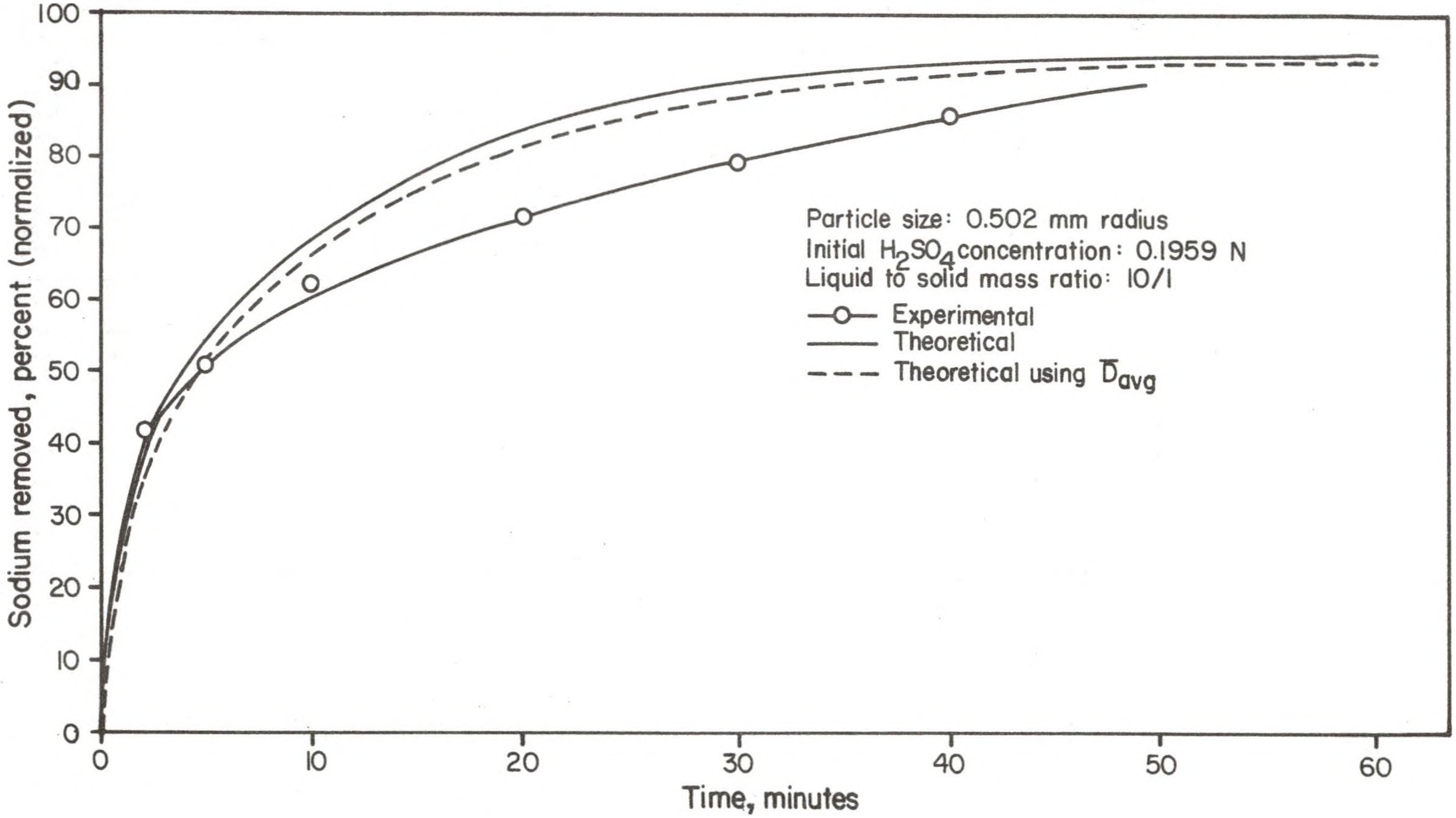

FIGURE 26 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1959 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

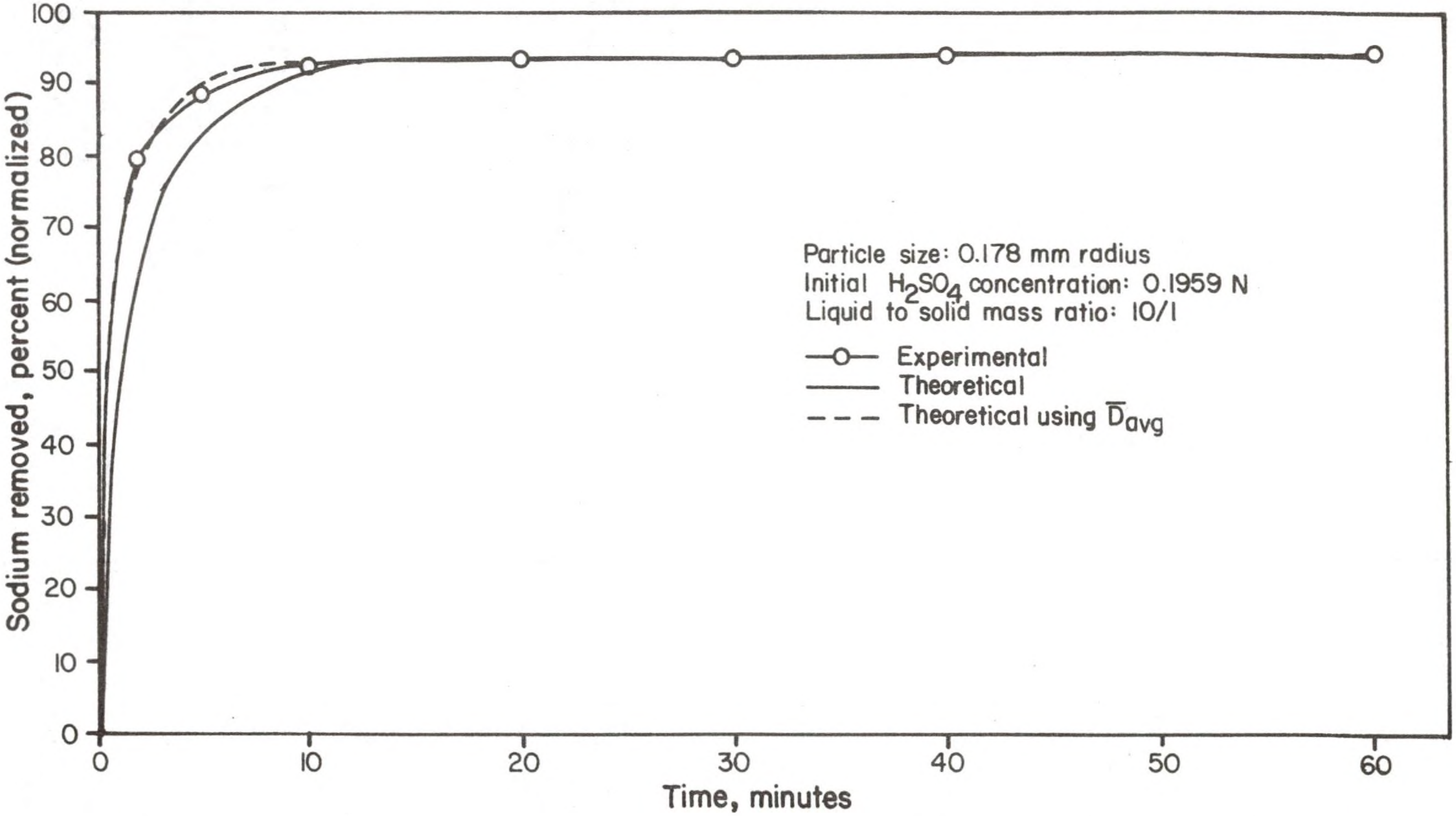

FIGURE 27 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1959 N H_2SO_4 Solution, 0.178 mm Particle Radius (35-48 Mesh Size), and 10/1 Liquid to Solid Mass Ratio

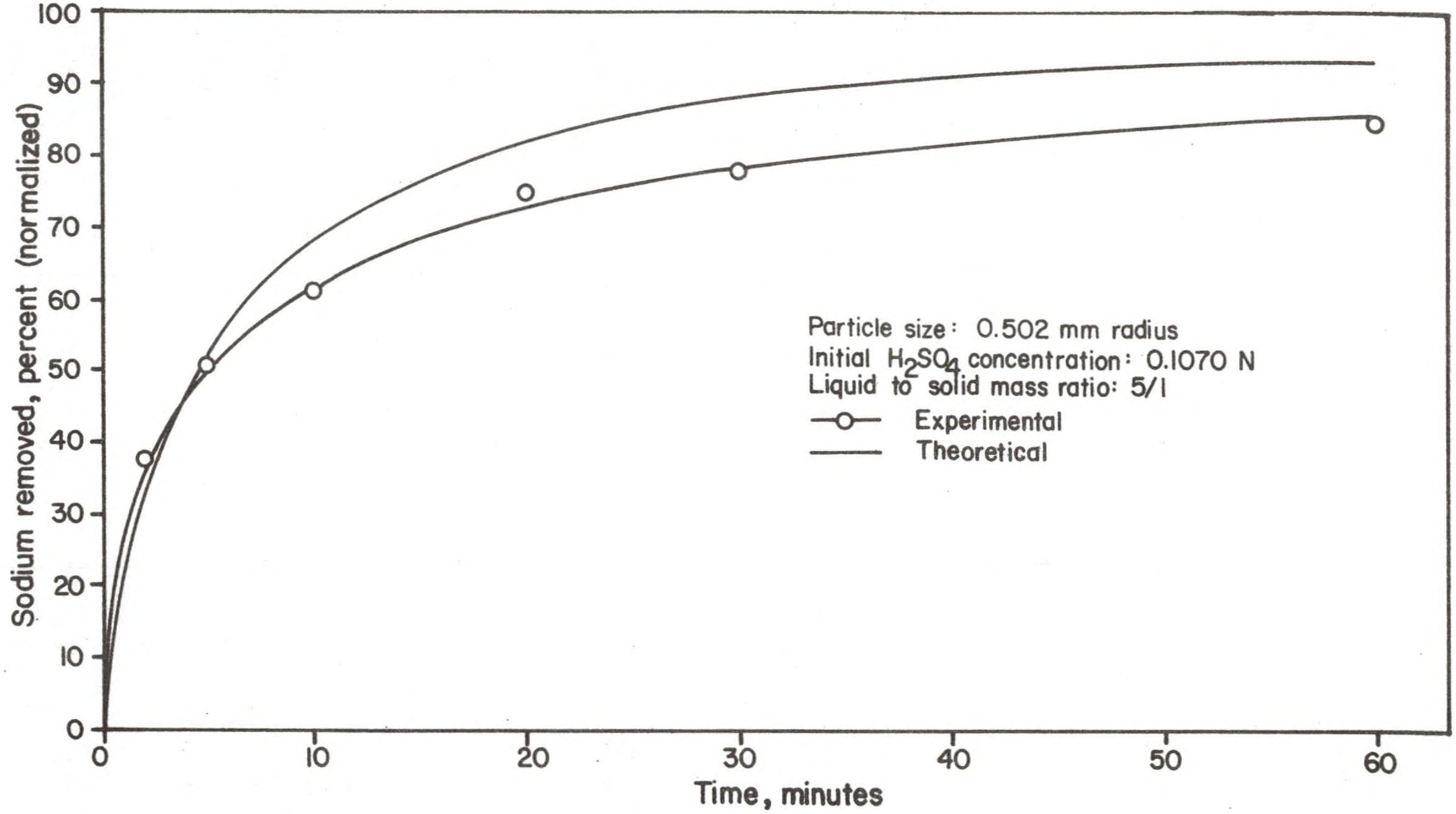

FIGURE 28 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 5/1 Liquid to Solid Mass Ratio

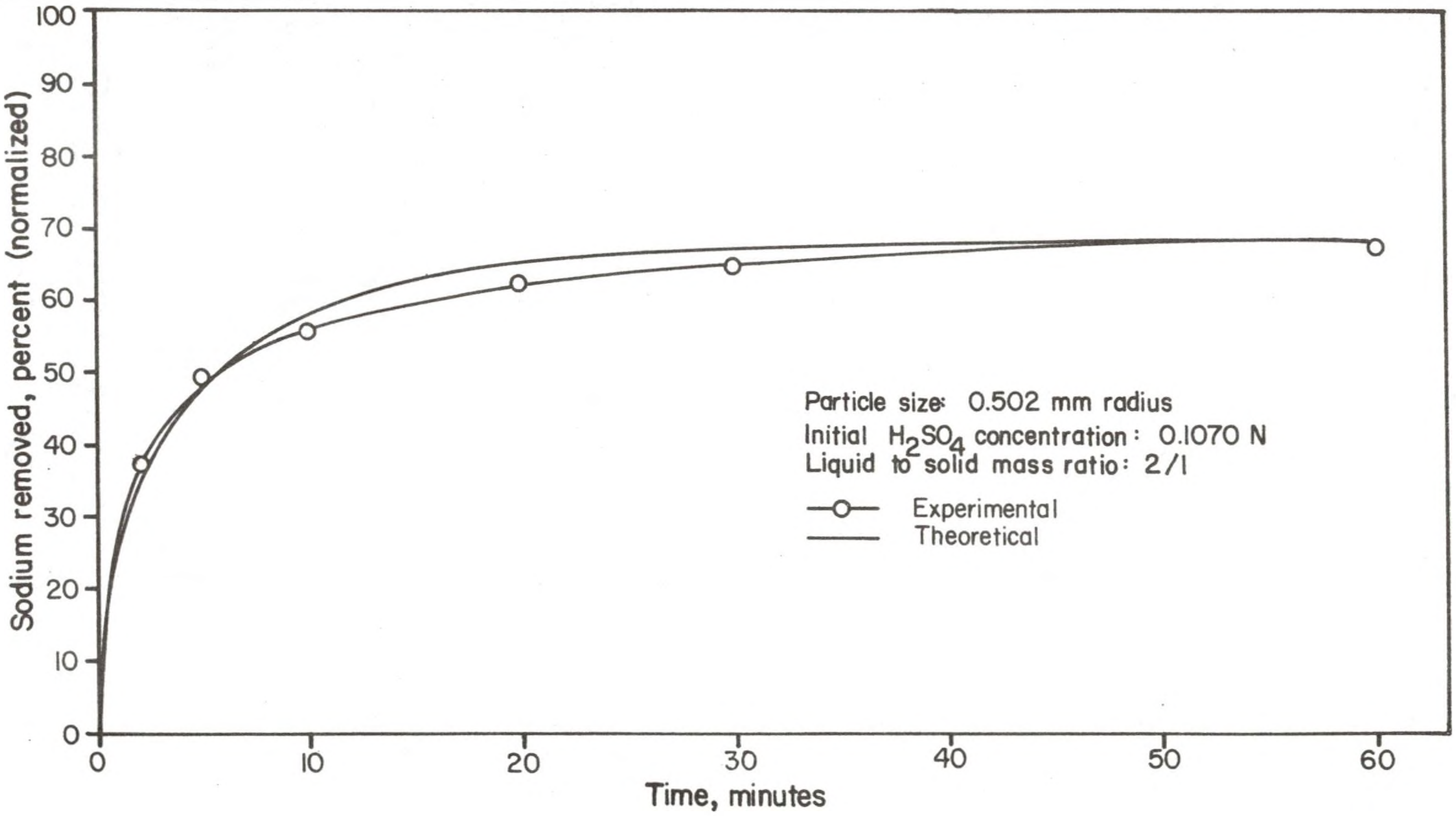

FIGURE 29 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1070 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 2/1 Liquid to Solid Mass Ratio

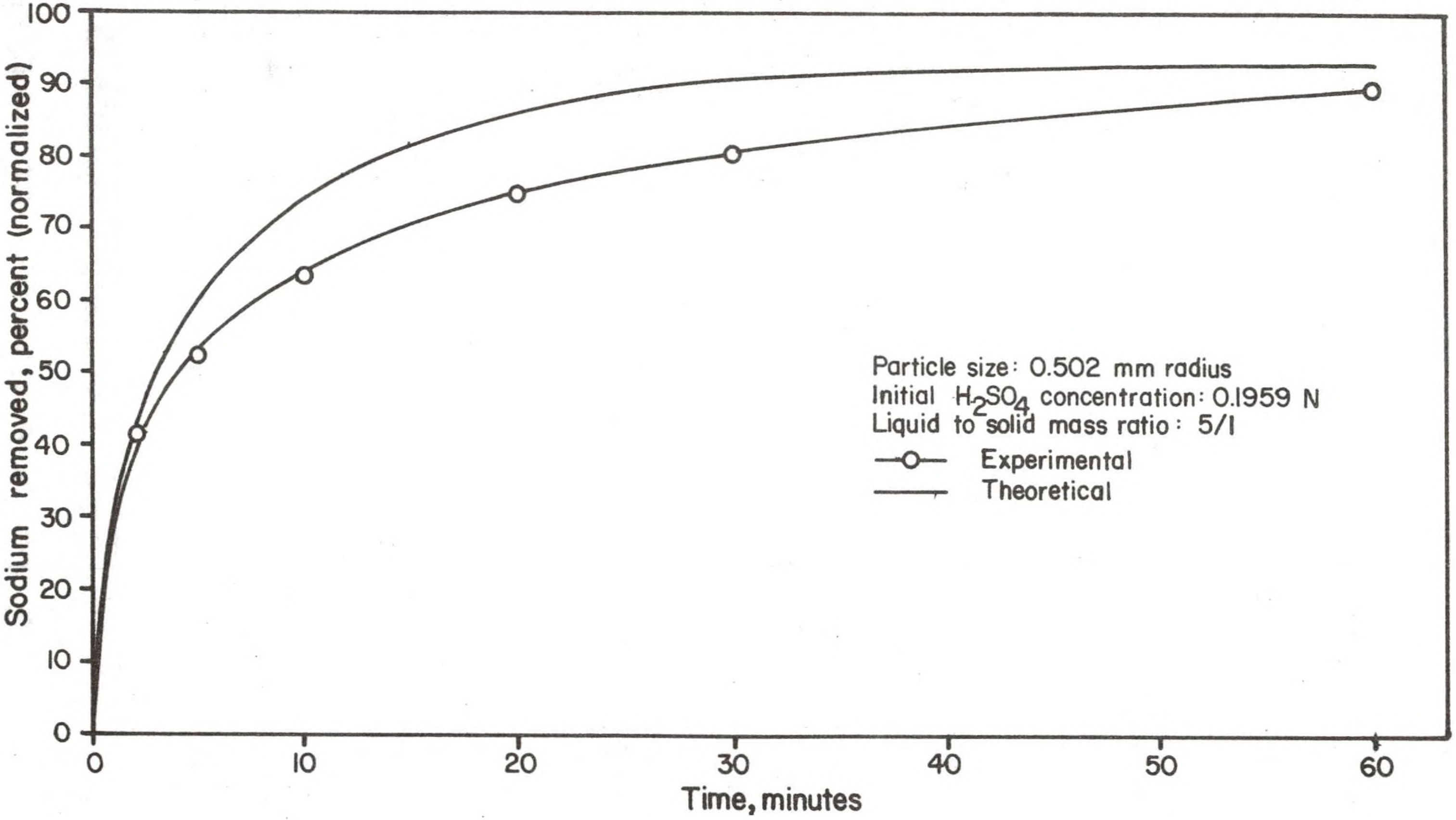

FIGURE 30 - Experimental and Predicted Sodium Removal as a Function of Time Using 0.1959 N H_2SO_4 Solution, 0.502 mm Particle Radius (14-20 Mesh Size), and 5/1 Liquid to Solid Mass Ratio

TABLE 4

SELF DIFFUSION COEFFICIENTS

Particle Radius, mm	H_2SO_4 Conc., N	Liquid/Solid Mass Ratio	Self-Diffusion Coefficient, mm^2/min	
			Actual	Average
2.013	0.1070	10/1	0.0014	
0.502	0.1070	10/1	0.0029	
0.178	0.1070	10/1	0.0012	0.0018
0.082	0.1070	10/1	0.0004	
2.013	0.1565	10/1	0.0016	
0.502	0.1565	10/1	0.0021	
0.178	0.1565	10/1	0.0014	0.0017
2.013	0.1959	10/1	0.0024	
0.502	0.1959	10/1	0.0019	
0.178	0.1959	10/1	0.0010	0.0018
0.502	0.1070	5/1	0.0020	
0.502	0.1959	5/1	0.0026	0.0023
0.502	0.1070	2/1	0.0036	

Half time for the ion exchange was found by using multiple linear regression on the experimental data and solving for the time in which half of the equilibrium amount of Na removal is achieved. Again a computer program was used for the determination of both the half time of the ion exchange and the H-ion:Na-ion self-diffusion coefficient in the coal particle. This program is also shown in Appendix A.

The self-diffusion coefficient for H-ion:Na-ion ion exchange in the coal particle should be constant over all particle sizes of coal at a common initial H_2SO_4 concentration and liquid to solid mass ratio. Using this idea, the individual H-ion:Na-ion self-diffusion coefficients found for each particle size at a common H_2SO_4 solution concentration and mass ratio, were averaged and used to get predicted values for percent Na removed from the Vermuelen's approximation. Further, it was found that the averaged values for the self diffusion coefficient were also constant over the 3 different initial H_2SO_4 concentrations. The constant value for the H-ion:Na-ion self-diffusion coefficient was found to be 0.0018 mm^2/min (3.0×10^{-7} cm^2/sec).

Along with the experimental values in Figures 18-30, graphs were drawn for both Vermuelen's approximation with the actual H-ion:Na-ion self-diffusion coefficient calculated for that specific set of runs, as well as with the overall average value for the H-ion:Na-ion self-diffusion coefficient. In some cases, such as in Figures 18 and 19, the overall average value for the H-ion:Na-ion self-diffusion coefficient had a correcting effect on the predicted values from the Vermuelen's approximation as compared to using the H-ion:Na-ion self-diffusion coefficient for that specific series of runs. The overall average value for the H-ion:Na-ion self-diffusion coefficient helped little to improve the accuracy of the predicted values for the data presented in Figures 25 and 26.

Taken as a whole, with the assumptions made for the derivation of Vermuelen's approximation as well as the use of coal (the crude, nonideal ion exchanger), the results obtained for the prediction of the kinetic response of the ion exchange of Na were quite good. Though not an exact equation, Vermuelen's approximation is a good representation of what was found experimentally for the ion exchange of Na, and the predicted information is readily determined. With the heterogenous nature of coal and the inherent experimental error, averaging of specific H-ion:Na-ion self-diffusion coefficients should be done to help improve the accuracy of the theoretical equation used.

5.5 OTHER IONS

Four other ions, K, Mg, Ca, and Al, were investigated for their removal from coal by ion exchange. By the observation of Figures 2-14, the order of removal was Na> K> Mg> Ca> Al. This is similar to the order predicted by theory where a smaller ion with a lower charge has a higher affinity to displace a larger ion with a higher charge (23).

Chapter VI

CONCLUSIONS

1. Subbituminous coal can have approximately 95 % of its initial Na content removed by ion exchange with dilute aqueous H_2SO_4 on a batch, experimental basis at equilibrium.

2. The ion exchange of Na from subbituminous coal, using aqueous H_2SO_4 as the exchange medium, has particle diffusion as the rate controlling step, provided the agitation rate is set such that the clinging film layer about the coal particle is minimal.

3. The amount of H-ion, as controlled by the liquid to solid mass ratio, has no significant effect on the rate of Na removed, provided sufficient H-ion is present to complete the ion exchange within the range studied.

4. Initial H_2SO_4 concentration has no significant effect on the rate of Na removal over the range of concentration studied.

5. The H-ion:Na-ion self-diffusion coefficient was found to be nearly constant over the concentration range of

H_2SO_4 solution used, at a value of 0.0018 mm^2/min (3.0 x 10^{-7} cm^2/ sec).

6. Particle size was found to be the major factor in the removal of Na over the range of particle sizes studied.
7. Particle size had the most significant effect on Na removal by ion exchange, with residence time following in order.
8. Vermuelen's approximation is a good representation for predicting the ion exchange of Na from subbituminous coal on the batch basis, and the equation can be used to predict the kinetic response of the ion exchange. The self-diffusion coefficient used for Vermuelen's approximation should be an average value over all the particle sizes investigated to improve the accuracy of the theoretical equation.

Appendix A

SAMPLE CALCULATIONS

SAMPLE CALCULATIONS

CAR = Coal as Received
DB = Dry Basis
MAF = Moisture, Ash Free Coal
M = Moisture
MF = Moisture Free Coal
ppm = Part per Million

A) Determine weight of cation from AA analysis

$$\frac{\mu\text{g cation}}{\text{g MAF}} = \text{ppm}\ \frac{100\text{ ml solution}}{\text{Ash in Pellet, g}}\ \frac{\%\text{ Ash DB}}{(100 - \%\text{ Ash})\text{ g MAF}}$$

$$\frac{\mu\text{g Na}}{\text{g MAF}} = 65.5\text{ ppm}\ \frac{100\text{ ml solution}}{0.0997\text{ g Ash}}\ \frac{3.82\ \%\text{ Ash}}{(100 - 3.82\ \%)\text{ g MAF}}$$

$$= 2610\ \frac{\mu\text{g Na}}{\text{g MAF}}$$

$$\frac{\text{g cation}}{\text{g CAR}} = \frac{\mu\text{g cation}}{\text{g MAF}}\ \frac{100 - \%\text{ Ash DB}}{100\text{ g MF}}\ \frac{100 - \%\text{ M}}{100\text{ g CAR}}\ \frac{10^{-6}\text{ g}}{\text{g}}$$

$$\frac{\text{g Na}}{\text{g CAR}} = 2610\ \frac{\mu\text{g Na}}{\text{g MAF}}\ \frac{100 - 3.82\ \%\text{ Ash}}{100\text{ g MF}}\ \frac{100 - 24.0\ \%\text{ M}}{100\text{ g CAR}}\ \frac{10^{-6}\text{ g}}{\text{g}}$$

$$= 1.93 \times 10^{-3}\ \frac{\text{g Na}}{\text{g CAR}}$$

B) Determine % Oxide of the cation in the ash

$$\%\text{ Oxide} = \frac{\mu\text{g cation}}{\text{g MAF}}\ \frac{100 - \%\text{ Ash DB}}{\%\text{ Ash DB}}\ (K)(10^{-4})$$

where

	K
Na	1.35
K	1.20
Mg	1.66
Ca	1.40
Al	1.43

$$\%\ Na_2O = 2610\ \frac{\mu\text{g Na}}{\text{g MAF}}\ \frac{100 - 3.82\ \%\text{ Ash}}{3.82\ \%\text{ Ash}}\ (1.35)(10^{-4})$$

$$= 8.9\ \%\ Na_2O$$

MATERIAL BALANCE CLOSURE PROGRAM

The preceeding will demonstrate how to use the TI-59 program and also list out the complete program.

Input

Item	Storage Location
Weight ash in pellet, grams	09
Weight coal used, grams	10
Volume initial solution recovered, mls	11
Volume of rinse recovered, mls	12
% H_2O in coal as received (CAR)	13
% ash in coal as received	14
% ash in solid sample	15
Weight Na in CAR sample, grams	16
Weight K in CAR sample, grams	17
Weight Ca in CAR sample, grams	18
Weight Mg in CAR sample, grams	19
Weight Al in CAR sample, grams	20
Weight Fe in CAR sample, grams	21
Concentration Na from solid sample, ppm	22
Concentration K from solid sample, ppm	23
Concentration Ca from solid sample, ppm	24
Concentration Mg from solid sample, ppm	25
Concentration Al from solid sample, ppm	26
Concentration Fe from solid sample, ppm	27
Concentration Na from liquid sample, ppm	28
Concentration K from liquid sample, ppm	29
Concentration Ca from liquid sample, ppm	30
Concentration Mg from liquid sample, ppm	31
Concentration Al from liquid sample, ppm	32
Concentration Fe from liquid sample, ppm	33
Concentration Na from rinse solution, ppm	34
Concentration K from rinse solution, ppm	35
Concentration Ca from rinse solution, ppm	36
Concentration Mg from rinse solution, ppm	37
Concentration Al from rinse solution, ppm	38
Concentration Fe from rinse solution, ppm	39

Output

```
                                                  NA

% Ion Remaining in Coal                     7.1172066  01
% Ion Removed by Solution                   3.3957265  01
% Ion Removed by Rinse                      1.2820513  00
Material Balance Closure                    1.0641138  02

Normalized % Ion Remaining in Coal          6.6883885  01
Normalized % Ion Removed by Solution        3.1911309  01
Normalized % Ion Removed by Rinse           1.2048065  00
```

which means

% Closure = 106.4 %
% Na Removed = 31.9 % + 1.2 % = 33.1 %

COMPLETE LISTING OF TI-59 PROGRAM FOR MATERIAL BALANCE CLOSURES

```
000 43 RCL          051 94 +/-          101 00  0
001 15  15          052 95  =           102 00  0
002 65  X           053 42 STO          103 95  =
003 53  (           054 07  07          104 42 STO
004 01  1           055 43 RCL          105 05  05
005 00  0           056 12  12          106 99 PRT
006 00  0           057 65  X           107 43 RCL
007 75  -           058 01  1           108 08  08
008 43 RCL          059 82 EE           109 65  X
009 13  13          060 06  6           110 43 RCL
010 54  )           061 94 +/-          111 34  34
011 65  X           062 95  =           112 55  ÷
012 53  (           063 42 STO          113 43 RCL
013 01  1           064 08  08          114 16  16
014 00  0           065 98 ADV          115 65  X
015 00  0           066 03  3           116 01  1
016 75  -           067 01  1           117 00  0
017 43 RCL          068 01  1           118 00  0
018 14  14          069 03  3           119 95  =
019 54  )           070 69 OP           120 42 STO
020 65  X           071 02  02          121 06  06
021 43 RCL          072 69 OP           122 99 PRT
022 10  10          073 05  05          123 43 RCL
023 65  X           074 98 ADV          124 04  04
024 01  1           075 43 RCL          125 85  ÷
025 52 EE           076 01  01          126 43 RCL
026 08  8           077 65  X           127 05  05
027 94 +/-          078 43 RCL          128 85  ÷
028 55  ÷           079 22  22          129 43 RCL
029 53  (           080 55  ÷           130 06  06
030 43 RCL          081 43 RCL          131 95  =
031 09  09          082 16  16          132 42 STO
032 65  X           083 65  X           133 03  03
033 53  (           084 01  1           134 99 PRT
034 01  1           085 00  0           135 98 ADV
035 00  0           086 00  0           136 01  1
036 00  0           087 95  =           137 00  0
037 75  -           088 99 PRT          138 00  0
038 43 RCL          089 42 STO          139 55  ÷
039 15  15          090 04  04          140 53 RCL
040 54  )           091 43 RCL          141 03  03
041 54  )           092 07  07          142 95  =
042 95  =           093 65  X           143 42 STO
043 42 STO          094 43 RCL          144 02  02
044 01  01          095 28  28          145 65  X
045 43 RCL          096 55  ÷           146 43 RCL
046 11  11          097 43 RCL          147 04  04
047 65  X           098 16  16          148 95  =
048 01  1           099 65  X           149 99 PRT
049 52 EE           100 01  1           150 43 RCL
```

```
050 06   6
151 02   02
152 65   X
153 43  RCL
154 05   05
155 95   =
156 99  PRT
157 43  RCL
158 02   02
159 65   X
160 43  RCL
161 06   06
162 95   =
163 99  PRT
164 98  ADV
165 02   2
166 06   6
167 69  OP
168 02   02
169 69  OP
170 05   05
171 98  ADV
172 43  RCL
173 01   01
174 65   X
175 43  RCL
176 23   23
177 55   ÷
178 43  RCL
179 17   17
180 65   X
181 01   1
182 00   0
183 00   0
184 95   =
185 99  PRT
186 42  STO
187 04   04
188 43  RCL
189 07   07
190 65   X
191 43  RCL
192 29   29
193 55   ÷
194 43  RCL
195 17   17
196 65   X
197 01   1
198 00   0
199 00   0
200 95   =
201 99  PRT
202 42  STO
203 05   05
204 53  RCL
205 08   08
206 65   X
207 43  RCL
208 35   35
209 55   ÷
210 43  RCL
211 17   17
212 65   X
213 01   1
214 00   0
215 00   0
216 95   =
217 99  PRT
218 42  STO
219 06   06
220 85   ÷
221 43  RCL
222 05   05
223 85   ÷
224 43  RCL
225 04   04
226 95   =
227 99  PRT
228 42  STO
229 03   03
230 98  ADV
231 01   1
232 00   0
233 00   0
234 55   ÷
235 43  RCL
236 03   03
237 95   =
238 42  STO
239 02   02
240 65   X
241 43  RCL
242 04   04
243 95   =
244 99  PRT
245 43  RCL
246 02   02
247 65   X
248 43  RCL
249 05   05
250 95   =
251 99  PRT
252 43  RCL
253 02   02
254 65   X
255 43  RCL
256 06   06
257 95   =
258 99  PRT
259 98  ADV
260 01   1
261 05   5
262 01   1
263 03   3
264 69  OP
265 02   02
266 69  OP
267 05   05
268 98  ADV
269 43  RCL
270 01   01
271 65   X
272 43  RCL
273 24   24
274 55   ÷
275 43  RCL
276 18   18
277 65   X
278 01   1
279 00   0
280 00   0
281 95   =
282 42  STO
283 04   04
284 99  PRT
285 43  RCL
286 07   07
287 65   X
288 43  RCL
289 30   30
290 55   ÷
291 43  RCL
292 18   18
293 65   X
294 01   1
295 00   0
296 00   0
297 95   =
298 42  STO
299 05   05
300 99  PRT
```

```
301 43 RCL
302 08  08
303 65  X
304 43 RCL
305 36  36
306 55  ÷
307 43 RCL
308 18  18
309 65  X
310 01  1
311 00  0
312 00  0
313 95  =
314 42 STO
315 06  06
316 99 PRT
317 85  +
318 43 RCL
319 05  05
320 85  +
321 43 RCL
322 04  04
323 95  =
324 99 PRT
325 35 1/X
326 65  X
327 01  1
328 00  0
329 00  0
330 95  =
331 42 STO
332 02  02
333 98 ADV
334 65  X
335 43 RCL
336 04  04
337 95  =
338 99 PRT
339 43 RCL
340 02  02
341 65  X
342 43 RCL
343 05  05
344 95  =
345 99 PRT
346 43 RCL
347 02  02
348 65  X
349 43 RCL
350 06  06
351 95  =
352 99 PRT
353 98 ADV
354 03  3
355 00  0
356 02  2
357 02  2
358 69 OP
359 02  02
360 69 OP
361 05  05
362 98 ADV
363 43 RCL
364 01  01
365 65  X
366 43 RCL
367 25  25
368 55  ÷
369 43 RCL
370 19  19
371 65  X
372 01  1
373 00  0
374 00  0
375 95  =
376 42 STO
377 04  04
378 99 PRT
379 43 RCL
380 07  07
381 65  X
382 43 RCL
383 31  31
384 55  ÷
385 43 RCL
386 19  19
387 65  X
388 01  1
389 00  0
390 00  0
391 95  =
392 42 STO
393 05  05
394 99 PRT
395 43 RCL
396 08  08
397 65  X
398 43 RCL
399 37  37
400 55  ÷
401 43 RCL
402 19  19
403 65  X
404 01  1
405 00  0
406 00  0
407 95  =
408 42 STO
409 06  06
410 99 PRT
411 85
412 43 RCL
413 05  05
414 85  ÷
415 43 RCL
416 04  04
417 95  =
418 99 PRT
419 98 ADV
420 35 1/X
421 65  X
422 01  1
423 00  0
424 00  0
425 95  =
426 42 STO
427 02  02
428 65  X
429 43 RCL
430 04  04
431 95  =
432 99 PRT
433 43 RCL
434 02  02
435 65  X
436 43 RCL
437 05  05
438 95  =
439 99 PRT
440 43 RCL
441 02  02
442 65  X
443 43 RCL
444 06  06
445 95  =
446 99 PRT
447 98 ADV
448 01  1
449 03  3
450 02  2
```

```
451 07  07         501 95   =          551 43 RCL
452 69 OP          502 42  STO         552 01  01
453 02  02         503 06   06         553 65  X
454 69 OP          504 99  PRT         554 43 RCL
455 05  05         505 85   +          555 27  27
456 98 ADV         506 43  RCL         556 55  ÷
457 43 RCL         507 05   05         557 43 RCL
458 01  01         508 85   +          558 21  21
459 65  X          509 43  RCL         559 65  X
460 43 RCL         510 04   04         560 01  1
461 26  26         511 95   =          561 00  0
462 55  ÷          512 99  PRT         562 00  0
463 43 RCL         513 98  ADV         563 95  =
464 20  20         514 35  1/X         564 42 STO
465 65  X          515 65   X          565 04  04
466 01  1          516 01   1          566 99 PRT
467 00  0          517 00   0          567 43 RCL
468 00  0          518 00   0          568 07  07
469 95  =          519 95   =          569 65  X
470 42 STO         520 42  STO         570 43 RCL
471 04  04         521 02   02         571 33  33
472 99 PRT         522 65   X          572 55  ÷
473 43 RCL         523 43  RCL         573 43 RCL
474 07  07         524 04   04         574 21  21
475 65  X          525 95   =          575 65  X
476 43 RCL         526 99  PRT         576 01  1
477 32  32         527 43  RCL         577 00  0
478 55  ÷          528 02   02         578 00  0
479 43 RCL         529 65   X          579 95  =
480 20  20         530 43  RCL         580 42 STO
481 65  X          531 05   05         581 05  05
482 01  1          532 95   =          582 99 PRT
483 00  0          533 99  PRT         583 43 RCL
484 00  0          534 43  RCL         584 08  08
485 95  =          535 02   02         585 65  X
486 42 STO         536 65   X          586 43 RCL
487 05  05         537 43  RCL         587 39  39
488 99 PRT         538 06   06         588 55  ÷
489 43 RCL         539 95   =          589 43 RCL
490 08  08         540 99  PRT         590 21  21
491 65  X          541 98  ADV         591 65  X
492 43 RCL         542 02   2          592 01  1
493 38  38         543 01   1          593 00  0
494 55  ÷          544 01   1          594 00  0
495 43 RCL         545 07   7          595 95  =
496 20  20         546 69  OP          596 42 STO
497 65  X          547 02   02         597 06  06
498 01  1          548 69  OP          598 99 PRT
499 00  0          549 05   05         599 85
500 00  0          550 98  ADV         600 43 RCL
```

```
601 05  05
602 85
603 43 RCL
604 04  04
605 95  =
606 99 PRT
607 98 ADV
608 35 1/X
609 65  X
610 01  1
611 00  0
612 00  0
613 95  =
614 42 STO
615 02  02
616 65  X
617 43 RCL
618 04  04
619 95  =
620 99 PRT
621 43 RCL
622 02  02
623 65  X
624 43 RCL
625 05  05
626 95  =
627 99 PRT
628 43 RCL
629 02  02
630 65  X
631 43 RCL
632 06  06
633 95  =
634 99 PRT
635 98 ADV
636 91 R/S
637 00  0
638 00  0
639 00  0
```

MULTIPLE LINEAR REGRESSION PROGRAM

The following will both show how to use the multiple linear regression program and list out the complete program. The regression program fits experimental data into the form of the following equation.

$$(\%\ \text{Na Removed}) = \text{Bzero} + \exp(\text{Bone}*\text{Time}) + \text{Time}^{\text{Btwo}}$$

The interactive program will run as follows:

Input

```
THE NUMBER OF RUNS IN THIS SERIES OF RUNS IS, REAL NUMBER?
?
THE TIME FOR THIS RUN IS, MIN.?
?
THE PERCENT REMOVED IN THIS TIME IS?
?
THE PERCENT NA REMOVED AT EQUILIBRIUM IS?
?
THE MEAN RADIUS FOR THE MESH SIZE IS, MM?
?
```

Output

```
    (Bone)        (Btwo)       (Bzero)
  -0.005958     0.341283     3.396585

     TIME           % REMOVED

     60.0            84.47
     40.0            82.86
     30.0            79.73
     20.0            73.69
     10.0            61.73
      5.0            50.20
      2.0            37.38

THE CORRELATION INDEX, R**2, IS 0.997599

THE T HALF VALUE IS 3.8 MIN.

THE DIFFUSIVITY VALUE IS 0.0036 MM**2/MIN
```

COMPLETE LISTING OF THE FORTRAN 77 INTERACTIVE MULTIPLE LINEAR REGRESSION PROGRAM

```
1*  THIS IS A INTERACTIVE LINEAR REGRESSION PROGRAM
2*
10  DIMENSION X(20), Y(20), YLN(20), TV(10), XLN(20)
15*
20         TV(1) = 60.0
30         TV(2) = 40.0
40         TV(3) = 30.0
50         TV(4) = 20.0
60         TV(5) = 10.0
70         TV(6) =  5.0
80         TV(7) =  2.0
85*
90  WRITE(6,1000)
100  READ*,N
105*
110 DO 5 I = 1,N
120        WRITE(6,1010)
130         READ(5,1011) X(I)
140        WRITE(6,1020)
150         READ(5,1011) Y(I)
160 5 CONTINUE
165*
170 WRITE(6,1030)
180  READ(5,1011) YEQUIL
190 WRITE(6,1040)
200  READ(5,1011) RAD
205*
210 DO 20 IA = 1,N
220        YLN(IA) = ALOG(Y(IA))
230        XLN(IA) = ALOG(X(IA))
240 20 CONTINUE
245*
250* MAIN CALCULATIONS
255*
260   XTOT = 0.0
270   XBTOT = 0.0
280   YTOT = 0.0
285*
290 DO 30 IB = 1,N
300        YTOT = YTOT + YLN(IB)
310        XBTOT = XBTOT + XLN(IB)
320        XTOT = XTOT + X(IB)
330 30 CONTINUE
330*
340   XAVE = XTOT/ FLOAT(N)
350   XBAVE = XBTOT/ FLOAT(N)
360   YAVE = YTOT/ FLOAT(N)
365*
370   SXY = 0.0
380   SBY = 0.0
390   SBB = 0.0
```

```
400   SXB = 0.0
410   SXX = 0.0
420   SYY = 0.0
425*
430 DO 40 IC = 1,N
440   SXY = (X(IC)-XAVE)*(YLN(IC)-YAVE) + SXY
450   SXB = (X(IC)-XAVE)*(XLN(IC)-XBAVE) + SXB
460   SBY = (XLN(IC)-XBAVE)*(YLN(IC)-YAVE) + SBY
470   SBB = (XLN(IC)-XBAVE)*(XLN(IC)-XBAVE) + SBY
480   SXX = (X(IC)-XAVE)*(X(IC)-XAVE) + SXX
490   SYY = (YLN(IC)-YAVE)*(YLN(IC)-YAVE) + SYY
500 40 CONTINUE
505*
510 BONE = (SBB*SXY-SXB*SBY)/(SXX*SBB-(SXB**2.0))
520 BTWO = (SXX*SBY-SXB*SXY)/(SXX*SBB-(SXB**2.0))
530 BZERO = YAVE - BONE*XAVE - BTWO*XBAVE
535*
540   WRITE(6,1800) BONE,BTWO,BZERO
550   WRITE(6,1800)
555*
560 DO 100 JA = 1,7
570   AVAL = BZERO + BONE*(TV(JA)) + BTWO*(ALOG(TV(JA)))
580   YPRINT = EXP(AVAL)
590   WRITE(6,1900) YPRINT
600 100 CONTINUE
605*
610   RSQUAR = (BONE*SXY + BTWO*SBY)/SYY
620   WRITE(6,2030) RSQUAR
630   YHALF = YEQUIL/2.0
640   YVAL = ALOG(YHALF)
650   THALF = 50.0
660 140 AVAL = BZERO + BONE*THALF + BTWO*(ALOG(THALF))
670   ADIFF = AVAL - YVAL
680 IF(ADIFF .GT. .0001) GO TO 150
690 IF(ADIFF .LT. (-.0001)) GO TO 150
700      WRITE(6,2010) THALF
710 GO TO 160
715*
720 150 THALF = THALF - ADIFF*THALF
730 GO TO 140
735*
740 160 D = (.03*RAD**2.0)/THALF
750   WRITE(6,2020) D
755*
760* THIS IS THE FORMAT SECTION
765*
770 1000 FORMAT(/2X,'THE NUMBER OF RUNS IN THIS SERIES
IS,REAL')
780 1010 FORMAT(/2X,'THE TIME FOR THIS RUN IS, MIN.')
790 1011 FORMAT(F15.6)
800 1020 FORMAT(/2X,'THE PERCENT REMOVED IN THIS TIME IS')
810 1030 FORMAT(/2X,'THE PERCENT NA REMOVED AT EQUILIBRIUM
IS')
```

```
820 1040 FORMAT(/2X,'THE MEAN RADIUS FOR THIS MESH SIZE IS,
MM')
830 1800 FORMAT(/2X,'           TIME       %REMOVED')
840 1900 FORMAT(/2X, F10.1,' ',F10.5)
850 2010 FORMAT(/2X,'THE T HALF VALUE IS ',F10.1,' MIN.')
860 2020 FORMAT(/2X'THE DIFFUSIVITY VALUE IS ',F10.4,'
MM**2/MIN')
870 2030 FORMAT(/2X,'THE CORRELATION INDEX, R**2, IS
',F10.6)
880 2040 FORMAT(/2X,F13.6,' ',F13.6,' ',F13.6)
890 STOP
900 END
```

VERMUELEN'S APPROXIMATION PROGRAM

The precceeding shall show how to use the Vermuelen's approximation program as well as listing the complete program.

The interactive program runs as follows:

Input

```
THE VALUE FOR THE D VALUE IS, MM**2/MIN
?
THE VALUE FOR THE MEAN RADIUS IS, MM
?
THE EQUILIBRIUM VALUE FOR THE % NA REMOVED IS
?
```

Output

```
 RESIDENCE   FRACTION OF EQUILIBRIUM  % CATION REMOVED

  60.0                0.99989            67.29291
  40.0                0.99823            67.18085
  30.0                0.99272            66.81007
  20.0                0.96980            65.26781
  10.0                0.86955            58.52060
   5.0                0.71144            47.88014
   2.0                0.49587            33.37193
```

COMPLETE LISTING OF THE FORTRAN 77 INTERACTIVE VERMUELEN'S APPROXIMATION PROGRAM

```
1*    THIS PROGRAM IS A ESTIMATION OF FICKS LAW
5*
10    DIMENSION ATIME(10), AREMOV(10), UVAL(10)
15*
20        ATIME(1) = 60.0
30        ATIME(2) = 40.0
40        ATIME(3) = 30.0
50        ATIME(4) = 20.0
60        ATIME(5) = 10.0
70        ATIME(6) =  5.0
80        ATIME(7) =  2.0
85*
90  WRITE(6,99)
100 WRITE(6,100)
110  READ(5,101) DVAL
120 WRITE(6,110)
130  READ(5,101) ARVAL
140 WRITE(6,115)
150  READ(5,101) AEQUIL
155*
160* MAIN CALCULATIONS
165*
170 DO 50 IJOB = 1,7
180   ANUM = (DVAL*ATIME(IJOB)*((22.0/7.0)**2.0))/
(ARVAL)**2.0
190   AENUM = EXP(-(ANUM))
200   UVAL(IJOB) = (1.0 - AENUM)**0.5
210   AREMOV(IJOB) = AEQUIL*UVAL(IJOB)
220 50 CONTINUE
225*
230 WRITE(6,125)
240 WRITE(6,126)
245*
250 DO 95 IPRINT 1,7
260   WRITE(6,130) ATIME(IPRINT),UVAL(IPRINT),AREMOV(IPRINT)
270   WRITE(6,131)
280 95 CONTINUE
285*
290   WRITE(6,200)
300    READ(5,101) AEND
310 IF(AEND .LT. 2.0) GO TO 1
320 GO TO 900
325*
330* THIS IS THE FORMAT SECTION
335*
340 99  FORMAT(/2X,'THIS PROGRAM IS A ESTIMATION OF FICKS
LAW')
350 100 FORMAT(/2X,'THE VALUE FOR THE D VALUE IS,
MM**2/MIN')
360 101 FORMAT(F15.10)
370 110 FORMAT(/2X,'THE VALUE FOR THE MEAN RADIUS IS, MM')
```

```
380 115 FORMAT(/2X,'THE EQUILIBRIUM VALUE FOR THE % NA
REMOVED IS')
390 125 FORMAT(//2X,'RESIDENCE  FRACTION OF EQUILIBRIUM
FRACTION CATION REMOVED')
400 126 FORMAT(50('*'))
410 130 FORMAT(/2X,F6.2,'  *   ',F8.5,'     *    ',F8.5)
420 131 FORMAT(/50('*'))
430 200 FORMAT(///2X,'IF YOU WANT TO CONTINUE PRINT 1.0,
ELSE 5.0')
435*
440 900 STOP
450  END
```

Appendix B

RAW DATA

TABLE 5

AS RECEIVED COAL ANALYSIS

Particle Radius, mm	Moisture, %	Ash, (Dry Basis) %	Sulfur, %	Ions Present									
				µg/g MAF Coal					% Oxide in Ash				
				Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
2.013	23.5	4.26	0.07	2670	159.0	1390	6240	2950	8.2	0.4	5.2	19.7	9.6
	24.5	3.46	0.14	2360	99.8	1280	6240	2560	8.9	0.3	5.9	24.4	10.2
	24.3	3.56	0.17	2750	134.0	1160	5440	3350	10.1	0.4	5.3	20.7	13.1
0.502	24.3	3.59	0.04	3150	186.0	1610	6010	4460	11.4	0.6	7.2	22.6	17.2
	22.8	3.62	0.15	3120	169.0	1030	5250	2990	11.3	0.6	4.6	19.6	11.4
	23.2	3.67	0.16	2670	175.0	1160	5410	3660	9.4	0.6	5.1	19.9	13.7
	23.5	3.71	0.15	2790	166.0	1180	5750	3250	9.9	0.6	5.1	21.2	12.2
0.178	24.1	3.81	0.08	2930	215.0	1310	6510	3990	10.0	0.7	5.5	23.1	14.4
	23.3	3.86	0.16	2780	196.0	1130	5530	3590	9.5	0.6	4.7	19.3	12.8
	23.3	3.90	0.18	2910	160.0	1180	5460	3930	9.7	0.5	4.8	18.9	13.9
0.082	22.6	3.99	0.05	2530	180.0	1170	5750	3690	8.2	0.5	4.7	19.3	12.6
Ave	23.4	3.70	0.13	2790	167.0	1240	5780	3490	9.6	0.5	5.2	20.7	12.7

MAF = Moisture, Ash Free
Ave = Arithmetic Average Value

TABLE 6

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 2.013 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
1440	S	0.0780	6	0.7	10	90	74
	LR	55.0	220	4.5	60	224	33
	RR	4.0	25	4.55	1.5	14	0.5
180	S	0.1069	40	1.5	25	126	84
	LR	55.0	140	3.6	30	126	10.9
	RR	4.5	15	1.4	2.5	12	0.5
150	S	0.1007	41.5	2.0	29	137	82
	LR	53.7	140	3.4	28	113	9.3
	RR	4.0	12.5	0.55	2.5	15	0.65
120	S	0.0923	41	1.1	24	120	88
	LR	57.0	110	2.4	26	103	6.9
	RR	4.5	15	0.65	3.5	14.5	1.0
80	S	0.0975	41	1.8	26	125	100
	LR	54.0	90	2.6	24	97	5.2
	RR	4.2	12	0.4	3.6	15	0.5
40	S	0.0932	42	4.0	25	114	76
	LR	57.0	100	3.50	16	68.4	2.2
	RR	5.6	30	0.75	4.5	21.4	1.0
30	S	0.0962	53	4.2	26	122	83
	LR	58.0	68.5	1.2	13	56	7.8
	RR	5.0	30	0.95	4.0	19.5	2.9
20	S	0.0921	55	1.9	22.0	128	69
	LR	57.5	58	1.1	9.5	41	5.9
	RR	5.0	35	0.55	4.5	11.8	4.9
10	S	0.0999	65	4.2	42	177	77
	LR	57.0	50	0.85	2	9	2
	RR	5.5	45	0.9	6	25.5	2.5

TABLE 6 continued

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
7	S	0.0851	59	2.4	31	136	79
	LR	57.0	44	0.9	6	34.5	3
	RR	5.0	45	0.6	6	25.5	5
5	S	0.0943	60	2.8	30	146	71
	LR	58.2	40	0.9	1.5	7	1.5
	RR	5.0	60	1.1	9.5	36	2.5

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 7

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
	S	0.0435	2.5	0.3	3	27	57
1440	LR	55.0	220	4.8	74	350	55
	RR	5.0	30	0.7	6.5	32	5
	S	0.0804	9	1.1	10	63	85
150	LR	54.0	230	5.3	58	252	26
	RR	4.0	32.5	1.3	8	35.5	3
	S	0.0867	8	1.2	12	76	100
80	LR	57.0	200	4.2	66	262	18.7
	RR	5.0	30	1.65	11.5	48.5	3.5
	S	0.0708	13.5	1.4	9	71	77.2
40	LR	55.0	230	5.8	44.3	222	13.9
	RR	5.8	32.5	0.85	7.3	38	2
	S	0.0751	16	0.75	14	66	84
30	LR	58.0	260	5.8	36	182	5
	RR	6.0	40	0.8	8.5	48	5
	S	0.0788	22	1.6	16	91	112
20	LR	57.0	220	8.8	60	188	12
	RR	5.8	45	1.85	20	36.5	10
	S	0.0841	29	1.5	18	92	102
10	LR	59.0	160	8.6	28	148	4
	RR	5.9	65	2.8	15	66.5	10
	S	0.0887	36.6	2.1	34.6	122	85.3
5	LR	59.0	160	4.15	23.6	136	4.8
	RR	5.7	95	1.8	21	95	0

TABLE 7 continued

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
2	S	0.0927	41.9	2.8	42.0	132	99.1
	LR	59.0	120	3.6	17.2	96.3	2.8
	RR	6.0	145	4.6	14.5	61.5	9

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 8

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.178 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
	S	0.0650	2.6	0.4	2	40	91.6
40	LR	55.5	250	4.7	77.6	297	26.6
	RR	5.9	50	0.5	10.9	54	18.7
	S	0.0625	1.7	1.2	9	54	101
30	LR	58.0	240	4.4	80	388	18.8
	RR	6.6	65	1.25	9	63	0.5
	S	0.0727	1.6	0.9	8	49	83.4
20	LR	57.5	270	4.6	72	346	16.6
	RR	5.9	45	1.1	12.5	62.5	0.5
	S	0.0707	5	1.2	9	49	88
10	LR	56.9	270	4.7	70	288	17.8
	RR	5.9	65	1.3	22	96.5	12.5
	S	0.0778	8	2.3	12	61	84
7	LR	56.7	250	6.5	56	272	11.1
	RR	5.3	75	5.3	25	118	1.0
	S	0.0793	8	4.2	11	61	86
5	LR	57.9	260	4.8	58	250	8.8
	RR	6.0	75	1.8	30	125	0

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 9

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.082 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
	S	0.0510	4.5	0.8	6	25	68
40	LR	56.0	280	4.9	72	344	28
	RR	5.2	77.5	1.4	17.8	85	20
	S	0.0700	5	1.0	9	41	91.5
30	LR	52.0	270	5.2	79	326	31
	RR	6.0	105	1.7	30	128	10
	S	0.0718	7	1.1	6	36.5	86
20	LR	58.9	280	4.9	72	306	11
	RR	5.4	95	1.5	27.5	120	10
	S	0.0645	7	0.6	6	39	79
10	LR	52.0	240	4.0	50	324	13
	RR	6.0	155	2.4	47.5	200	13.5
	S	0.0637	5	0.6	5	33	76
5	LR	53.5	280	4.9	80	344	21.6
	RR	5.5	107.5	2.25	35	172.5	21.6
	S	0.0735	7	0.56	6.5	49	87
2	LR	54.5	230	4.9	55	252	9.6
	RR	6.0	90	1.6	40	188	16

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 10

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1565 N
Mean Particle Radius = 2.013 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
	S	0.0852	6	0.5	9	93	86
1440	LR	54.0	230	4.7	72	238	40
	RR	5.0	25	3.0	3.5	21	1.5
	S	0.1017	40	2.3	29	142	90
150	LR	54.0	130	2.9	28	132	10.2
	RR	6.0	5	0.55	3	14.5	1.5
	S	0.1037	39.5	2.9	24	121	79
120	LR	55.0	120	3.55	26	106	9.8
	RR	4.0	12.5	3.3	2.5	10	0.5
	S	0.1095	35.5	2.9	20	99	57
80	LR	55.0	100	3.3	24	90	8
	RR	4.5	12.5	2.35	2	14	0.5
	S	0.0998	53	3.2	28	129	81
40	LR	56.0	95	2.25	16.5	81	6.7
	RR	4.1	10	2.25	2.5	10.5	0.5
	S	0.1008	54	3.2	29	137	71
30	LR	56.7	75	2.2	14	69	4.8
	RR	4.5	25	3.25	3.5	17.5	1.5
	S	0.1088	60.5	1.5	26.5	141	108
20	LR	55.0	58	1.3	11	60	3.6
	RR	4.0	20	0.9	2.5	15	1.3
	S	0.1021	42	2.3	20	109	63
10	LR	57.0	43	1.2	7.5	40	2.4
	RR	4.0	30	1.5	5.5	25	0.5

TABLE 10 continued

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
5	S	0.1016	56	3.4	25	136	97
	LR	57.0	32	0.8	6	32	1.6
	RR	4.3	30	1.35	5.8	22.5	0.5
2	S	0.0937	51	1.7	21	114	75.5
	LR	57.0	28	0.9	4.5	25	2.6
	RR	4.0	35	1.85	6.5	31	1.3

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 11

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1565 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
	S	0.0697	3.5	0.7	8	51	78
240	LR	55.0	230	5.8	60	282	39
	RR	4.4	35	1.5	7	34.5	5.5
	S	0.0755	8.5	1.7	8	60	75
120	LR	53.0	220	4.9	60	264	31
	RR	3.5	25	2.15	6	25	2.5
	S	0.0788	10	0.5	9.5	63	77
80	LR	54.0	210	4.7	54	230	27
	RR	4.0	25	1.1	5.5	28	2.5
	S	0.0852	11.5	0.9	11	69	82
60	LR	56.0	210	4.9	50	226	24
	RR	4.0	40	2.55	11.5	47	5
	S	0.0972	17	1.4	16.5	91	91
40	LR	56.5	235	4.8	48	206	20
	RR	4.0	30	3.2	7	20.5	3
	S	0.0870	18	0.8	16	81	84
30	LR	55.0	190	4.7	50	196	14.7
	RR	4.5	25	0.8	7.5	35	0.5
	S	0.1014	24	1.1	19	97	82
20	LR	56.5	180	4.6	36	154	13
	RR	4.5	45	1.65	13	53	3.5
	S	0.0968	32	3.9	21	108	84
10	LR	57.0	140	3.0	30	126	8.1
	R	4.5	50	1.7	13.5	59	5

TABLE 11 continued

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
5	S	0.0942	35	2.9	24	112	81
	LR	58.0	130	2.8	26	122	7
	RR	4.3	65	1.8	17	73.5	5.5
2	S	0.1018	46	3.2	25	121	90
	LR	56.0	95	2.2	17	80	4.1
	RR	4.5	90	1.8	19.5	83	6.0

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 12

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1565 N
Mean Particle Radius = 0.178 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
	S	0.0616	4	0.5	6.5	40	82
40	LR	55.0	220	4.95	72	338	33
	RR	4.5	45	1.7	12	60	10
	S	0.0684	8	0.9	9	62	87
30	LR	56.0	230	4.55	66	290	25
	RR	4.0	380	1.05	9.5	44.5	5
	S	0.0800	6	0.6	8	54	91
20	LR	57.0	220	4.4	62	210	22
	RR	4.0	325	0.8	10	43	4.5
	S	0.0774	5	0.5	7	44	76
10	LR	57.0	195	4.0	64	280	16.1
	RR	4.0	45	2.8	18.5	78.5	9.5
	S	0.0859	15	2.2	13	73	82
5	LR	56.0	155	3.5	48	204	10.1
	RR	5.0	70	2.15	27	123.5	11.5
	S	0.1000	26	2.1	17	97	91
2	LR	57.5	150	3.1	36	153	7.3
	RR	4.0	55	1.5	21.5	75	4.5

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 13

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 2.013 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
1440	S	0.1015	5	0.5	10	104	92
	LR	54.5	220	5.9	62	204	41
	RR	4.7	10	1.55	3	14	1
180	S	0.1000	34	1.4	22	117	69
	LR	55.0	140	3.5	30	126	12.9
	RR	4.5	20	1.9	4	17	1.5
40	S	0.1022	34	2.2	23	93	77
	LR	57.0	75	2.6	18	84	4.9
	RR	4.5	20	1.05	3.5	16.5	1.5
30	S	0.1017	40	2.6	22	111	86
	LR	56.0	71	1.5	14	71	4.3
	RR	4.0	30	1.75	5.5	26	1.5
20	S	0.1063	44	2.0	26	106	66
	LR	56.2	63	1.7	11	59	3.7
	RR	4.2	40	1.85	5.5	24.5	0.5
10	S	0.1026	69	2.7	29.5	143	99
	LR	57.5	50	1.4	9	47	2.3
	RR	4.0	35	1.6	5	24	0.2
5	S	0.1000	70	1.8	30	145	102
	LR	57.0	36	1.2	12	61	1.6
	RR	4.0	35	1.6	4	21.5	0.2
2	S	0.1000	74	2.5	30	149	90
	LR	58.0	28	0.7	6	27	1.1
	RR	4.5	35	1.55	5	24.5	0.1

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[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 14

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
240	S	0.0642	5	0.5	5	53	70
	LR	52.5	240	5.1	70	304	47
	RR	4.3	20	0.85	7.5	38.5	4
80	S	0.0818	8	0.6	10	65	80
	LR	53.8	220	5.5	62	249	31
	RR	4.1	25	0.8	6	28.5	4.5
40	S	0.0816	14	1.3	15	82	80
	LR	55.5	210	4.7	54	221	22
	RR	4.5	35	1.0	9	42.5	4.5
30	S	0.0890	19	0.8	16	84	100
	LR	56.5	169	4.1	44	196	16.5
	RR	4.0	30	1.25	9	38.6	4
20	S	0.0971	27	0.9	18	89	100
	LR	56.5	156	3.8	40	182	13.5
	RR	4.5	35	1.55	9.5	36.5	1.0
10	S	0.0931	35	1.7	18	90	90
	LR	56.0	150	3.5	32	145	9.6
	RR	4.0	50	1.2	13.5	56.5	4
5	S	0.1039	44	1.6	20	102	90
	LR	57.0	120	2.8	26	116	6.3
	RR	4.0	55	1.25	15.5	63	6.5
2	S	0.0972	44	2.6	21	106	80
	LR	57.0	84	2.1	18	87	6.5
	RR	4.0	105	1.85	11.5	90.5	4

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 15

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 0.178 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
	S	0.0696	6	0.5	5	34	80
60	LR	55.7	230	4.9	72	338	42
	RR	4.6	30	1.25	9	43.5	6.5
	S	0.0706	6	0.6	8	44	80
40	LR	53.0	230	6.8	70	318	32
	RR	5.0	45	1.1	14	65.5	8.5
	S	0.0700	6	1.0	8	45	80
30	LR	56.0	230	5.3	58	262	30
	RR	3.5	15	2.7	4	21	3.5
	S	0.0801	6	1.2	8	48	100
20	LR	55.0	230	5.7	68	292	26
	RR	3.5	15	1.3	5.5	24.5	3.5
	S	0.0691	7	0.6	7	46	85
10	LR	56.0	270	5.3	68	306	27
	RR	4.2	40	1.55	18	84	12.2
	S	0.0820	9	0.7	9	59	96
5	LR	56.0	184	4.2	48	218	16
	RR	4.4	95	1.95	42	202	14.5
	S	0.0910	16	1.4	12	76	104
2	LR	56.0	162	3.7	38	176	9.6
	RR	4.8	110	2.15	80	247.5	15.5

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 16

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 5/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
1440	S	0.0678	9	0.6	5	43	78
	LR	39.8	520	17.4	160	705	96
	RR	4.4	95	9.55	26	137	13.5
240	S	0.0685	8	0.9	7	53	86
	LR	44.0	450	94	108	516	60
	RR	4.0	40	1.05	4.5	22.5	0.5
60	S	0.0849	15	1.4	14	80	88
	LR	44.0	420	8.5	100	472	30
	RR	4.0	50	2.05	12.5	59.5	1.5
30	S	0.0998	22	1.3	18	101	106
	LR	46.0	360	7.5	76	352	23
	RR	4.0	40	1.15	8	39.5	1
20	S	0.1048	27	2.5	21	115	101
	LR	44.8	360	7.6	85	380	20
	RR	4.2	65	1.7	16	70.5	4.5
10	S	0.1023	38	3.3	26	127	99
	LR	47.0	280	5.5	64	268	13
	RR	4.5	65	2.05	16.5	60	0
5	S	0.0706	31	1.7	18	90	68
	LR	48.0	230	4.7	32	160	10
	RR	4.0	35	1.15	8	33.5	0
2	S	0.1013	49	3.9	26	131	92
	LR	48.0	150	4.1	28	128	5
	RR	4.0	55	1.4	13	44	0

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[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 17

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 2/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
1440	S	0.1084	34	3.0	19	139	112
	LR	28.0	1000	17.4	250	600	22
	RR	4.0	230	16.5	46	226	4.5
60	S	0.1024	32	2.5	22	127	105
	LR	32.5	840	15.8	200	790	31
	RR	4.75	160	3.1	27	119.5	2.5
30	S	0.1056	35	1.8	22	127	109
	LR	35.0	720	23.6	160	700	31
	RR	4.0	90	3.2	20	76.5	2.5
20	S	0.1080	39	2.2	24	131	108
	LR	36.0	700	16.2	140	920	30
	RR	4.5	70	2.15	16	75.5	2.5
10	S	0.1116	45	2.9	24	129	101
	LR	33.0	700	11.8	130	540	22
	RR	4.0	185	3.15	40	138	5.5
5	S	0.1091	48	3.5	26	134	98
	LR	33.9	580	10.2	80	410	14
	RR	3.8	175	2.8	35	117	4
2	S	0.1046	55	3.5	27	140	96
	LR	33.8	440	8.4	60	320	12.8
	RR	3.6	190	3.3	40	135	3

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 18

RAW DATA FOR CATION REMOVED

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 5/1

Residence Time, min		Amount Used[1], g,ml,ml	Ion Concentration in Sample, ppm				
			Na	K	Mg	Ca	Al
1440	S	0.0583	8	0.6	5	46	68
	LR	41.0	500	13.5	160	640	142
	RR	4.2	85	10.5	27	178	2.4
60	S	0.0892	11	1.1	12	75	95
	LR	44.7	440	9.2	115	490	44
	RR	4.1	110	9.3	31	126	8
30	S	0.0969	20	1.4	16	93	99
	LR	44.5	380	7.7	110	405	34
	RR	4.0	155	2.6	17	68	8
20	S	0.0979	24	1.5	16	94	101
	LR	45.4	340	7.0	95	360	28
	RR	4.0	135	2.5	26	100	14
10	S	0.1086	35	1.9	22	121	105
	LR	46.0	270	6.6	75	285	18
	RR	4.0	90	1.9	28	106	11.5
5	S	0.0994	39	2.7	22	115	96
	LR	45.5	220	5.1	60	220	13
	RR	4.2	90	1.9	37	141	7
2	S	0.1078	53	3.3	26	137	97
	LR	46.0	170	4.1	45	170	9
	RR	4.0	65	1.95	37	130	6.5

[1] S = Processed Coal in grams
LR = Liquid Recovery in ml
RR = Rinse Recovery in ml

TABLE 19

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 2.013 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			μg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
1440	29.9	2.23	175	19.6	292	2630	2160	1.0	0.1	2.1	16.1	13.5
180	29.5	3.17	1220	45.9	766	3860	2570	5.0	0.2	3.9	16.5	11.2
150	28.4	3.00	1270	61.4	891	4210	2520	5.5	0.2	4.8	19.1	11.7
120	29.3	3.25	1490	40.0	873	4370	3200	6.0	0.1	4.3	18.2	13.6
80	29.4	3.49	1520	68.2	964	4640	3710	5.7	0.2	4.4	18.0	14.7
40	29.1	3.55	1660	158.0	987	4500	3000	6.1	0.5	4.5	17.1	11.6
30	28.4	3.32	1890	150.0	928	4360	2960	7.4	0.5	4.5	17.8	12.3
20	32.2	3.05	1880	64.9	751	4370	2360	8.1	0.3	4.0	19.4	10.7
10	29.1	3.29	2180	141.0	1410	5930	905	8.8	0.5	7.0	24.8	3.9
7	29.5	3.24	2320	94.4	1220	5350	3110	9.4	0.3	6.1	22.4	13.3
5	29.5	3.21	2110	98.5	1060	5130	2500	8.6	0.4	5.3	21.7	10.8

MAF = Moisture, Ash Free

TABLE 20

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			µg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
1440	32.7	1.40	82	9.5	98	881	1860	0.8	0.1	1.1	8.7	18.7
150	30.7	2.26	259	32.8	288	1810	2440	1.5	0.2	2.1	11.0	15.1
80	34.0	2.39	226	35.0	339	2150	2820	1.3	0.2	2.3	12.3	16.5
40	33.7	2.68	525	55.2	350	2760	3000	2.6	0.2	2.1	14.0	15.6
30	31.0	2.68	559	26.2	489	2310	2940	2.7	0.1	3.0	11.7	15.2
20	31.9	2.85	819	60.7	596	3390	4540	3.8	0.2	3.4	16.2	22.1
10	31.6	2.81	997	51.2	619	3160	3510	4.7	0.2	3.6	15.3	17.3
5	31.0	3.07	1310	74.6	1240	4540	3050	5.6	0.3	6.5	20.1	13.8
2	31.2	3.11	1450	95.9	1450	4570	3430	6.1	0.4	7.5	19.9	15.3

MAF = Moisture, Ash Free

TABLE 21

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.178 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			μg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
40	36.7	2.22	62	42.9	327	1960	3670	0.4	0.2	2.4	12.1	23.1
30	35.6	2.20	90	14.2	69	1380	3160	0.5	0.1	0.5	8.6	20.1
20	37.6	2.45	145	17.1	193	1470	3040	0.8	0.1	1.3	8.2	17.3
10	35.7	2.47	179	44.1	322	1760	3150	1.0	0.2	2.1	9.7	17.8
7	35.5	2.48	261	75.2	392	1990	2750	1.4	0.4	2.6	11.0	15.5
5	35.8	2.71	281	148.0	386	2140	3020	1.4	0.6	2.3	10.8	15.5

MAF = Moisture, Ash Free

TABLE 22

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.082 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			µg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
40	44.1	2.12	191	34.0	255	1060	2890	1.2	0.2	2.0	6.9	19.1
30	36.0	2.13	155	31.1	280	1270	2840	1.0	0.2	2.1	8.2	18.7
20	43.0	2.35	235	37.2	201	1220	2880	1.3	0.2	1.4	7.1	17.1
10	44.3	2.49	277	24.9	238	1540	3130	1.5	0.1	1.6	8.4	17.5
5	47.6	2.35	189	22.7	189	1250	2870	1.1	0.1	1.3	7.3	17.1
2	43.9	2.89	283	22.7	263	1980	3520	1.3	0.1	1.5	9.3	16.9

MAF = Moisture, Ash Free

TABLE 23

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1565 N
Mean Particle Radius = 2.013 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			μg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
1440	28.2	2.31	167	13.3	250	2580	2390	1.0	0.1	1.8	15.3	14.5
150	28.5	2.98	1210	69.5	876	4290	2720	5.3	0.3	4.7	19.6	12.7
120	29.0	3.20	1260	92.4	765	3160	2520	5.2	0.3	3.8	13.4	10.9
80	29.4	2.97	1420	116.0	800	3960	2280	4.4	0.3	3.0	12.7	7.4
40	27.9	3.19	1750	106.0	924	4260	2670	7.2	0.4	4.7	18.1	11.6
30	30.6	3.21	1780	105.0	954	4510	2340	7.3	0.4	4.8	19.0	10.1
20	28.9	3.90	2260	56.0	988	5260	4028	7.5	0.2	3.5	18.1	14.2
10	28.5	4.20	1800	98.8	859	4680	2700	5.5	0.3	3.3	14.9	8.8
5	29.9	3.67	2100	127.0	937	5100	3640	7.4	0.4	4.1	18.7	13.7
2	29.0	3.90	2220	74.1	915	4970	3290	7.4	0.2	3.7	17.1	11.6

MAF = Moisture, Ash Free

TABLE 24

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1565 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			µg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
120	33.7	2.29	264	52.8	248	1860	2330	1.5	0.3	1.8	11.1	14.2
80	32.4	2.36	307	14.7	291	1930	2360	1.7	0.1	2.0	11.2	14.0
60	32.5	2.68	372	28.1	356	2230	2650	1.8	0.1	2.2	11.3	13.8
40	30.6	2.63	472	38.1	459	2530	2530	2.4	0.2	2.8	13.1	13.4
30	31.9	2.76	587	26.8	522	2640	2740	2.8	0.1	3.1	13.1	13.9
20	32.5	2.78	677	31.9	536	2740	2310	3.2	0.1	3.1	13.4	11.6
10	33.1	3.03	1030	126.0	678	3490	2710	4.5	0.5	3.6	15.6	12.4
5	32.9	3.16	1210	100.0	831	3880	2810	5.0	0.4	4.2	16.6	12.3
2	31.8	3.21	1500	104.0	814	3940	2930	6.1	0.4	4.1	16.6	12.6

MAF = Moisture, Ash Free

TABLE 25

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1565 N
Mean Particle Radius = 0.178 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			µg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
40	34.2	2.01	133	15.3	216	1330	2730	0.9	0.1	1.8	9.1	19.0
30	33.6	2.06	246	35.3	277	1910	2680	1.6	0.2	2.2	12.7	18.2
20	32.9	2.44	188	19.1	250	1690	2840	1.0	0.1	1.7	9.5	16.2
10	34.3	2.45	162	17.2	227	1430	2470	0.6	0.1	1.5	8.0	14.1
5	37.1	2.71	486	71.3	422	2370	2660	2.4	0.3	2.5	11.9	13.7
2	32.7	3.12	837	67.6	547	3120	2930	3.5	0.3	2.8	13.6	13.0

MAF = Moisture, Ash Free

TABLE 26

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 2.013 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			µg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
1440	30.1	2.92	148	14.8	296	3080	2730	0.7	0.1	1.6	14.3	13.0
180	29.9	3.16	163	44.7	718	3820	2250	0.7	0.2	3.7	16.4	9.9
40	27.6	3.63	1250	81.1	848	3430	2840	4.5	0.3	3.7	12.7	10.8
30	29.5	3.50	1430	92.7	785	3960	3070	5.3	0.3	3.6	15.3	12.1
20	28.8	3.36	1440	65.4	850	3470	2160	5.6	0.2	4.1	14.0	8.9
10	29.9	3.28	2280	89.2	975	4730	3270	9.1	0.3	4.8	19.5	13.8
5	29.6	3.27	2370	60.8	1010	4900	3450	9.5	0.2	5.0	20.3	14.6
2	29.4	3.54	2720	91.7	1100	5470	3300	10.0	0.3	5.0	20.9	12.9

MAF = Moisture, Ash Free

TABLE 27

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			µg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
240	32.5	1.88	149	14.3	149	1580	2090	1.1	0.1	1.3	11.5	15.6
80	33.8	2.26	226	17.8	283	1840	2260	1.3	0.1	2.0	11.1	14.0
40	34.6	2.42	425	38.3	456	2490	2430	2.3	0.2	3.0	14.1	14.0
30	31.7	2.58	565	23.8	476	2500	2980	2.9	0.1	3.0	13.2	16.1
20	32.1	2.77	792	26.4	528	2610	2930	3.8	0.1	3.1	12.8	14.7
10	33.0	3.03	1170	57.1	604	3020	3020	5.1	0.2	3.2	13.5	13.8
5	31.5	3.37	1480	53.7	671	3420	3020	5.7	0.2	3.2	13.7	12.4
2	33.2	3.45	1620	95.6	772	3900	2940	6.1	0.3	3.6	15.3	11.8

MAF = Moisture, Ash Free

TABLE 28

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 0.178 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			μg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
60	37.5	1.79	157	14.1	131	890	2090	1.2	0.1	1.2	6.8	16.4
40	35.0	2.29	199	19.6	266	1460	2660	1.2	0.1	1.9	8.7	16.2
30	35.1	2.44	214	35.0	286	1610	2860	1.2	0.2	1.9	9.0	16.4
20	32.6	2.40	184	36.2	246	1470	3070	1.0	0.2	1.7	8.4	17.9
10	39.2	2.52	262	23.6	262	1720	3180	1.4	0.1	1.7	9.3	17.6
5	37.0	2.71	306	24.5	306	2000	3260	1.5	0.1	1.8	10.1	16.7
2	37.8	3.07	557	47.3	418	2650	3620	2.4	0.2	2.2	11.7	16.3

MAF = Moisture, Ash Free

TABLE 29

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 5/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			μg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
1440	34.7	1.81	245	16.3	136	1170	2120	1.8	0.1	1.2	8.9	16.4
240	29.8	1.96	233	25.4	204	1550	2510	1.6	0.2	1.7	10.9	18.0
60	32.7	2.49	451	41.5	421	2410	2650	2.4	0.2	2.7	13.2	14.8
30	31.6	2.81	637	36.8	521	2930	3070	3.0	0.2	3.0	14.2	15.2
20	33.1	2.79	739	68.5	575	3150	2770	3.5	0.3	3.3	15.4	13.8
10	31.6	3.03	1160	101.0	794	3880	3020	5.0	0.4	4.2	17.4	13.8
5	30.8	3.21	1460	79.9	846	4230	3190	5.9	0.3	4.2	17.9	13.8
2	30.2	3.41	1710	136.0	906	4570	3210	6.5	0.5	4.3	18.1	13.0

MAF = Moisture, Ash Free

TABLE 30

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 2/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			µg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
1440	34.9	3.00	970	85.6	542	3970	3200	4.2	0.3	2.9	18.0	14.8
60	33.0	2.82	907	70.8	623	3600	2980	4.2	0.3	3.6	17.4	14.7
30	32.9	2.78	948	48.7	596	3440	2950	4.5	0.2	3.5	16.8	14.8
20	32.4	2.83	1050	59.3	647	3530	2910	4.9	0.2	3.7	17.0	14.3
10	34.7	3.06	1270	82.0	679	3650	2860	5.4	0.3	3.6	16.2	13.0
5	33.5	3.12	1420	103.0	767	3960	2890	6.0	0.4	4.0	17.2	12.8
2	34.0	3.27	1780	113.0	873	4520	3100	7.1	0.4	4.3	18.7	13.1

MAF = Moisture, Ash Free

TABLE 31

ANALYSIS OF PROCESSED COAL

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 5/1

Residence Time, minute	Moisture Content, %	Ash Content, (Dry Basis) %	Ions Present									
			µg/g MAF Coal					% Oxide in Ash				
			Na	K	Mg	Ca	Al	Na_2O	K_2O	MgO	CaO	Al_2O_3
1440	33.8	1.71	239	17.9	149	1370	2030	1.9	0.1	1.4	11.0	16.7
60	34.6	2.49	315	31.5	344	2150	2720	1.7	0.1	2.2	11.8	15.2
30	33.9	2.71	575	40.2	460	2670	2850	2.8	0.2	2.7	13.4	14.6
20	33.8	2.87	724	45.3	483	2840	3050	3.3	0.2	2.7	13.5	14.8
10	33.4	2.97	986	53.6	620	3410	2960	4.4	0.2	3.4	15.6	13.8
5	34.0	3.14	1270	88.1	717	3750	3130	5.3	0.3	3.7	16.2	13.8
2	33.2	3.01	1530	95.0	749	3940	2790	6.7	0.4	4.0	17.8	12.9

MAF = Moisture, Ash Free

Appendix C

RESULTS

TABLE 32

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 2.013 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
1440	111.9	94.0	81.0	75.2	79.5	71.7	89.7	51.3	71.7	15.8
180	102.0	58.6	89.1	49.8	110.2	32.6	95.2	28.8	90.5	5.0
150	113.9	57.3	104.8	40.4	94.2	27.8	92.1	24.7	72.9	4.3
120	111.9	48.8	73.0	43.9	92.9	27.8	94.4	23.4	91.7	2.7
80	99.6	42.5	100.9	32.3	95.2	23.7	95.0	20.7	103.0	1.7
40	130.1	44.4	111.9	22.6	109.0	16.8	110.0	16.8	95.2	1.0
30	113.2	33.1	104.3	9.9	100.2	15.9	102.3	14.9	95.8	3.5
20	107.1	29.8	50.1	18.8	79.9	14.7	98.4	11.2	76.1	3.4
10	122.2	24.1	84.6	7.8	132.2	2.3	124.7	2.4	29.6	3.1
7	117.9	22.5	55.2	12.4	111.3	7.0	111.3	8.8	92.5	1.5
5	108.2	22.1	69.6	11.4	98.4	2.8	105.3	2.5	99.1	0.9

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 33

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
1440	97.0	97.1	40.8	86.3	98.3	90.4	91.6	83.1	84.8	26.8
150	105.4	91.6	57.6	66.6	96.3	71.1	85.7	63.0	92.0	11.5
80	108.1	92.1	91.0	61.1	91.6	71.9	91.5	61.6	81.9	5.1
40	132.3	88.1	64.9	78.8	62.1	50.4	79.8	55.8	72.8	6.1
30	128.6	85.7	57.9	74.0	61.4	48.8	80.6	50.6	69.9	2.3
20	117.9	78.1	97.7	65.8	86.2	57.5	97.0	42.4	96.5	3.9
10	102.0	69.2	93.9	70.0	62.4	39.1	86.4	39.7	79.4	1.9
5	113.0	63.5	73.1	43.8	96.7	21.4	99.1	27.5	69.5	2.5
2	103.0	55.6	82.8	36.4	104.2	14.3	94.5	20.3	77.5	1.6

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 34

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.178 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
40	112.9	97.3	40.7	81.3	73.5	93.5	82.6	73.5	92.2	10.2
30	113.2	98.3	53.4	60.3	94.7	78.0	111.4	74.3	94.9	6.9
20	109.9	94.3	82.8	76.1	80.6	79.2	91.3	69.8	103.9	7.8
10	127.2	95.2	49.2	58.7	95.4	74.4	85.6	68.8	84.2	7.3
7	121.0	92.6	76.8	54.2	87.3	65.5	86.5	64.4	72.6	4.9
5	128.8	92.6	98.6	30.8	90.5	67.5	85.6	61.8	78.0	3.7

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 35

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.082 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
40	154.0	94.9	61.9	64.8	109.8	78.3	111.3	80.5	112.7	11.4
30	139.7	95.5	59.7	66.9	114.2	77.4	106.4	75.7	101.9	11.6
20	164.1	94.2	65.9	64.1	109.0	83.0	109.7	77.4	96.1	5.2
10	133.2	91.6	47.5	66.6	81.3	73.1	113.4	72.4	104.4	5.1
5	149.2	94.9	53.5	73.0	109.6	84.1	113.3	77.6	100.0	9.0
2	130.3	91.2	53.8	73.2	90.9	73.4	107.7	62.7	115.7	3.8

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 36

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1565 N
Mean Particle Radius = 2.013 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
1440	114.6	94.4	75.3	81.8	86.6	77.7	91.1	52.9	80.3	16.9
150	106.7	57.1	106.7	34.4	92.7	28.5	96.8	27.7	78.1	4.5
120	105.0	54.5	141.2	33.9	82.9	29.9	85.0	25.7	72.7	4.7
80	101.8	47.1	161.2	27.3	83.6	27.4	83.4	22.3	65.5	4.2
40	112.6	41.1	137.6	22.5	86.2	18.7	86.7	19.6	75.9	3.1
30	104.9	35.9	138.0	23.2	86.2	16.2	88.4	16.8	65.8	2.6
20	113.5	24.8	73.5	23.4	85.3	12.4	98.2	12.7	111.6	1.1
10	90.2	24.6	116.1	14.6	72.6	10.7	85.0	10.4	74.9	1.2
5	95.6	17.1	140.2	8.7	77.1	8.2	90.1	7.9	100.1	0.6
2	99.3	15.1	87.7	6.6	74.3	6.6	87.0	6.6	91.5	1.0

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 37

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1565 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
240	111.6	96.4	54.4	76.9	86.1	75.1	94.5	69.0	79.2	16.8
120	99.4	90.9	66.2	53.1	93.3	74.3	87.8	62.9	90.5	14.3
80	98.6	89.3	42.6	79.7	91.8	69.2	83.1	59.2	89.9	12.2
60	104.5	87.8	54.0	69.4	96.0	64.2	89.4	56.3	98.5	10.3
40	119.3	86.4	59.8	62.5	103.7	57.2	90.3	50.9	92.9	9.2
30	102.8	80.4	50.2	68.7	110.8	54.4	57.0	74.9	97.5	6.2
20	103.0	77.4	53.9	65.2	97.1	46.6	83.0	42.2	82.7	6.8
10	98.6	64.0	97.5	24.1	103.8	36.9	90.2	32.4	93.9	3.8
5	108.7	55.3	91.2	24.5	127.9	26.7	108.2	26.8	112.3	2.9
2	95.5	46.1	78.5	22.0	101.2	22.2	88.1	21.6	99.6	1.9

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 38

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1565 N
Mean Particle Radius = 0.178 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
40	104.0	95.4	40.2	80.5	99.5	80.7	101.1	76.2	87.8	13.3
30	113.9	92.3	43.6	68.6	99.2	75.3	101.4	66.1	83.4	10.7
20	108.9	93.8	39.1	75.1	93.6	76.3	80.0	61.9	87.2	9.2
10	96.9	94.0	39.0	77.5	94.5	78.7	92.2	72.0	74.6	8.1
5	90.2	80.7	60.4	39.6	93.8	60.2	91.9	53.5	78.0	5.0
2	101.2	70.5	55.8	38.1	91.6	47.1	93.6	39.7	84.3	3.3

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 39

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 2.013 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
1440	104.0	94.9	60.8	83.5	94.6	72.3	100.9	45.3	95.3	15.8
180	105.5	60.5	62.6	49.6	99.9	33.7	99.9	28.6	74.0	6.5
40	80.8	44.3	78.4	30.1	95.9	21.9	81.1	24.5	85.4	2.2
30	85.1	39.5	77.1	18.2	85.9	18.9	87.8	19.0	92.3	1.8
20	83.0	36.6	60.7	25.8	89.4	14.5	77.7	18.1	66.0	2.1
10	106.0	23.1	73.9	18.2	97.1	11.2	96.1	11.9	97.3	0.9
5	103.0	17.4	52.5	21.5	103.8	13.6	102.3	14.2	102.1	0.6
2	112.3	13.0	69.6	10.7	104.9	7.1	104.8	6.5	97.7	0.4

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 40

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
240	112.2	95.0	43.1	81.0	84.9	84.8	96.5	69.6	72.2	21.1
80	108.8	92.2	48.6	79.1	89.6	72.8	90.3	62.4	71.9	14.4
40	115.2	96.2	56.0	60.9	98.3	60.0	98.1	52.9	73.9	10.3
30	102.3	79.4	44.0	69.1	90.0	54.4	93.0	50.3	86.9	6.7
20	104.9	71.8	43.6	65.4	90.2	49.6	91.8	47.4	84.6	5.6
10	116.1	62.3	58.4	44.2	87.9	40.8	90.6	38.4	85.7	4.0
5	114.4	51.8	51.8	40.8	87.9	34.1	91.9	31.1	84.6	2.8
2	104.1	42.0	70.9	23.1	87.3	23.8	94.2	23.5	82.4	2.9

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 41

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 0.178 mm
Liquid/Solid Mass Ratio = 10/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
60	106.6	94.9	48.2	81.6	89.1	87.4	95.0	82.8	67.1	20.9
40	103.9	93.4	63.9	80.9	95.3	76.3	97.7	72.7	77.5	12.9
30	108.5	93.2	65.3	66.5	87.1	72.1	90.4	67.5	82.4	11.8
20	105.6	94.0	67.6	66.5	93.2	77.6	93.7	71.2	86.4	9.6
10	128.5	92.9	57.6	74.3	97.1	76.9	103.9	69.6	90.2	10.0
5	94.3	88.8	49.7	69.2	81.3	68.0	90.8	59.6	88.5	6.2
2	94.3	79.6	60.4	50.9	83.9	57.6	94.1	48.5	95.7	3.7

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 42

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 5/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
1440	111.2	92.1	70.0	85.9	86.4	86.6	87.9	76.9	81.3	19.9
240	105.3	92.1	49.6	69.0	72.5	76.0	80.7	66.6	88.1	12.5
60	106.8	84.9	56.4	55.6	87.2	58.9	91.3	54.2	86.7	6.4
30	104.0	78.1	50.9	56.3	85.1	47.9	89.4	43.1	98.8	4.5
20	106.2	75.0	69.9	40.6	93.9	47.7	95.8	42.6	89.1	4.3
10	106.8	61.1	82.8	26.5	103.1	34.5	97.7	31.1	95.3	2.7
5	106.3	51.0	67.1	28.1	90.0	20.1	91.9	20.1	100.0	2.0
2	97.1	37.1	98.6	16.8	93.2	17.3	94.2	15.8	99.4	1.0

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 43

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1070 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 2/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
1440	105.0	66.9	74.5	30.4	87.8	47.3	89.9	23.2	99.6	1.3
60	100.5	67.7	64.4	33.6	91.4	41.9	93.5	33.1	93.4	2.3
30	95.9	64.7	63.8	53.9	83.6	39.3	89.0	32.9	92.9	2.5
20	99.5	65.2	60.1	40.4	84.7	34.9	100.8	39.1	91.7	2.5
10	103.4	56.0	66.0	25.0	83.6	30.8	85.1	25.5	89.3	1.7
5	100.0	49.3	77.1	19.0	81.9	20.1	85.7	19.7	89.9	1.1
2	101.3	37.3	80.4	15.1	86.9	14.5	92.0	14.5	96.2	1.0

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

TABLE 44

NORMALIZED CATION REMOVED AND MATERIAL BALANCE CLOSURES

H_2SO_4 Concentration = 0.1959 N
Mean Particle Radius = 0.502 mm
Liquid/Solid Mass Ratio = 5/1

Residence Time, minutes	Cation									
	Na		K		Mg		Ca		Al	
	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN	Clos	%RN
1440	109.8	92.2	59.8	81.9	89.8	85.9	87.5	72.7	86.6	28.1
60	109.0	89.6	55.9	65.8	90.2	67.4	90.4	58.5	92.2	9.0
30	105.7	80.5	53.3	54.3	96.5	59.4	89.6	48.1	93.8	6.9
20	103.5	75.0	54.2	49.6	92.1	55.4	88.8	44.5	99.2	5.6
10	97.3	63.7	57.9	44.1	93.9	43.8	91.2	35.0	94.5	3.9
5	95.9	52.6	72.8	27.0	94.4	35.3	90.2	27.7	98.7	2.6
2	93.9	41.8	73.5	78.1	89.4	18.7	88.3	22.3	87.7	2.1

Clos = Material Balance Closure
%RN = Percent Cation Removed (Normalized)

Appendix D

CALCULATIONS FOR STATISTICAL ANALYSIS

CALCULATIONS FOR THE ANOVA TABLE

Particle Radius, mm	% Na Removal, X Residence Time, minutes 40	30	20	10	5	ΣX
2.013	a 44.4	a 33.1	a 29.8	a 24.1	a 22.1	
	b 41.1	b 35.9	b 24.8	b 24.6	b 17.1	
	c 44.3	c 39.5	c 36.6	c 23.1	c 17.4	
	Σ129.8	Σ108.5	Σ 91.2	Σ 71.8	Σ 56.6	457.9
0.502	a 88.1	a 85.7	a 78.1	a 69.2	a 63.5	
	b 86.4	b 80.4	b 77.4	b 64.0	b 55.3	
	c 86.2	c 79.4	c 71.8	c 62.3	c 51.8	
	Σ260.7	Σ245.5	Σ227.3	Σ195.5	Σ170.6	1099.6
0.178	a 97.3	a 98.3	a 94.3	a 95.2	a 92.6	
	b 95.4	b 92.3	b 93.8	b 94.0	b 80.7	
	c 93.4	c 93.2	c 94.0	c 92.9	c 88.8	
	Σ286.1	Σ283.8	Σ282.1	Σ282.1	Σ262.1	1396.2
ΣX	676.6	637.8	600.6	549.4	489.3	2953.7

a = 0.1070 N H_2SO_4
b = 0.1565 N H_2SO_4
c = 0.1959 N H_2SO_4

i = columns (time)
j = rows (particle radius)
k = groups (acid concentration)

$$\Sigma\Sigma\Sigma X^2_{ijk} = 227{,}958.17$$

$$\begin{aligned}\Sigma(\text{column}^2) &= (676.6)^2 + (637.8)^2 + (600.6)^2 \\ &\quad + (549.4)^2 + (489.3)^2 \\ &= 1{,}766{,}551.6\end{aligned}$$

$$\begin{aligned}\Sigma(\text{row}^2) &= (457.9)^2 + (1099.6)^2 + (1396.2)^2 \\ &= 3{,}368{,}167\end{aligned}$$

$$\begin{aligned}\Sigma(\text{group}^2) &= (129.8)^2 + (260.7)^2 + (286.1)^2 + (108.5)^2 \\ &\quad + (245.5)^2 + (283.8)^2 + (91.2)^2 + (227.3)^2 \\ &\quad + (282.1)^2 + (71.8)^2 + (195.5)^2 + (282.1)^2 \\ &\quad + (56.6)^2 + (170.6)^2 + (262.1)^2 \\ &= 598{,}051.64\end{aligned}$$

A) Determine the Sum of Squares

$$\text{SSMean} = \frac{(\Sigma\Sigma\Sigma X_{ijk})^2}{ijk} = \frac{(2953.7)^2}{5*3*3} = 193{,}874.3$$

$$\text{SSTime} = \frac{\Sigma(\text{column}^2)}{jk} - \text{SSMean} = \frac{1{,}766{,}551.6}{3*3} - 193{,}874.3$$
$$= 2409.2$$

$$\text{SSRadius} = \frac{\Sigma(\text{row}^2)}{ik} - \text{SSMean} = \frac{3{,}368{,}167}{5*3} - 193{,}874.3$$
$$= 30{,}670.17$$

$$\text{SSConc} = \frac{\Sigma(\text{group}^2)}{ij} - \text{SSMean} = \frac{598{,}051.64}{5*3} - 193{,}874.3$$
$$= 102.0$$

$$\begin{aligned}\text{SSError} &= 227{,}958.17 - \text{SSTime} - \text{SSRadius} - \text{SSConc} - \text{SSMean}\\ &= 227{,}958.17 - 2409.2 - 30{,}670.17 - 102.0\\ &\quad - 193{,}874.3\\ &= 902.5\end{aligned}$$

B) Determine the F value for the F test

$$F_{\text{Time}} = \frac{\text{MSTime}}{\text{MSError}} = \frac{602.3}{25.07} = 24.02$$

$$F_{\text{Radius}} = \frac{\text{MSRadius}}{\text{MSError}} = \frac{15{,}335.09}{25.07} = 611.7$$

$$F_{\text{Conc}} = \frac{\text{MSConc}}{\text{MSError}} = \frac{51.0}{25.07} = 2.03$$

CALCULATIONS FOR THE "STUDENT T-TEST"

Time, min	% Na Removed, X Liquid to Solid Mass Ratio[1] A	 B	 C
60	92.0	84.9	89.6
30	85.7	78.1	80.5
20	78.1	75.0	75.0
10	69.2	61.0	63.7
5	63.5	51.0	52.6
2	55.6	37.1	41.8
ΣX	444.1	387.2	403.2
Mean, $\bar{X}$	74.0	64.5	67.2
ΣX^2	33,820.35	26,643.24	28,705.1

[1] Column headings A, B, and C represent the liquid to solid mass ratios of 10/1, 5/1, and 5/1, respectively. Headings A and B used a H_2SO_4 concentration of 0.1070 N, while heading C used a 0.1959 N H_2SO_4 concentration. A mean particle radius of 0.502 mm was used for all 3 mass ratios.

A) Determine the Variance for the 3 Mass Ratios

$$S^2_x = \frac{X^2 - (\Sigma X)^2/n}{(n-1)}$$

where n is the number of observations in the sample and S^2_x is the calculated variance of the sample.

$$S^2_A = \frac{33{,}820.35 - (444.1)^2/6}{(6-1)} = 189.9$$

$$S^2_B = \frac{26{,}643.24 - (387.2)^2/6}{(6-1)} = 333.19$$

$$S^2_C = \frac{28{,}705.1 - (403.2)^2/6}{(6-1)} = 322.01$$

B) Determine if the Variances Differ

Null Hypothesis:

$H_o: \sigma^2_A = \sigma^2_B$ $H_o: \sigma^2_A = \sigma^2_C$ $H_o: \sigma^2_B = \sigma^2_C$

Alternate Hypothesis:

$H_a: \sigma^2_A \neq \sigma^2_B$ $H_a: \sigma^2_A \neq \sigma^2_C$ $H_a: \sigma^2_B \neq \sigma^2_C$

where σ^2 is the true sample variance

If $F_{calc} > F_{5,5,0.975}$, reject H_o $F_{5,5,0.975} = 7.15$ (22)

$$F_{calc} = \frac{s^2_A}{s^2_B}$$

$$F_{AB} = \frac{331.19}{189.9} = 1.74 \qquad F_{AC} = \frac{322.01}{189.9} = 1.70 \qquad F_{BC} = \frac{331.9}{322.01} = 1.03$$

Comparing A-B: Since $F_{calc} < 7.15$, fail to reject H_o
Comparing A-C: Since $F_{calc} < 7.15$, fail to reject H_o
Comparing B-C: Since $F_{calc} < 7.15$, fail to reject H_o

The variance for the three mass ratios are not significantly different, and thus pairs of the variances can be pooled for use in the "t-test".

C) Pool the Variances

$$s^2_p = \frac{(n_A-1)s^2 + (n_B-1)s^2}{n_A + n_B - 2}$$

where s^2_p is the pooled variance.

<u>Comparing A-B</u>

$$s^2_p = \frac{(6-1)*189.9 + (6-1)*331.19}{(6+6-2)} = 260.6$$

<u>Comparing A-C</u>

$$s^2_p = \frac{(6-1)*189.9 + (6-1)*322.01}{(6+6-2)} = 255.96$$

Comparing B-C

$$s^2_p = \frac{(6-1)*331.19 + (6-1)*322.01}{(6+6-2)} = 326.6$$

D) Complete the "t-test"

Null hypothesis:

H_o: $\overline{X}_A = \overline{X}_B$ H_o: $\overline{X}_A = \overline{X}_C$ H_o: $\overline{X}_B = \overline{X}_C$

Alternate hypothesis:

H_a: $\overline{X}_A \neq \overline{X}_B$ H_a: $\overline{X}_A \neq \overline{X}_C$ H_a: $\overline{X}_B \neq \overline{X}_C$

If t_{calc} $t_{5,0.975}$, reject H_o $t_{5,0.975} = 2.571$ (22)

$$t_{calc} = \frac{X_A - X_B}{(s^2_p)^{1/2}*(1/n_A + 1/n_B)^{1/2}}$$

Comparing A-B

$$t_{calc} = \frac{74.0 - 64.5}{(260.6)^{1/2}*(1/6 + 1/6)^{1/2}} = 0.72$$

Comparing A-C

$$t_{calc} = \frac{74.0 - 67.2}{(260.6)^{1/2}*(1/6 + 1/6)^{1/2}} = 0.52$$

Comparing B-C

$$t_{calc} = \frac{67.2 - 64.5}{(322.01)^{1/2}*(1/6 + 1/6)^{1/2}} = 0.18$$

Comparing A-B: Since $t_{calc} < 2.571$, fail to reject H_o
Comparing A-C: Since $t_{calc} < 2.571$, fail to reject H_o
Comparing B-C: Since $t_{calc} < 2.571$, fail to reject H_o

Since for all combinations of mass ratios $t_{calc} < 2.571$, there is no significant difference between the 3 liquid to solid mass ratios.

Appendix E

ABBREVIATIONS USED

AA	atomic absorption spectrophotometry
ANOVA	analysis of variance
ASTM	American Society for Testing and Materials
Ave	Arithmetic Average
C_A	concentration of A
CAR	coal as received
cm	centimeter
Conc.	concentration
D	self-diffusion coefficient
D_{avg}	overall average value for the self-diffusion coefficient
DB	dry basis
D.F.	degrees of freedom
g	grams
i.e.	in other words
J_A	flux of ion exchange
M	moisture
MAF	moisture, ash free
MF	moisture free
min	minutes
ml	milliliter
mm	millimeter
MW	megawatt
N	normality
ppm	part per million
Q_{Ao}	initial amount of A in ion exchanger

$Q_{A\infty}$	amount of A in ion exchanger at equilibrium
$Q_A(t)$	amount of A in ion exchanger at time t
rpm	revolutions per minute
s^2	variance
sec	seconds
t	time of ion exchange
U(t)	fraction of attainment of equilibrium
V	volume
X	fraction of Na removed at equilibrium
SS	sum of squares
^{o}C	degrees centigrade
^{o}F	degrees fahrenheit
%	percent
μg	microgram
∂	partial derivative
Δ	change in
π	pi, with a value of approximately 3.1416
μ	true sample mean
σ	true sample variance
ε	random experimental error

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