

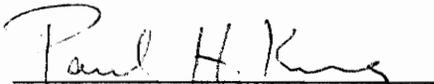
IRON RECOVERY
FROM A REPRESENTATIVE
WATER TREATMENT PLANT SLUDGE/

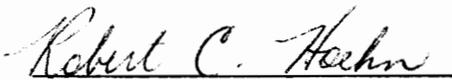
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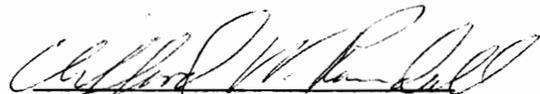
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Chapter I

INTRODUCTION

Iron salts have been used as coagulants in water treatment for nearly a century. Their initial use dates back to the patent of Hyatt in 1884, which discussed the pretreatment of influent water with ferric salts prior to filtration (1). One year later this process was first applied to municipal water treatment by the water companies of Somerville and Raritan, New Jersey, where ferric chloride was employed (2).

More widespread use of iron salts as coagulants began shortly after the turn of the century. The original impetus was an effort by the United States Steel Corporation to dispose of ferrous sulfate, a by-product of the pickling of steel by sulfuric acid. Several water purification plants in the Midwest were pioneers in the early use of "pickle liquor" (3). The combination of ferrous sulfate and lime, first used in this country in 1898 by Bull in Quincy, Illinois (4), came to be extensively applied in coagulation processes in ensuing years. Ferrous sulfate was also used with chlorine gas to form what is known today as chlorinated copperas, a mixture of ferric sulfate and ferric chloride. Subsequently ferric sulfate, an excellent coagulant, was introduced. When manufacturers began to produce this chemical more economically as a by-product, its use began to grow rapidly (5). Ferric sulfate is presently the most popular iron salt used in coagulation. While ferric chloride is recognized as an efficient coagulant in waste treatment, it is rarely employed in the treatment of potable water.

Today, except in the case of water softening, iron salts and alum are used almost exclusively as coagulants in the water utility industry. The use of iron (III) salts in water treatment has increased to about 100,000 tons per year. This level compares with an annual consumption of approximately 600,000 tons of alum (1). Many comparisons can be made between alum and ferric salts as coagulants. However, they perform in much the same manner, alum being more widely used chiefly because of cost. Both produce sludges which are characteristically resistant to drainage and concentration, although in most situations iron sludges show better dewatering properties.

In the past the normal practice has been to discharge these sludges directly into the nearest watercourse. A survey conducted in 1952 showed that 96 per cent of some 1500 water treatment plants across the country were discharging sludges into surface waters (6). This practice is still common. However, it is undergoing a very rapid change. As a result of public pressure for better quality water, the attention of the water utility industry has been increasingly focused on the economic disposal of the sludges produced by water treatment plants. Many states have established standards for the treatment of waste discharges from water purification plants. Others now consider such wastes to be in the category of industrial wastes. The guidelines division of the Environmental Protection Agency is presently developing effluent standards for the waterworks industry (7). Water treatment plant sludge disposal is a timely problem to consider, especially in light of current "zero-discharge" requirements.

Many investigators have sought an economic solution to the sludge disposal problem, and several conditioning and disposal methods have been suggested. In a report by the American Water Works Association Research Foundation (8), the following treatment and disposal methods are described for alum sludges: direct discharge to surface waters, discharge to sanitary sewers, lagooning, sand bed drying, vacuum filtration, filter pressing, centrifugation, freezing, poly-electrolyte aided thickening, land disposal, and alum recovery.

While many investigations have been directed toward the alum sludge disposal problem, fewer studies have encompassed the less common ferric sludges, or more specifically, the possibility of recovering the ferric coagulant. In recent years alum recovery has received increased attention, owing to its potential for cost savings resulting from reuse of the reclaimed coagulant. Magnesium carbonate, another reusable coagulant, has been advocated in a new system of water treatment (9). Indeed, coagulant recovery appears to be the only sludge disposal method which stands to gain an ultimate economic advantage. A recovery process offers the added benefit of conserving natural resources through coagulant reuse. Thus a closer look at the possibility of iron recovery seems well warranted.

The purposes of this investigation were to determine the extent of iron recovery achievable using a water treatment plant sludge, to characterize the properties of the remaining sludge after chemical addition and separation of the recovered iron, and to determine the effectiveness of recovered iron as compared to a fresh iron salt in coagulation.

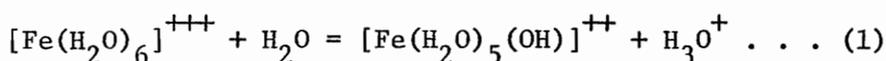
Chapter II

REVIEW OF LITERATURE

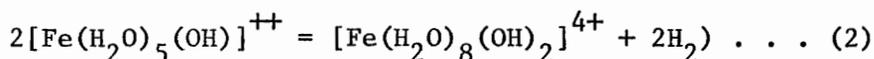
Iron in Aqueous Solutions

The hydrolysis of trivalent iron ion in aqueous solutions has been studied in detail. Most investigators agree that the effects of iron salts on coagulation are brought about by the hydrolysis products of the salts and not by the simple aquo-metal ions (10).

The hydrolytic reactions which occur take the form:



Additional ferric hydroxo complexes may be produced by similar reactions. These complexes have a pronounced tendency to polymerize, and the simplest reaction leading to a dimeric species is:



This dimer, which contains hydroxo bridges between the metal ions, may undergo further hydrolytic reactions and form higher hydroxide complexes. As the ferric iron is progressively coordinated with hydroxo groups, the repulsion between ions decreases and the tendency toward polymerization increases. Eventually, colloidal hydroxo polymers, and ultimately, insoluble hydrous ferric oxide precipitates are formed (11).

Singley and Black (1) studied the distribution of the hydrolyzed species of iron (III) in dilute solutions. They showed that the equilibrium system is dominated by the insoluble neutral form $\text{Fe}(\text{OH})_3$. But under non-equilibrium conditions existing in water treatment, they

demonstrated that several positively charged species could exist. The number of hydroxides bound to each ferric ion varied with the concentration of iron and the system pH. Figure 1 illustrates this phenomenon. The figure indicates that the iron will leach out in the form of Fe^{3+} at an iron concentration of 0.01 moles per liter when the pH of the system drops to about 2.0. When the system pH is raised to about 12.0, the soluble iron, in the form of $\text{Fe}(\text{OH})_4^-$, is present at a concentration of only 10^{-7} moles per liter. This effect seems to favor an acid method of recovery.

Matejevic and Janauer (13) reported systematic investigations on coagulation and reversal of charge of silver bromide sols by ferric nitrate as a function of pH and the age of aqueous solutions. The entire domain of stability in the plot of the log of ferric salt concentration versus pH was established. The investigation led to the formulation of the hydrolyzed species responsible for reversal of charge. For the pH range of 2 - 3.3 the species $\text{Fe}_3(\text{OH})_4^{5+}$ was suggested. The species $\text{Fe}_2(\text{OH})_2^{4+}$ probably caused charge reversal over the pH range 3.4 - 4.4, although $\text{Fe}(\text{OH})^{2+}$ was mentioned as a possibility. Temperature effects on reversal of charge were shown to be minimal.

O'melia and Stumm (14) studied the aggregation of silica dispersions by iron (III) as a function of pH, applied ferric iron concentration, and the surface area of the dispersed phase. It was postulated that sorption of a hydrolyzed iron (III) species is partially responsible for the great influence of the surface area

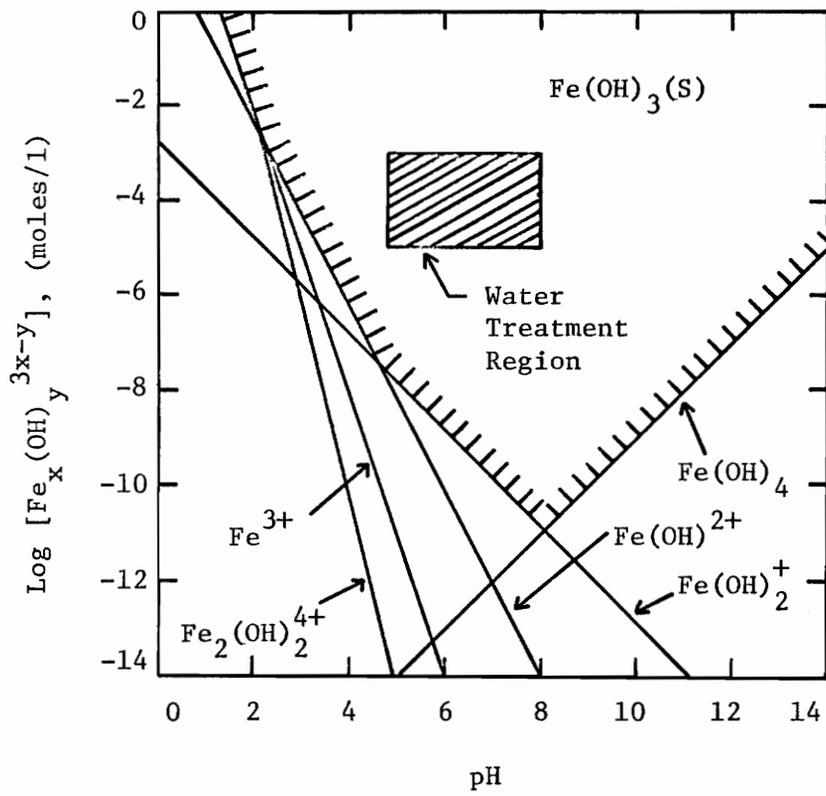


Figure 1. Solubility of Iron Hydroxide as a Function of pH

of the solid phase on coagulation and restabilization. Iron (III) equilibria were established using a 10^{-3} molar, pH adjusted solution. As shown in Figure 2, appreciable concentrations of soluble, stable hydroxo-ferric complexes are present only in the pH range 2 - 3. Consequently, adsorption of these species onto solid surfaces was expected to be considerable in this range. It is interesting to note that a breakpoint in iron (III) solubility occurs when the pH is lowered to about 2.7. This phenomenon suggests that recovery of soluble iron at low pH levels would become a process of diminishing returns.

Stumm and Morgan (15) reported alkalimetric titration curves for 10^{-3} molar iron (III) perchlorate solutions using sodium hydroxide. The presence of coordinating anions affected the shape of the curves, the extent of which was indicative of the degree of complex formation. Phosphate ions, for example, displaced the aquo groups of the ferric ions. Other coordinating anions, particularly sulfate and chloride ions, substituted for OH^- ions in the charge neutralization reaction, lowering the pH of optimum precipitation of iron oxides.

A polymeric structure for hydrous ferric oxide has been suggested on the basis of the observation that the magnetic moment of iron oxide gel is much lower than that of Fe(III) salts. Since cationic ferric-hydroxo complexes prevail in slightly acid or neutral solutions, positively charged ferric oxide colloids are formed in this pH range. Alternately, the polymerization of anionic hydroxo ferrates, $\text{Fe}(\text{OH})_4^-$, produces negatively charged colloids (11).

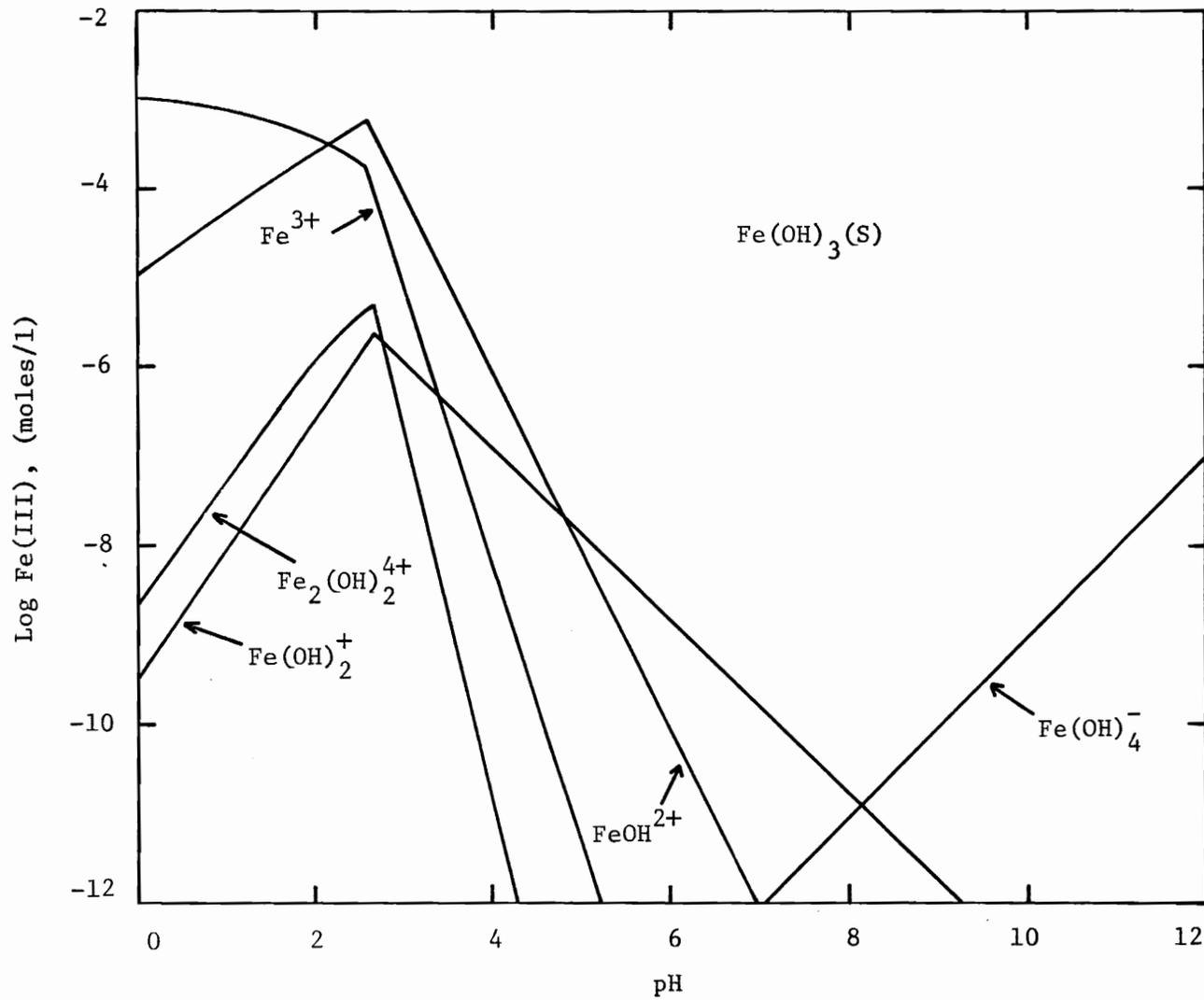


Figure 2. Iron (III) Equilibria Found by O'melia and Stumm (14)

Stumm and Morgan (15) also discussed the effects of aging ferric salt solutions. Ultraviolet absorption spectra of a dilute $\text{Fe}(\text{ClO}_4)_3$ solution at low pH indicated a change in absorbance with hydrogen ion concentration due to the equilibrium shown in Equation (1). Upon standing, a ferric salt solution gradually changes in color from yellow-orange to red, the pH decreasing simultaneously. The solution soon becomes turbid, and eventually hydrous ferric oxide precipitates. It is evident that during the aging process there is an increased coordination of OH^- ions with $\text{Fe}(\text{III})$. The investigators correlated the structural changes which occur to changes in spectra over time. Their results showed an increase in the iron to hydroxide ratio from 1:0.98 to 1:1.95 after 113 hours. This result may be related to the reaction previously described in Equation (2).

Characteristics of Iron Sludge

Sludges can vary significantly from plant to plant and even within the same plant due to seasonal influences. However, the basic characteristics of iron sludges are generally similar. Ferric sulfate sludge is a bulky substance, reddish-brown in color, composed of hydrous ferric oxides, color colloids, microorganisms, and the suspended matter, both organic and inorganic, removed from the water being treated. The sludge may also contain body feeds, added to aid coagulation, and powdered carbon, used for taste and odor control (7, 16).

Solids. Sludge solids content varies widely and is difficult to predict. Generally, the solids level is a function of the raw water turbidity, ferric sulfate dosage, and time of concentration.

The total solids content of sedimentation basin iron sludge is usually higher than that of alum systems. From various sources, total solids values from 0.26 per cent to 3.5 per cent have been reported (16, 17). Suspended solids typically account for about 80 per cent of total solids.

pH. The pH of iron sludge produced in water treatment is normally in the neutral range and approximately that of the treated water.

Oxygen Demands. Although ultimate BOD values of iron sludges from water treatment plants may be in the hundreds of milligrams per liter range, reported BOD₅ values are usually very low, perhaps due to toxicity associated with the metal. These sludges may exhibit relatively high values of COD (18). In one study COD values of a ferric sulfate sludge at a solids concentration of 3.5 per cent were found to be on the order of 5500 milligrams per liter (17). However, the composition of these sludges is such that the rate of decomposition is quite slow. Consequently, iron sludges from water treatment plants are stable and do not normally create critical deficits of dissolved oxygen when discharged to streams.

Settleability. There is not sufficient data to characterize the settleability of iron sludge. Generally, an inverse relationship exists between the solids content and the initial settling rate.

Filterability. Filterability of sludges can be measured using vacuum filter techniques. The analytical method chosen for this

study was the specific resistance test. Specific resistance can be described quantitatively as:

$$r = \frac{2PA^2b}{\mu C} \cdot g \quad \dots \dots \dots (3)$$

where r = specific resistance, m/kg

P = pressure of vacuum applied to filtration, kg/m^2

A = filter area, m^2

b = slope of T/V versus V curve, sec/m^6

T = time of filtration, sec

V = volume of filtrate in time T , m^3

μ = filtrate viscosity, $\text{kg/m}\cdot\text{sec}$

C = solids concentration of sludge, kg/m^3

g = acceleration of gravity, m/sec^2

The specific resistance of iron sludges varies depending on the sludge characteristics and experimental conditions, particularly solids concentration and volume of the sludge sample used. Thus it is difficult to translate specific resistance data from one study to another. The dewatering characteristics of ferric sludges are normally superior to those of alum sludges (17).

Coagulant Recovery From Water Treatment

Plant Sludges

The concept of using a coagulant recovery system within a water treatment plant is by no means new. Recovery of alum was attempted as early as the turn of the century. However, widespread interest in the process did not develop until about 1950 (19).

In 1954 Palin (20) reported little success in acidification of wet alum sludge. He stated that the aged alum precipitates did not readily dissolve in acid and that the sludge volume was not appreciably diminished. He found, however, that almost full alum recovery could be accomplished by adding sulfuric acid to a dried sludge containing Al_2O_3 .

Vahidi and Issac (21) found that the amount of aluminum hydroxide dissolved by sulfuric acid was a function of the quantity of acid added to the sludge. They further reported that the remaining sludge dewatered more rapidly than untreated sludge. Results of centrifugation of acid-treated sludge showed that the volume of remaining sludge decreased with increasing percentage of alum recovered. The investigators concluded that the coagulation efficiency of the treated supernatant was lower than that of fresh alum due to the organic color recovered.

Several alum recovery investigations have been conducted on a pilot plant scale and, in a few cases, extended to full-scale plant operations. During the 1960's Roberts and Roddy (22) worked on a sludge acidification process in Tampa, Florida. From batch studies they determined that the optimum pH range for recovery of alum was between 1.5 and 2.5. The reclaimed alum was nearly as efficient a coagulant as fresh alum. In actual plant operations there was no noticeable interference from any acid-insoluble material or any change in the treated water quality whatsoever. The operational scheme utilized is shown in Figure 3.

Other large-scale recovery systems include the facilities at Daer, Scotland, and Tokyo, Japan. At Daer Water Treatment Works,

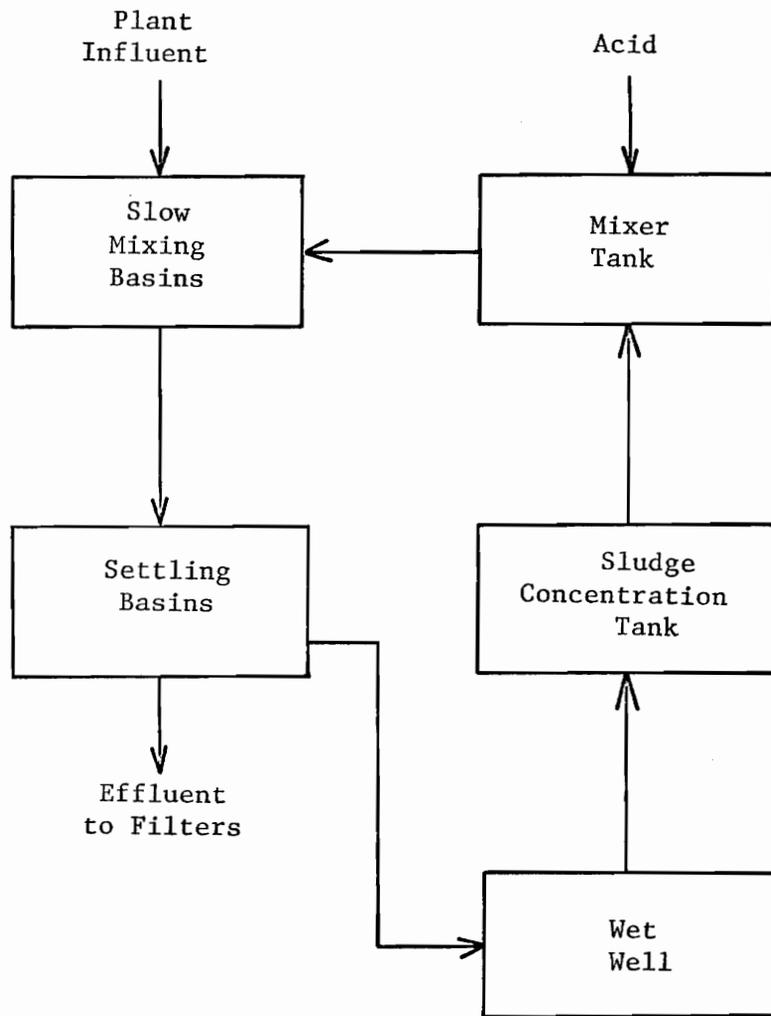


Figure 3. Operational Scheme for Alum Recovery Process at Tampa, Florida

Webster (23) operated a pilot plant on a batch basis, where the sludge and supernatant were pumped from their units every 24 hours. After acid treatment only about 5.6 per cent of the sludge was unreclaimed Al_2O_3 , and sludge solids were increased to 10 per cent (16). The Asaka Purification Plant near Tokyo has been recovering alum on a continuous flow basis for some time (24). Chen (25), whose economic analysis indicated that the break-even plant size for alum recovery from water treatment sludge is about 25 MGD, estimated that the recovery process employed at this 450 MGD facility is not only economical but also profitable.

Chen's (25) recent investigation showed that the amount of alum recovered is stoichiometrically related to the quantity of acid added. Sludge samples were obtained from a variety of sources, and both sulfuric and hydrochloric acids were used. Because this stoichiometric relationship existed, Chen maintained that control of the alum recovery process is not difficult and that pH measurements may be used to prevent an overdose of acid. The coagulation efficiency of recovered alum was not appreciably reduced from that of fresh alum, and the remaining sludge after acidification showed better filterability and drainability properties.

As previously mentioned, little work has been done on iron recovery from sludges. Cities Service Company, Atlanta, Georgia, claims to have attempted recovery of iron at low pH from water treatment plant sludge, but no experimental data is available. So far as can be discovered, no studies on iron recovery from water treatment

plant sludge have been published. One would predict, however, that iron coagulants could be reclaimed in somewhat the same manner as alum. The hydrolyzed species of aluminum and iron are not dissimilar, and the solubilities as a function of pH follow the same general trend.

Chapter III

EXPERIMENTAL METHODS AND MATERIALS

Source of Iron Sludge

The sludge samples were collected from the sedimentation basins of the 11 MGD water treatment plant serving the city of Kingsport, Tennessee. A tabulation of typical raw and finished water quality is given in Table 1. As indicated by the flow diagram in Figure 4, the Kingsport plant is a typical water filtration facility. A ferric sulfate coagulant, "Ferri-floc" (Cities Service Company, Atlanta, Georgia), is employed for treatment of a surface water supply. Lime is used for pH adjustment, and activated carbon is added in small amounts for the removal of organic color. The plant is equipped with flash-mixing and baffled slow-mixing basins, sedimentation basins, and both conventional and high-rate rapid sand filters. Chlorine is added in the flash mix unit for disinfection and odor and algae control. Yearly water production and coagulant consumption are shown in Table 2.

Sludge disposal at the Kingsport plant is performed twice yearly by direct discharge into the Holston River. At the time of collection of samples, the iron sludge had accumulated over a period of five months. Withdrawal of the sludge from the sedimentation basin was accomplished using a small pump. The sludge samples obtained had a relatively low solids content due to dilution during pumping.

TABLE 1
TYPICAL CHEMICAL ANALYSIS OF KINGSPORT WATER

	<u>Raw Water</u>	<u>Treated Water</u>
pH	7.5	8.0
Alkalinity M.O. (mg/l)	85	90
Alkalinity P. (mg/l)	0	0
Hardness (mg/l)	95	100
Iron (mg/l)	.15	.05
Manganese (mg/l)	.02	0
Color units		
Apparent	28.3	0
Centrifuge	10	0
Turbidity	6	.04

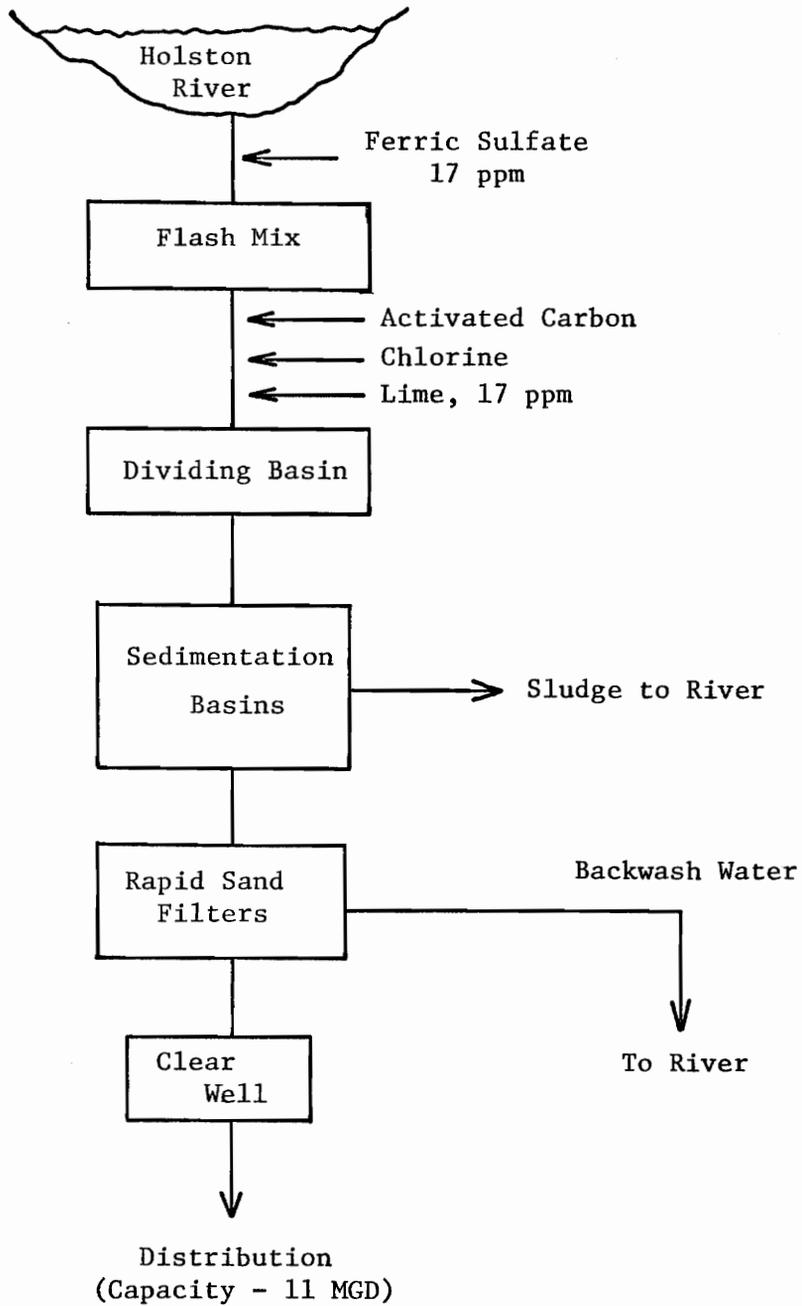


Figure 4. Flow Diagram Kingsport Water Treatment Plant

TABLE 2
ANNUAL WATER PRODUCTION AND COAGULANT USE AT
KINGSPORT, TENNESSEE, WATER TREATMENT PLANT

<u>Year</u>	<u>Total Water Gallons</u>	<u>Ferric Sulfate Pounds</u>	<u>Lime Pounds</u>
1967	2,405,000,000	251,000	295,000
1968	2,405,000,000	241,000	346,000
1969	2,406,000,000	280,000	422,000
1970	2,620,000,000	300,000	361,000
1971	2,624,000,000	310,000	314,000
1972	2,876,000,000	399,000	376,000

Settleability Test

Sludge samples were allowed to settle for an indefinite period, the supernatant liquid being decanted intermittently. Concentration of samples was continued until there was no longer an appreciable daily change in total solids. The sludge was then stored in five-gallon polyethylene carboys at 20 degrees Centigrade.

A particular total solids concentration was chosen for all sludge samples to be chemically treated. After thorough mixing to assure uniformity, the stored sludge was diluted to this solids concentration using the decanted supernatant. Various amounts of acid or alkali of known purity were added to the sludge in order to achieve iron recovery. Although the reactions are exothermic, the mixtures were stirred at 30 rpm for 30 minutes. The mixture was poured into 500-milliliter graduated cylinders until reaching the 500-milliliter mark. The position of the solid-liquid interface was recorded at various time intervals. After 30 minutes the final sludge volume was recorded, and the supernatant was carefully decanted for pH, solids, and iron content determinations. The remaining settled sludge was used for the filterability measurement using the specific resistance test. The decanted supernatant was preserved for coagulation testing.

Iron Determination

After the iron sludge was chemically treated, portions of the decanted supernatant were digested in preparation for measurement of iron content. Iron concentration was determined using the

phenanthroline method as outlined in Standard Methods, 13th edition (26). In this procedure the iron is separated from interfering substances by extraction into isopropyl ether. After the addition of phenanthroline indicator, the absorbance of the orange complex was measured using a Klett-Summerson photoelectric colorimeter. This method is subject to many interferences and was accurate to within about 15 per cent. Consequently, iron concentrations were verified using the Atomic Absorption Spectrophotometric Method as outlined in Standard Methods (26).

Solids Determinations

Total and volatile solids and total and volatile suspended solids concentrations were determined prior to chemically treating the sludge samples. After acidification and settling, the decanted supernatant was analyzed for dissolved and volatile dissolved contents. The remaining sludge was tested for total and volatile solids concentrations. All tests were performed using the procedures outlined in Standard Methods (26).

Filterability Test

Filterability of the chemically treated sludges was determined using the Buchner funnel specific resistance test. A diagram for the apparatus is shown in Figure 5. Testing procedures were as follows:

1. One piece of Whatman Number 1 filter paper was placed on the bottom of the Buchner funnel. A proportion of distilled water

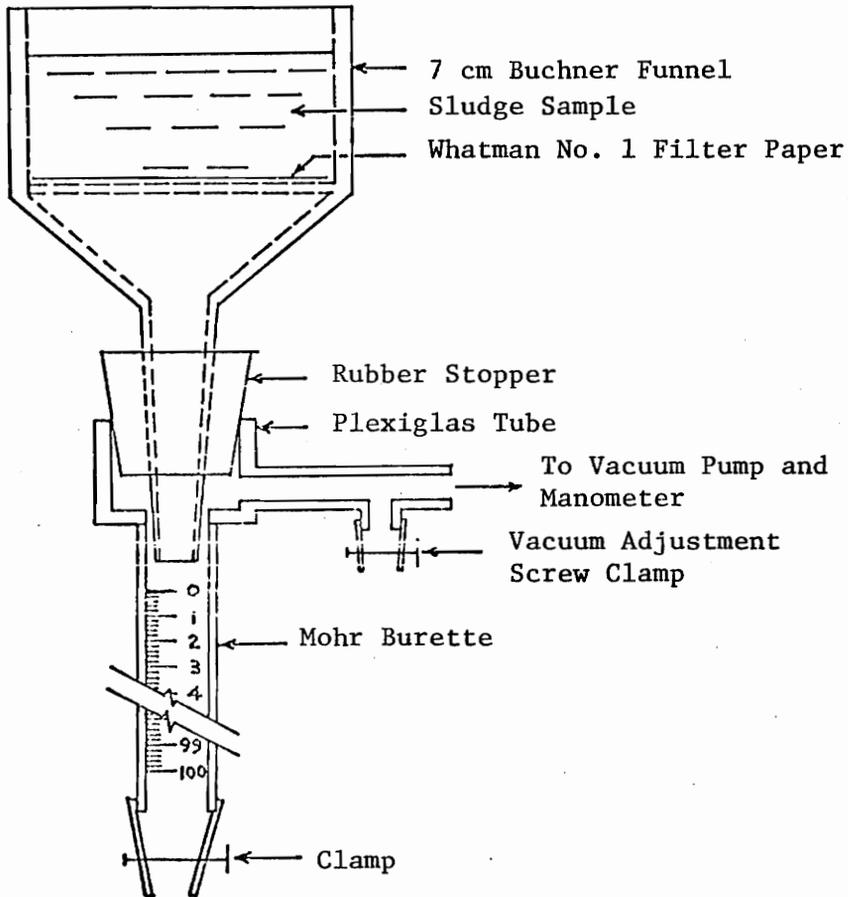


Figure 5. Apparatus for Buchner Funnel Specific Resistance Test

was poured into the funnel, and vacuum was applied to drain off the liquid to properly seat the filter paper.

2. The vacuum was relieved and the Mohr burnette was emptied.

3. A 50-milliliter sample was poured into the funnel. The vacuum was applied, and the stopwatch was started as the filtrate reached the 100-milliliter mark. Time readings were taken at various volumes for about two minutes. The applied pressure was recorded from the manometer.

The values of specific resistance to dewatering were then calculated according to Equation (3) previously shown.

Evaluation of the Coagulation Efficiency of Recovered Iron

The supernatant above the acidified sludge was preserved for coagulation tests. Prior to testing, samples were again carefully decanted to insure the removal of all settleable solids. Two raw waters were used in coagulation testing, one natural and the other artificial. The natural water was taken from the New River, a source which normally maintains sufficient alkalinity for iron coagulation. The artificial water samples were made from distilled water to which various amounts of finely ground kaolin were added. The alkalinity was adjusted to 100 milligrams per liter by adding sodium bicarbonate.

Both the recovered iron solution and a fresh reagent ferric sulfate solution were used as coagulants. Different amounts of either coagulant were added to 1000-milliliter raw water samples in 1500-milliliter beakers, which were placed on a six-phase stirring apparatus. One minute of rapid-mixing was followed by 30 minutes

of flocculation at 20 rpm. The stirring paddles were then removed, and the flocculated solution was allowed to settle quiescently for another 30 minutes before the supernatant was withdrawn. Residual turbidity of the supernatant was measured using a Hach Model 2100A turbidimeter.

The reagent grade ferric sulfate ($\text{Fe}_2(\text{SO}_4)_3 \cdot n\text{H}_2\text{O}$) used was labeled greater than 99 per cent pure, but no indication was given as to the amount of bound water contained in the chemical. Therefore, known amounts of the reagent were dissolved in distilled, deionized water and analyzed for Fe(III) concentration. Two samples were analyzed, yielding an average purity of 74 per cent as $\text{Fe}_2(\text{SO}_4)_3$. This result approximately corresponds to the chemical formula, $\text{Fe}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$.

CHAPTER IV

EXPERIMENTAL RESULTS

Sludge Characteristics

It is imperative for accurate correlation with other research that sludges be properly characterized. Hence, initial work was concerned with characterization of the selected iron sludge.

The characterization results obtained are summarized in Table 3. As previously described, the sludge was allowed to settle for several days. The final total solids concentration was 40,800 milligrams per liter. In recovery experiments the concentrated sludge was diluted 1:3, resulting in a total solids content of 13,600 milligrams per liter. Characteristics of both sludges are shown in the table for purposes of comparison.

Solids analyses indicated that the greatest percentage of the total solids existed as suspended solids. Independent of the solids contents of the samples, the volatile fraction was consistently between 15 to 20 per cent of the total solids. This result does not necessarily imply that all the measured volatile portion is organic matter. Some of the measured volatile solids may be due to previously bound water of hydration. The temperature of 103 degrees Centigrade, which is used in the total solids test, is probably not sufficient to release all the water bound by iron oxide or hydroxide compounds or by the clay or silt particulates. Judging from the appearance of the cake remaining after heating, this feature may be particularly true in the case of suspended solids determinations.

TABLE 3
 CHARACTERISTICS OF FERRIC SULFATE SLUDGE
 FROM KINGSPORT WATER TREATMENT PLANT

<u>Parameter</u>	<u>Concentrated Samples</u>	<u>Samples Diluted for Chemical Addition</u>
Total Solids (mg/l)	40800	13600
Volatile Solids (mg/l)	6895	2310
Per cent Volatile (%)	16.9	17.0
Total Suspended Solids (mg/l)	40540	13520
Volatile Suspended Solids (mg/l)	6690	2240
Per cent Volatile (%)	16.5	16.6
pH	7.6	7.6
Total Iron (mg/l as Fe)	7050	2350
Per cent Iron (%)	17.3	17.3
Specific Resistance (m/kg)	13.7×10^{12}	19.5×10^{12}
Initial Settling Velocity (cm/sec)	very slow	.007

Difficulty was encountered in weighing the dried residues during solids analyses. Once removed from the desiccator, containers continuously gained weight owing to the hygroscopic iron present. To avoid variability in weighings, the dish or crucible was quickly moved from the desiccator to the balance pan, and the reading was taken when the balance first stabilized.

The total iron content of the sludge was found to be 17.3 per cent by weight as iron using the Atomic Absorption (A.A.) Spectrophotometric Method on acid-digested samples. Analysis of identical samples by the phenanthroline method resulted in a concentration of 15.3 per cent iron by weight, indicating that an interference existed in one of the methods used. It was suspected that iron adsorbed by colloidal particles may have constituted the difference in readings. Therefore, digested samples were filtered prior to analysis by both methods. The AA determination resulted in 15.2 per cent iron while the phenanthroline method gave 14.8 per cent, suggesting that suspended iron had indeed accounted for the variance. Consequently, all subsequent samples were digested and passed through glass fiber filters to remove any suspended material prior to the iron determination. This technique resulted in the measurement of dissolved iron only, and the data were adjusted accordingly.

From the comparative data obtained by A.A. analysis of filtered and unfiltered supernatant samples, it appears that between 12 and 15 per cent of the recoverable iron remained associated with colloidal particles even after strong acid digestion. The author suspects that this

adsorbed iron was only partially extracted in the phenanthroline procedure. The decision to filter all samples was based on the assumption that any adsorbed iron which dissolves will do so gradually as the pH is lowered.

The pH of the untreated sludge was 7.6, or approximately that of the raw and treated water (see Table 1). Specific resistance values as measured by the Buchner funnel specific resistance test were initially on the order of 10^{13} meters per kilogram. As indicated in Table 3, the specific resistance of samples diluted for chemical addition was greater than that of the concentrated sludge. The initial settling velocity of the untreated sludge was too low for practical measurement.

Recovery of Iron from the Sludge

A preliminary study of iron recovery from the sludge was performed using various chemicals. Selected concentrations of sulfuric acid, hydrochloric acid, and sodium hydroxide were added to sludge samples. After settling occurred, iron contents of the supernatants were determined.

It was found that both sulfuric acid and hydrochloric acid solubilized the iron present, while sodium hydroxide did not. The poor recovery of iron by sodium hydroxide is probably due to the formation of ferric hydroxide precipitates. Thus, the alkaline technique was eliminated from further recovery studies. As shown in Figure 6, iron recovery was best achieved using sulfuric acid. Since the use of hydrochloric acid offered no apparent chemical advantage,

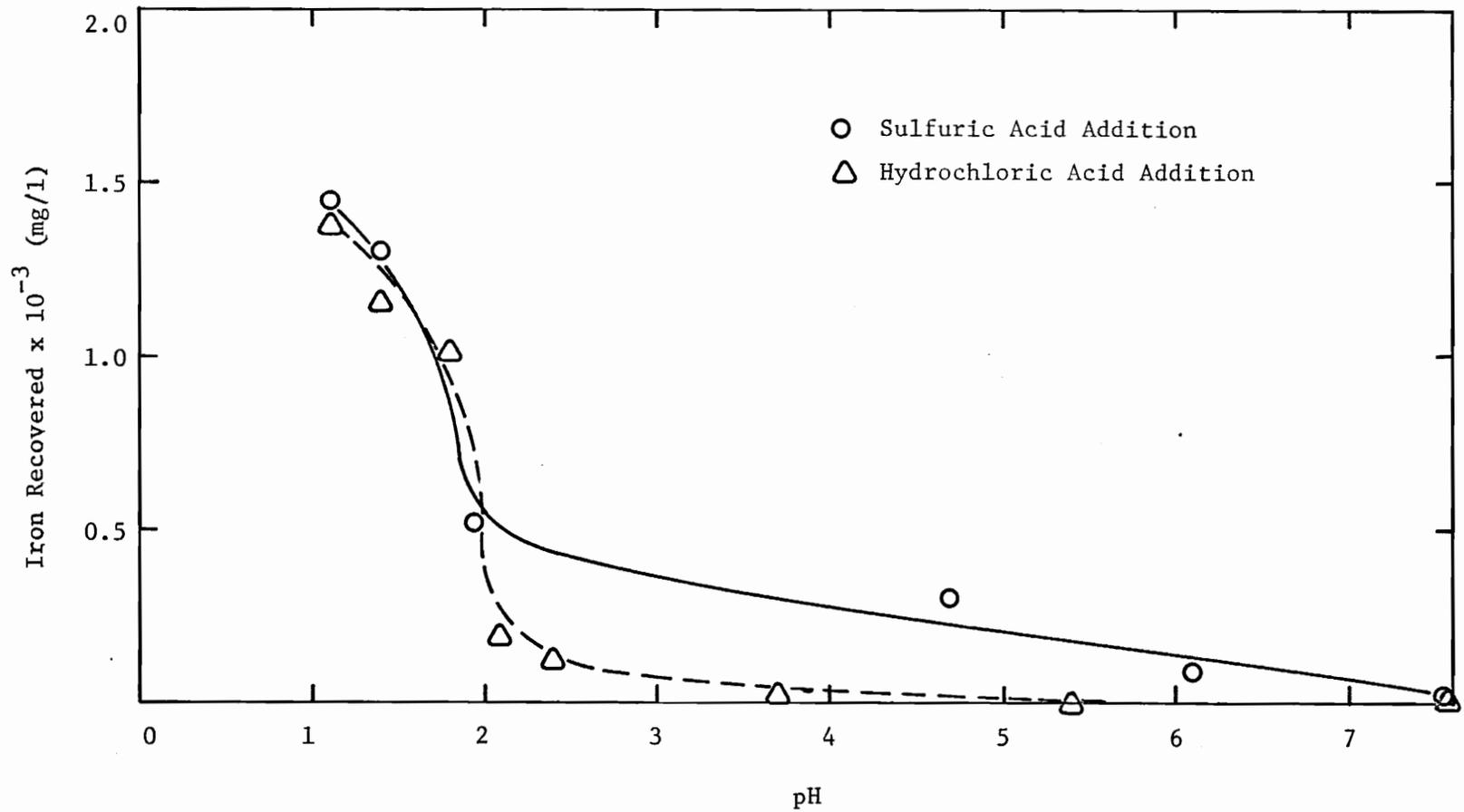


Figure 6. Iron Recovered as a Function of pH using Two Acids

and its cost is prohibitively higher than that of sulfuric acid, it was also eliminated from further studies.

Additional data relating iron recovered using sulfuric acid as a function of pH are shown in Figure 7. Recovery of iron began to be accomplished as soon as the pH was lowered. The titration curve given in Figure 8 indicates that the sludge pH immediately responded to acid addition and that a breakpoint occurred at a pH value between 2.0 and 2.5. This pH level closely corresponds to the pH at the point of inflection of the recovery curve. The maximum portion of soluble iron recovered was 74 per cent, occurring at a pH value of 0.85.

Settleability of the Remaining Sludge

As previously described, the acidified sludge was settled in 500-milliliter graduated cylinders. The initial settling velocity, defined as the steepest slope of the height of the solids-liquid interface versus time curve, was used to evaluate the settleability of the remaining sludge. Some typical settling data are shown in Figure 9. The relationship found between the initial settling velocity and the sample pH is given in Figure 10. As noted therein, the observed settling velocity consistently increased with decreasing pH.

After 30 minutes of settling, the final volume of the remaining sludge was recorded. As illustrated in Figure 11, the final sludge volume decreased as the pH was lowered, the most substantial volume reduction occurring at pH values between 2.0 and 3.0.

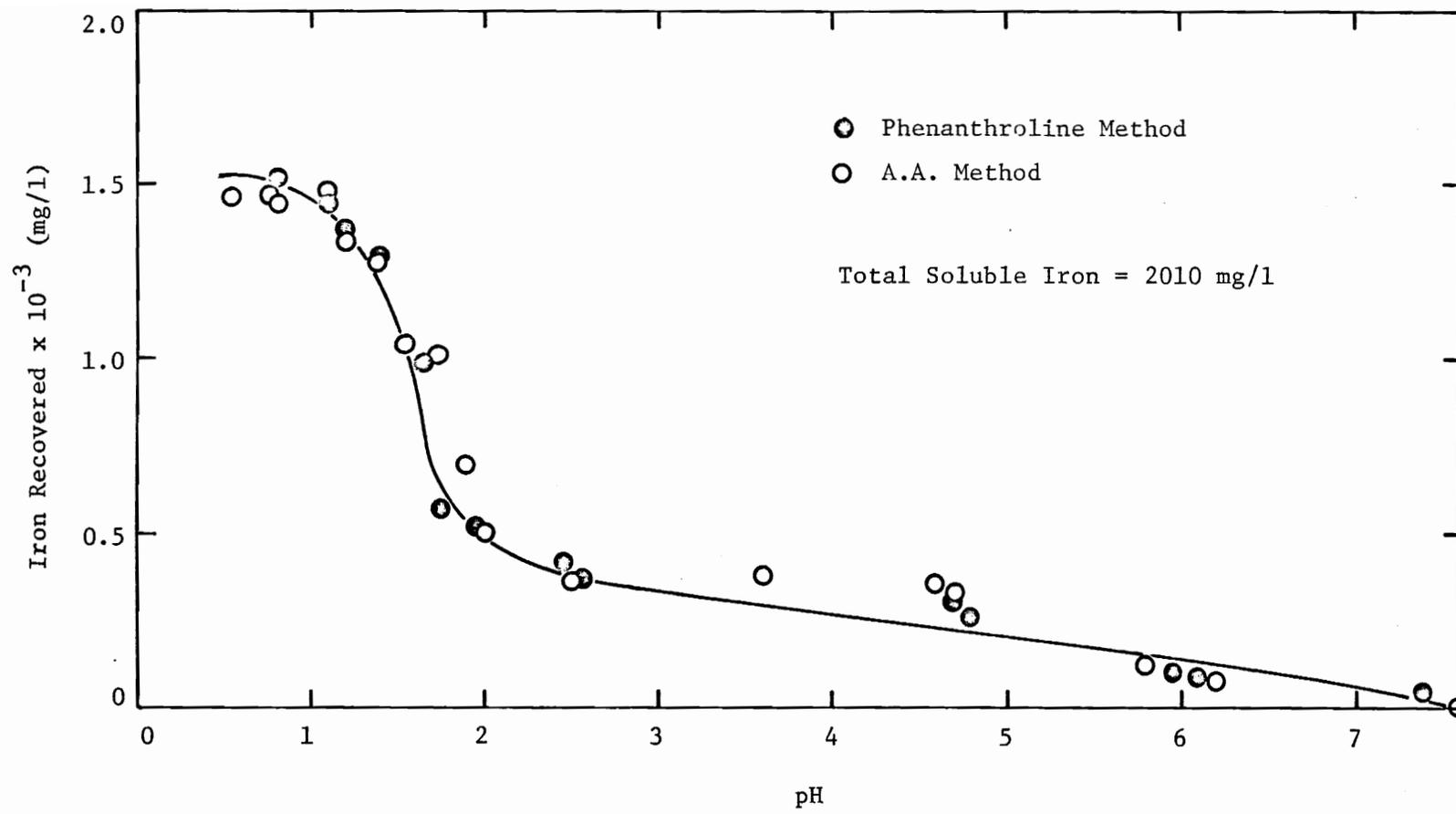


Figure 7. Iron Recovered as a Function of pH using Sulfuric Acid

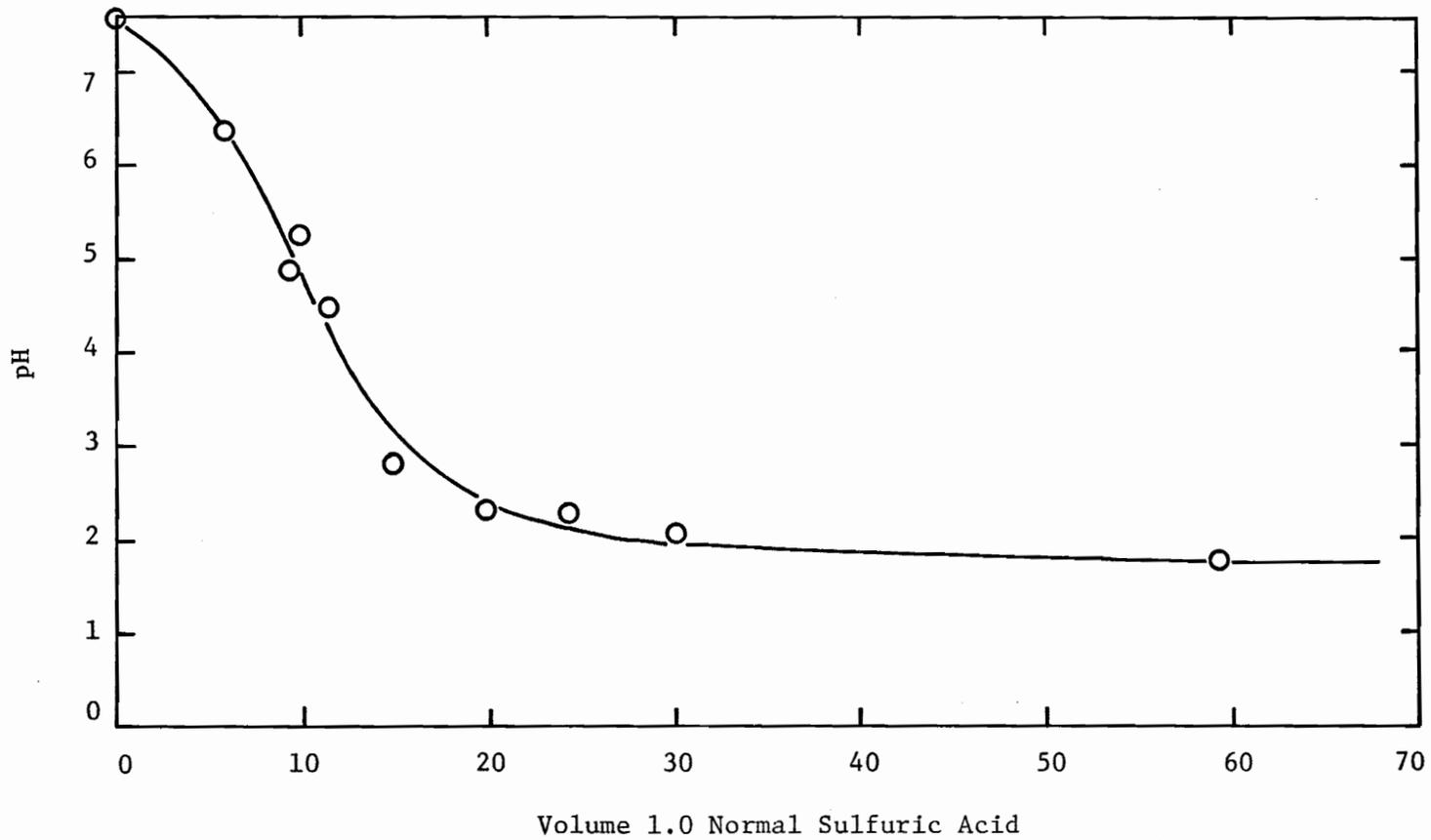


Figure 8. Titration Curve for Recovery Experiments

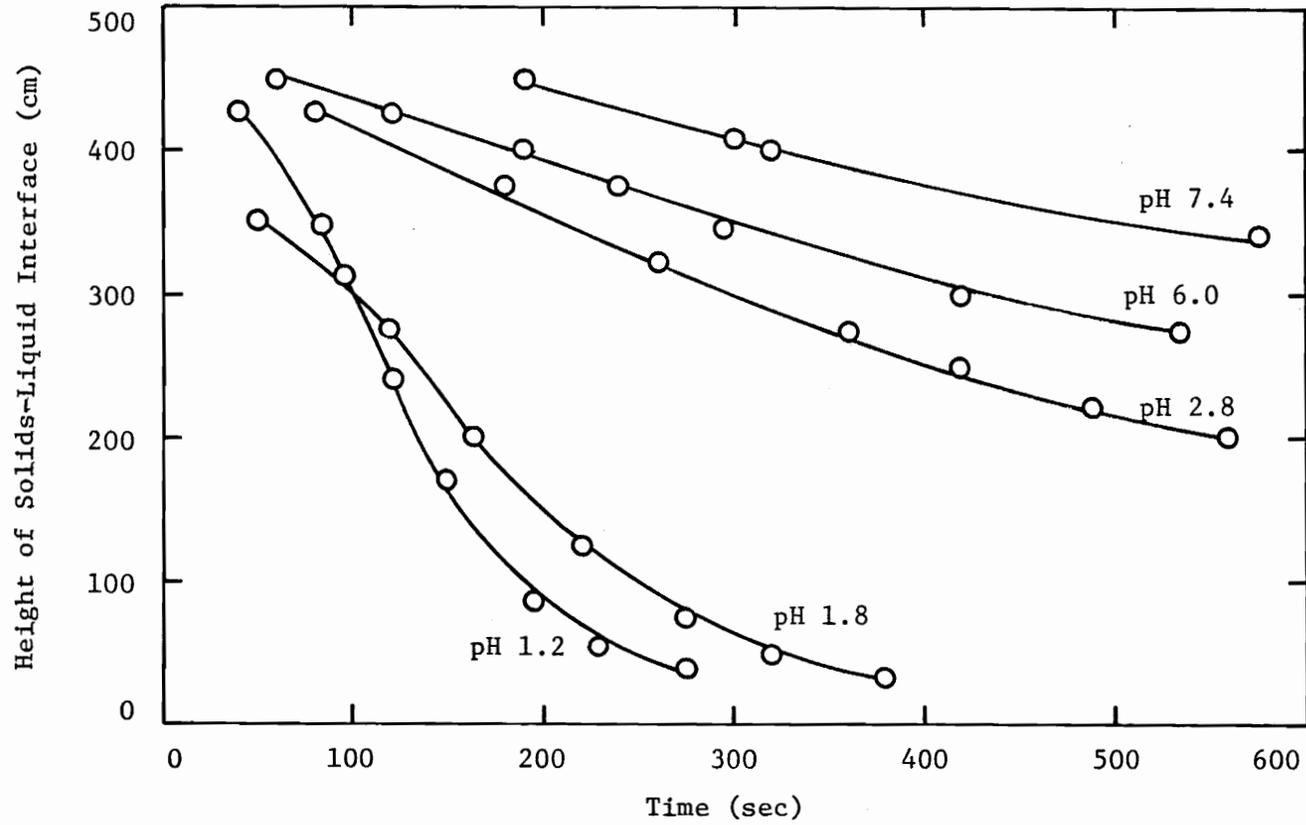


Figure 9. Settling Curves of Acidified Sludge Samples

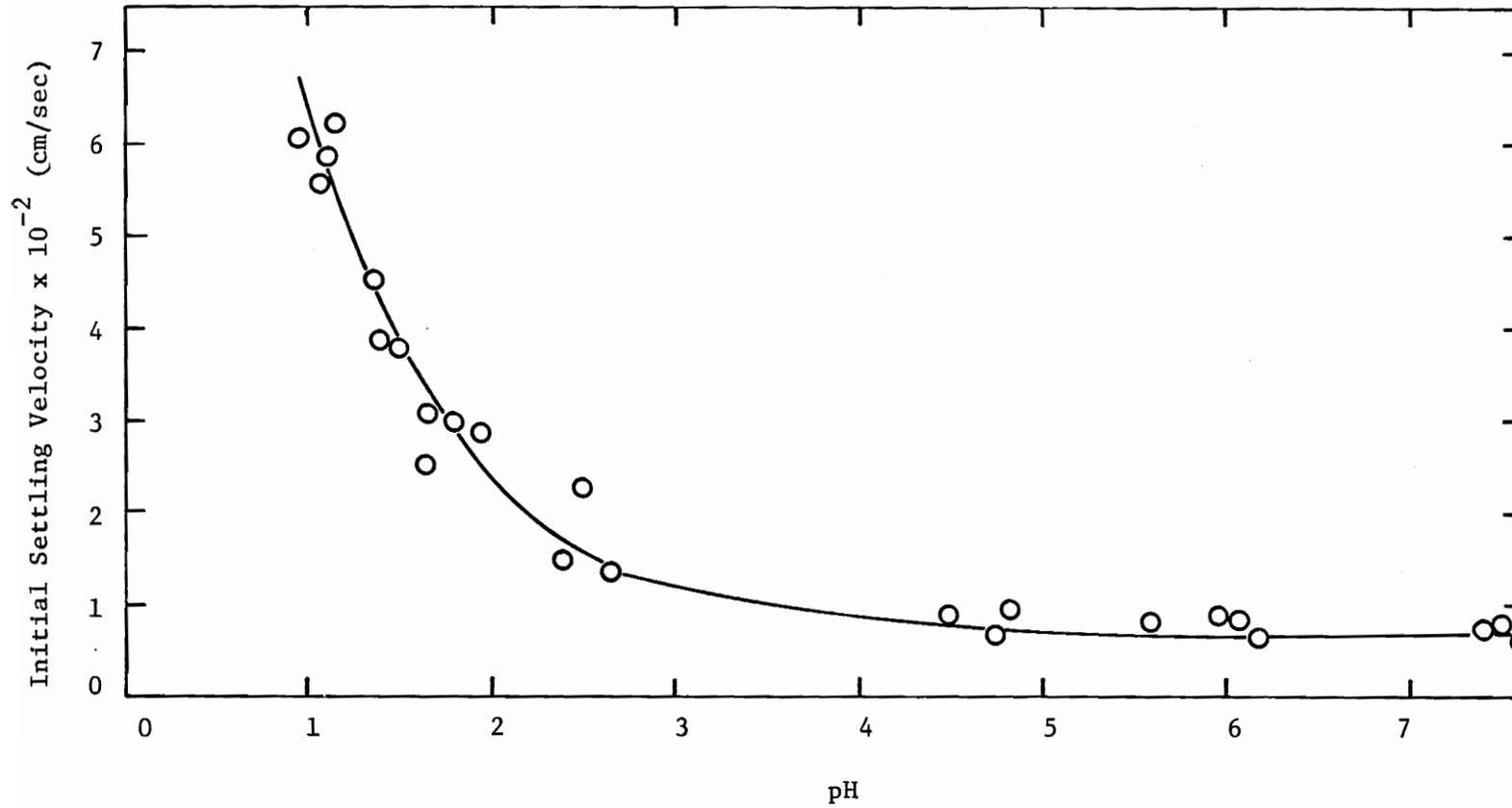


Figure 10. Initial Settling Velocity of Remaining Sludge After Acidification

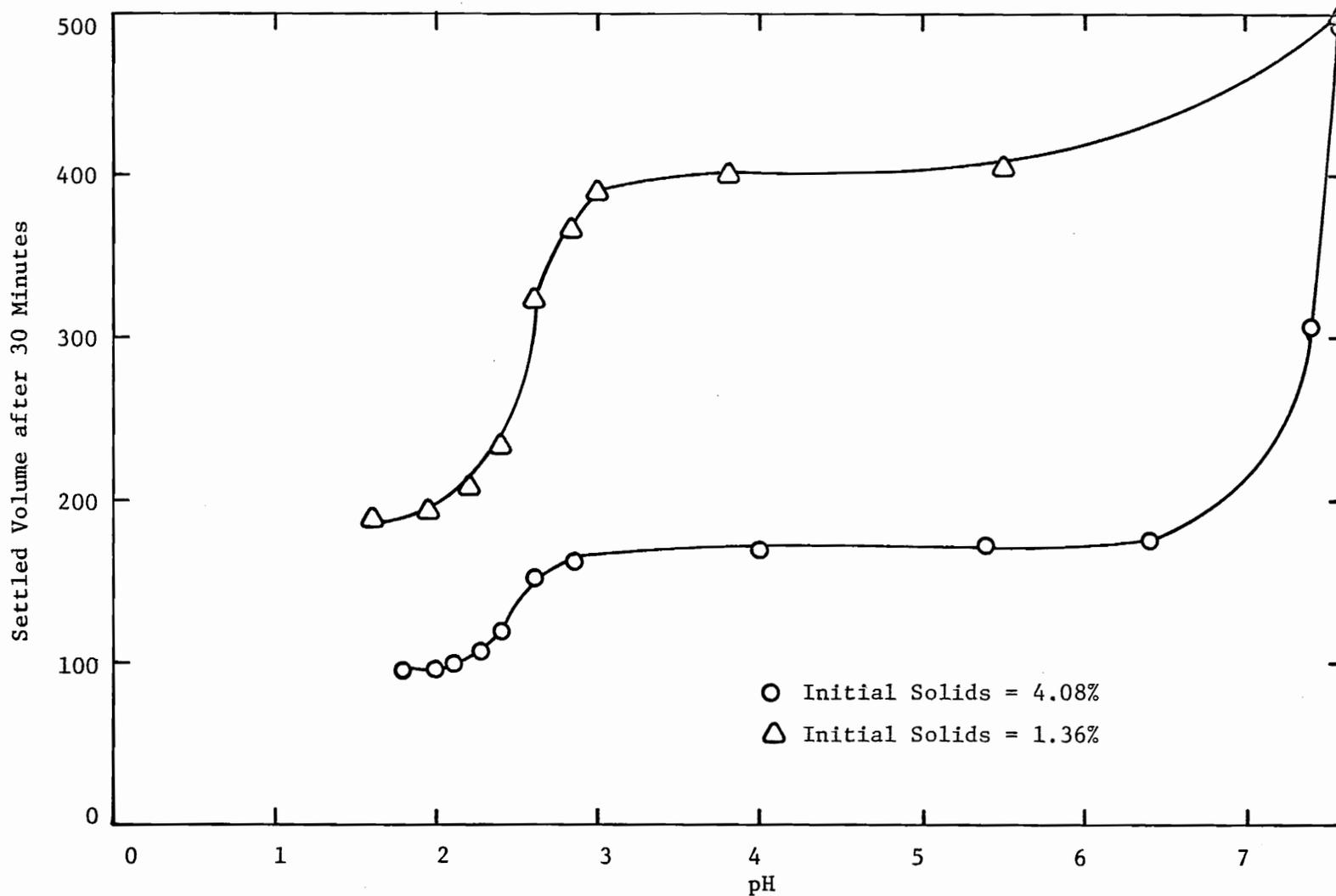


Figure 11. Settled Volume of Sludge as a Function of pH

Solids Characteristics of the Remaining Sludge

After the supernatant was decanted, portions of the remaining sludge samples were analyzed for total solids content. The relationships between total solids concentration and the pH level is depicted in Figure 12. Independent of the solids concentration of the sample, the volatile portion was always between 13 and 17 per cent of the total solids.

Filterability of the Remaining Sludge

The specific resistance of the settled sludge was determined for all acidified samples. Figure 13 indicates that acid addition sharply improved the filterability of the sludge, although deterioration was noted when the system pH was decreased below 2.0.

Solids Characteristics of the Decanted Supernatant

Supernatant samples were filtered to remove suspended matter, and the filtrates were analyzed for total dissolved and volatile dissolved solids contents. The results are shown in Figure 14. Conductivity measurements, taken for verification of the solids data, are included in the figure. The data indicate that dissolved solids increased with decreasing pH, the increase becoming substantial at pH values below 2.0.

Coagulation Efficiency of the Recovered Iron

Coagulation tests were performed using reagent grade ferric sulfate and supernatant samples as coagulants. Although the pH value and volume of coagulant added varied, the raw water pH level

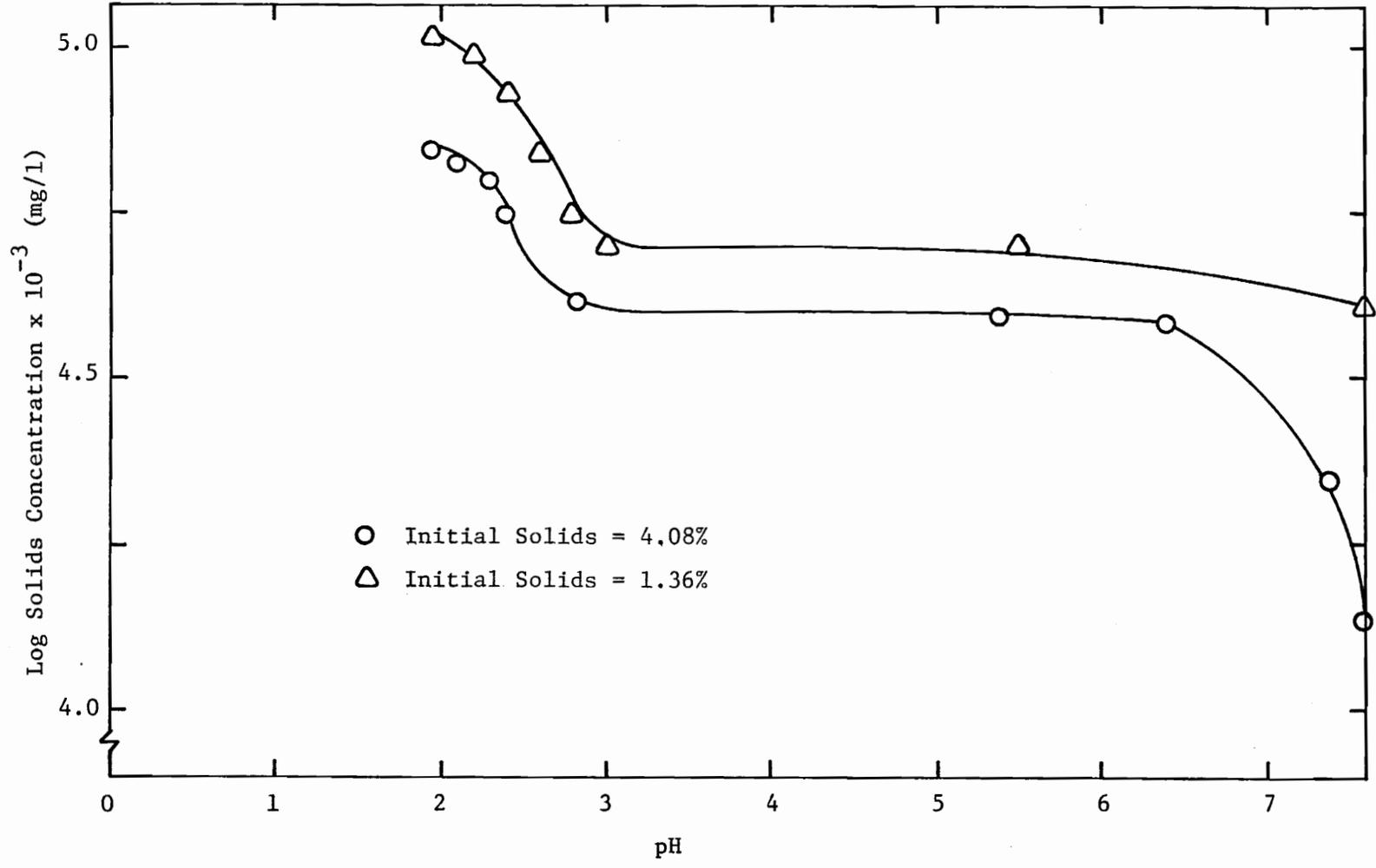


Figure 12. Solids Concentration of Remaining Sludge as a Function of pH

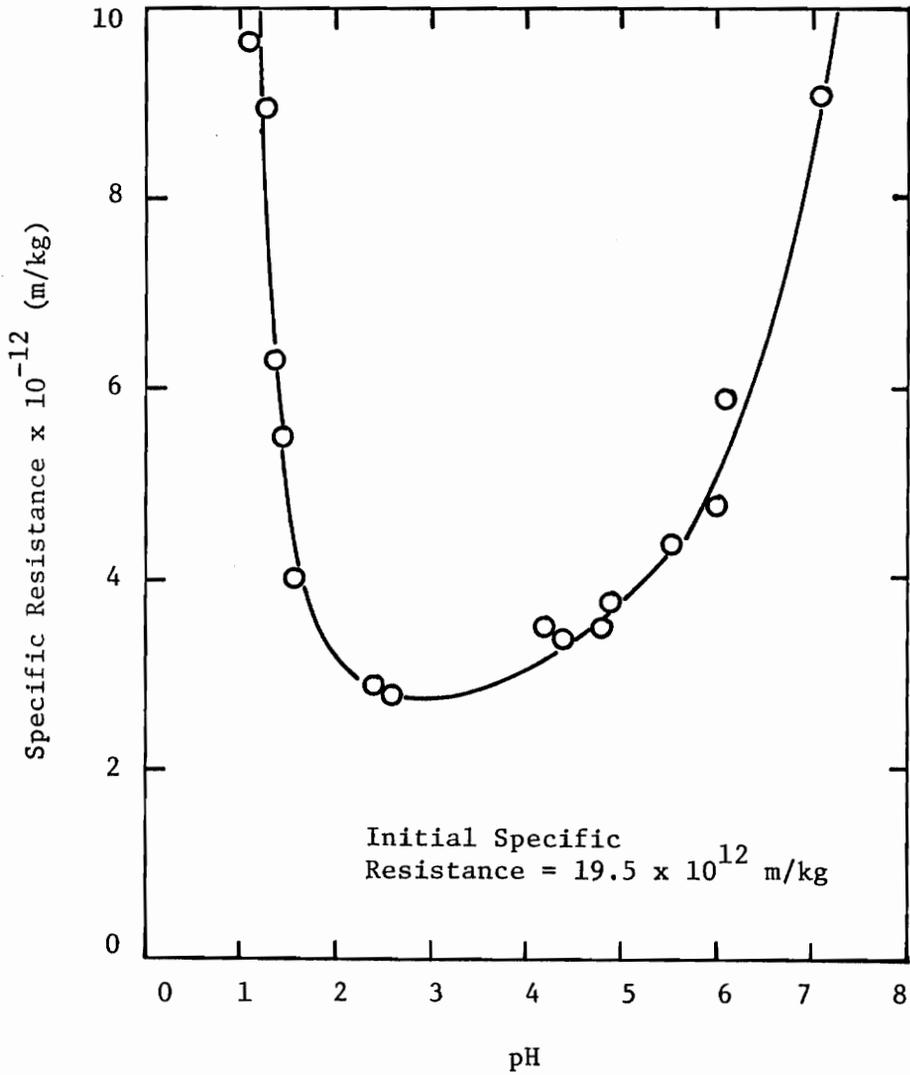


Figure 13. Specific Resistance of Remaining Sludge as a Function of pH

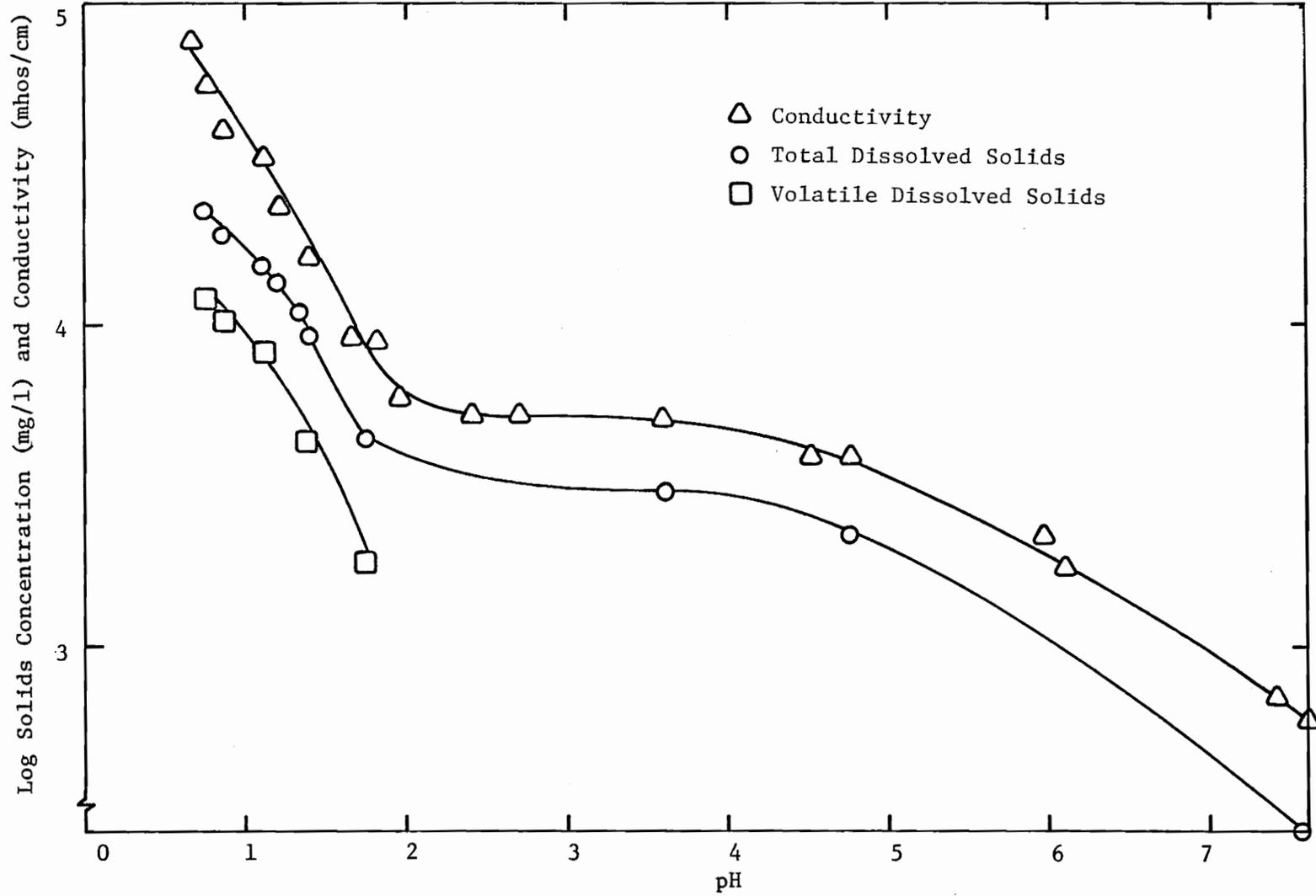


Figure 14. Dissolved Solids in the Supernatant as a Function of pH

never decreased by more than one-half of a pH unit. The data given in Figure 15 are representative of the results obtained. As noted therein, the raw water used in this series was a kaolinite suspension having moderate turbidity and alkalinity. The critical coagulation concentration (c.c.c.) was obtained by extrapolation of the steepest portion of the residual turbidity versus iron dosage curve to zero turbidity, as shown in Figure 16. From the data presented it may be noted that while fresh iron coagulant resulted in a slightly lower residual turbidity, the critical coagulation concentration was less when recovered iron was used.

Because of the great influence of colloid concentration on coagulation, raw water samples of various turbidity levels were prepared for use in coagulation tests. The results obtained are illustrated in Figure 17, where raw water turbidity is plotted against critical coagulation concentration of iron. The data indicate that for low turbidity waters, the recovered iron was superior to fresh iron.

As previously described, a natural raw water sample was also used in coagulation tests. The data given in Figure 18 are typical of those collected. The characteristics of the New River water are somewhat different from those of the artificial samples. Even so, the results are quite similar to those previously shown, except that residual turbidity levels are higher. Again, the use of recovered iron resulted in the lower critical coagulation concentration and a higher residual turbidity.

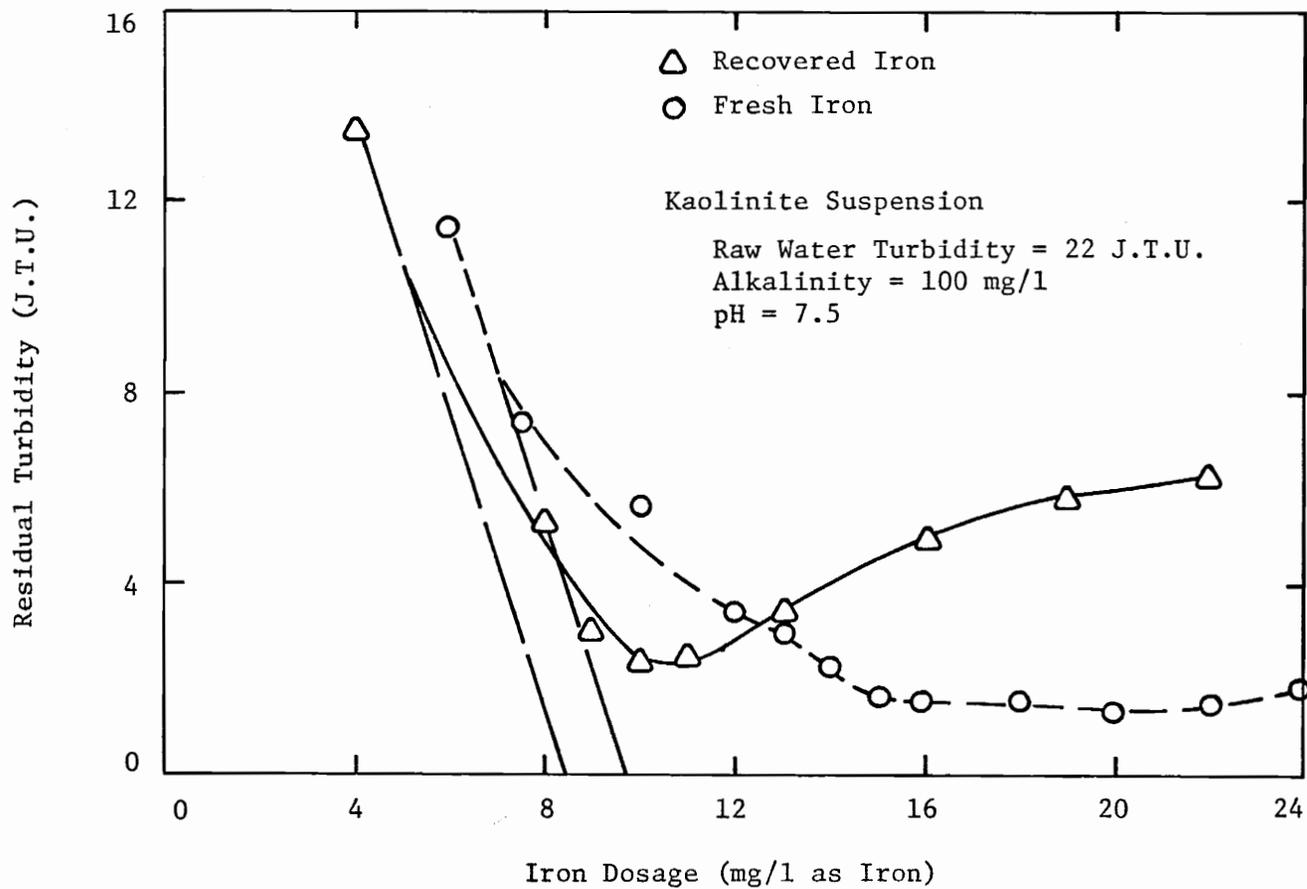


Figure 15. Residual Turbidity as a Function of Iron Dosage

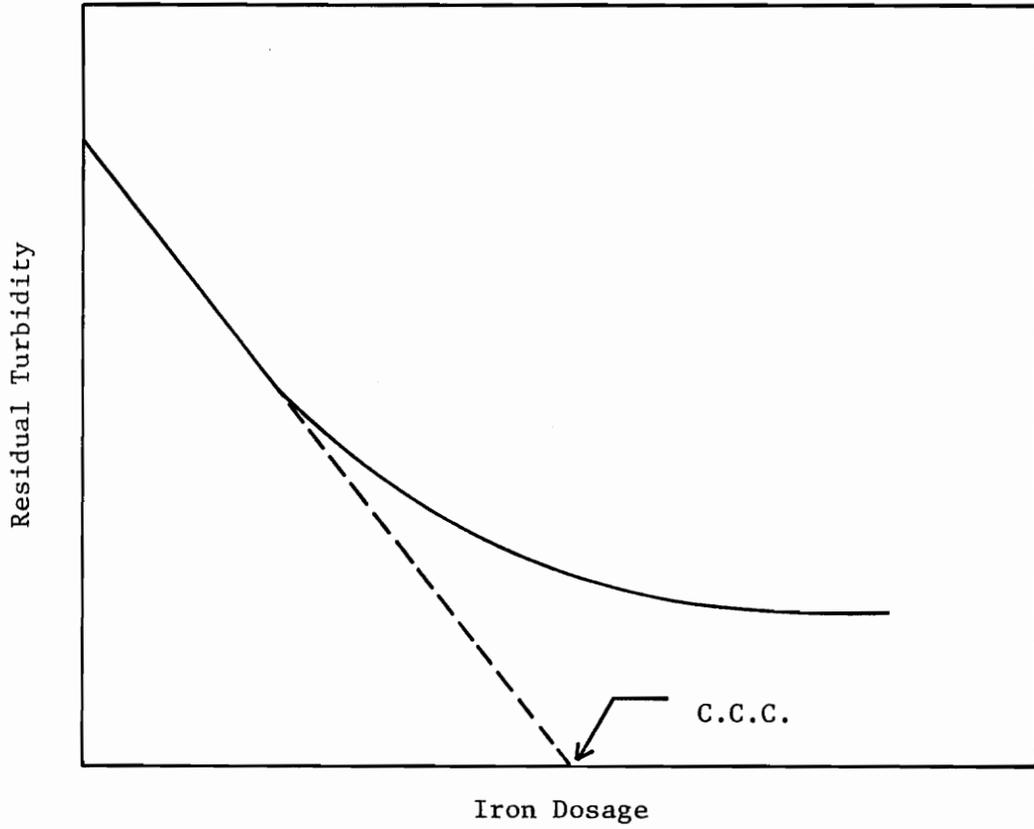


Figure 16. Typical Residual Turbidity versus Iron Dosage Curve

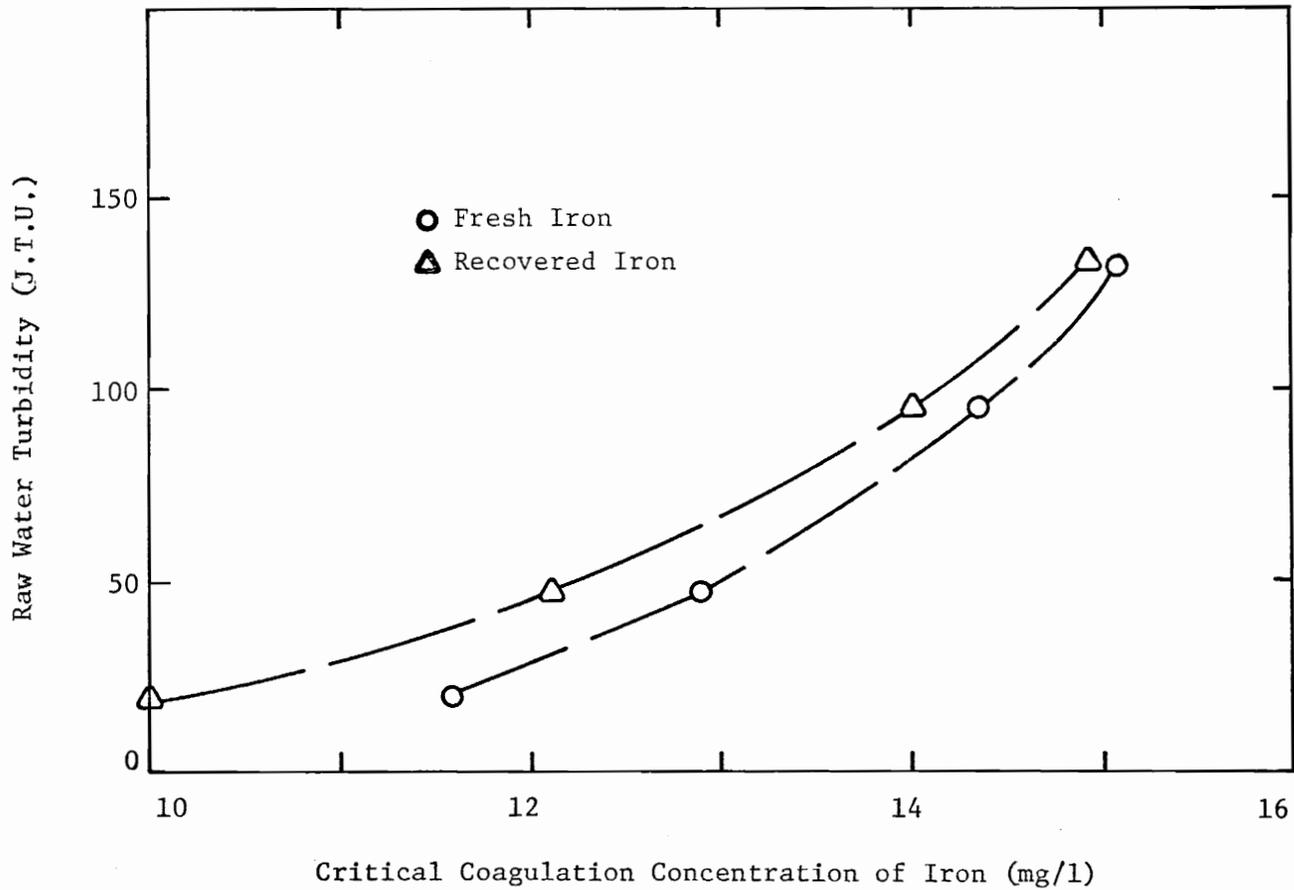


Figure 17. Critical Coagulation Concentration as a Function of Turbidity Level of Raw Water

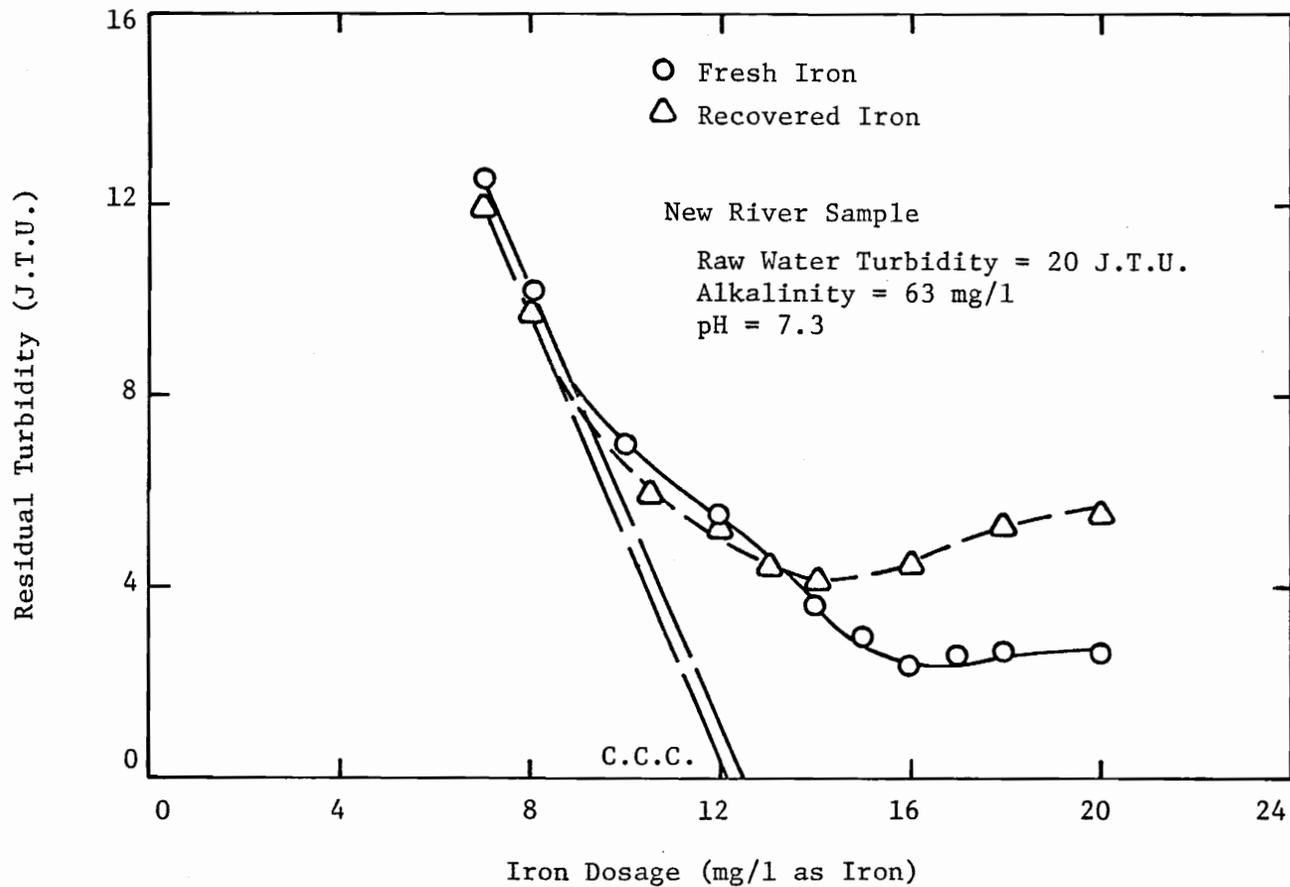


Figure 18. Residual Turbidity as a Function of Iron Dosage Using New River Raw Water Sample

CHAPTER V

DISCUSSION OF RESULTS

The purposes of this investigation were to furnish experimental information concerning the feasibility of recovering iron from water treatment sludge. The results aid in understanding the effects of acid addition to sludge on recovery and reuse of iron coagulant and on sludge dewatering properties. The following analysis points out those results considered particularly significant and seeks to interpret them in terms applicable to an actual recovery operation.

Sludge Characteristics

The characteristics of the Kingsport sludge, as listed in Table 3, are similar to those found in other studies concerning ferric sulfate water treatment sludges (17, 27). The solids content of the concentrated sample was 4.1 per cent, which compares to the 3.6 per cent sedimentation basin sludge reported by Olver (27). The volatile fraction of approximately 17 per cent of the total solids is also consistent with other results. The activated carbon added at the Kingsport plant probably accounts for a portion of the measured volatile solids. Gates and McDermott (28) noted in their investigation of alum sludge that activated carbon increased the volatile solids content of the sludge by as much as 30 per cent.

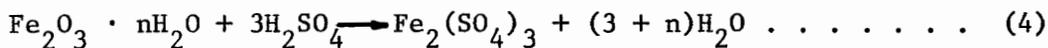
Specific resistance values were comparable to those reported in the literature. As indicated in Table 3, the specific resistance of the 4.1 per cent solids sample was 13.7×10^{12} meters per kilogram. Olver (27) reported a specific resistance of 4.9×10^{12} meters per

kilogram for a 3.6 per cent solids iron sludge. Gates and McDermott (28) and Chen (25) showed that an inverse relationship exists between total solids content and specific resistance. In accordance with this reverse relationship, the specific resistance of the sludge under investigation increased upon dilution, as shown in the table.

All untreated sludge samples had pH values of 7.6. The pH of the stored sludge samples remained constant throughout the study, and there was no indication of any occurrence of biological decomposition. However, the presence of bacteria was noted in several of the acidified sludge samples after storage.

Recovery of Iron From Ferric Sulfate Sludge

As previously described, the preliminary study resulted in the elimination of both sodium hydroxide and hydrochloric acid from further recovery studies. Because the best iron recovery occurred after sulfuric acid addition to the sludge, more extensive data were developed using this acid (see Figure 7). The following chemical reaction is anticipated:



It may be predicted that the amount of iron recovered is stoichiometrically related to the quantity of acid added. Equation (4) indicates that one and one-half moles of sulfuric acid would be required to recover one mole of iron. In order to determine whether the recovery experimented followed a stoichiometric relationship, the iron concentrations of supernatant samples were compared to the different

amounts of sulfuric acid added to the sludge samples. The data presented in Figure 19 appear to confirm to some degree the stoichiometric relationship.

A closer look at the figure reveals some interesting trends in the recovery process. Initially, as small quantities of acid were added, the per cent iron recovered was less than anticipated. The points below the line predicted by stoichiometry indicate that the sludge may have had an immediate acid demand due to impurities. However, such a demand is not confirmed by the titration curve, as the system pH quickly responded to acid addition (see Figure 8). More likely, stoichiometric recovery is limited by solubility considerations at pH values above 2.0 (see Figures 1 and 7).

Returning to Figure 19, it is noted that other points lie above the stoichiometric line. These data begin to occur at about 30 per cent of the stoichiometric amount of acid added, or at a pH value of 2.0. Previously insoluble iron appears to dissolve quite readily, a trend which continues until 70 per cent of the stoichiometric amount of acid has been added. Since different hydrolyzed species dominate at various pH levels, this sudden increase in soluble iron may indicate the formation of a particular soluble hydroxo complex. An alternate explanation could be that previously adsorbed iron was released at low pH values, but it is not anticipated that this phenomenon would occur so dramatically.

As larger amounts of sulfuric acid were added, recovery of iron was again marginally less than the stoichiometric quantity predicted.

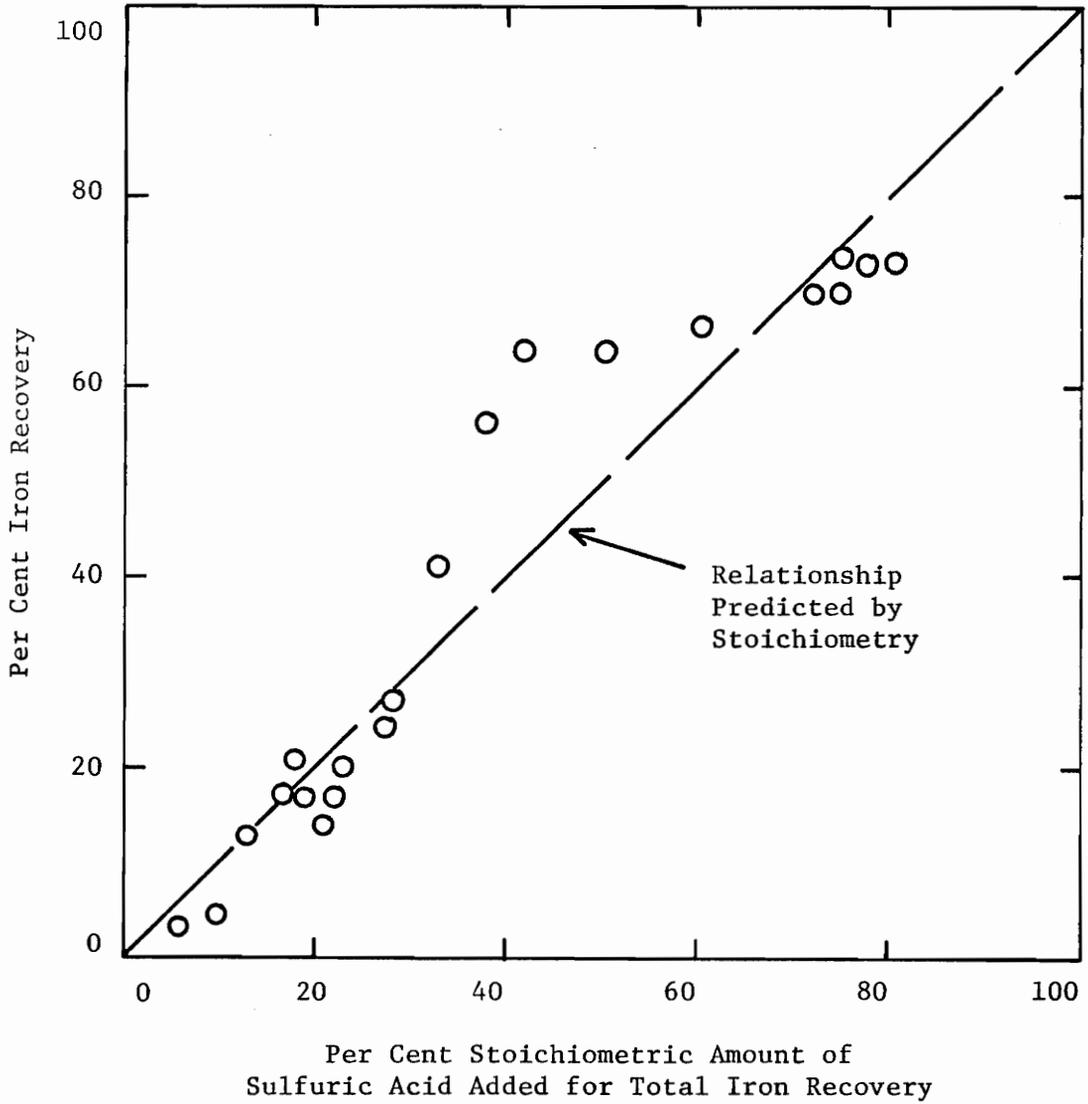


Figure 19. Per Cent Iron Recovered as a Function of Amount of Sulfuric Acid Added

The iron (III) equilibria developed by O'melia and Stumm (14), as shown in Figure 2, indicate that the solubilities of ferric hydroxide compounds decrease below a pH level of 2.7. Since the solubility of Fe^{3+} increases only slightly at low pH values, recovery might be expected to diminish as the pH is lowered. The optimum recovery pH value observed in this study was 1.4.

It is possible that a chemical reaction other than that given in Equation (4) may result in a better alignment of the data with the stoichiometric relationship. Surely there are a number of reactions that take place, and Equation (4) expresses only an approximate net reaction.

The maximum percentage of soluble iron, which was about 85 per cent of the total iron, transferred to the supernatant in recovery experiments was 74 per cent. This was achieved at a pH value of 0.85. Because iron recovery becomes a process of diminishing returns at very low pH values, it would not be desirable to operate a recovery process at such a low pH value. A pH of 1.5 can yield about 60 per cent recovery of soluble iron. In addition, all the iron adsorbed by colloidal particles, approximately 15 per cent of the total iron could be recycled. Thus, in a system operated at this pH, the coagulant is estimated to be 75 per cent recoverable.

Characteristics of the Remaining Sludge

The solids data obtained in the analysis of the sludge remaining after acid addition, as presented in Figure 12, indicate that the total solids content consistently increased with decreasing pH. The

increase was substantial when the pH was lowered to a value of 2.8, this effect being independent of the initial solids concentration of the sample. As mentioned earlier, the measured volatile portion of the remaining sludge solids ranged from 13 to 17 per cent, or somewhat less than the 15 to 20 per cent existing in the untreated sludge samples. This result indicates that some volatile matter was solubilized upon acid addition to the sludge.

In any practical coagulation recovery process, separation of the remaining solids from the supernatant following acidification must be accomplished expeditiously. The relationship found between the initial settling velocity and the pH of the remaining sludge is shown in Figure 10. As noted therein, the observed initial settling velocity increased with decreasing pH. Particularly favorable settling velocity was achieved at pH values below 2.0.

As noted in Figure 11, the final settled volume of the sludge decreased with decreasing pH. Independent of the initial solids concentration of the sample, the greatest reduction in volume occurred between pH values of 2.0 and 3.0. The per cent volume reduction is plotted against per cent iron recovered in Figure 20. These data reveal that sludge volume reduction due to acid addition occurred in stages. Initially, the volume decreased with no simultaneous recovery of iron. This effect appears to indicate that the acid was at first consumed by other materials in solution. Next, the per cent iron recovered increased dramatically with little accompanying volume reduction, perhaps signifying a release of adsorbed iron. When

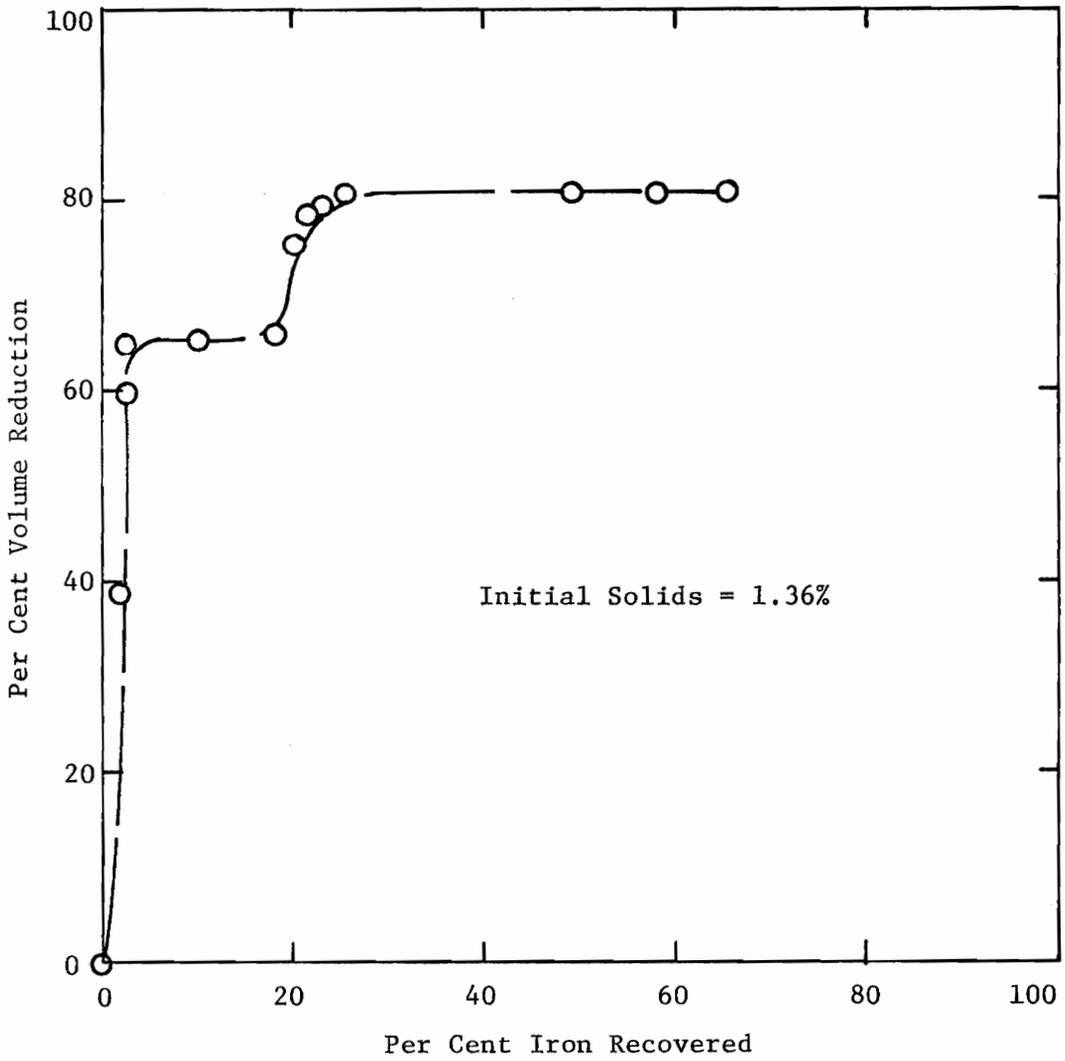


Figure 20. Per Cent Volume Reduction Versus Per Cent Iron Recovered

recovery reached 20 per cent, substantial volume reduction again occurred, and iron ceased to be dissolved. The author suspects that in this range the acid preferentially participates in destabilization reactions. Finally, sludge volume reduction no longer increased, and iron was optimally solubilized.

An economical process for iron recovery must result in a remaining sludge that can be readily dewatered. Figure 13 illustrates the decrease in specific resistance which occurred upon acidification of the sludge. The specific resistance reached a minimum at a pH value between 2.5 and 3.0. This pH corresponds to the pH at the point of inflection of the remaining sludge solids versus pH data given in Figure 12. It is reasonable to conclude that the improvement in filterability of the acidified sludge becomes limited by the increasing solids concentration.

Solids Characteristics of the Decanted Supernatant

The dissolved solids and conductivity data obtained in analyses of the supernatant samples are shown in Figure 14. As noted therein, a consistent increase in inorganic dissolved solids, as evidence by the conductivity data, occurred as the system pH decreased. This effect is attributed to the increase in Fe^{3+} and $\text{SO}_4^{=}$ ions in solution. However, volatile dissolved solids also increased with decreasing pH. An accumulation of organics in a recovery operation would no doubt be undesirable.

Coagulation Efficiency of the Recovered Iron

The effectiveness of the recovered coagulant is certainly of major importance in a recovery system. The coagulation data presented in Figure 15 show that the recovered iron was nearly as efficient as fresh iron in coagulation. The critical coagulation concentration of iron was less when recovered iron was used, although residual turbidity was greater.

It was suspected that the particulates in the recovered iron solution were responsible for the differences noted in the figure. Packham (29) described an inverse relationship between the optimum coagulant dose and the concentration of material in suspension, resulting from nucleation by the colloidal particles. The data given in Figure 17 appear to lend support to this phenomenon as an explanation of the results obtained using recovered iron. As noted therein, the difference between critical coagulation concentrations was greatest for low turbidity water, where the effect of additional solids would be most pronounced. In order to ascertain the effect of the suspended material, supernatant samples were filtered prior to coagulation tests. The results are shown in Figure 21. The filtered solution gave coagulation data similar to that obtained using the fresh reagent, indicating that suspended solids had indeed affected the efficiency of the recovered coagulant.

Perhaps the most distinct result observed in coagulation with recovered iron occurred at dosages greater than the critical coagulation concentration. The recovered coagulant tended to quickly

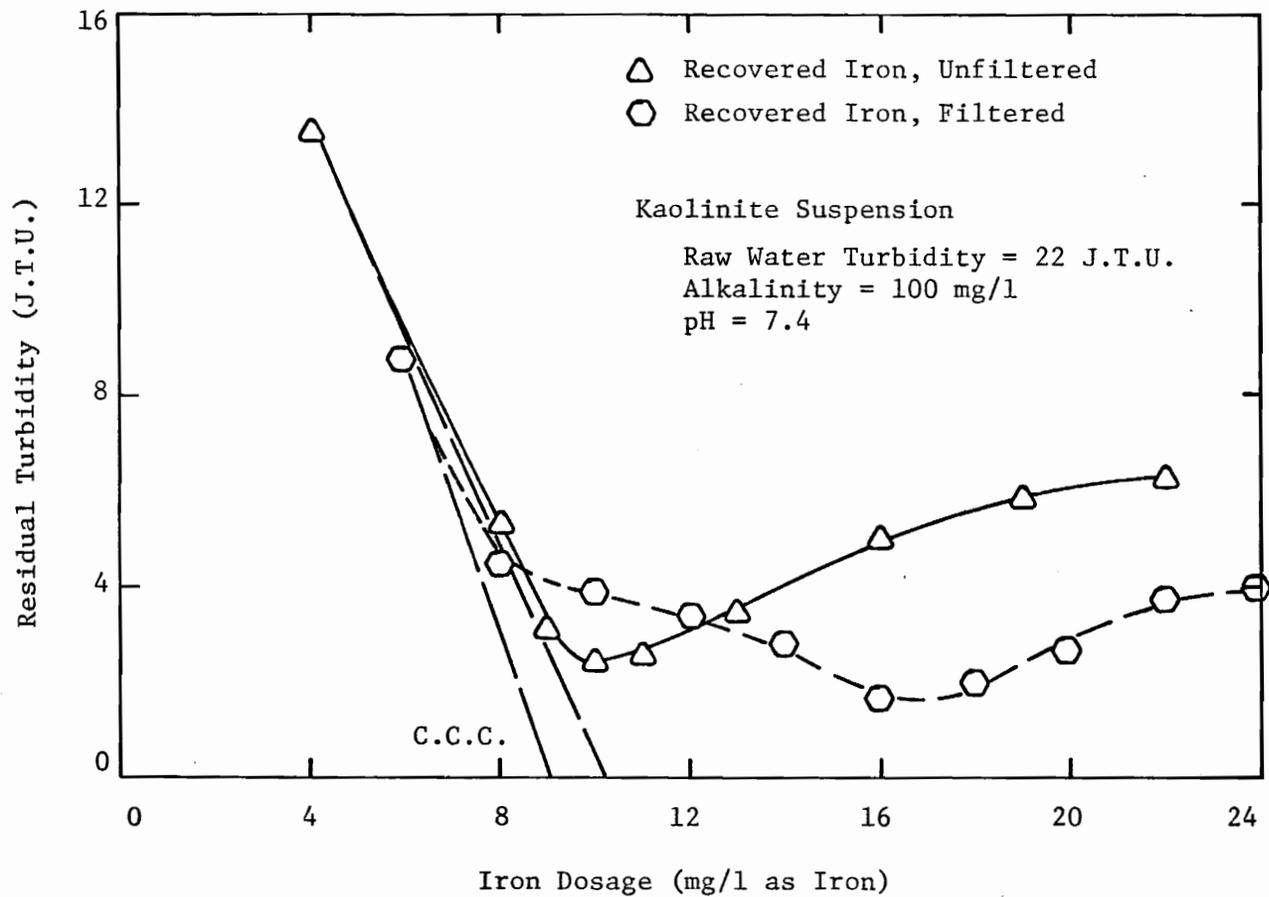


Figure 21. Residual Turbidity as a Function of Iron Dosage Using Filtered and Unfiltered Supernatant Samples

restablize the raw water samples at residual turbidity levels substantially higher than the minimum level attained. A possible explanation for this result is that the average positive charge of the iron species present in the recovered solution is higher than that of the fresh iron species. Because fresh coagulant would be used to replenish the recovered solution in an actual operation, it is anticipated that the effectiveness of the mixture would not be appreciably reduced from that of fresh ferric sulfate.

CHAPTER VI

CONCLUSIONS

The results of the investigation of iron recovery from water treatment sludge support the following conclusions:

1. The acidic method of iron recovery is technically feasible. Both sulfuric acid and hydrochloric acid can be used for this process, but for economic reasons sulfuric acid is superior.
2. Acid addition resulted in a dramatic reduction in sludge volume. However, volume reduction and iron recovery did not occur simultaneously at a given pH.
3. Acid addition greatly improved the settleability and the filterability of the remaining sludge.
4. The efficiency of recovered iron in coagulation of suspended solids is not appreciably reduced from that of fresh iron.

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VITA

Robert Kenneth Weeks, Jr., was born November 13, 1948, in Augusta, Georgia. In 1952 the family moved to Norfolk, Virginia, where he was educated in the public school system. He was graduated from Granby High School in June, 1966. In September of 1966 he entered Wake Forest University, Winston-Salem, North Carolina, where he was awarded the Bachelor of Science degree in Physics in June, 1970. After military service, he assumed a teaching position with the Chesapeake Public Schools.

After one year of teaching at the high school level, the author elected to continue his education. He entered Virginia Polytechnic Institute and State University in September of 1972 to pursue the Master of Science degree in Sanitary Engineering. Upon completion of requirements, he will assume a position with R. Kenneth Weeks Engineers, Norfolk, Virginia.

R. Kenneth Weeks, Jr.

IRON RECOVERY
FROM A REPRESENTATIVE
WATER TREATMENT PLANT SLUDGE

by

R. Kenneth Weeks, Jr.

(ABSTRACT)

Water treatment plant sludge disposal has become a timely problem to consider. While many methods of sludge treatment and disposal have been suggested, coagulant recovery may prove to be the most economical. This investigation was concerned with recovery of iron coagulant by chemical addition. Sulfuric acid, hydrochloric acid, and sodium hydroxide were added to a water treatment plant sludge containing precipitated iron. The acidic method of recovery proved to be technically feasible.

Iron dissolved upon addition of acid to the sludge. The concentration of soluble iron increased substantially when the pH was lowered to a level of 2.0 and then diminished at pH values below 1.0. Iron recovery appeared to generally follow the stoichiometry predicted.

Acid addition to the sludge resulted in an improvement in sludge dewatering properties. Settling rates increased consistently with greater amounts of acid added, causing substantial reductions in sludge volume. The filterability of the sludge was also improved. Specific resistance decreased as the pH was lowered, reaching a minimum value at a pH level of about 3.0, and then increased due to compaction.

Sludge volume reduction and iron recovery did not occur simultaneously at a given pH. It appeared that the acid acted to either solubilize the iron or to destabilize the colloidal particles, depending on the system pH.

The recovered iron proved to be superior to fresh iron for coagulation of low turbidity waters. The suspended solids in the recovered iron solutions appeared to aid in nucleation. While coagulation with fresh iron resulted in a lower raw water residual turbidity, the critical coagulation concentration was less when recovered coagulant was used.