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NUTRIENT REMOVAL FROM SECONDARY EFFLUENT BY ALUM FLOCCULATION AND LIME PRECIPITATION*

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Abstract—The removal of phosphorus and nitrogen compounds from biochemically treated wastewater effluents by alum flocculation and lime precipitation was investigated. Samples of secondary effluent were flocculated or precipitated in accord with conventional Jar Test procedures. The phosphorus removal was found to be highly pH dependent with an optimum pH of 5.57 ± 0.25. At this pH an alum dose of 250 mg/l. removed 95 per cent of total phosphorus, 55 per cent of the chemical oxygen demand, 60 per cent of the organic nitrogen, 25 per cent of the NO_3^--N and NO_2^--N, 17 per cent of the apparent ABS and none of the ammonia-N.

A dose of 600 mg/l. $Ca(OH)_2$ raised the pH of the sample to 11.0 and removed 99 per cent of the total phosphorus.

The estimated chemical costs for removal of 95 per cent of the phosphorus by lime and alum were 32 and 73 $MG (dollars per million gallons), respectively.

1. INTRODUCTION

ONE of the major problems caused by discharging biochemically treated wastewater effluents into lakes and streams is the eutrophication of these waters. This eutrophication is generally attributed to the discharge of fertilizing elements such as phosphorus and nitrogen. The removal of phosphorus and nitrogen from sewage effluents appears to be a practical way of limiting algal blooms in the receiving waters.

The removal of these elements by biochemical or chemical means appears to be feasible. Various laboratory and field studies, SAWYER(1944), FENG (1950), WUHRMANN (1960), LUDZACK and ETTINGER (1962), have shown that the biochemical wastewater treatment processes can be modified such that significant removal of nitrogen and/or phosphorus can be attained.

Chemical precipitation or flocculation of biochemically treated wastewater effluents have been investigated by RUDOLF (1947), OWEN (1953), LEA *et al.* (1954). These investigations have been discussed by STUMM (1962). In general these investigations have shown that significant amounts of phosphorus can be removed from secondary effluents by alum and iron flocculation or lime precipitation. ROHLICH (1963) has recently reviewed methods of phosphorus and nitrogen removal from sewage plant effluent.

In the past 10-15 years since these studies were conducted, the composition of treated sewage effluent has changed considerably. The proportion of the condensed phosphate in the total phosphorus as well as the total phosphorus has increased. In addition, the concentration of organics, and in particular, surfactants, e.g. ABS, has increased significantly during this period.

Since there is growing concern with respect to eutrophication of natural waters by

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wastewater effluents, and since the character of the present-day effluents has changed since the previous chemical removal studies were conducted, a detailed investigation on the removal of nutrients from secondary effluents by alum flocculation and lime precipitation was initiated.

2. EXPERIMENTAL PROCEDURE

2.1. *Samples*

Grab samples of final effluent from the activated sludge part of the Nine Springs Madison, Wisconsin Metropolitan Sewage Treatment Plant were used in all studies. This activated sludge plant treats domestic sewage plus meat packing wastes. These samples were immediately transported to the laboratory for processing. In order to minimize the effect of varying amounts of suspended matter in the effluent on the flocculation process, the effluent was vacuum filtered through Whatman No. 1 filter paper. The temperature of the samples was brought to 20±1° C. Six 1 1. samples of the filtered effluent were taken per run, one of which served as the control. Where necessary, pH adjustment was made on each sample by addition of 1.0 N H_2SO_4 or 1.0 N NaOH using Bechman model H-2 pH meter.

2.2. *Typical jar test*

Stirring apparatus for the Jar Test consisted of a gang stirrer (Phipps and Bird, Inc., Richmond, Va.). This stirrer enabled the simultaneous stirring of six samples at any speed up to 200 rpm. Six beakers each of two liter capacity were used as jars for each experiment. All experiments were conducted at 20±1° C.

A freshly prepared alum solution using reagent grade $AL_2(SO_4)_3.18\ H_2O$ was placed in each of five test tubes. Coagulant was added to each one liter sample of the effluent. The sample was mixed for 12 minutes at about 200 rpm followed with slow stirring at 20 rpm for 12 min. After stirring, the floc was allowed to settle for 30 min and the supernatant from each jar was filtered through Whatman No. 1 filter paper.

The filtrate from the treated samples and the control was used for the various analyses described below.

2.3. *Analysis of samples*

Unless otherwise stated, reagent (ACS) grade chemicals were used in this investigation. All chemical analyses were conducted in accord with procedures described by Standard Methods (1960).

The orthophosphate content of the sample is defined as that phosphorus that yields a positive color with the ammonium molybdate-strong acid solution using stannous chloride as the reducing agent. The total phosphorus is defined for this investigation as the sum of the orthophosphate plus phosphorus compounds that are hydrolyzed to orthophosphate by boiling the sample with acid in an autoclave at 15 psi for 30 min. The apparent ABS was determined by the "methylene blue method".

3. EXPERIMENTAL STUDIES AND RESULTS

The total amount of plant nutrients and other pollutants present in a sewage plant effluent is subjected to seasonal, daily, and hourly variation. TABLE 1 summarizes the composition and variability of the effluent under study in this investigation.

The reported percentage removals in this study refer to the concentration of the variable in the sample as compared to the concentration in the control.

Selected studies on the controls have shown that about 15 per cent of the total phosphorus is removed by the preliminary filtration of the sample through Whatman No. 1 filter paper. These results indicate that a significant amount of the phosphorus present in the treated effluent is associated with easily filterable particulate matter. Additional phosphorus removal was observed in the stirred, settled and refiltered control. The mean overall percentage removal of ortho- and total phosphate in the controls, based on their original concentration in the effluent, was 9.6 and 22 per cent respectively.

3.1. *Effect of alum dose on removal of nutrients*

One of the first investigations was a study of the effect of alum dose on the removal of various nutrients by flocculation. Filtered effluent without pH adjustment (pH 8.0)

TABLE 1. COMPOSITION OF THE TREATED EFFLUENT-ACTIVATED SLUDGE PLANT, MADISON, WISCONSIN, SUMMER AND FALL 1962

	Concentration mean	Concentration range
Total Phosphorus—mg P/l.	7.6	3.1–11.2
Orthophosphate—mg P/l.	6.5	2.1–9.5
pH	—	7.95–8.05
NO_2^-—N mg N/l.	trace	—
NO_3^-—N mg N/l.	trace	—
Ammonia-N—mg N/l.	22.1	18.7–24.8
Organic-N—mg N/l.	4.3	3.2–5.6
Apparent ABS—mg/l.	3.7	3.0–4.3
Alkalinity mg/l. as $CaCO_3$	—	440–460
Chemical Oxygen Demand mg/l.	91	71–102
Chloride mg Cl^-/l.	310	242–335
Calcium mg Ca^{++}/l.	71	45–82
Flow, million gallons per day	12.6	9.75–16.2

was flocculated using alum doses of 100, 130, 180, 200 and 250 mg/l. The percentage removals of total phosphorus, nitrate-N, nitrite-N, organic-N and COD versus alum dose are plotted in FIGS. 1, 2, 3, 4 and 5, respectively. For the purpose of comparison, the data for the removal of various nutrients for samples that had their initial pH adjusted to 6.0 are shown on the same figures. In each of these figures, the heavy line represents the average of the results obtained from replicate runs on different samples of effluent. The band between the thin lines shows the range of values obtained. Examination of FIGS. 1-5 shows that in any one run with a single sample of effluent there is a well-defined trend for the removal of the material as a function of alum dose. However, marked differences are observed when samples of effluent taken on different days are treated in an identical manner. For example, FIG. 1 shows that for runs 10 and 11 with pH adjusted to 6.0, an alum dose of 250 mg/l. removed 85 and 96 per cent of the total phosphorus. This variability observed between runs can possibly be explained by a variation in the concentrations of one or more unknown components of the treated effluent. Part of the variation in these two runs is attributed to the different total phosphorus concentrations. The effect of the phosphorus concentration on the alum dose will be discussed in a subsequent section of this paper.

Examination of FIG. 1 shows that significantly better removals of total phosphorus were achieved when the initial pH of the effluent was adjusted to pH 6.0 as compared to the removals at its natural pH of 8.0. Also, these data show that at pH 6.0 an alum dose of approximately 200 mg/l. is needed to remove 90 per cent of the total phosphorus, while only 80 per cent removal was attained at pH 8.0 with an alum dose of 250 mg/l. These experiments have also shown that the removals of orthophosphate followed a similar pattern as was observed for total phosphorus.

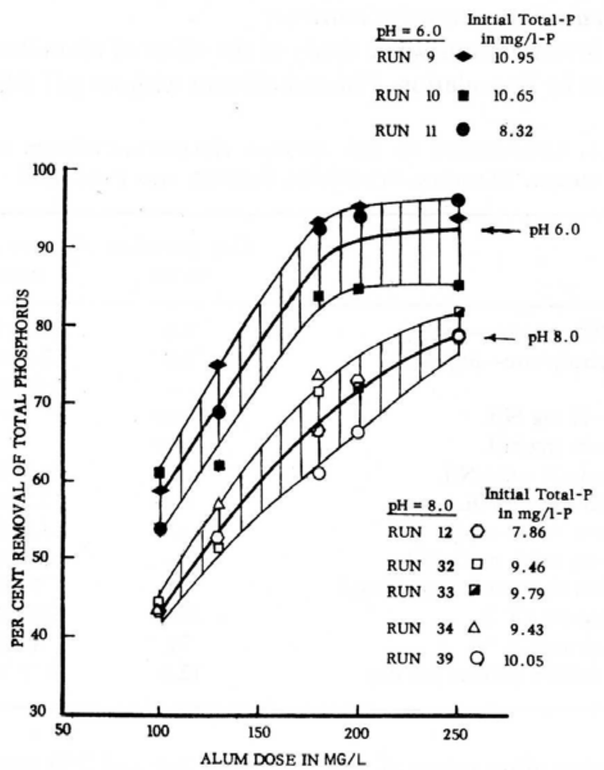

FIG. 1. Removal of total phosphorus by alum flocculation.

The removal of N-compounds by alum flocculation is presented in FIGS. 2, 3 and 4. Examination of these figures shows that the removal of nitrate and nitrite nitrogen with an alum dose of 200 mg/l. was on the order of 10-20 per cent, while at this same dose approximately 50 per cent of the organic nitrogen was removed. The studies on the removal of NO_3^- and NO_2^- are based on effluent samples that were fortified with these chemicals since only traces of these compounds were present in the effluent. Studies on the removal of ammonia-N have shown that no removal was achieved at pH 6.0 or 8.0 with an alum dose of 250 mg/l.

The removals of COD are presented in FIG. 5. Examination of this figure shows that significantly better removal of COD was achieved when the initial pH was adjusted to 6.0 as compared with its natural pH of 8.0. Also it is shown that approximately 50 per

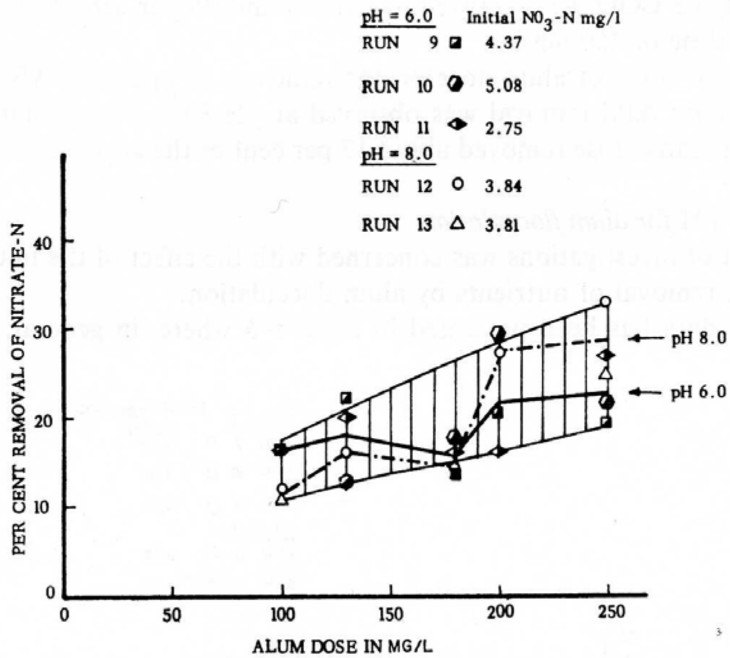

FIG. 2. Removal of nitrate nitrogen by alum flocculation.

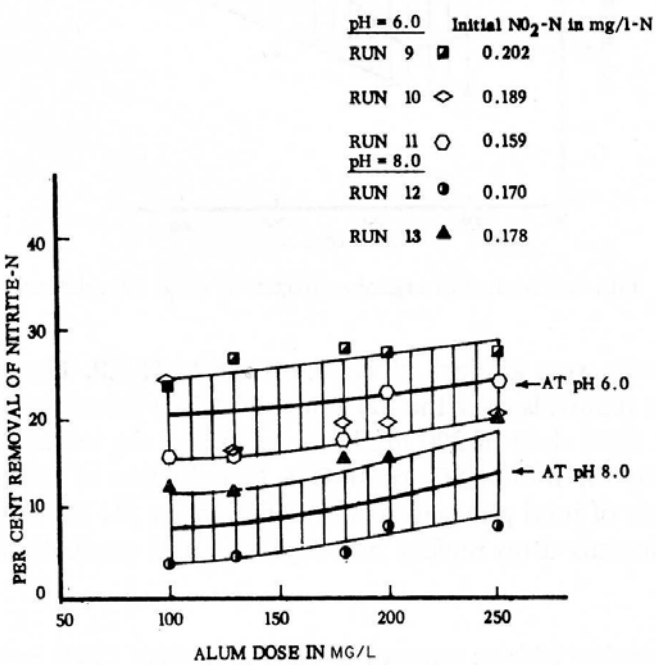

FIG. 3. Removal of nitrite nitrogen by alum flocculation.

cent of the COD was removed at pH 6.0 and 25 per cent removal at pH 8.0 with an alum dose of 250 mg/l.

Studies on the effect of alum dose on the removals of apparent ABS have shown that essentially no ABS removal was observed at pH 8.0 with an alum dose of 250 mg/I., while the same dose removed about 17 per cent of the apparent ABS at pH 6.0.

3.2. *Optimum* pH *for alum flocculation*

The next set of investigations was concerned with the effect of the initial pH of the sample on the removal of nutrients by alum flocculation.

Part of this data has been presented in FIGS. 1-5 where, in general, it was found that better

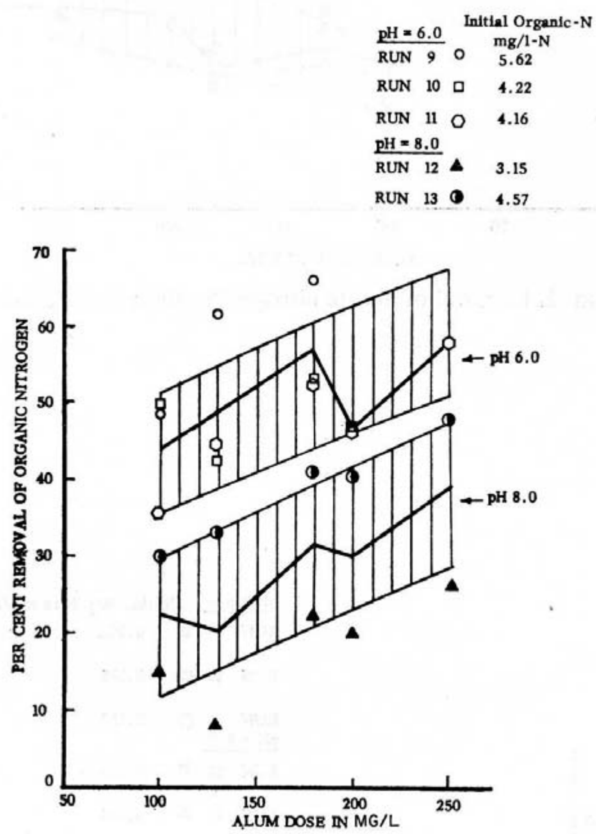

FIG. 4. Removal of organic nitrogen by alum flocculation.

removals were attained at pH 6.0 than at pH 8.0. The data obtained for total phosphorus removals over the pH range of 4.5 to 9.0 are presented in FIG. 6. In these runs an alum dose of 200 mg/l. was used and the initial pH of the sample was adjusted by addition of H_2SO_4 or NaOH. Examination of this figure shows that optimum removals of total phosphorus were obtained at pH 5.5 to 6.0. As would be expected, the removals drop rapidly below pH 5.5 and gradually in the pH 6.0 to 9.0 region.

3.3. *Effect of coagulant aids on removal of phosphorus and COD by alum flocculation*

Since the clarification of waters by flocculation is often improved by addition of coagulant aids, the effect of Bentoinite and Separan NP-10 on the removal of phosphorus and COD was investigated.

In general these experiments showed that the addition of 300 mg/l. of Bentonite and up to 25 mg/l. of Separan NP-10 (Dow Chemical Co.) at an alum dose of 100 mg/l. do not markedly improve the removals of the various nutrients.

3.4. *Effect of phosphorus concentration on its removal by alum flocculation*

Some of the studies reported thus far point to the general observation that some of the variability between different samples of effluent could possibly be explained on the basis of the varying amounts of phosphorus in the sample. It was decided that this

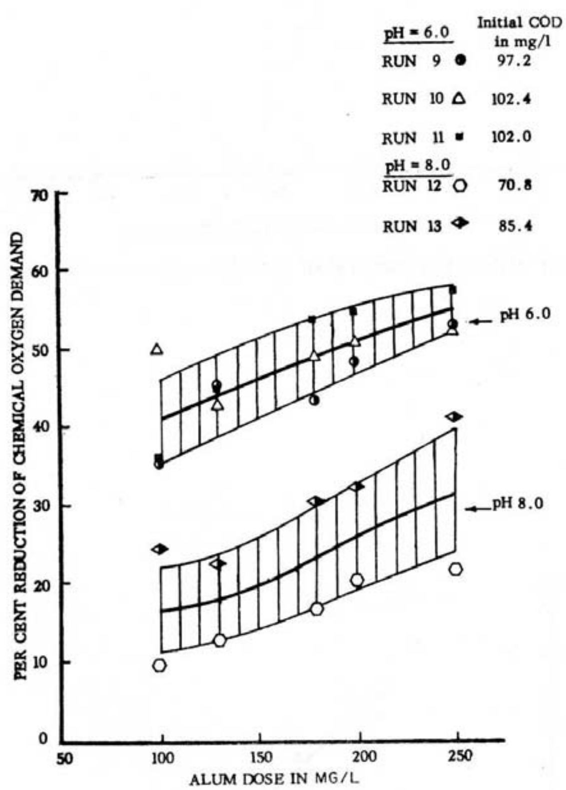

FIG. 5. Removal of chemical oxygen demand by alum flocculation.

observation could be confirmed by taking a sample of effluent and varying the phosphorus content of the sample by adding phosphates to the sample.

Filtered effluent at its natural pH of 8.0 with added amounts of phosphate was flocculated with 200 ppm of alum. Most of the added phosphorus was in the form of KH_2PO_4. Percentage reduction of total phosphorus versus initial total phosphorus concentration of the sample is plotted in FIG. 7.

Examination of FIG. 7 shows that the percentage removal of total phosphorus decreases almost linearly with increasing initial phosphorus concentration of the sample. An increase in total phosphorus concentration from 9.0 to 26.0 mg/1.-P decreases its removal from about 75 to 44 per cent. Every 1.0 mg/1.-P increase in total

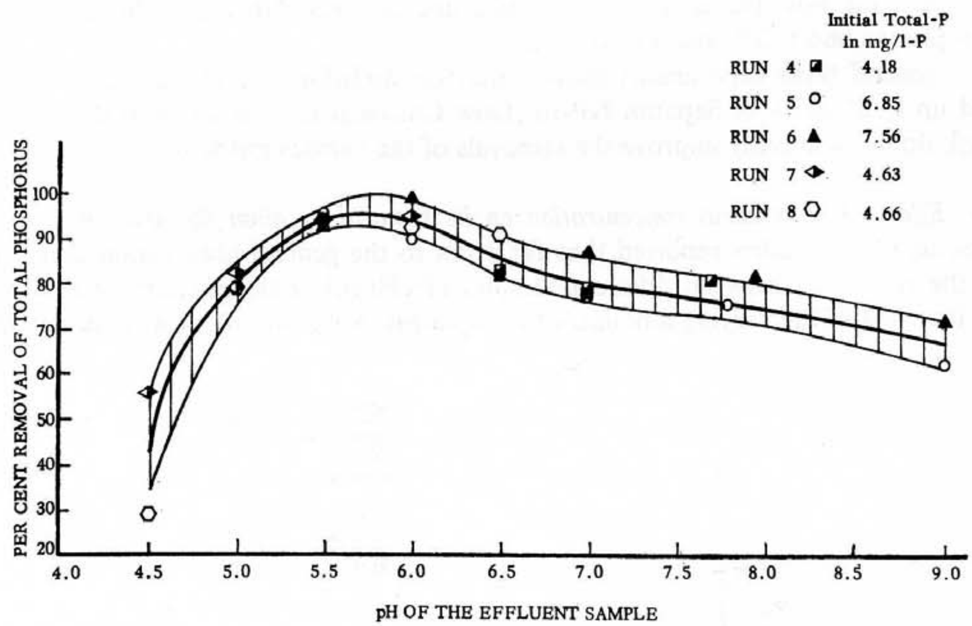

FIG. 6. Effect of pH on the removal of total phosphorus with 200 mg/l. of alum.

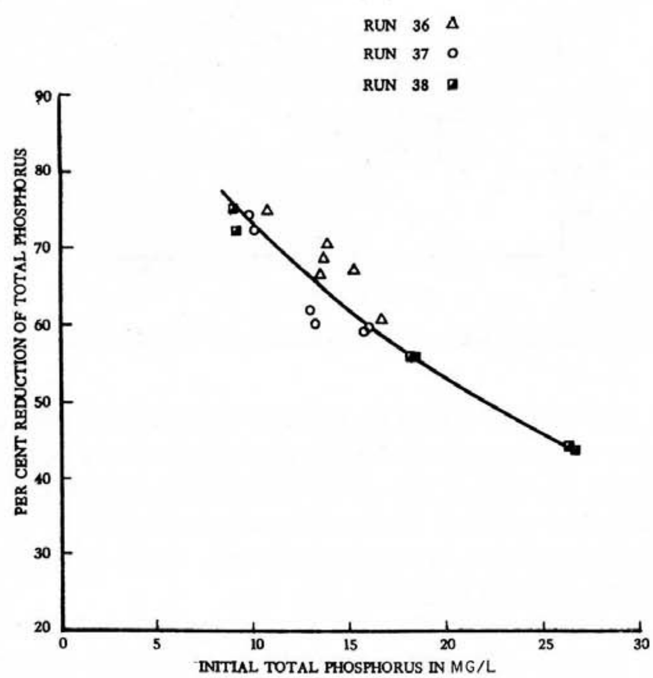

FIG. 7. Effect of initial total phosphorus concentration on its removal by alum flocculation with 200 mg/l. of alum.

phosphorus decreased its removal by approximately 2 per cent. From FIG. 1 it is noted that for alum flocculation of the sample at pH 8.0 in the alum dose range of 100 to 200 ppm, an alum dose of about 6 mg/l. is needed for every 2 per cent increase in the removal of total phosphorus. Thus, every 1 mg/1.-P increase in total phosphorus is equivalent to about 6 mg/l. increase of alum dose.

3.5. Removal of phosphorus by raising pH *of the effluent*

While studying the effect of pH of the effluent sample on the removal of phosphorus with 100 ppm of alum plus 25 ppm of Separan NP-10, it was observed that at pH values above 10.0 better percentage removal of phosphorus is achieved than at pH 5.5.

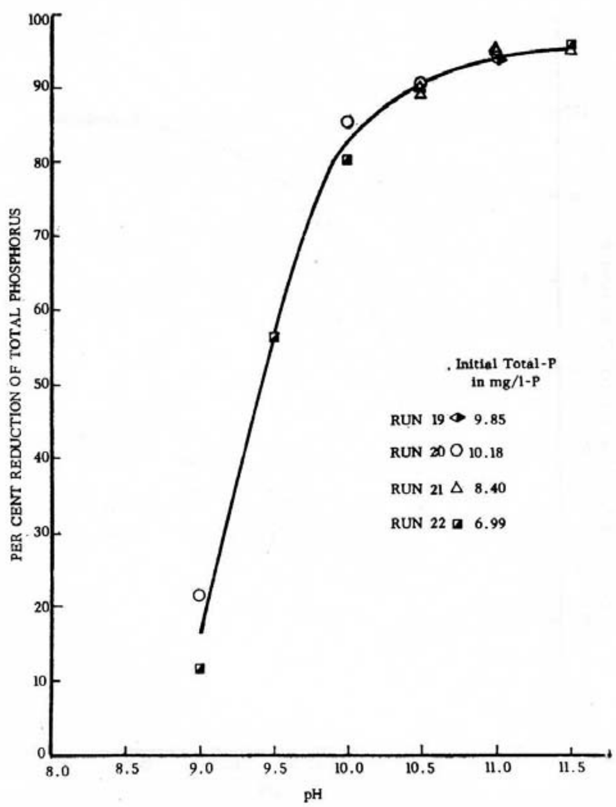

FIG. 8. Removal of total phosphorus by pH adjustment with NaOH.

Because aluminum hydroxide floc is fairly soluble at such high pH values, it was felt that the use of alum was unnecessary at such high pH values. The removal of phosphorus from the effluent was then studied by simply raising the pH of the sample with the use of sodium hydroxide or lime as described in the following paragraphs.

3.5.1. *Removal of phosphorus by raising* pH *with sodium hydroxide.* Effluent from the activated sludge plant was placed in six jars. The pH of the effluent in these jars was adjusted from 9.0 to 11.5 with 1.0 N NaOH. These samples were then subjected to standard Jar Tests. Percentage removal of total phosphorus versus pH is plotted in FIG. 8.

Examination of FIG. 8 shows that percentage removal of phosphorus increases linearly with increase in pH up to a pH of 10.0. From pH 10.0 to 11.0, the gain in percentage removal per unit increase in pH, decreases appreciably. A further increase in pH of the sample does not improve percentage removal of phosphorus by a significant amount. At pH 11.0 about 95 per cent phosphorus removal is obtained. From the data previously presented, it is observed that unlike alum flocculation, the initial phosphorus concentration of the sample seems to have almost no effect on its percentage removal by raising the pH.

3.5.2. *Removal of phosphorus by raising* pH *by lime.* The high cost of NaOH leads to consideration of the use of lime for the precipitation of calcium phosphates (hydroxy-

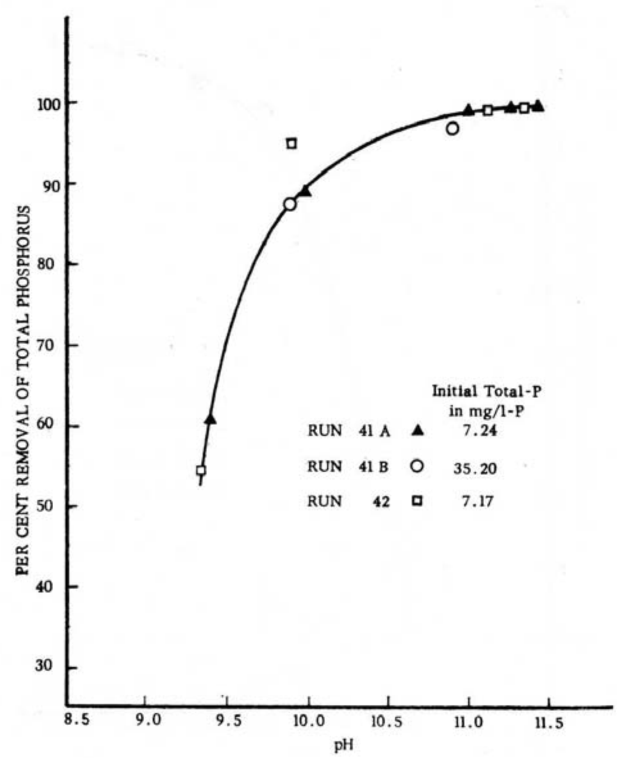

FIG. 9. Removal of total phosphorus by pH adjustment with lime.

lapatite). Powdered $Ca(OH)_2$, U.S.P. grade, was added to filtered effluent in doses of 200, 400, 600, 700 and 800 mg/l. Each jar was stirred and its pH was measured. These five jars with lime additions and the control were subjected to the Jar Test. Percentage removal of total phosphorus versus pH of the effluent after adding lime is plotted in FIG. 9.

Examination of FIGS. 8 and 9 shows that the percentage of removals of phosphorus obtained by raising the pH of the effluent with NaOH or with lime follows approximately the same pattern of removal with increasing pH. However, at any pH value slightly better removals are obtained with lime than with NaOH. Raising the pH to 11.0 with lime removed about 99 per cent of the total phosphorus. At this pH, using NaOH for pH adjustment, about 95 per cent of the total phosphorus was removed.

OWEN (1953) has reported that lime at a dose of 720 mg/l. at a pH of 11.0 removed about 95 per cent of the phosphorus from samples of effluent from a low-rate trickling filter treatment plant.

3.6. *Comparison of different chemical sludges*

In selecting any of the chemical processes for use on a plant scale, one has to consider not only its cost and effectiveness in removing nutrients, but also the volume and the nature of the sludge produced.

To determine the volume and the settleability of any sludge, the 1 liter effluent sample that had been flocculated was thoroughly mixed, and transferred to a 1 liter

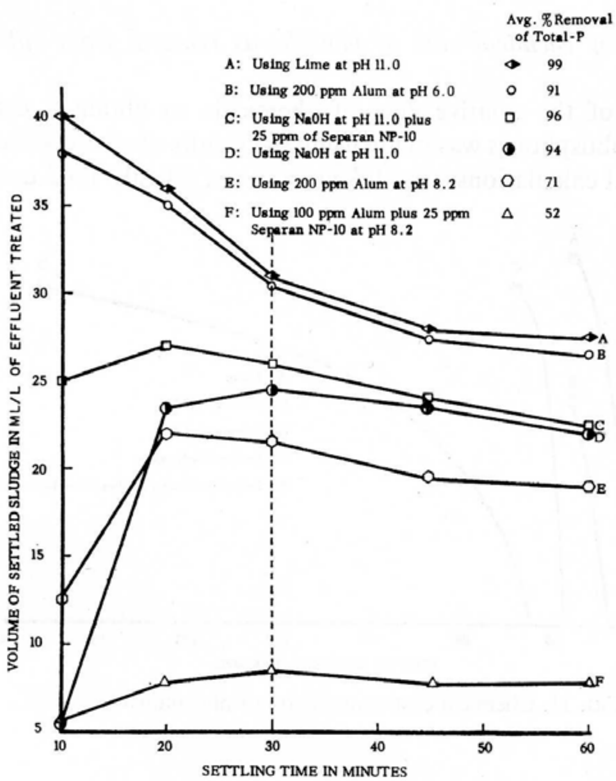

FIG. 10. Settleability of sludges.

Imhoff Cone. A duplicate of each chemical dose was run in parallel. The volume occupied by the sludge was noted at intervals of 10, 20, 30, 45 and 60 min. After taking a sludge volume reading, the sides of the cone were gently stirred by spinning to dislodge the floc particles from the sides of the cone. The average of these volume readings against time have been plotted in FIG. 10.

Examination of FIG. 10 shows that in all the six investigations reported, most of the sludge settled in 30 min. The volume of the sludge that settled in 60 min varied from 8 to 27 ml./1. of the effluent treated. In general, the larger volume of sludge was associated with the sample showing the greater percentage removals of phosphate. An alum dose of 200 mg/l. at pH 6.0 and 600 mg/l. of $Ca(OH)_2$ at pH 11.0 gave approximately equal volumes of sludge and of similar settling rates. The treatment of effluent with 600 mg/l. of $Ca(OH)_2$ at pH 11.0 resulted in the maximum volume of sludge. Also, with this dose of lime the maximum phosphorus removal is obtained. In general, slightly better removals of phosphorus from effluent samples treated with NaOH at pH 11.0 are obtained than with 200 mg/l. of alum at pH 6.0, but the volume of sludge in the former treatment is 22 m1/1. as compared to 27 m1/1. with 200 mg/l. alum at pH 6.0. The use of 25 mg/l. of Separan NP-10 at pH 11.0 (obtained with NaOH) results in a slight increase in the volume of sludge. A minimum volume of about 8 m1/1. of sludge is obtained with 100 mg/l. of alum plus 25 mg/l. of Separan NP-10 at pH 8.0. This combination of chemicals also results in the minimum percentage removal of phosphorus out of the six conditions compared in this investigation.

3.7. *Comparison of chemical cost of phosphorus removal from effluent by different chemicals*

A calculation of the relative cost of chemicals to obtain a certain percentage removal of total phosphorus was made using the results obtained with these chemicals. Prices used for all calculations were taken from the 30 July 1962 quarterly report on

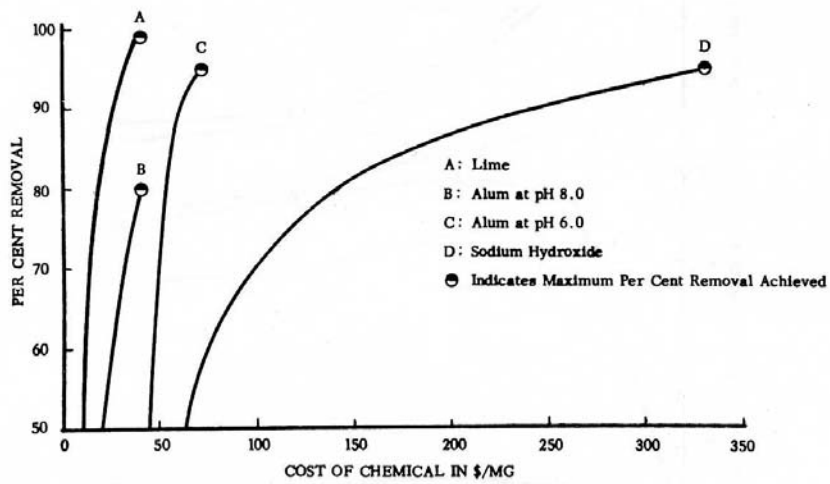

FIG. 11. Chemical cost comparison of phosphorus removal.

current prices, published by *Chemical and Engineering News.* Percentage removal of total phosphate against chemical cost in $ /MG (dollars per million gallons) is plotted in FIG. 11.

Examination of FIG. 11 shows that to obtain any significant percentage removal of total phosphorus, lime is the cheapest, and NaOH is the most expensive chemical. To obtain a removal of 50 per cent in the total phosphate with lime, alum at pH 8.0, alum at pH 6·0, and NaOH, the respective chemical costs are 11, 21, 44 and 63 $ /MG. At 80 per cent removal which is the maximum percentage removal obtained with alum at pH 8.0, the corresponding cost figures are 20, 42, 54 and 138 $ /MG. At 95 per cent removal which is the maximum obtained with alum at pH 6.0 and with NaOH, the cost of lime, alum, and NaOH are 32, 73 and 330 $ /MG respectively. A removal of 99 per cent was obtained with the lime treatment only and its chemical cost is 41 $ /MG.

Subsequent to the completion of this study, two papers have been published by

HENRIKSEN (1962; 1963) on the mechanism of the removal of phosphate by various coagulants.

4. SUMMARY AND CONCLUSIONS

Lime appears to be the most suitable chemical for the removal of soluble phosphorus from the effluent of the activated sludge treatment plant at Nine Springs, Madison, because the maximum percentage removal is achieved at the minimum chemical cost. A dose of 600 ppm of $Ca(OH)_2$ raises the effluent to a pH of 11.0 and gives about 90 per cent removal of total phosphorus at a chemical cost of approximately 40 $/MG. The percentage removal of phosphorus by lime precipitation is independent of the initial phosphate concentration of the sample in the concentration range of 7 to 35 mg/1.-P. A dose of 400 ppm of lime raises the pH of the effluent sample to a pH value of 10.0, and precipitates approximately 10 per cent of the total phosphorus. This dose of lime produces the least volume of sludge, which is 6 to 7 ml./I., after one hour of settling in an Imhoff Cone.

The major disadvantage for the use of lime is the high pH of the effluent. In general, it was found that the final pH of the effluent was about 1 pH unit less than its initial pH in the pH 9-11 range. pH adjustment may have to be considered in those cases where the buffer capacity of the receiving water is not adequate.

The removal of soluble phosphorus by alum flocculation appears to be the second choice on the basis of the chemical cost. The optimum pH zone for alum flocculation of effluent from the activated sludge treatment plant is 5.75+0.25. An alum dose of 250 ppm removes approximately 78 per cent of total phosphorus from the effluent at its natural pH of 8.0, but the same dose removes about 94 per cent of total phosphorus from effluent samples adjusted to pH 6.0. At any alum dose the percentage removal of total phosphorus from effluent samples at pH 8.0 decreases with the increase in the initial phosphate concentration of the sample. In general, at any specific chemical dose, the percentage removals of orthophosphate and total phosphorus obtained are approximately equal.

The use of coagulating aids such as Bentonite and Separan NP-10 increase the removal of phosphorus. But the high doses required offset the benefit derived from their use in conjunction with alum. In general, the addition of Separan increases the size and settleability of the floc, but also increases the COD and organic-N concentration of the sample.

A reduction of about 55 per cent in the COD concentration of an effluent sample is obtained by using 250 ppm of alum with the effluent sample adjusted to pH 6.0.

A removal of approximately 60 per cent in the concentration of organic-N is obtained by using 250 ppm of alum with the effluent sample adjusted to pH 6.0.

The concentration of nitrites or nitrates of an effluent sample can be reduced by about 25 per cent by flocculating the sample, adjusted to pH 6.0, with 250 ppm of alum.

Alum flocculation of effluent samples with an alum dose up to 250 ppm does not change the concentration of chlorides or ammonia-N by a significant amount. However, approximately 20 per cent of ammonia-N is driven off when the pH of the effluent sample is increased to a pH value of 11.0.

ABS is not significantly removed by alum flocculation of effluent samples at pH 8.0; however, a removal of about 17 per cent in the concentration of apparent ABS is obtained by using 200 ppm of alum with the effluent adjusted to pH 6.0.

ROHLICH (1963) proposed an alum recovery method in which a considerable reduction in chemical costs could be achieved. Since some of the lime demand is the result of a reaction of the calcium with the carbonate species in the effluent, it is conceivable that a lime recovery method could be developed whereby the chemical costs could be reduced. Studies are under way on the feasibility of lime recovery.

Acknowledgements—The authors wish to acknowledge the support given to this investigation by the U.S. Agency for International Development, the Public Health Service Water Chemistry Training Grant No. ITI-WP-22-01, and the Department of Civil Engineering and Engineering Experiment Station of the University of Wisconsin. Also, special recognition is given to WAYNE GARDNER for performing many of the analyses and also the cooperation of the Madison Metropolitan Sanitary District was gratefully appreciated.

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