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THE USE OF MAGNESIUM FOR THE CLARIFICATION
OF ALKALINE INDUSTRIAL WASTES

A THESIS

Presented to
the Faculty of the Graduate Division
by
Olen Eben Morgan

In Partial Fulfillment
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THE USE OF MAGNESIUM FOR THE CLARIFICATION
OF ALKALINE INDUSTRIAL WASTES

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TABLE OF CONTENTS

	Page
ACKNOWLEDGMENTS	ii
LIST OF TABLES	iv
LIST OF ILLUSTRATIONS	v
SUMMARY	vi
Chapter	
I. INTRODUCTION	1
II. LABORATORY INVESTIGATIONS	3
III. PILOT PLANT CONSTRUCTION AND OPERATION	13
IV. CONCLUSIONS AND RECOMMENDATIONS	25
BIBLIOGRAPHY	28

LIST OF TABLES

Table	Page
1. Cost Comparison for Treating Waste	8
2. Clarification of Waste Obtained in the Laboratory	9
3. Clarification of Waste Obtained with the Pilot Plant	24

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LIST OF ILLUSTRATIONS

Figure	Page
1. pH Required to Dissolve the Magnesium Hydroxide	6
2. Daily Gas Production of the Digester	11
3. Daily pH's of the Digestion Mixture	12
4. Flow Diagram of Pilot Plant	14
5. Picture of the Pilot Plant	15
6. Picture of the Mixing System	17
7. Picture of the Constant Head Orifice	18
8. Picture of the Dual Chemical Feed Pump	19

SUMMARY

The purpose of this thesis is to develop a method to treat alkaline industrial wastes economically and efficiently. The method though developed for the treatment of textile waste is applicable to most alkaline wastes.

The fact that most chemical methods of treating industrial wastes require large quantities of coagulating chemicals and produce large volumes of sludge makes them expensive. The method developed in this thesis, however, reduces the quantity of the coagulating chemical required and reduces the large volume of the sludge produced by dissolving the coagulating chemical and thus enables it to be reused. By reducing these paramount cost factors, this method becomes more economical than most methods presently used.

The method consists of precipitating the hydroxide ion already in the alkaline waste with magnesium. A solution containing dissolved magnesium ions is mixed with the alkaline waste, giving a magnesium hydroxide precipitate. This magnesium hydroxide precipitate entraps the solids in the waste and leaves a clarified effluent.

After sedimentation, the magnesium hydroxide-sludge mixture is drawn off and mixed with sulfuric acid to a pH of 7.0 or below. The sulfuric acid dissolves the magnesium hydroxide and leaves only the insoluble solids from the original waste. When these solids settle, the dissolved magnesium supernatant is remixed with more raw waste and thus completes the cycle.

The method was developed in the laboratory to find the optimum

amounts of chemicals required, the amount of sludge produced, and the amount of clarification obtained. Next, a pilot plant was constructed at the Dundee Mills of Griffin, Georgia, actually to test the method.

The results show that alkaline waste can economically be treated by this method. The chemicals required cost only about one-fifth the amount that chemicals required in presently used methods cost. The sludge volume, likewise, was reduced to approximately one-tenth of the volume of sludge before redissolving of the magnesium floc.

The amount of clarification obtained was as follows: 38-64 per cent reduction in the five-day Biochemical Oxygen Demand; 43-67 per cent reduction in the Chemical Oxygen Demand; 82-94 per cent reduction in the turbidity; and 30-70 per cent reduction in the color.

The results show that the weakness in the process is the long sedimentation period required for the magnesium floc to settle. This long sedimentation period must be taken into consideration in the design of the sedimentation basin.

CHAPTER I

INTRODUCTION

The problem of alkaline industrial wastes has long plagued the textile industry, pulp mills, chemical plants, tanneries, and laundries. This waste is usually highly polluttional and very toxic to fish and other biological life. Nevertheless, the waste is commonly discharged into receiving streams without prior treatment. The reason for not treating this waste is not entirely the lack of restrictive pollution laws but, at least in part, the high cost and accompanying operational difficulties which exist in the present treatment methods (1). Therefore, it was believed that the development of a more efficacious and more economical method to treat this type of waste should be developed.

Chemical methods have long been used to treat alkaline waste, with alum being the most common precipitating agent. Other chemicals sometimes used as the precipitating agent consist of chlorinated copperas, ferric chloride, ferric sulphate, and ferrous sulphate (2).

For neutralization, the most inexpensive commercially available acid to use is sulphuric acid (3). However, flue gas has been successfully used to reduce the pH, but at a high pumping cost, since flue gas contains only 12-14 per cent carbon dioxide and requires about double the theoretical amount of this gas to obtain the desired pH 8.5, (4).

With most chemical treatment methods the cost of treatment usually is exorbitant because of the enormous amounts of chemicals required and the voluminous amounts of sludge produced that must be disposed of. Nevertheless, chemical treatment methods have an advantage over biological

treatment methods in that they are insensitive to toxic material, flexible because the process can be used when stream conditions demand and, in general, more economical (2).

Biological treatment, too, has long been used to handle alkaline waste. This treatment consisted of mixing the waste with domestic sewage or adjusting the pH with an acid to make the waste amenable to biological treatment. The biological method most in vogue is that which uses the high rate trickling filter and the activated sludge process (5). These two processes, however, have been only partially successful since an enormous domestic flow or an expensive amount of acid for neutralization is required in either.

Since there was not at the beginning of this study a very economical method to treat this alkaline industrial waste, the purpose of this investigation was to try to develop a feasible and an economical method that had the flexibility of a chemical method and that would produce a reasonably clarified effluent.

CHAPTER II

LABORATORY INVESTIGATIONS

Equipment.--The equipment used in the laboratory investigation consisted of a stirrer used in the Standard Jar Test. This piece of equipment is essentially a unit containing six small individual stirrers, geared to mix very slowly. Besides the Standard Jar Test Apparatus, the other equipment consisted of glassware normally found in a chemical laboratory, such as beakers, pipettes, and graduate cylinders.

Procedure.--The first and paramount fact to ascertain is the amount of magnesium required to produce a relatively clear effluent. Since it was decided that magnesium sulphate was to be the chemical used, it was tested first.

A series of beakers, each containing one liter of the textile waste was measured out. To each of these beakers containing a liter of waste, a constantly increasing increment of a 10 per cent solution of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ was added and stirred for five minutes. After this mixing and flocculating, the beakers were allowed to remain in a quiescent state for fifteen minutes while the floc settled. The amount of magnesium sulphate required to give a clear supernatant was found to be 1200 parts per million of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. The experiment was performed with seven different samples of the waste. Five samples required the 1200 p.p.m.; one gave a clear supernatant at 1100 p.p.m.; and another required 1300 p.p.m. to give a clear supernatant.

The next step was to determine how much of this magnesium could be reclaimed and reused. This amount would depend on the amount of the

original magnesium hydroxide-sludge mixture compared with the amount of sludge after the magnesium had been dissolved with acid, since the dissolved magnesium in the sludge would be lost when the sludge was discarded.

A magnesium solution was added to several beakers each containing a liter of the waste. Each sample was allowed to settle for two hours. The volume of magnesium hydroxide-sludge mixture obtained varied between 160 milliliters and 210 milliliters. Every sample of this waste was different; consequently every test gave slightly different results.

After the magnesium hydroxide-sludge mixture had settled, the supernatant was poured off leaving only the magnesium hydroxide-sludge mixture. This sludge mixture was acidified to a pH of 7.0 or below and then allowed to settle for two hours. The sludge remaining after the magnesium dissolved varied between 14 and 21 milliliters. This indicated that 8 to 10 per cent of the dissolved magnesium will be lost in the sludge or that 90 to 92 per cent of the original magnesium can be dissolved and reused.

Next, the amount of sulphuric acid required to dissolve the magnesium needed to be determined. To find the minimum amount of acid that could be used and that would still bring about complete dissolving of the magnesium hydroxide, a series of beakers containing magnesium hydroxide-sludge mixture was set up. A different amount of acid was added to each beaker in order to get various pH values from the series of beakers. After the remaining sludge in these beakers was allowed to settle, it was measured. The results are plotted on the graph in

Figure 1.

The graph shows that the minimum sludge or the complete dissolving of the magnesium hydroxide was first attained near pH 7.0. This means that enough acid must be added to the magnesium hydroxide-sludge mixture to lower its pH to 7.0 or below in order to dissolve the magnesium completely.

The amount of sulphuric acid required ranged between 150 p.p.m. and 300 p.p.m. based on the original amount of waste. The large range of sulphuric acid required was the result of both the concentration of the magnesium hydroxide-sludge mixture obtained and the original pH of the waste. The more the sludge mixture can be concentrated before the acid is added the less acid is required. Moreover, the original pH of the waste had a preponderant effect on the amount of the acid required to dissolve the sludge mixture. When the pH of the original waste was not too high, less acid was required to lower the pH to the required 7.0. The 150 p.p.m. stated above was required when the original pH of the raw waste was 10.8.

After it was found that the method worked reasonably well, the next step was to determine exactly how much chemicals for treating waste by this method would cost. At the beginning of the study magnesium sulphate was used as the precipitating agent, but since the magnesium ion was the only constituent needed, a search was made for a cheaper source of the magnesium ion. This search revealed that magnesium chloride was a much cheaper source. Magnesium chloride was then tried in the laboratory and was found to work just as well as the magnesium sulphate. The amount of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ required was 950 p.p.m.; the amount of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ required was 1200 p.p.m. If the magnesium chloride were bought in the anhydrous form as would normally be done, the amount required would be

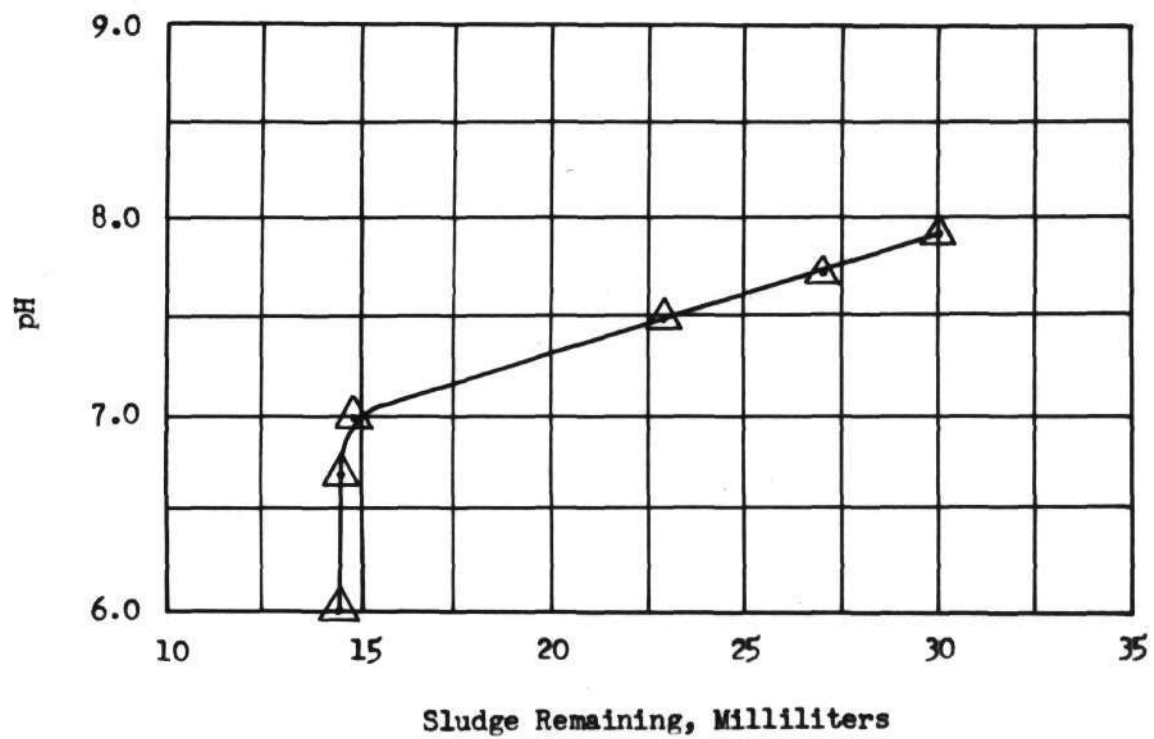


Figure 1. pH Required to Dissolve the Magnesium Hydroxide.

470 p.p.m. and its cost would be considerably less than that of the original magnesium sulphate.

The finding led to an actual comparison of the cost of treating one sample of the waste when magnesium chloride was used; when magnesium sulphate was used; and when alum plus acid was used. The cost of the chemicals was taken from the Chemical News, June, 1958, edition. This cost estimate is tabulated in Table 1.

The results of this comparison indicate that the waste can be treated by the method using magnesium chloride at less than 20 per cent of the cost of the alum treatment. Even the treatment using the magnesium sulphate, as was originally planned, was only about 40 per cent of the cost of the alum treatment.

Now that the method using magnesium chloride was found to be both feasible and economically desirable, a knowledge of the exact degree of clarification of waste that it would give was desired. This degree of clarification was determined by ascertaining the Biochemical Oxygen Demand, the Chemical Oxygen Demand, the Turbidity, and the Color reduction both before and after the waste had been submitted to the magnesium treatment. The results are tabulated in Table 2.

The large range of values obtained in the above tests was due to the variable characteristics of the waste tested as previously indicated. The actual degree of clarification is about what one would anticipate with a chemical method of treatment of this type of waste. The 56 per cent five-day B.O.D. removal compares favorably with the clarification obtained with a similar waste when alum was used as the flocculating agent where a 47 per cent five-day B.O.D. removal occurred (6).

Table 1
Cost Comparison For Treating of Waste

Chemicals Used For Flocculation			
	MgCl_2	$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	$\text{Al}_2(\text{SO}_4)_3$
<u>Precipitating Agent</u>			
Amount of Chemicals Required	47 ppm	120 ppm	300 ppm
Pounds per Million Gallons	392 lbs.	1000 lbs.	2500 lbs.
Cost per Pound	\$.025	\$.022	\$.05
Cost per Million Gallons	\$9.80	\$22.00	\$125.00
<u>Acid for Neutralization</u>			
Amount Sulfuric Acid Used	240 ppm	240 ppm	333 ppm
Pounds per Million Gallons	2000 lbs.	2000 lbs.	2770 lbs.
Cost per Million Gallons	\$20.00	\$20.00	\$27.70
Total Cost per Million Gallons	\$29.80	\$42.00	\$152.70

Table 2
Clarification of Waste Obtained in the Laboratory

Test	Original Amount (p.p.m.)	Amount Remaining (p.p.m.)	Reduction (%)	Average Reduction
5-day B.O.D.	943	405	57	52
	786	485	38	
	1046	376	64	
	904	460	49	
C.O.D.	1141	422	63	58
	968	550	43	
	1123	372	67	
	1037	425	59	
Turbidity Removal (Relative)			89	89
			94	
			91	
			82	
Color (Relative)			45	50
			70	
			55	
			30	

The conclusion of the laboratory investigation consisted of trying to find a way by which the final sludge, after the magnesium had been dissolved, could be disposed of. When the sludge was examined under the microscope, it was found to be primarily starch, for it had the characteristic shape of starch. Its being starch was verified when it was mixed with an iodine solution and found to form a dark bluish-black precipitate. But besides starch, this sludge was seen under the microscope to contain some fibers.

Therefore, since this sludge material was primarily organic matter, the choice of using sludge digestion for disposing of it was investigated. This process consisted of mixing one hundred milliliters of the sludge material with nine hundred milliliters of digested domestic sludge and pouring the whole into a sludge-digestion apparatus. This sludge digester was maintained at a constant temperature of 28°C in a water bath. The gas produced by this digester and the change in pH were read and recorded daily. The results are plotted in Figure 2 and Figure 3.

The plot of the gas production indicates that this sludge is amenable to the biological action in the sludge digester when the sludge material is mixed at this 10 per cent ratio with digested sludge.

The plot of the pH reading indicates that the methane organisms were able to multiply and volatilize the organic material and, consequently, to produce favorable sludge digestion.

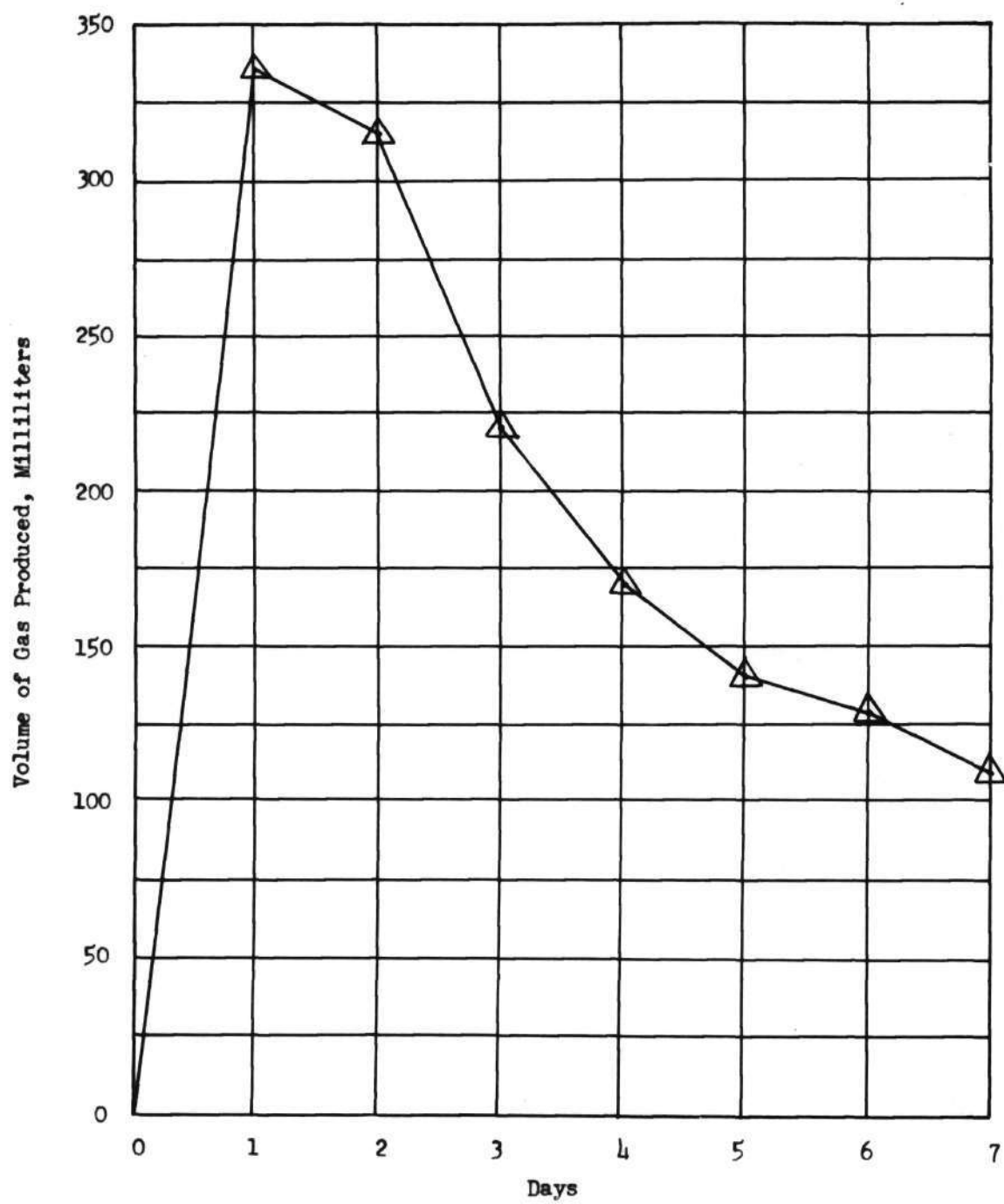


Figure 2. Daily Gas Production of the Digester.

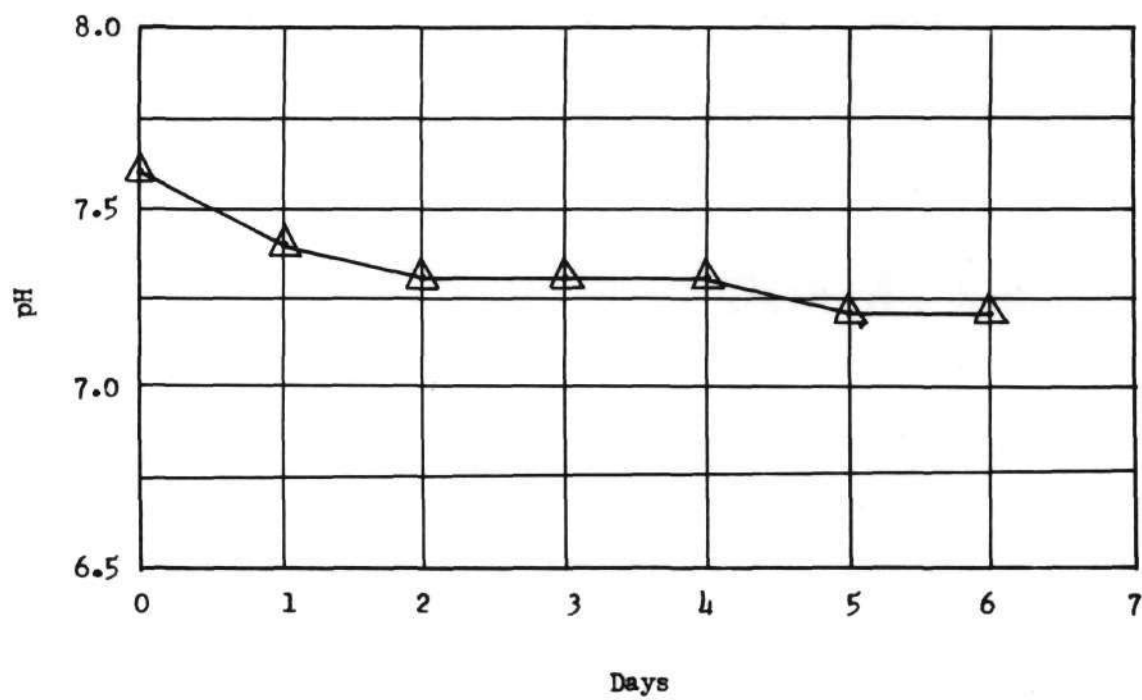


Figure 3. Daily pH's of the Digestion Mixture.

CHAPTER III

PILOT PLANT CONSTRUCTION AND OPERATION

General.—The laboratory investigations having given promising results, the next step was to construct a pilot plant and actually test the method on a larger scale and to use a continuous process of treatment.

The design of the pilot plant was based on the information obtained from the laboratory work. The plant was designed to operate with a flow of 10 gallons per minute.

Preliminary plans were drawn of the proposed pilot plant. The fact that there was only limited time for construction and the fact that the most economical design possible was desirable were taken into consideration. It was finally decided that a modified Potter-Rayfield Water Treatment Plant would be the most economical design for the pilot plant, since a plant of such design could be constructed most quickly. Therefore, the Potter and Rayfield Company of Atlanta was given the contract to construct this pilot plant.

The flow diagram of this pilot plant is given in Figure 4, and a picture of the pilot plant is shown in Figure 5.

Description.—The system consists first of a centrifugal pump on a sump at the outlet of the waste sewer. This pump is connected to the sump with a $1\frac{1}{2}$ -inch diameter pipe with a check valve. The pump has a minimum capacity of ten gallons per minute and a minimum head of fifty feet. The waste is fed to the plant from this pump by a one-inch galvanized pipe approximately 120 feet long.

The waste first enters the pilot plant at the large Potter-

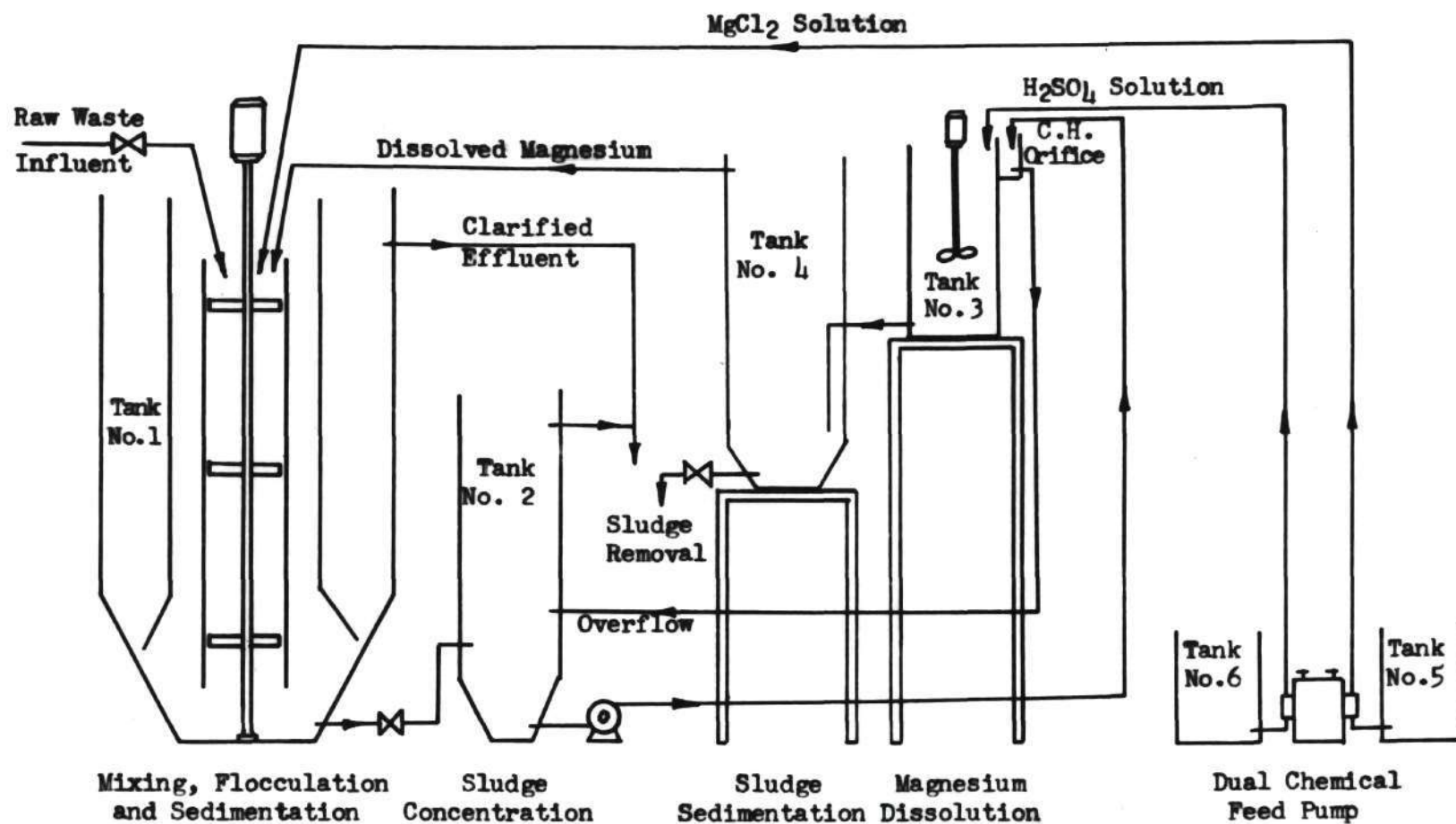


Figure 4. Flow Diagram of the Pilot Plant.



Figure 5. Picture of the Pilot Plant

Rayfield Water Treatment tank, Tank No. 1 (Figure 4). This tank consists essentially of three tanks in one -- a mixing chamber, a flocculating chamber, and a sedimentation chamber. A sketch of this tank is given in Figure 4, Tank No. 1.

When the waste enters the center chamber -- the mixing chamber -- a magnesium solution is mixed with it. This magnesium is obtained from the dissolved magnesium solution in Tank No. 4 (Figure 4) plus a small amount of make-up magnesium to replace the amount lost. The magnesium solution and raw waste are thoroughly mixed and flocculated by the mixer system depicted in Figure 6.

After the mixing and flocculation, the magnesium-sludge floc settles in the outer compartment of the large tank with the clarified effluent discharging out the over-flow pipe.

The magnesium-sludge floc is periodically drawn out of the bottom of this tank by a quick-opening valve into Tank No. 2 (Figure 4). In Tank No. 2 the magnesium-sludge floc is further allowed to settle with a certain amount of clarified effluent flowing out the overflow. From the bottom of this tank the concentrated magnesium-sludge floc is pumped to Tank No. 3 (Figure 4) through a constant-head orifice that is calibrated to discharge two gallons per minute. This constant-head orifice is shown in Figure 7.

To the magnesium-sludge floc in Tank No. 3 is fed sulfuric acid by one side of the dual chemical feed pump. This dual chemical feed pump is shown in Figure 8. The sulfuric acid and magnesium-sludge floc are mixed by a stirrer which dissolves the magnesium hydroxide of the floc and leaves the insoluble material of the original waste. The

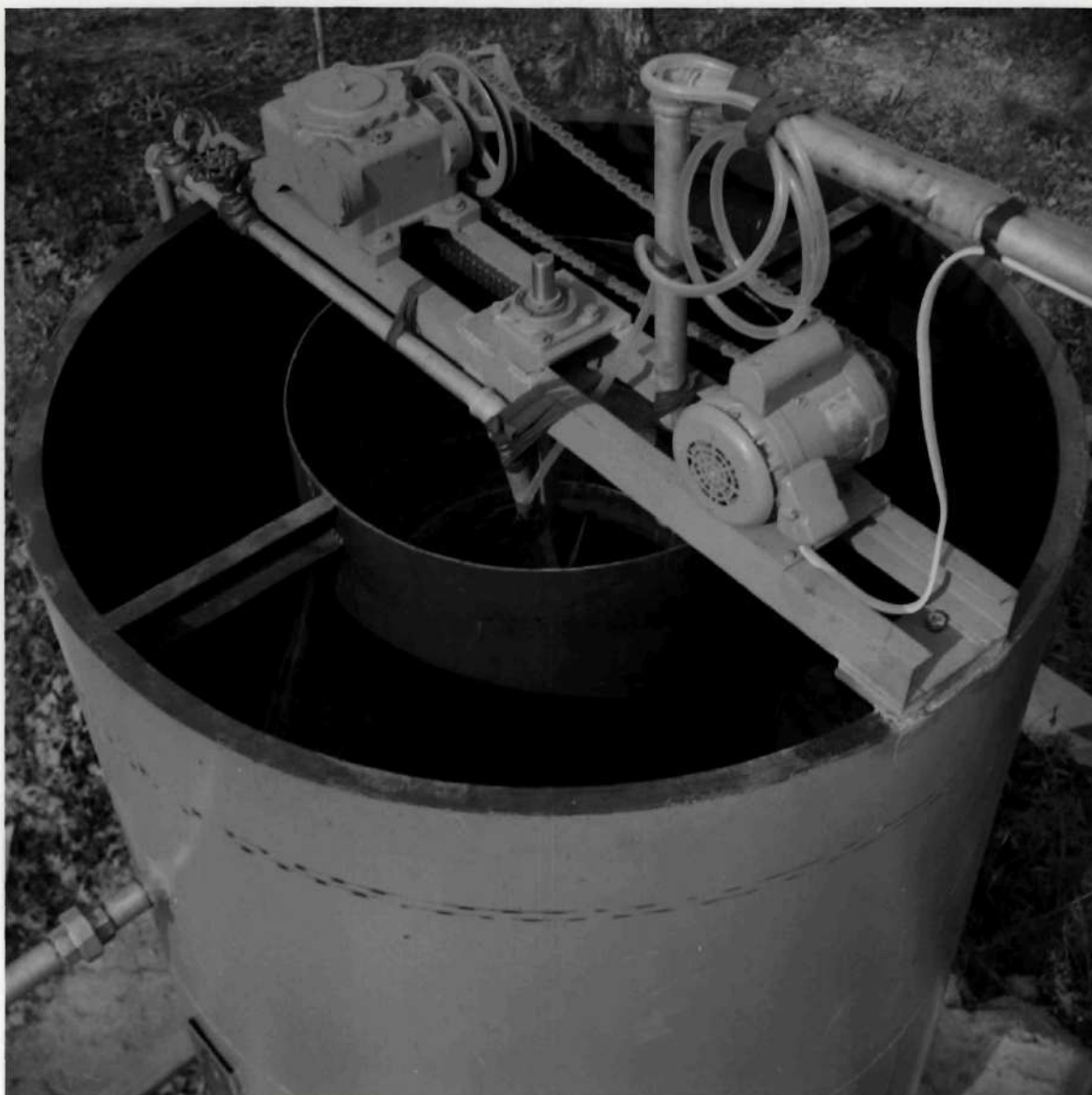


Figure 6. Picture of the Mixing System



Figure 7. Picture of the Constant Head Orifice



Figure 8. Picture of the Dual Chemical Feed Pump

mixture of dissolved magnesium and insoluble textile waste flow into Tank No. 4 (Figure 4) by gravity.

In Tank No. 4 the insoluble material of the textile waste settles and leaves a dissolved magnesium supernatant. This dissolved magnesium supernatant flows by gravity into Tank No. 1 (Figure 4), precipitates more incoming alkaline waste by forming more magnesium hydroxide and thus completes the cycle.

Operation.—The first step in the operation of this pilot plant was to calibrate the individual pumps of the dual chemical feed pump. These pumps have manual knob controls with readings on the dials of gallons fed per twenty-four hours. The controls were set at five gallon increments, and the amount pumped at these various settings was measured in milliliters per minute. The graphs of setting versus the milliliters of solution fed per minute are shown in the Appendix.

The next step was to adjust the incoming waste to the desired amount - ten gallons per minute. Since a water meter calibrated in gallons per minutes was connected to the effluent of Tank No. 1 (Figure 4), this adjustment consisted of merely the opening and closing of a valve on the incoming line until the desired ten gallons per minute of flow was indicated by the meter on the effluent line.

The third step was the adjusting of the constant-head orifice on Tank No. 3 (Figure 4) to obtain the desired flow of two gallons per minute, which was the amount of flow desired as shown by the laboratory investigations. This consisted of a trial-and-error process of drilling a hole in the orifice plate, gradually increasing its size by one-sixteenth inch increments until the approximate flow of two gallons per minute was obtained. A slight adjustment could then be obtained by

turning the plate which raised or lowered the height of the hole and enabled a precise adjustment of the desired flow to be made.

After the necessary adjustments were made, the chemical solutions were mixed and poured into the chemical tanks, No. 5 and No. 6 (Figure 4). Tank No. 5 was filled with a 20 per cent solution by weight of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$. Tank No. 6 was filled with a 10 per cent solution by volume of sulfuric acid.

Since at the beginning of the operation no redissolved magnesium was available, it was imperative to set the magnesium feed pump to feed 950 parts per million of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ until a magnesium floc could be built up. The plant was then started up; the influent pump, the stirrer, and the magnesium chemical feed pump were switched on. The plant was permitted to operate for about twenty minutes at the end of which time the magnesium floc began to rise to the top of the sedimentation tank and to flow out with the effluent. Then the plant was switched off for about one-half hour and the floc was allowed to settle. The plant was again switched on and allowed to run for about twenty minutes more at the end of which time the floc again rose to the surface and overflowed out with the effluent.

It was reasoned from the rising floc that the upward velocity in the sedimentation was too great and that its being too great caused the floc to break up and rise to the surface of the tank. Therefore, it was decided to reduce the capacity of the system to five gallons per minute rather than to keep it at the original ten gallons per minute.

The necessary adjustments were made so the plant could operate at five gallons per minute and then it was started again. By this

time enough magnesium floc had been accumulated so that it could be redissolved and reused. Therefore, the magnesium chemical feed pump was adjusted to feed 95 parts per million of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ with the remaining 855 parts per million of magnesium required being obtained from dissolving the magnesium hydroxide in the previous precipitated magnesium floc. To dissolve this magnesium, it was necessary to set the sulphuric acid feed pump to feed enough acid to reduce the pH in Tank No. 3 (Figure 4) to 7.0 or below. The amount of sulphuric acid required to do this varied between 150 and 200 parts per million, depending upon the initial pH of the incoming waste.

With all units of the pilot plant in full operation, the entire plant was switched on and allowed to run for about one and one-half hours, at the end of which time the magnesium floc again started to rise to the surface of the sedimentation tank. The plant was switched off and allowed to remain quiescent for about one-half hour before it was started up again. The system operated again for about one and one-half hours before the floc again rose to the surface.

It was concluded that the floc rose to the top of the tank because the design of the baffling system inside of the tank broke up the floc and because the sedimentation time in this tank was too short.

Because the oncoming of freezing weather limited the available time for operation and the time for modifying the baffling system, it was decided to operate the plant for one and one-half hour intervals with a one-half hour of shut down between operating periods.

By using the one and one-half hour operational periods, the amount of chemicals required to produce a clarified effluent were

determined. The amount of make-up $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ required varied between 95 and 110 parts per million. The amount of sulphuric acid required varied between 150 and 200 parts per million. These variances compare very favorable with the laboratory investigations, which indicated slightly more magnesium and slightly less sulfuric acid as being required.

Next, the amount of clarification of the waste was determined by finding the percentage removal of the Biochemical Oxygen Demand, the Chemical Oxygen Demand, the Turbidity, and the Color of the clarified effluent. These results are reported in Table 3.

Table 3

Clarification of Waste Obtained with the Pilot Plant

Test	Original Amount (p.p.m.)	Amount After Treatment (p.p.m.)	Reduction (%)	Average Reduction (%)
5-day B.O.D.	938	458	51	53
	811	423	48	
	1121	461	59	
	981	463	53	
C.O.D.	1004	432	57	58
	934	456	51	
	1145	413	64	
	1062	437	59	
Turbidity Removal (Relative)			83	87
			90	
			91	
			85	
Color (Relative)			45	49
			40	
			60	
			50	

CHAPTER IV

CONCLUSIONS AND RECOMMENDATIONS

Conclusions.—The method appears as a very promising one for chemically treating alkaline industrial waste. The pilot plant investigations gave results very similar to those obtained from the laboratory investigation. The pilot plant work merely required only slightly more magnesium and slightly less sulphuric acid than the laboratory work. The pilot plant required approximately 53 parts per million of anhydrous magnesium chloride; the laboratory study requires approximately 47 parts per million. The sulphuric acid required for the pilot plant operation was 150-200 parts per million as compared with 150-300 parts per million required in the laboratory. These small differences can be attributed to the variable nature of the waste, the purity of the chemicals used, the accuracy of measuring and weighing the chemicals, or the design features of the pilot plant. The cost of the chemicals used based on either the laboratory investigations or the pilot plant investigations was about \$30 per million gallons of waste treated.

The amount of clarification of the waste by the pilot plant and the laboratory investigations was also very similar with slightly better clarification being obtained in the laboratory. The pilot plant investigation showed a reduction of 48-59 per cent in B.O.D., 51-64 per cent in C.O.D., 83-91 per cent in Turbidity, and 40-60 per cent in Color. Comparatively, the laboratory investigations showed a reduction of 38-64 per cent in B.O.D., 43-67 per cent in C.O.D., 82-94 per cent in Turbidity, and a 30-70 per cent in Color. The wide range of values for each test

was due to the constantly changing characteristics of this waste.

The disadvantage of this method is the long detention period required for the sedimentation of the magnesium floc because it is a light floc and because the other insolubles in the waste are primarily organic compounds with a low specific gravity. Any full size treatment plant would in its design stage have to take into consideration this long three-to-six hour detention period for the sedimentation.

Recommendations.—Because of the limited time available for the pilot plant study it was not possible to make the necessary modifications to the pilot plant to ascertain the full potentialities of the magnesium-chloride method of treating alkaline wastes. Therefore, further investigations should be made and the pilot plant modified. The modifications believed to be imperative for the most efficacious operation of this pilot plant are:

1. A constant-head orifice should be installed on the incoming line to the pilot plant to replace the present valve regulator. The present valve is not a dependable means of controlling the incoming flow since it is easily clogged by the cotton fiber in the textile waste.

2. The outer baffle in the large tank, Tank No. 1 (Figure 4), should be completely removed as the fifteen-minutes detention for mixing and flocculation is not necessary. Removing of this baffle would also increase the sedimentation time and reduce the upward rising velocity.

3. The inner baffle in the large tank, Tank No. 1 (Figure 4), should have its height reduced by cutting off the bottom one-third. This would give about two thirds of the present mixing time, would keep the magnesium floc from breaking up, and would direct this floc downward

rather than upward as it is presently being directed.

4. The effluent overflow pipe in Tank No. 1 (Figure 4) should be replaced by a circular overflow weir to prevent short-circuiting.

5. A device should be built to observe the type of sludge drawn from Tank No. 1 into Tank No. 2 (Figure 4) in order to reduce the amount of sulfuric acid required to redissolve the magnesium in this sludge. This device could either be a piece of clear plastic tube or a small sludge observation box.

6. The size of Tank No. 4 (Figure 4) should be increased in order to permit a longer detention time and a slower upward velocity. This is deemed necessary since most of the undissolved solids are organic compounds and need a longer detention time for sedimentation than the present design permits.

It is believed that with the above modifications this plant will operate continuously and successfully at the original design capacity of ten gallons per minute.

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