PHYSICAL - CHEMICAL TREATMENT OF RAW WASTEWATER

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PHYSICAL-CHEMICAL TREATMENT

OF RAW WASTEWATER

Ву

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INTRODUCTION

Much work, as illustrated by the referenced papers (1 through 9), has been done recently to evaluate the feasibility of applying physical-chemical treatment techniques, such as chemical coagulation, filtration and activated carbon adsorption, directly to raw wastewaters or primary effluents to eliminate entirely the need for biological processes. Chemical coagulation and filtration are used to remove the raw wastewater suspended matter, whereas activated carbon is used to adsorb the remaining soluble organics. Phosphorus removal normally occurs with chemical coagulation. If nitrogen removal is also required, physical-chemical processes such as ion exchange and break-point chlorination are adaptable to the Rocky Mountain climatic conditions. Special sludge disposal and recovery considerations, dissimilar to biological systems, are included in the physical-chemical approach.

The purpose of this paper is to discuss typical design parameters for the unit processes involved in physical-chemical treatment of raw wastes and how the design engineer may determine the design criteria best suited for a given wastewater. The emphasis of the paper will be directed toward those processes particularly suited to smaller cities and cold climates.

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TREATMENT REQUIREMENTS

For purposes of providing an illustrative example, the raw waste characteristics and effluent requirements shown in Table 1 have been assumed. The effluent requirements should not be considered as recommended levels for any particular location.

TABLE 1

	INFLUENT, MEAN ANNUAL (mg/l)	DESIRED EFFLUENT, MEAN MONTHLY (mg/l)
BOD	180	15
COD	520	30
SUSPENDED SOLIDS	250	10
HARDNESS, AS CaCO ₃	170.5	-
PHOSPHORUS, TOTAL	11.5	1
PHOSPHORUS, ORTHO	10	-
NITROGEN, TOTAL	20	5
NITROGEN, AMMONIA	15	-
ALKALINITY AS CaCO ₃	220	-

WASTEWATER CHARACTERISTICS AND EFFLUENT QUALITY REQUIREMENTS

The effluent standards cannot be met with secondary treatment alone as chemical coagulation would be required to meet the phosphorus standard and, at least, filtration of a secondary effluent to meet the BOD and SS requirements. On the other hand, the effluent standards are not so stringent as to require for certain that physical-chemical techniques must be used in series with biological treatment.

A design engineer faced with the above situation should conduct the tests necessary to determine if these standards could be met by physical-chemical treatment alone and, if so, what design criteria should be used. The unit processes involved are proven

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to the degree that extensive, on-site pilot tests are not normally necessary for most wastewaters and design criteria can usually be obtained in laboratory tests. Should on-site pilot studies be necessary, pilot equipment for liquid and solids handling studies is available from several sources including consultant groups and equipment manufacturers.

PRELIMINARY DATA COLLECTION

In order to proceed with the design on a rational basis, a characterization of the raw wastewater in terms of its amenability to physical-chemical treatment must be made. The following description of tests on a wastewater illustrate techniques which may be used.

The goals of these tests are to answer the following major questions which must be known before the design can proceed:

- What is the best coagulant?
- How much sludge is produced?
- How well does the sludge dewater?
- Is coagulant recovery practical?
- What is the particulate, colloidal, soluble, and nonadsorbable fraction of organics in the raw wastewater?
- What is the fraction of soluble organic phosphorus and nitrogen in the raw wastewater?
- How much carbon contact time will be required?
- What effluent quality can be expected?

Physical-chemical processes are limited in their ability to remove colloidal and non-adsorbable organics, and soluble organic phosphorus and nitrogen. If these latter constituents are present in high concentrations, various combinations of biological/ physical/chemical treatment may be required.

CHEMICAL TREATMENT

Selection of Coagulant

There are four major classes of coagulants which may be considered singularly or in combination:

- 1. <u>Polymers</u>. Some investigators have reported successful coagulation of raw sewage with polymers alone. When used as the primary coagulant, polymers do not provide phosphorus removal. One of the following inorganic coagulants is required if phosphorus removal is of concern. Polymers used in conjunction with an inorganic coagulant are effective settling and filtration aids.
- 2. Iron Salts. Ferric chloride or ferric sulfate may be used for both suspended solids and phosphorus removal. Experience has shown that efficient phosphorus removal requires the stoichiometric amount of iron (1.8 mg/l Fe per mg/l of P) to be supplemented by at least 10 mg/l of iron for hydroxide formation. Typically, 15-30 mg/1 as Fe is required to provide phosphorus reductions of 85-90 percent. When considering iron for coagulation of raw wastes, it must be remembered that in an anaerobic environment, as may be encountered in a downstream carbon column, iron sulfide may be formed. This black precipitate is obviously not desirable in the final effluent. Depending on the iron salt, the optimum pH for coagulation or phosphorus removal will vary between 4.5 and 8.0.
- 3. <u>Aluminum Salts</u>. Both aluminum sulfate (alum) and sodium aluminate have been used for coagulation of wastewaters. Alum is generally a much more effective coagulant than sodium aluminate. Alum doses of 200-300 mg/l are

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typically required for 85-90 percent phosphorus removal (22 mg/l alum per mg/l phosphorus). Disadvantages of both iron and aluminum salts are (1) both form gelatinous hydroxide flocs which are difficult to dewater in many cases; (2) no practical techniques are yet available for recovery and reuse of the coagulant when phosphorus removal is required; and (3) large amounts of anions (chlorides or sulfates) are added to the wastewater. The optimum pH will vary between 5.5 and 8.5.

4. Lime has been successfully used in several locales Lime. for wastewater coagulation and phosphorus removal. The amount of lime required is usually independent of the amount of phosphorus present; rather it is a function of the wastewater alkalinity and hardness. When the pH reaches 9.5 to 10.5 due to the addition of lime, the orthophosphate is converted to an insoluble form. In some cases, additional quantities of lime may be required to form a readily settleable floc. Lime has been recalcined and reused in some cases when used to coagulate secondary effluent. However, recalcining and reuse may often not be practical when it is used to coagulate raw wastewaters due to the large amount of inert materials present in the combined raw sewage-chemical sludges. In any case, lime sludges nearly always dewater more readily than those resulting from iron or aluminum coagulation.

The choice of coagulant can usually be made rather quickly by laboratory jar tests. The following illustrative example is based on data collected on a raw wastewater from a community in the midwest.

In the technique used, six one-liter samples are dosed with the coagulants being studied while being rapidly mixed with a jar test device. In this example, 0.5 mg/l of an anionic polymer was added as a settling aid. Following a 60-second rapid mix (100 rpm), the samples are slowly mixed for about 5 minutes (30-40 rpm). They

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are then allowed to dynamically settle (5 rpm) for 30 minutes. Samples of the supernatant are then obtained by a pipette from a point just below the liquid surface in the jar. This is done to avoid including any of the floating solids which are invariably found in raw sewage. This supernatant sample is then analyzed for turbidity and phosphorus.

Lime Coagulation. By plotting the jar test data, it was determined that the lime dosages required to achieve a phosphorus concentration of one mg/l was less than that required for optimum coagulation and suspended solids removal. One mg/l phosphorus was achieved at a pH of 10.5-11.0. The lime dosage required for optimum solids removal varied from 200-400 mg/l. In general, a somewhat higher dose of lime was required for optimum solids removal than was required for phosphorus removal. A lime dose of 400 mg/l as Ca(OH)₂ achieved adequate solids removal for all samples and this dose will be used in subsequent calculation of the cost of lime coagulation.

Suspended solids analyses showed that settled supernatant contained less than 5 mg/l suspended solids at this dose and the filtered supernatant generally contained no measurable suspended solids. The lime and polymer dosage produced a rapidly settling floc, as it does in most wastewaters.

Alum Coagulation. The alum dosages required to achieve a phosphorus concentration of one mg/l averaged 200 mg/l.

Adequate solids removal was achieved at alum doses equal to or less than that required for phosphorus removal.

<u>Iron Coagulation</u>. The ferric chloride dosages required to achieve a phosphorus concentration of one mg/l averaged 80 mg/l. Adequate solids removal was achieved at ferric chloride dosages less than that required for phosphorus removal.

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Once the proper chemical dose is obtained, the test is repeated at that dose. The supernatant sample is then analyzed for suspended solids, COD, BOD, pH, turbidity and, if required, organic nitrogen and ammonic nitrogen. The supernatant from the lime jar tests should be recarbonated to pH 6.5-8.5 before performing the above analyses.

Past experience has shown that the filtrate quality (Whatman No. 2) obtained with filter paper will be about the same as that which will be achieved when mixed media filtration is applied after chemical clarification.

The ultimate choice of chemical will depend on the chemical cost, the amount of sludge produced, and the method of sludge disposal or recovery.

Chemical Costs

The approximate chemical costs, f.o.b. Seattle, are illustrated in Table 2.

TABLE 2

APPROXIMATE CHEMICAL COSTS F.O.B. SEATTLE

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CHEMICAL		COST PER LB.	CHEMICAL DOSE	COST PER MG
LIME	50 LB. Ca(OH) ₂ SACK	\$0.025	400	\$ 80
ALUM	100 LB. DRY ALUM SACK	\$0.075	200	\$120
FERRIC CHLORIDE	350 LBS. FeCI ₃ DRUM	\$0,20	80	\$130

Chemical Process Design

<u>Preliminary Treatment</u>. Comminution and grit removal facilities designed in accordance with standard sewage treatment design practices should be provided.

Chemical Feed, Rapid Mix, and Flocculation. These functions may all be carried out in accordance with standard practices followed in the water treatment field for years.

Proper rapid mixing is important for efficient utilization of the coagulating chemicals. The use of a mechanical rapid mixing device in the basins with a total of 2 minutes detention time at the average flow is recommended. When using lime as coagulant, scaling of the mixer shaft will occur and may cause excessive bearing wear if not cleaned regularly.

A mechanically mixed flocculator with 15 minutes detention is generally adequate for wastewaters. In many cases, the flocculation resulting from the large coagulant doses added to wastewaters results in very rapid flocculation and even shorter detention times may be feasible. Provisions should be made to add up to one mg/l polymer at the rapid mix or at the flocculator inlet or outlet or split among these points.

<u>Clarifier Sizing</u>. The critical design parameter is the <u>peak hourly</u> <u>surface overflow rate</u>. Gross carryover of solids can cause the downstream filter or adsorption processes to fail due to excessive headloss which, in turn, will result in a total failure of the plant. A maximum peak hourly rate of 1,400 gpd/ft² for conventional horizontal or radial flow clarifiers is recommended when using lime as a coagulant unless pilot tests indicate that other rates should be used. A maximum average rate of 900 gpd/ft² is recommended.

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Several attempts have been made to use sludge blanket type clarifiers on coagulated primary or secondary effluents. Difficulty in holding a sludge blanket has been reported in many cases. Successful operation has been achieved with these units by lowering the overflow rate to conventional clarifier rates and eliminating the sludge blanket.

Provision should be made for recirculation of controlled amounts of sludge from the bottom of the clarifier to the rapid mix inlet. The high pH of lime-treated water will form deposits of calcium carbonate on structures and in pipelines which it contacts. Lime sludge suction lines should be glass lined to facilitate cleaning. Provisions must also be made for regular cleaning of all other pipelines which carry the high pH effluent. Use of the new polyurethane cleaning pigs should be compatible with the layout of the pipelines. Mechanical sludge collection equipment used in lime settling basins should be of the bottom scraper type rather than the vacuum pickup style because of the dense sludge to be handled.

<u>pH Adjustment</u>. Lime treatment of wastewaters for phosphorus removal often raises the pH to values of 10-11. At this pH, the water is unstable and calcium carbonate floc will precipitate readily. This floc is very tenacious and would encrust any downstream filters or carbon particles to a serious degree. The pH may be lowered by injecting CO₂ gas obtained from the incinerator stack gases or from special generation equipment.

It is possible to reduce the pH of a treated wastewater from 11 to 7 or to any other desired value in one stage of recarbonation. Single-stage recarbonation eliminates the need for the intermediate settling basin which is used with two-stage systems. However, by applying sufficient carbon dioxide in one step for the total pH reduction, little, if any, calcium is precipitated with the bulk of calcium remaining in solution, thus increasing the calcium hardness of the finished water. Until equipment is available

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to separate raw organic sludge from the lime sludge, 2-stage recarbonation and lime recovery is probably not practical. The theory of 2-stage recarbonation is discussed by Culp and Culp⁽¹⁰⁾.

Alum or iron coagulation may also require pH adjustment. The optimum pH for coagulation with these metal salts can vary between pH 4.5 and 8.5. Depending on the raw waste characteristics and the discharge standards, pH adjustment may be required both before and after coagulation. Lime, caustic, soda ash have been used to raise the pH. Whereas, mineral acids or carbon dioxide have been used to lower the pH.

FILTRATION

Whether or not filtration is needed prior to activated carbon adsorption is subject to debate. There is little question that filtration ahead of a downflow granular carbon adsorption bed will reduce the rate at which the pores of the activated carbon become plugged with inert materials. Also, the use of an efficient filter permits downstream use of upflow, packed carbon beds which may be operated in the more efficient countercurrent mode discussed later. The question is whether or not the cost of providing the filtration exceeds the benefits mentioned above. Only long-term operating data from plants using granular carbon with and without prior filtration will answer this question. In the interim, a conservative design will include filtration prior to carbon adsorption. In addition to protecting carbon pores from plugging by inerts, filtration also provides a more efficient means of solids removal than carbon alone, resulting in a higher effluent quality.

Filtration equipment is available which will provide simple, reliable, and automatic operation. Carbon is not a particularly effective filter because it acts as basically a surface type filter and as such, is subject to all the shortcomings of other surface filters applied to wastewaters. Any high solids loading

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will blind a surface type filter in short order. The use of dual media or mixed, tri-media filters provides a much more efficient filtration device which is capable of tolerating a much higher solids loading than is a surface type filter. A discussion of alternate filtration devices and a detailed discussion of filter system design are discussed by Culp and Culp⁽¹⁰⁾.

In instances where an upflow expanded bed carbon contactor is used, the filter may be located downstream of the carbon column to remove the bacterial floc which is flushed from the carbon.

For the removal of the trace amounts of chemical floc which one will expect from the chemical clarifier, a properly designed dual media or mixed tri-media bed may operate at rates of 5-10 gallons per minute per square foot. The use of 5 gpm/ft² will provide a conservative basis for design. Surface wash is a must when filtering sewage.

The remaining question is whether the filter structure should be of the gravity or pressure type. The pressure system offers significant advantages in wastewater applications. In many instances, the applied solids loading will be higher and more variable than in a water treatment application. Thus, it is desirable to have higher head available than practical with gravity filter designs, preferably up to 20 feet of head when operating at 5 gpm/ft². In many physical-chemical treatment processes, the filtration step will be followed by a granular carbon adsorption step. The filter effluent from the pressure filter can pass through the downstream carbon columns without having to be repumped, often eliminating a pumping step which would be required with a gravity filter. All filter wash waters must be reprocessed in sewage applications. The use of the pressure filter will reduce the amount of wash water because of its ability to operate to higher headlosses.

The backwashing of the filter is accomplished by reversing the flow at a rate of 3-4 times the normal through-put rate of 5

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gpm/ft². Direct return of the wash waters directly to the head of the plant would create a very substantial hydraulic surge which may cause the upstream clarifier to fail. Therefore, the backwash wastewater should be collected in a storage tank and recycled to the head of the plant at a controlled rate. The surge storage tank should be sized adequately to handle successive backwashes from two to three filters.

It is desirable to precede the filtration step with a flow equalization pond so that the filters may be operated at essentially a constant rate. Provisions should be made for a feed of polymer or alum directly to the filter influent as a filter aid. Filter effluent turbidity and head loss should be monitored continuously with high filter head loss being used to initiate an automated backwash program. Polymers (0.1 - 1.0 mg/l) will probably be most effective when used on waste streams coagulated with iron or aluminum salts. Whereas alum (5-20 mg/l) will probably be the choice when following lime coagulation.

CARBON ADSORPTION

General

There are organics (i.e., sugars) which may be readily biodegradable but which are very difficult to adsorb on carbon. The amount of these nonadsorbable materials will vary greatly from wastewater to wastewater and their presence will be the governing factor concerning the quality of effluent which can be achieved by carbon adsorption. The same physical-chemical process may produce a BOD of 10 mg/l in one locale and 30 mg/l in another due to this fact.

Carbon Evaluation

A quick method for determining the nonadsorbable fraction of organics consists of contacting chemical treated wastewater with

1000 mg/l dose of a powdered carbon for one hour. Alternately, a sample of the granular carbon under consideration may be ground and applied to the sample. The sample is then settled and the supernatant filtered through Whatman No. 2 filter paper prior to analysis. The analyses should include the BOD and COD tests.

The column tests may be conducted using a granular carbon in five 3/4-inch diameter columns in series. The columns are sized so that cumulative contact times of 7.5, 15, 30, 45, and 60 minutes are provided at the end of the respective columns. Four to five gallons of raw sewage are coagulated with either lime, iron, or alum, settled, the supernatant decanted (the pH adjusted as required), and the clarified wastewater pumped through the columns. This quantity of sewage will provide several days of operation in columns of this size. The tests should be continued as long as possible to accurately determine the effects of biological activity.

The reasons for preferably conducting both the powdered carbon and column tests are to determine if the effluent from the columns could be lower in BOD than that achieved by adsorption alone due to the biological growth in a column and to determine the effects of contact time on column performance.

Figures 1 and 2 summarize the data collected from the laboratory columns for the midwestern waste. As can be seen from the figures, the benefits achieved by contact times greater than 30 minutes are slight. The carbon column effluent BOD values after 60 minutes contact ranged from 5 to 15 mg/l and averaged 11.0 mg/l. The BOD samples collected at a 30-minute contact time averaged 12.5 mg/l.

An estimate of the required carbon dosage can be made by assuming that carbon will be withdrawn for regeneration when the carbon loading is 0.5 pounds of COD removed per pound of carbon. This

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loading has been achieved in several studies. An average soluble influent COD of 86 mg/l was achieved with lime clarification in the four series of jar tests. The average COD achieved in the four powdered carbon tests was 23 mg/l and averaged 24 mg/l after 30 minutes contact in the columns. Thus, an average COD removal of about 62 mg/l would be expected from these tests. The corresponding carbon dosage is 1,030 pounds/mg. Carbon dosages calculated from short-term laboratory column tests are usually conservatively high, as biological action usually results in greater permissible loadings in a continuous, plant scale operation.

Process Design

Adsorption. Because of the unproven economics of recovery and reuse of powdered carbon, the use of granular carbon is the only current practical technique available, except for very small systems, for removal of soluble organics from coagulated raw wastewater. Granular carbon recovery has been demonstrated at Pomona, California, to be practical at 0.3 mgd⁽¹¹⁾.

With powdered carbon the grain size increases the kinetics of adsorption such that 90% of its adsorption equilibrium is attained in less than 10 minutes. Powdered carbon is dosed in slurry form, after which it is separated by sedimentation following polymer flocculation and/or filtration. Powdered carbon has the advantage over granular in that its initial cost is about 1/3 as great.

Determination of the technical and economic feasibility must await the result of contracts with Eimco Corp. and Infilco.

The major design decisions facing the engineer are the selection of a contact time, carbon dosage, and the configuration of carbon contactor to be used.

The two major alternate contactor configurations to consider are open vessels of either an upflow or downflow type or upflow or

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downflow counter-current pressure columns. The counter-current approach (see Figure 3) offers a more efficient utilization of the carbon as only the most saturated carbon is withdrawn for generation. This results from the fact that as the carbon becomes saturated with organics, it becomes heavier. When the carbon column is backwashed, the more saturated, heavier carbon migrates to the bottom of the column where it is withdrawn for regenation.

A semi-counter-current approach can also be achieved by using two downflow columns in series. As indicated on Figure 4, water is first passed down through Column A, then down through Column When the carbon in Column A is exhausted, the carbon in Column в. B is only partially spent. At this time, all carbon in Column A is removed for regeneration, and is replaced with fresh carbon. Column B then becomes the lead column in the series. When the carbon in Column B is spent, the carbon is removed for regeneration and is replaced with fresh carbon. This type of operation gives only some of the advantages of counter-current operation, because only the carbon near the inlet of the lead bed is fully saturated with impurities removed from the water, and some capacity is unused in much of the rest of the carbon sent to regeneration. Also, the piping and valving is more complex and costly than for an upflow, counter-current column. Unless one is attempting to use the carbon for the dual purpose of filtration and adsorption (which the author does not recommend for most cases). there is no advantage to using the downflow approach while there are the disadvantages discussed above.

The choice of contactor design is also dependent upon the method selected for control of hydrogen sulfide generation in the carbon columns. The upflow, expanded bed with a downstream filter has been used with injection of oxygen into the carbon influent for hydrogen sulfide control. The prolific biological growth resulting from this approach would result in excessive head loss in a downflow, packed bed. Frequent backwashing of a downflow, packed bed has been reported effective at Rocky River in con-

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trolling hydrogen sulfide. Also, breakpoint chlorination, prior to downflow beds, although expensive, has been reported effective at Blue Plains in controlling hydrogen sulfide. References 1 through 7 provide additional information on hydrogen sulfide generation and control.

The technology transfer manuals published by EPA and the book by Culp and Culp⁽¹⁰⁾ present detailed carbon contactor design alternates. However, there are a few other points related to contactor design which I would like to call to your attention in addition to the comments above.

When using steel contactors, it is imperative that the interior be properly protected from the very corrosive effects of partially dewatered activated carbon. Two 8-mil thick coatings of a coal tar epoxy have proven to be effective at Tahoe over 4 years of continuous operation. Fiberglas-polyester coatings would also be effective, although more costly than the coal-tar epoxy coatings.

Another point to consider is the effect of the pH, of the upstream coagulation step, on the efficiency of the carbon process. One available process is based on a claim that use of extremely high pH (11.8 - 12.2) in the lime coagulation process will hydrolyze some organic materials making them more readily adsorbable. Before one incurs the disadvantages of the high pH approach (massive quantities of sludge plus greatly increased carbon dioxide requirements for pH adjustment), he should carefully evaluate the effects of pH on the specific wastewater involved.

<u>Carbon Regeneration</u>. As granular activated carbon adsorbs organics from wastewater, the carbon pores eventually become saturated and the carbon must be regenerated for reuse. The best way to restore the adsorptive capacity of the carbon is by means of thermal regeneration (by heating the carbon in a lowoxygen steam atmosphere in a multiple-hearth furnace). At

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temperatures of 1,650-1,750 degrees F, the dissolved organics are volatilized and released in gaseous form. The regenerated carbon is cooled by water quenching. By proper treatment, carbon can be restored to near virgin adsorptive capacity while limiting burning and attrition losses to 5-10 percent. Regeneration furnace off-gas odors can be controlled by after-burning, if necessary, and particulates and soluble gases can be removed by use of Venturi or jet impingement type scrubbers. Figure 5 illustrates a typical regeneration system.

The carbon furnace should be sized with recognition of the fact that substantial downtime may be required for maintenance of the furnace. An allowance of 40 percent downtime in selecting the furnace size provides a conservative basis for furnace selection.

NITROGEN REMOVAL

Ammonia Removal by Selective Ion Exchange

A demonstration project at the Battelle Memorial Institute -Pacific Northwest (Hanford) Laboratories, 1969 showed that certain zeolites, including the naturally occurring mineral clinoptilolite, had a high selectivity for ammonium in natural and wastewaters.

Clarifier effluent is passed downward through columns containing clinoptilolite. When a column becomes loaded with ammonia, it is regenerated with limewater containing sodium chloride to speed up the rate of regeneration. The high pH of the limewater converts the ammonium ion to ammonia gas in solution. The ammonia laden limewater is then pumped through a packed column through which heated air is blown to remove the ammonia.

Ammonia in the regenerant solution may also be converted to nitrogen gas by reaction with chlorine which is generated electrolytically

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from the regenerant solution. This process can be carried out with a regenerant of neutral pH so that the problem of precipitation of Mg(OH)₂ and CaCO₃ within the bed during regeneration is eliminated. Also, cold weather does not affect the process. The regenerant solutions used are rich in NaCl and CaCl₂ which provide the chlorine produced at the anode of the electrolysis cell. The reactions for the destruction of ammonia by chlorine are the same as for breakpoint chlorination.

In pilot tests of the electrolytic treatment of the regenerant at Blue Plains, Battelle Northwest found that about 50 watt hours of power were required to destroy one gram of ammonia nitrogen. When related to the treatment of water containing 15 mg/l NH₃-N, the energy consumed would be 2.9 kWh/l,000 gallons. Thus, power costs are not prohibitive. Overall costs for a 10 mgd plant using the electrolytic technique were estimated at 12.7 cents/ 1,000 gallons by Battele Northwest.

Effluent ammonia concentrations below 1 mg/l are easily achieved. Figure 6 illustrates the pilot tests at South Lake Tahoe.

Ammonia Removal by Breakpoint Chlorination

When chlorine is added to water containing ammonia-nitrogen, the ammonia reacts with the chlorine (hypochlorous acid) to form various chloramines.

Several investigators, including the author, have found that breakpoint chlorination requires approximately 10 mg of chlorine per mg of NH_3 -N. An alkaline material such as NaOH or Ca(OH)₂ may have to be added in order to prevent pH depression and subsequent nitrogen trioxide formation. Another consideration is that a substantial increase in effluent chlorides will result from the addition of such large quantities of chlorine.

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FIGURE 6

AMMONIA BREAKTHROUGH CURVES FOR A 6 FT. CLINOPTILOLITE BED AT VARIOUS FLOW RATES Tests at Blue Plains showed that eductors do not give adequate chlorine-wastewater mixing, resulting in localized low pH regions in which objectionable quantities of NCl₃ form. Violent, mechanical mixing is required to minimize NCl₃ formation. The NaOH quantity added to neutralize the acidic effects of the chlorine addition was 0.9-1.7 pounds of NaOH/Pound of chlorine, corresponding to adjusting a pH of 6 to 7.9. One run was made with lime, which indicated about one pound of lime would be required per pound of chlorine for a pH of 7.0.

Breakpoint chlorination may be particularly well suited to small treatment systems with part-time staffs.

The economics of the process are not attractive when removing the relatively large quantities of ammonia found in an un-nitrified effluent. For example, at a chlorine cost of 3.5 cents/pound (which is the lowest cost one could anticipate), the cost of chlorine to remove 15 mg/l of ammonia nitrogen (150 mg/l of chlorine) would be \$43.80/mg. According to the Blue Plains work, about 195 mg/l of NaOH would also be required which would, at a cost of \$70/ ton, add another \$57/mg to the chemical cost. Thus, without capital and labor costs, the cost of this approach is about \$100/mg using NaOH for pH control. If lime proves satisfactory for pH control, the chemical cost would be reduced substantially to about \$60/mg at a lime cost of \$25/ton.

PHYSICAL-CHEMICAL TREATMENT OF SMALL WASTE FLOWS

General

The basic unit processes discussed earlier are directly applicable to any size treatment plant including the less than 500,000 gal/day package plant range. A number of manufacturers are developing or have developed package physical-chemical wastewater treatment plants. Examples of different types of small physical-chemical processes are discussed in the following paragraphs. Some of the information has been taken from reference 13. Mention of commercial products does not imply endorsement by the EPA or by ourselves.

Clarification-Filtration

Johns-Manville Corporation has developed a process referred to as the "Moving Bed Filter". Figure 7 shows a schematic of the process. Recently Peabody Wells has been marketing the process.

Raw waste is dosed with alum and an anionic polymer and flows into a tank which provides head for the filter operation. The sewage filters downward through the inclined packed bed of sand to a screened pipe and thence flows to a collector. Sewage solids and floc collect primarily on the filter face although some depth filtration is obtained. When the head loss exerted by the accumulated solids becomes excessive the sand bed is pushed upward and a cutter slices off the top layers of sand and suspended solids. The sand-sludge mixture is collected in the bottom of the head tank and is then pumped to a sand washer. Clean sand is returned to a hopper and eventually to the bottom of the sand bed. At the time of the study report (reference 13) the feasibility of adding powdered carbon was still to be evaluated. Without carbon treatment BOD efficiency is limited to particulate BOD removal.

Clarification-Carbon Treatment

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A process employing the clarification and granular activated carbon unit in series is the Met-Pro system. This system is a package plant utilizing an upflow solids contact clarifier ahead

Met-Pro Water Treatment Company of Lansdale, Pennsylvania.

of two-stage 12x40 mesh granular activated carbon treatment. Different chemical coagulants have been evaluated by the EPA (reference 13). The two-stage granular carbon system consists of a downflow packed bed followed by upflow bed. Partially spent carbon from upflow bed is used to replace the spent carbon in the preceding downflow bed. Completely spent carbon from the downflow bed is discarded.

Clarification-Filtration-Carbon Treatment

Two physical-chemical systems using clarification, filtration, and activated carbon adsorption are the package units developed by Neptune MicroFLOC⁽¹⁾ and AWT Systems Inc.⁽²⁾ The MicroFLOC system employs powder activated carbon whereas the AWT System uses granular activated carbon.

In the MicroFLOC system powdered carbon and a coagulant are introduced into the raw waste stream just prior to coagulation. The waste stream then flows through a two stage flocculator, inclined settling tubes for clarification, and through a mixed (tri-media) filter. Alum or lime may be used as the primary coagulant. With alum, a soda ash feed system is provided for pH control. If lime is used, pH adjustment is provided following the tube settlers. Polymer can be fed at both the flocculation and filtration steps.

In the AWT System's physical-chemical unit a metal salt coagulant and an acid-alkaline control additive are added to the raw waste before coagulation. Following coagulation, a polymer is introduced to improve clarification. The effluent from the clarifier is treated with a magnetic additive and fed through a magnetic filter for further solids removal. An upflow carbon contactor with granular activated carbon is used after filtration to remove dissolved organics.

- Neptune MicroFLOC, 1965 S.W. Airport, Corvallis, Oregon.
 NET Systems Inc. 010 Market Streat Wilminster Delegen
 - AWT Systems Inc., 910 Market Street, Wilmington, Delaware.

Sludge Production

It is difficult to obtain an accurate gravimetric measurement of sludge quantities in a laboratory test due to loss of solids during decanting, etc. However, it is possible to estimate the quantities of sludge from the chemistry involved and the data collected from the jar tests.

The basic equations required for these calculations may be simplified as follows:

1.
$$3PO_4^{3-} + 5Ca^{2+} + 0H^{1-} \rightarrow Ca_5 OH(PO_4)_3 \forall$$

2. $Mg^{2+} + 2OH^{1-} \rightarrow Mg(OH)_2 \forall$
3. $Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3 \forall$
4. $CaCO_3 \xrightarrow{1800°F} CaO + CO_2^{\uparrow}(incineration)$
5. $CaO + H_2O \rightarrow Ca(OH)_2$
6. $Al^{3+} + PO_4^{3-} \rightarrow AlPO_4 \forall$
7. $Al^{3+} + 3OH^{1-} \rightarrow Al(OH)_3 \forall$
8. $2Al(OH)_3 \xrightarrow{1400°F} Al_2O_3 + 3H_2O$ (incineration)
9. $Fe^{3+} + PO_4^{3-} \rightarrow FePO_4 \forall$
10. $Fe^{3+} + 3OH^{1-} \rightarrow Fe(OH)_3 \forall$
11. $2Fe(OH)_3 \xrightarrow{1400°F} Fe_2O_3 + 3H_2O$ (incineration)
12. Σ Coagulant in = Σ Coagulant out

Tables 3, 4 and 5 describe the computations used to estimate the quantities of sludge produced. The total quantities of raw and chemical sludges produced are:

- ° Lime @ 400 mg/l [Ca(OH)₂] = 6290 lbs/mg
- Alum @ 200 mg/l $[Al_2(SO_4)_3 \cdot 14H_2O] = 2648 \text{ lbs/mg}$
- Ferric Chloride @ 80 mg/l [FeCl₃] = 2662 lbs/mg

Sludge Disposal

Sludge disposal is perhaps the most important factor governing the choice of chemical coagulants. Unhappily, the least is known about this particular facet.

Alum and iron sludges can normally be added to existing anaerobic digesters. The higher digester loadings resulting from additional sludge production will not usually be detrimental to operation unless an organic overloading condition exists. Release of soluble phosphorus from the sludge during digestion is considered to be minimal. Final disposal of the digested sludge can be on land or by dewatering and incineration.

Alum iron and lime sludges can be disposed of directly onto land. Depending on temperature requirements, alum and iron sludges could need lime treatment to prevent odors.

In larger systems sludge thickening or dewatering prior to lagooning or incineration can be considered. Here the type of sludge becomes important. Alum and iron sludges are much harder and expensive to thicken or dewater than are lime sludges.

ESTIMATE OF LIME SLUDGE QUANTITIES

RAW SEWAGE SU RAW SEWAGE VO RAW SEWAGE PO RAW SEWAGE TO RAW SEWAGE Ca RAW SEWAGE Mg EFFLUENT PO ₄ EFFLUENT Ca ²⁺ EFFLUENT Mg ²⁺	SPENDED SOLIDS LATILE SUSPENDED SOLIDS 3- 4 TAL HARDNESS 2+ 2+		250 MG/L 150 MG/L 11.5 MG/L AS P 170.5 MG/L AS CaCO ₃ 60 MG/L 5 MG/L 0.3 MG/L AS P 80 MG/L 0
LIME DOSAGE	400 MG/L AS Ca(OH) ₂ OR 216 MG/L AS Ca ²⁺		
FROM EQ. (1);	$Ca_{5}OH(PO_{4})_{3}$ FORMED IS 1 $\frac{11.2}{30.97} = 0.365$ MOLES P REI 0.122 MOLES $Ca_{5}OH(PO_{4})_{3}$ / THEREFORE WT. IS 0.122×5	MOLE PER 3 MOI MOVED; THEREFO ARE FORMED – F 02 = 61 MG/L AS $Ca_5 OH(PO_4)$	-ES P RE <u>0.365</u> OR .W. IS 502 3
FROM EQ. (2);	$Mg(OH)_2 \text{ FORMED IS 1 MOL}$ $\frac{5}{24.31} = 0.206; \text{ THEREFORE}$.E PER MOLE Mg ² 0.206x58.31 = 12 m Mg(d	+ MG/L AS DH) ₂
FROM EQ. (12);	Ca^{2+} IN = Ca^{2+} OUT; Ca^{2+} I Ca^{2+} CONTENT OF Ca_5 OH(P Ca^{2+} LOST IN EFFLUENT = Ca^{2+} NOT ACCOUNTED FOF	N = 60+216 = 276 O_4 ₃ FORMED = 5 80 MG/L, THEREF R = 276 - (80+24)	x40x0.122 = 24 MG/L ORE = 172 MG/L
FROM EQ. (3);	CaCO ₃ FORMED IS 1 MOLE	PER MOLE Ca^{2+}	
	THEREFORE $\frac{172}{40}$ = 4.3 MOL	ES CaCO ₃ – F.W.	= 100
	SO WT. OF $C_aCO_3 = 430$ MG	i/L	
	SLUDGE COMP	OSITION	
SLUDGE SPECI	ES TOTAL	WEIGHT	ASH
RAW SEWAGE SO	LIDS 250 MG/L =	2,080 LBS./MG	832 LBS./MG
$Ca_{5}OH(PO_{4})_{3}$	61 MG/L =	510 LBS./MG	510 LBS./MG
Mg(OH) ₂	12 MG/L =	100 LBS./MG	100 LBS./MG
CaCO ₃	430 MG/L =	3,600 LBS./MG	2,020 LBS./MG
TOTALS		6,290 LBS./MG	3,462 LBS./MG

ESTIMATE OF ALUM SLUDGE QUANTITIES

RAW SEWAGE SUSPE	ENDED SOLIDS	250	MG/L	
RAW SEWAGE VOLA	TILE SUSPENDED SOLIDS	150	MG/L	
RAW SEWAGE PO43-		11.5	MG/L AS	Ρ
RAW SEWAGE TOTA	L HARDNESS	170.5	MG/L AS	CaCO ₃
RAW SEWAGE Ca ²⁺		60	MG/L	Ũ
RAW SEWAGE Mg ²⁺		5	MG/L	
EFFLUENT PO4		0.3	MG/L AS	Р
EFFLUENT Ca2+		60	MG/L	
EFFLUENT Mg ²⁺		5		
EFFLUENT AI ³⁺		0		
ALUM DOSAGE	200 MG/L AS $AI_2(SO_4)_3$ 14 $H_2O - F.W$. = 594		
FROM EQ. (6); =	AI PO4 FORMED IS 1 MOLE PER MOLE	OF P;		
	$\frac{11.2}{30.97}$ = 0.365 MOLES P REMOVED; THE	REFORE	0.365	
	MOLES OF AI POA ARE FORMED - F.W.	IS 122		
	THEREFORE WT. IS 0.365x122 = 44 MG/	L		
FROM EQ. (12);	AI^{3+} IN = AL^{3+} OUT; AI^{3+} IN = 18.1 MG	ì/L		
	AI^{3+} CONTENT OF AL PO = 0.365×27 =		,	
		5.5 MG/1	-	
	Al ^{\circ} NOT ACCOUNTED FOR = 18.1 – 9.	9 = 8.2 N	/IG/L	
FROM EQ. (7);	AI(OH)3 FORMED IS 1 MOLE PER MOLE	аі ³⁺		
	THEREFORE $\frac{8.2}{27}$ = 0.31 MOLES AI(OH) ₃	— F.W. =	78	
	SO WT. OF AI(OH)3 IS 0.31x78 = 24 MG/	/L		

SLUDGE COMPOSITION

SLUDGE SPECIES	TOTAL WEIGHT	ASH
RAW SEWAGE SOLIDS	250 MG/L = 2,080 LBS./MG	832 LBS./MG
AI PO ₄	44 MG/L = 368 LBS./MG	368 LBS./MG
AI(OH) ₃	24 MG/L = 200 LBS./MG	133 LBS./MG
TOTALS	2,648 LBS./MG	1,333 LBS./MG

ESTIMATE OF IRON SLUDGE QUANTITIES

RAW SEWAGE SU RAW SEWAGE VO RAW SEWAGE TO RAW SEWAGE TO RAW SEWAGE CA RAW SEWAGE MA EFFLUENT PO4 EFFLUENT Ca ²⁺ EFFLUENT Ca ²⁺ EFFLUENT Fe ³⁺ FeCl ₃ DOSAGE =	USPENDED SOLIDS DLATILE SUSPENDED SOLIDS 04 07 07 07 07 07 07 07 07 07 07 07 07 07	250 150 11.5 170.5 60 5 0.3 60 5 0	MG/L MG/L AS MG/L AS MG/L MG/L MG/L AS MG/L	P CaCO ₃ P
FROM EQ. (9);	FePO ₄ FORMED IS 1 MOLE PER MOLE P; $\frac{11.2}{30.97}$ = 0.365 MOLES P REMOVED, THEREFOR MOLES OF FePO ₄ ARE FORMED - F.W. = 151 THEREFORE WT. IS 0.365x151 = 55 MG/L	RE 0.36	3 5	
FROM EQ. (12);	Fe^{3+} IN = Fe^{3+} OUT; Fe^{3+} IN = 28 MG/L Fe^{3+} CONTENT OF $FePO_4$ = 0.365x55.8 = 20.4 Fe^{3+} NOT ACCOUNTED FOR = 28 - 20.4 = 7.6	MG/L 5 MG/L		
FROM EQ. (10);	Fe(OH) ₃ FORMED IS 1 MOLE PER MOLE Fe^{3+} THEREFORE 7.6 = 0.136 MOLES $Fe(OH)_3 - I_5$ SO WT. OF $Fe(OH)_3 = 0.136 \times 107 = 15$ MG/L	F.W. =	107	

SLUDGE COMPOSITION

SLUDGE SPECIES	TOTAL WEIGHT	ASH
RAW SEWAGE SOLIDS	250 MG/L = 2,080 LBS./MG	832 LBS./MG
FePO ₄	55 MG/L = 460 LBS./MG	460 LBS./MG
Fe(OH) ₃	15 MG/L = 122 LBS./MG	105 LBS./MG
TOTALS	2,662 LBS./MG	1,397 LBS./MG

The following data (Table 6), although only an educated guess, should serve to demonstrate the magnitude of the problem.

TABLE 6

PROBABLE SLUDGE CONCENTRATIONS

	CHEMICAL COAGULANT	% SOLIDS	
GRAVITY THICKENING	ALUM AND IRON	2–5	
	LIME	10–25	
DEWATERING	ALUM AND IRON	10–20	
	LIME	20-40	

Sludge incineration, particularly for larger cities could be an integral part of physical-chemical processes. The advantages of converting organic solids to ash and thereby reducing the weight and volume of solids cannot be ignored. Alum, iron and lime sludges can be incinerated. The relative amounts of water and solids described earlier control the incinerator size. Table 7 illustrates the weight reduction achieved by incineration.

TABLE 7

PHYSICAL-CHEMICAL SOLIDS REDUCTION BY INCINERATION

	DRY WEIGHT, LBS./MG		
COAGULANT	BEFORE INCIN.	AFTER INCIN	
ALUM	2,648	1,333	
IRON	2,662	1,397	
LIME	6,920	3,462	

Generally speaking, only lime, of the three coagulants listed, can be recovered using current technology. Even lime recovery may not be economically practical when used to coagulate raw wastewater. An effective means must first be found to separate the lime from the inert organic raw sewage ash.

Lime recovery involves the conversion of calcium carbonate to carbon dioxide and calcium oxide (quick lime).

When lime recovery systems are employed, recycling solids necessarily appear as a part of the reclaimed coagulant feed. Again pursuing the previous example it may be seen from Table 3 that if recalcination for coagulant reuse is employed, each cycle of coagulant recovery will increase the total dry solids to be processed by the amounts shown in Table 8.

TABLE 8

THEORETICAL BUILD-UP OF INERTS IN A RECYCLING COAGULANT RECOVERY SYSTEM

	MGT. OF INERTS/CYCLE
PARAMETER	(LBS./MG)
ASH (FROM RAW SEWAGE SOLIDS)	832
HYDROXYAPATITE	510
MAGNESIUM HYDROXIDE	100
TOTAL INERTS/CYCLE	1,442

Following this line of reasoning then, unless blowdown of inerts from the system occurs, regardless of plant size, coagulant recovery systems must in time approach an infinite capacity. Purely as a coarse approximation, equation (13) can be used to illustrate this. eq (13) Feed = CaCO₃ + Organics + [Inerts X (C-1)] where CaCO₃ = #CaCO₃/mg Note: eq (13) usable only for > 1 cycle Organics = #Organics/mg Inerts = #Inerts/mg and C = the number of cycles starting with the initial feed as No. 1

Table 9 illustrates for our example what would occur at the 5th, 10th, and 20th cycle of such a system.

TABLE 9

INCINERATOR FEED RATES THEORETICALLY REQUIRED FOR A NON BLOW DOWN COAGULANT RECOVERY SYSTEM

CYCLES	FEED LB./MG (DRY SOLIDS)
1	6,290
5	11,440
10	18,640
20	33,040

Clearly such a buildup of inerts as indicated in Table 9 is unacceptable in the design of solids handling systems. This has spurred research into better techniques of separating or classifying chemical sludges one from another. Several techniques for reducing the buildup of inert solids within a coagulant recovery system are available. These include the following:

- ° Direct blowdown of unprocessed sludges
- Blowdown of dewatered chemical sludges

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- ° Classification of solids content
- Chemical treatment of unprocessed sludges
- Indirect blowdown of recovered coagulant
- Combinations of the above methods

Regardless of the methodology employed for blowdown of unwasted constituents, some fraction of inert materials will be present as a recycle in any solids handling system employing coagulant recovery and reuse. Therefore, the design engineer must be able to determine what this fraction is as well as its characteristics prior to design of a proper solids handling system. This is most easily accomplished by calculation of mass balance under conditions when equilibrium is reached in the system. In our example system, from Table 3, equilibrium would occur when blowdowns of inerts are:

- 2080 #/mg organics
- 510 #/mg hydroxyopatite
- 100 #/mg magnesium hydroxide

Continuing our example, assume a coagulant recovery system employing the following unit processes is used:

Centrifugal dewatering and classification Recalcination Dry blowdown of 25% of calciner output

Assuming the following test results are available, calculate the theoretical centrifuge feed, cake output, calciner output and blowdown of solids required and enumerate by type:

Assume 30% of Hydroxyapatite is wasted in centrate Assume 25% of Magnesium Hydroxide is wasted in centrate Assume 25% of Organics are wasted in centrate Assume 10% of Calcium Carbonate is lost in centrate Assume 10% of Ash is wasted in centrate Assume 25% of Calciner output is blown down.

Solution:

eq (14) Apatite to waste = $0.3\chi + 0.25$ (0.7χ) $510 = 0.3\chi + 0.175\chi$ = 1075 #apatite/mg reports in centrifuge feed

eq (15) Mag. hydx to waste =
$$0.25\chi + 0.25$$
 (0.75 χ)
100 = $0.25\chi + 0.19\chi$
= 227 #magnesium hydroxide/mg reports in centrifuge feed
feed

eq (16) organics to waste = 2080 #/mg
This is wasted in two forms i.e., ash and organics
organic equivalent as ash = 0.40 (2080) = 830 #/mg
centrate wasteage = 0.25 x 830 = 208 #/mg
622 #/mg remain and are wasted as ash
ash to waste =
$$0.1\chi + (0.25)(0.9\chi) + (0.25)(622)$$

622 = $0.1\chi + 0.225\chi + 156$
= 1440 # of actual ash/mg report in centrifuge feed

eq (17) Calcium Carbonate to waste =
$$0.1\chi + (0.25)(0.9\chi)$$

 $\chi = 3600 \ \#/mg$ (from Table 1)
Calcium Carbonate wasted = 3600 x 0.325
 $= 1170 \ \#/mg$

By use of equations 14 through 17, Tables 10, 11, and 12 may be constructed.

The example assumes that there will be a net positive blowdown of the inert solids itemized in Table 12. The inerts <u>cannot</u> be recycled.

THEORETICAL FEED, CENTRATE AND CAKE CONTENT AT EQUILIBRIUM IN A COAGULANT RECOVERY SYSTEM

	ALL SLUDGES EXPRESSED IN LBS./MG (DRY SOLIDS)				
PARAMETER	CaCO3	$Ca_5OH(PO_4)_3$	ORGANICS	ASH	Mg(OH) ₂
CENTRIFUGE FEED	3,600	1,075	2,080	1,440	227
CENTRATE	360	323	520	144	57
CAKE	3,240	752	1,5 60	1,296	170

TABLE 11

THEORETICAL CALCINER OUTPUT AT EQUILIBRIUM IN A COAGULANT RECOVERY SYSTEM

	ALL PROD	UCTS EXPRESSED	IN LBS./MG	(DRY SOLIDS)
PARAMETER	CaO	Ca ₅ OH(PO ₄) ₃	ASH	Mg(OH)2
CALCINER OUTPUT	1,820	752	1,920	170
BLOWDOWN (25%)	455	187	480	43
REMAINDER TO REUSE	1,365	565	1,440	127

TABLE 12

COMPARISON OF INERTS ACTUALLY WASTED WITH THEORETICAL INERTS WASTAGE REQUIRED AT EQUILIBRIUM IN A COAGULANT RECOVERY SYSTEM

	SOURCE O	F WASTAGE		THEORETICAL REQUIRED TABLE 3 TOTAL
INERT	CENTRATE	BLOWDOWN	TOTAL	
Ca ₅ OH(PO ₄) ₃	323	187	510	510
Mg(OH) ₂	57	43	100	100
ASH	208+144	480	832	832

Table 13 compares solids handling and lime requirements for solids handling system with and without lime recovery.

The arrangement of the calculations required to determine equalibrium values for chemical sludges in the manner illustrated provides the design engineer with a concise tabulation of the amounts of each type of sludge under any condition he may choose to investigate. This in turn allows an orderly economic evaluation to be made. The designer may choose to evaluate several alternative methods of solids handling ranging from no recovery to sophisticated recovery systems and can, therefore, make a sound decision. In addition, the designer is assured that adequate capacity is provided for the system's needs. Weak points in the system can then be evaluated and standby capacity or redundancy added as may be required or deemed advisable.

TABLE 13

SOLIDS HANDLING AND LIME REQUIREMENTS WITH OR WITHOUT LIME RECOVERY AT EQUILIBRIUM

WITH LIME RECOVERY	WITHOUT LIME RECOVERY
8,422	6,920
1,442	3,462
1,135	2,500
	WITH LIME <u>RECOVERY</u> 8,422 1,442 1,135

SUMMARY

Relatively simple techniques for determining the efficiency of physical-chemical treatment of a given wastewater are available and are described. A brief discussion of design criteria for the major unit processes is presented. The physical-chemical techniques can be applied to any size waste flow including flows of less than 1 mgd. Examples of package physical-chemical units are included in the paper. Finally, procedures for estimating sludge production are illustrated.

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