

WASTEWATER TREATMENT METHODS

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Abstract

Alcoa's Point Comfort, Texas industrial facility is a combination of a bauxite refining plant utilizing the Bayer process and an aluminum fluoride production plant. Due to the location's use of dry stack technology for bauxite residue disposal, the pond surface areas for evaporation are minimal compared to the rainfall catchment areas. This results in the periodic need to reduce accumulated volumes of storm water at the Residue Disposal Area (RDA).

This paper describes the options for treating a combination of wastewaters from the RDA. The current water treatment method utilizes ferric sulfate for total organic carbon (TOC) and metallic ion adsorption. The precipitated solids are separated and the treated water neutralized prior to discharge. Experimental work will also be presented for the treatment of Bayer process water alone.

Introduction

Point Comfort Facility

The Alcoa World Alumina Atlantic (AWA Atlantic) facility at Point Comfort, Texas is located in the Gulf Coast region of the United States. The plant property is bordered by the brackish water bodies of Lavaca Bay to the west and Cox Bay to the south. Along the eastern perimeter lies the Cox Creek watershed. The water in this watershed is impounded for plant fresh water use prior to excess water overflowing into Cox Bay. The industrial facility includes a Bayer Processing plant for production of 2.3 million metric tonnes of alumina per year and a plant for converting alumina trihydrate into 64 thousand metric tonnes per year of aluminum fluoride.

Bayer Process

The Point Comfort Bayer plant processes primarily Boke' bauxite and utilizes a combination of low and high temperature digestion units. After digestion the bauxite residue is washed in a series of counter-current decantation washers. It is then pumped as thickened slurry from the last washer to waste disposal sites located within the facility's Residue Disposal Area (RDA).

At the RDA the bauxite residue is dry stacked at elevations above the exterior containment dikes. The water runoff from the dry stack areas is stored in a separate water reservoir for use in the Bayer Process or transferred to the aluminum fluoride wet disposal area as described below. Other inputs may include rainwater collected off the plant site, water released from the thickened bauxite residue slurry and leachate collected from the

dry stack underdrains. This water will be referred to as Bayer Lakewater.

Aluminum Fluoride Process

The first step in the aluminum fluoride process is to react fluorspar (CaF₂) with sulfuric acid (H₂SO₄) to produce hydrogen fluoride (HF) gas and byproduct gypsum (anhydrous CaSO₄). The next and final processing step is to react the HF gas with alumina trihydrate to form aluminum fluoride (AlF₃).

The gypsum waste material is slurried and transported to a separate area of the RDA using a recycling flow of water. The slurry returning to the RDA is pH 2 but the body of water in the disposal site is maintained above a pH of 7 by the addition of Bayer Lakewater. This transport water will be referred to as Chemicals Lakewater.

Background

Water Management Issues

With the conversion of older wet disposal lakes to dry stacking of bauxite residue, water management in the disposal area becomes an important issue because the rainfall catchment area is now much greater than the evaporative area. This imbalance results from the location objective to maximize the amount of residue stored in the former wet lake areas, creating more open area than is necessary solely for dry stack management. As a result it will be a number years before the site water balance becomes manageable without frequent periods of treatment and discharge of water.

Annual rainfall totals measured at the plant site have ranged from 13.8 to 75.9 inches, with an average of 43.7 inches. Annual pan evaporation has ranged from a minimum of 57.0 to 90.5 inches, with an average of 73.5 inches. Due to dry stacking, the rainfall catchment area in the RDA is 80% greater than the evaporative area. The evaporative area will be further diminished as more acreage is utilized for bauxite residue storage. Ultimately, 90% of the area will be catchment area. There are no typical annual wet or dry seasons for this area, but at any time significant amounts of rain may fall during brief, intense storm events. The Texas Gulf Coast is subject to occasional tropical storms and hurricanes.

Wastewater Discharge Permits

The Federal National Pollutant Discharge Elimination System (NPDES) and the Texas Pollutant Discharge Elimination System (TPDES) permits authorize the discharge of process wastewater and storm water from Point Comfort Operations to Lavaca Bay. The specific effluent characteristics, discharge limitations, and monitoring requirements are shown in Table I. In Table I, the

term “flow MGD” refers to the allowable daily discharge flow in millions of gallons. TOC and TSS are acronyms for total organic carbon and total suspended solids, respectively. Concentrations are stated in units of milligram per Liter (mg/L). The permit limit criteria given in mass loadings have been converted to mg/L based

Table I. Water Permit Limitations

	Daily Average	Daily Maximum
Flow MGD	<4.32	<6.48
pH	6.0-9.0	6.0-9.0
TOC mg/L	55	75
TSS mg/L	101.8	209.8
As mg/L	0.1	0.3
Cr mg/L	0.10	0.36
F mg/L	37.2	78.2
Ni mg/L	0.14	0.47
Se mg/L	0.1	0.3
Zn mg/L	0.37	1.20

on an outfall flow of 2.88 MGD.

The concentration ranges of the lake waters are shown below in Table II for comparison. From Table II it can be seen that the Bayer Lakewater is significantly higher in pH, alumina, TOC, As, Cr and Se. As the Bayer Lakewater is transferred to the Chemicals Lakewater for pH control, metallic ions and TOC are reduced through the mechanism of pH swings, alumina precipitation and gypsum precipitation occurring in that lake system.

Table II. Plant Solution Concentrations

Parameters	Chemicals Lake	Bayer Lake Water
pH	7.2-9.6	12.0-12.3
Al ₂ O ₃ g/L	0.004-0.10	1.5-2.5
TC g/L		2-7
TA g/L	0.2-1.0	12-22
SO ₄ g/L	20-60	0.6-3.1
TDS g/L	30-100	12-28
TOC mg/L	80-135	190-370
As mg/L	0-0.2	1.6-3.5
Ca mg/L	20-600	0.8-2.8
Cr mg/L	<0.01	0.05-0.09
F mg/L	30-70	30-90
Ni mg/L	0.05-0.15	<0.01
Se mg/L	0.09-0.10	0.40-0.70
Zn mg/L	<0.05-0.09	<0.05

Wastewater Treatment Methods

Historically, Point Comfort Operations has had to treat and discharge water to maintain water volumes in the RDA areas. In the early 1990’s Chemicals Lakewater was treated and discharged in order to convert a wet lake to a dry stack area. The parameters of concern were TOC, pH, and TSS. TOC levels in the water ranged from 100 to 120 mg/L. References [1,2,3] are related to treatment of wastewater for TOC removal. The treatment system consisted of injecting ferric sulfate solution into a stream of lake water in a pipeline. The amount of ferric sulfate was controlled to achieve a pH of 5.0. At that pH condition, a high percentage of the TOC absorbed onto the precipitated iron. The pipeline discharged into a series of three shallow settling ponds. Water from the last pond was blended with caustic prior to being pumped to a settler. The settler overflow water passed through media bed filters before being discharged through the designated outfall to Lavaca Bay.

In the late 1990’s, Chemicals Lakewater was treated and discharged due to an accumulation of water in the RDA system because of heavier than normal periods of rain. The element of concern was again TOC but this time the concentration was closer to the allowable discharge levels of 55 mg/L. The treatment system consisted of pumping the water through a series of two activated carbon beds and then into the settler. The settler overflow went directly to the designated outfall without any further filtration.

With the recognition of the water balance issues in the RDA, a permanent water treatment facility has been installed. Chemicals Lakewater is pumped into a stirred reactor where a combination of ferric sulfate and 50% caustic is added to maintain proper pH control for TOC with a target pH of 4.5. The iron precipitate generated is flocculated and allowed to separate in lamella plate settlers. The clear overflow water flows into a second reactor where the final pH control of 6.5 to 7.5 is achieved by addition of 50% caustic. The second reactor overflows into a holding tank and the water is pumped to the previously used settler before discharge to the Bay. The sludge from the lamella settler underflow is pumped back into the RDA.

Additional wastewater treatment methods that have proved unsuccessful with Chemicals Lakewater include pilot plant evaluations using organoclay absorptive media and ultra (micro) membranes for TOC removal and on a Dissolved Air Flotation (DAF) lamella settler for iron solids separation. Filter cartridges upstream of a reverse osmosis treatment process using nanofiltration membranes successfully removed the organic carbon but also removed the sulfate ion. Recycling this reject water back into the Chemicals Lakewater system would result in an unacceptable increase in sulfate concentration.

The purpose of this paper is to present laboratory data that supports the present method of Chemicals Lakewater treatment as well as data on treating Bayer Lakewater separately to meet the permitted outfall criteria presented in Table I. The treatment processes are primarily directed at lowering TOC using an iron precipitate as an adsorption media. Additional steps discussed for treatment of Bayer Lakewater include neutralization with either sulfuric acid or carbon dioxide, and both physical and chemical methods for enhancing alumina settling characteristics. Reductions of the arsenic and selenium ion concentrations are also discussed and references [4-8] are related to removal of these ions from wastewater.

Experimental

Commercial ferric sulfate solutions were used as a treatment chemical. These solutions nominally contained 12% Fe by weight with essentially no free acid. Additional reagent grade chemicals used included dry solid forms of CaO, CaCl₂, FeCl₃, NaOH, and FeSO₄•H₂O. Reagent grade sulfuric acid was used as well as CO₂ from a compressed gas cylinder.

A variety of anionic and cationic flocculants from Cytec were also tested for settling the precipitated solids.

In general, the experiments were carried out in stirred beakers containing an electronic pH probe to monitor solution pH. The volume of water to be treated in individual experiments varied from 250 mL to 2 Liters. Procedures specific to the individual experiments will be discussed along with the data for those experiments.

Point Comfort laboratory's automated titration equipment measured the concentrations of alumina (Al₂O₃), total caustic (TC), and total alkalinity (TA). TC and TA are the North American convention of expressing soda concentrations in terms of sodium carbonate. Also, the location's Dohrmann DC-80 was used to report TOC concentrations. Analyses of other ions were made by local accredited laboratories.

Data and Results

Chemicals Lakewater Treatment

1.0 Neutralization Experiments Using Ferric Sulfate - A series of beaker experiments were performed by adding incremental amounts of the acidic ferric sulfate solution to the initially basic Chemicals Lakewater to determine the TOC removal response through changing pH. Figure 1 plots the response of TOC as a function of pH. The g/L Fe needed for treatment is also shown on the secondary axis curve.

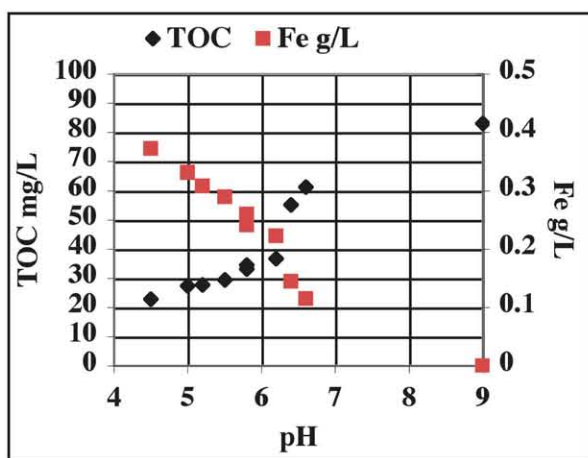


Figure 1. Chemicals Lakewater Iron Treatment Response Curve

TOC removal is the result of adsorption on the iron precipitate formed at the pHs shown. The point at which the maximum amount of TOC is removed is around a pH of 4.5 but with an initial TOC of 83 mg/L, the iron is effective at reducing the TOC to below 55 mg/L in the 4.5 to 6 pH range. Above pH 6.5 there is

a sharp upward break in the curve. The plotted curve for TOC implies, and further experiments confirmed, that the adsorption of TOC on the iron precipitate is reversible which necessitates removal of the iron precipitate before neutralization of the treated water back to pH 7 for discharge. Not shown on the graph is that below pH 4 the iron floc re-dissolves and releases all of the TOC back into solution.

2.0 Ferric Treatment For TOC Removal - The Chemicals Lakewater was treated in laboratory experiments with both ferric sulfate and ferric chloride. Over the timeframe of this experimental work, the TOC concentrations in the lake water decreased for various reasons. In Figure 2 data labeled A represent a starting TOC of 128 mg/L while those labeled B contained 98 mg/L. In these experiments, incremental amounts of iron were being added to the lake water solutions while maintaining the targeted pH with additions of either sulfuric acid or caustic solutions.

The data shown for Solution A at pHs of 4.5 and 5.0 have been combined in the second order polynomial correlation curve and indicate that the iron contained in either ferric chloride or ferric sulfate is equally responsive for TOC removal on a g/L Fe basis. It is also apparent that it is difficult to differentiate between the response data at pH 4.5 and 5.0. The curves for pH 6A and 7A show that the addition of larger amounts of iron at the same pH will result in lower levels of TOC in the treated water. From this it can be concluded that operating in the lower 4.5-5 pH range reduces the amount of the ferric sulfate needed to be below the limit of 55 mg/L TOC. The curve for pH 4.5 Solution B indicates that there is a significant shift in the iron response curve depending upon the start TOC concentration.

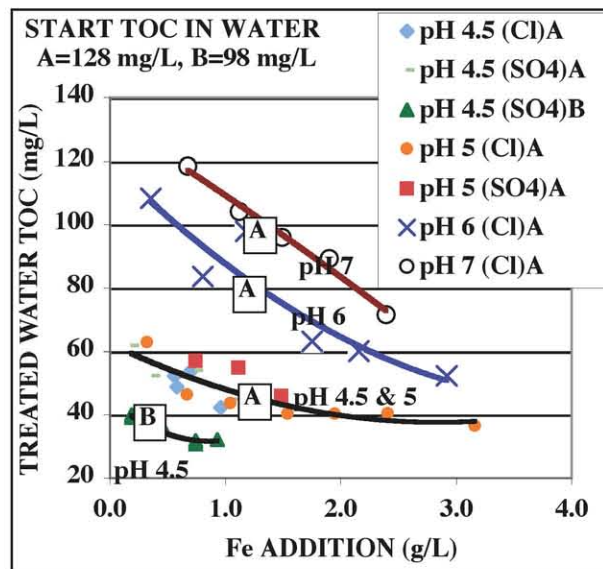


Figure 2. Chemicals Lakewater Treatment For TOC Removal With FeCl₃ & Fe₂(SO₄)₃

3.0 Correlation Based On TOC Difference - This change in response depending upon the start TOC led to the question of whether the TOC • (difference from start to finish) versus g/L Fe addition might be a better way of representing data for different TOC start water concentrations. Figure 3 shows a re-plot of the pH 4.5 and 5.0 data for the two waters and indicates that there is

still a shift in the response curve for the two starting TOCs. This shift can be explained by the fact that the response curves are not linear particularly at increments of very low iron addition.

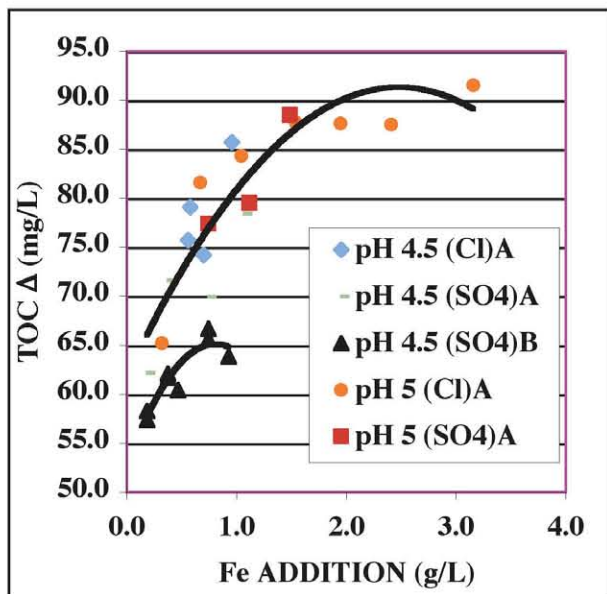


Figure 3. Chemicals Lakewater TOC Treatment

4.0 Flocculation of Iron Precipitates - Several flocculants from Cytec Industries were examined for use in settling the iron precipitate formed in the stirred tank reactor at the water treatment facility. Criteria for success included identifying a flocculant that would effectively settle the iron precipitate in the lamella clarifier, thus providing an overflow with minimal suspended solids while not adding organic carbon to the treated water concentration. Additionally, the flocculants had to be nontoxic in a marine environment. A third aspect for flocculant selection was the ability to develop a feasible system for flocc addition given the remote location of the water treatment facility. Preliminary laboratory screening of flocculants included cationic and anionic charges as well as products in the form of a dry powder and water-in-oil emulsion.

Flocculants were screened at a process pH range of 4.5 to 5.5. Results from this work showed that an anionic emulsion based product, SUPERFLOC® A-1849RS, was the most effective. Testing done between emulsions and dry powder flocculants showed that emulsions had a negligible contribution towards the TOC of the treated lake water, and therefore were suitable for use in the process.

Due to the remote location of the treatment system, there were limitations on the power available for flocculant equipment and choices of water for preparing a flocculant solution. To minimize power requirements, a decision was made to use an emulsion based product instead of a dry powder. The only options for water for the preparation of a flocculant solution were treated or untreated lake water. Use of this water was challenging due to the high level of salts, such as calcium, that would adversely affect the performance of the flocculant. The impact of these salts on the flocculant was minimized by the addition of a small amount of 50% NaOH to the lake water before it was used to invert the flocculant emulsion.

5.0 Plant Treatment Results - The results of ferric sulfate treatment at pHs of 4.5 -6.0 (depending upon start TOC) were successful in controlling TOC to less than 55 mg/L. Use of SUPERFLOC® A-1849RS successfully flocculated the iron precipitate and thus controlled TSS in the treated water. In general the concentrations of the remaining ions are below permit limits.

Bayer Lakewater Treatment

1.0 Neutralization Experiments Using Sulfuric Acid - The initial set of laboratory experiments with Bayer Lakewater tested the TOC response to pH changes caused by incremental additions of sulfuric acid. The experimental procedure used 2-Liter quantities of lake water at a starting pH of 12.2 with addition of sufficient acid to lower the pHs to various levels ranging from 9 to 4.5. Figure 4's top curve illustrates the TOC response from the initial concentration of 280 mg/L to a low point at pH 5, where 66% of the TOC has been removed. TOC is reduced over this pH range by the mechanism of adsorption of organic carbon on the precipitated alumina originally present in the lake water.

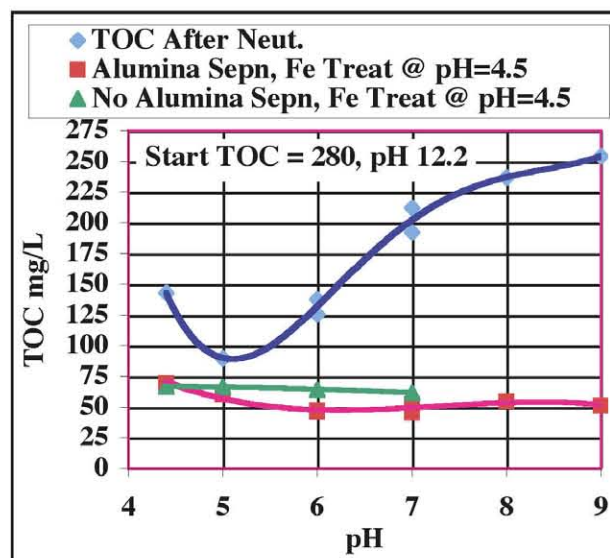


Figure 4. Bayer Lakewater Neutralization

2.0 Ferric Sulfate Treatment Of Neutralized Bayer Lakewater - The neutralized lake waters from the above experiments were further treated with ferric sulfate to a pH of 4.5 in an attempt to lower the remaining TOC to the target level of less than 55 mg/L. The water from each experiment was divided into two aliquots. In the first set of aliquots, the solids from the neutralization process were removed using vacuum filtration. The solids were left in the second set. As the two lower curves in Figure 4 demonstrate, TOC concentrations below 55 mg/L could be achieved only with solids removal. Without solids removal, the TOC remained above 60 mg/L and stayed constant at any of the treatment pHs.

The precipitated alumina formed by this neutralization is a high volume gel type precipitate that does not readily settle. Addition of SUPERFLOC® A-1849RS flocculated the solids but a very high volume of settled mass still remained; about 40% of original volume.

3.0 Experiments To Avoid Alumina Gel Formation - To avoid some or all of the alumina gel formation, a series of laboratory experiments were performed with 30-minute lime addition as a first step in the water treatment process. In this series of experiments variables included:

1. The form of lime added – CaCl₂, dry CaO or 100 g/L slaked CaO
2. Lime addition at room temperature (RT) or to heated lake water (HT)(80-95 °C)
3. Removal (by settling or filtration) or no removal of the lime solids prior to neutralization
4. Neutralization with sulfuric acid or CO₂
5. Removal (by filtration) or no removal of any alumina solids prior to iron treatment
6. Treatment at pH 5 with ferric sulfate

Figure 5 summarizes the results of the lime experiments. The legend sequence means:

1. RT or HT refers to whether the lake water was at room temperature or heated during the lime reaction.
2. The first Y or N refers to whether the lime solids were removed before neutralization.
3. The second Y or N refers to whether the alumina solids were removed prior to lime treatment.

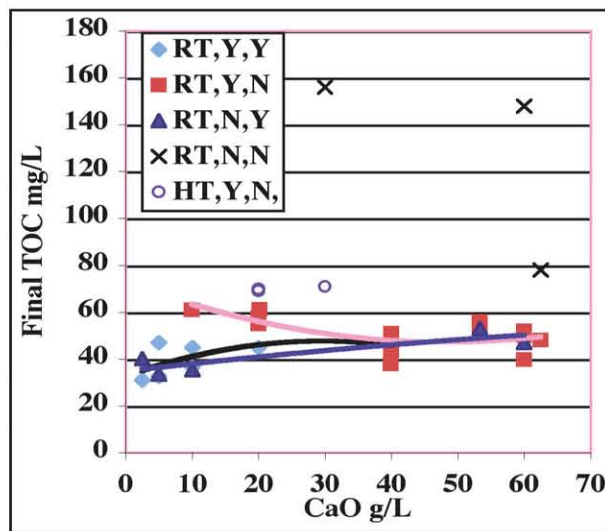


Figure 5. Bayer Lakewater Lime Pre-Treat Data (12 Fe g/L Addition)

Conclusions drawn from these and additional experiments were:

- On a g/L CaO basis, the form of the lime addition had no impact on the final results.
- Heating the water for the lime reaction (HT,Y,N) was not beneficial from a TOC removal standpoint.
- If the lime solids and the alumina solids were not removed prior to ferric treatment (RT,N,N), then the TOC values were unacceptably high.
- Between 20 and 40 g/L CaO additions all of the alumina reacts with the lime solids. Above 40 g/L, the trendlines merge. Below 40 g/L the trendlines diverge depending on whether the alumina was removed (RT,Y,Y; RT,N,Y) or not (RT,Y,N) with the TOC results going above 55 mg/L for those experiments with no alumina removal. These results

are similar to the previous ones obtained during no lime addition experiments.

- Neutralization with either sulfuric acid or CO₂ did not change the final TOC results. A problem with CO₂ is that all of the CO₂ is released with vigorous bubbling and foaming when the pH of CO₂ neutralized solutions are driven down into the 4.5-6 pH range for the final ferric sulfate treatment.

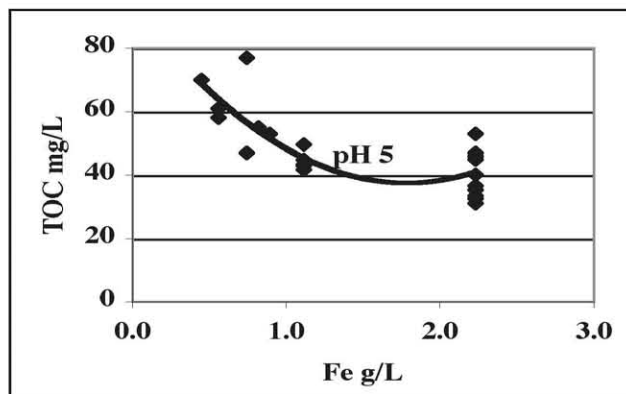


Figure 6. Bayer Lakewater (All solids removed prior to ferric treatment)

Figure 6 illustrates the results from a subset of experiments where all solids were removed prior to ferric treatment. Variable amounts of iron were added as well as sulfuric acid or sodium hydroxide to maintain a constant pH of 5. The curve in Figure 6 is very similar to the curve in Figure 2 for Solution A, the high TOC Chemicals Lakewater.

An overall conclusion reached from the above data is that the addition of lime was not effective enough to overcome the problem of alumina solids. The addition of lime also did not reduce the amount of iron needed to achieve less than 55 mg/L TOC.

4.0 Experiments To Change Alumina Settling Characteristics

A. Change In Physical Form - Additional laboratory experiments were made to attempt to change the high volume nature of the alumina gel. Fine alumina trihydrate seed was added prior to Bayer water neutralization to determine if the preformed seed would reduce the precipitate volume or act as a settling aid, with no positive results. Additionally, 50 g/L on a dry solids basis of last stage washer underflow slurry from the Bayer Plant was added to the lake water. The resultant solution was separated into two aliquots, with the sets neutralized to below pH 10 by either sulfuric acid or CO₂ addition. At this pH, essentially all of the alumina has precipitated. These experiments were moderately successful because the resultant bauxite residue/alumina gel formed in the pH 9 to 10 range would settle to a volume of 200 mL per Liter of solution after synthetic flocculant addition. After solids separation and ferric sulfate treatment, the TOC vs. Fe response curve was identical to Figure 6 above.

B. Laboratory Flocculation Studies - Synthetic flocculants from Cytec Industries were tested to improve the settling of the alumina gel precipitate. In treatment strategies that consisted of neutralizing the Bayer Lakewater with sulfuric acid or CO₂ to a pH of 7.0-9.0, a highly charged anionic flocculant, such as SUPERFLOC® 1227, or a highly charged cationic, such as

SUPERFLOC® 4518 were found to be the most effective in timed settling tests.

Synthetic flocculants were also tested in the treatment option involving the addition of last stage washer underflow to Bayer Lakewater prior to neutralization of the water. Testing focused on identifying a flocculant that would be effective as the pH of the water was reduced to a range of 9 to 10. Most of the flocculants tested were anionic, with varying degrees of charge. Test results indicated that the lower charged anionic flocculants were sensitive to pH and did not perform well as the pH was gradually reduced. SUPERFLOC® 1227 outperformed all the other flocculants tested.

Following solids separation and ferric sulfate treatment of the various options discussed above for Bayer Lakewater, it was found that SUPERFLOC® A-1849RS is also effective in settling the iron precipitate formed during this process.

5.0 Removal of Arsenic and Selenium - Table III portrays the resulting ion concentrations for arsenic and selenium after various laboratory treatments discussed above followed by ferric sulfate addition for TOC removal. Ferric sulfate reduces As to less than 0.01 mg/L but the Se ion is not reduced to the permit limit of 0.1 mg/L except with lime addition. Data from a limited number of additional experiments with a combination of ferrous and ferric sulfate are found in the right hand columns in Table III. These show that the ferrous addition will further reduce Se to well below 0.1 mg/L in all cases tested.

Table III
Treated Bayer Lakewater Ions Of Concern

	Ferric Sulfate Alone		Both Ferric & Ferrous Sulfate	
	As mg/L	Se mg/L	g/L FeSO ₄	Se mg/L
Neutralization to pH = 6	<0.01	0.07		
Neutralization to pH = 7	<0.01	0.17	1	<0.01
Lime Addition Neut to pH =7	<0.01	0.07	0.5	0.05
Last Stage Washer U'flow Residue to pH =9	<0.01	0.11	0.2	0.06

6.0 Laboratory Treatment Results - The Bayer Lakewater tested in these experiments could be treated to reduce TOC and other metallic ions to meet outfall criteria. The presence of alumina in this lake water, however, was a challenge because of the very high volume of precipitate that forms as the water is neutralized. Lime pre-treatment to remove the alumina was not promising and the use of flocculants or flocculating aids, such as bauxite residue, were of limited success because of the high volume of the

remaining solids. SUPERFLOC® 1227 was the most effective of flocculants tested for the alumina/residue mixtures. SUPERFLOC® A-1849RS successfully flocculated the iron precipitate as in the case of the Chemicals Lakewater.

Conclusions

Laboratory work and installation of a treatment plant have demonstrated that the Point Comfort Chemicals Lakewater can be successfully treated and discharged while meeting all permit ion parameters. Additional laboratory experiments also show that Bayer Lakewater can be treated to meet these same outfall standards. Future work includes laboratory and field testing of mechanical separation techniques for the alumina precipitate.

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