EVALUATION OF SLUDGE PRODUCED BY LIMESTONE NEUTRALIZATION OF AMD AT THE FRIENDSHIP HILL NATIONAL HISTORIC SITE[1]

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Abstract. A pulsed limestone bed (PLB) treatment plant for remediation of acid mine drainage (AMD) has been tested at the Friendship Hill National Historic site in southwestern Pennsylvania. The plant performed well, neutralizing over 50 metric tons of acidity over a 14-month period, but the cost of disposal of the 450 metric tons of sludge generated was high, because the sludge was hauled from the site and disposed of in a commercial landfill to minimize the impact on park property. Although the sludge was found to be non-hazardous based on the Toxicity Characteristics Leaching Protocol (TCLP), hauling and disposal costs were still elevated, at \$40 per m^3. Since sludge handling and disposal was a significant fraction of the operating cost of the facility, a study of the sludge characteristics and possible alternate handling methods was undertaken. Samples of AMD influent, treated water and settled sludge were used in a series of small-scale treatment studies. These tests showed that the sludge volume produced by limestone neutralization was less than half of that for lime, sodium hydroxide or ammonia. The settling rate was also greater for the limestone-based sludge. Vacuum filtration tests demonstrated that the limestone sludge was more readily filtered than sludges generated with sodium hydroxide or lime and that solids contents of up to 28% could be achieved. Because of lower maintenance costs, pressure filtration using a plate and frame filter is recommended for future plant operations. In this case, solids contents of as high as 31% were realized. As an alternative to the operating costs of mechanical filtration, a settling/percolation process was tested. Percolation tests showed that solids content of the settled sludge could be increased from 8% to 25%. These results show that sludge disposal costs for the treatment plant could be decreased through installation of a settling or filtration process. Moreover, the PLB process offers saving not only in reagent costs, but also in sludge disposal costs, through decreased sludge volume generated, rapid settling rates and ready filterability.

[1] Paper was presented at the 2003 National Meeting of the American Society of Mining and Reclamation and The 9th Billings Land Reclamation Symposium, Billings MT, June 3-6, 2003. Published by ASMR, 3134 Montavesta Rd., Lexington, KY 40502.

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Proceedings America Society of Mining and Reclamation pp 1151-1169
DOI: 10.21000/JASMR03011151

https://doi.org/10.21000/JASMR03011151

Introduction

Acid mine drainage (AMD) is a widespread environmental issue resulting from oxidation of exposed sulfide minerals, primarily pyrite, by air in the presence of moisture. In the state of Pennsylvania alone, over 5000 km of streams have been impaired by acidic discharges, with an estimated total remediation cost of over $5 billion (U.S. EPA, 2000). More economical methods of AMD treatment would enable more efficient use of scarce remediation funds in addressing this issue. A new process developed at the USGS Leetown Science Center uses pulsed fluidized beds of limestone to neutralize AMD. Although limestone is the most economical reagent available for acid neutralization, its use in AMD treatment has been limited due to two factors: slow dissolution rate, and formation of an impervious coating on the limestone surface, termed armoring. The new pulsed limestone bed (PLB) process overcomes these problems through the addition of carbon dioxide to enhance limestone reactivity, and through pulsing of the limestone in a fluidized bed, which results in a high attrition rate at the limestone surface. The PLB process has been described previously (Watten, 1999; Sibrell, et al., 2000). An important issue in the treatment of AMD is disposal of the waste sludge resulting from the precipitation of the metals contained in the AMD, especially for those sludges found to contain hazardous materials. Acid mine drainage sludges are notorious for their voluminous, low-density characteristics due to the floc-like nature of the iron oxide precipitates (Brown, et al., 1995). Over the years, several approaches have been identified that result in improved sludge handling characteristics. The use of staged pH neutralization as employed in the various high-density sludge processes results in a sludge that is easier and cheaper to handle (Murdock, et al., 1994). It has also been found that sludges from passive treatment systems using limestone as the neutralization agent tend to be more readily settled or filtered than lime sludges (Dempsey and Jeon, 2001). Since the PLB process is based on limestone and staged neutralization (due to pH buffering by CO_2), sludges produced by the PLB process should share these superior settling/dewatering characteristics. This paper examines the production and characteristics of the waste sludge produced by the PLB process at the National Park Service's Friendship Hill National Historic Site, near Point Marion in southwestern Pennsylvania.

The AMD at the Friendship Hill site is characterized as an oxidized acidic discharge, with a pH of 2.7, iron content of about 200 mg/L and acidity of over 1000 mg/L. Based on a survey of AMD sites in Pennsylvania (Rose and Cravotta, 1998), the acidity is greater than 90% of discharges in the state. A 230 liter per minute (L/min) pilot plant was operated at this site from June 2000 through August 2001 to evaluate the applicability of the PLB process, and to characterize the sludge produced as well as its impact on treatment costs. During the test period, the plant neutralized 50 million liters of AMD and produced over 450 metric tons of wet sludge. Due to site aesthetics, the sludge was hauled from the site and disposed of at a commercial landfill. The cost of this disposal method (\$40 per m^3) was high, representing 33% of the total treatment costs. In the present study, we conduct small-scale tests of the filterability and settleability of the sludge to establish the design and cost of an alternative on-site sludge disposal process.

Methods

The PLB system was sized to treat a maximum of 220 L/min of flow. The system consists of four 61-cm (24 in) diameter by 213 cm (84 in) tall fiberglass columns containing limestone, and one 46-cm (18 in) diameter by 213 cm tall column (the carbonator) functioning as a packed tower for CO_2 absorption into the water (Figure 1). Incoming AMD is routed to one set of two columns containing limestone. The limestone particle size is roughly 0.1 to 1.0 mm. The flow fluidizes a single limestone bed for a period of one minute. The flow is then diverted to the other column for the same time period, allowing the bed in the first column to settle. This rinse/recharge phase continues for four minutes. Treated water is discharged to the drain continuously. This pulsed-bed operation allows higher flow rates to be passed through the limestone bed, providing for better mixing and scouring of the particle surfaces. The flow path is directed by an electrically actuated ball valve operating on a time-based program. Meanwhile, in what is termed the treatment phase of the PLB process, the water in the second set of two columns is recirculated through the carbonator, where CO_2 can be added to the water from a pressurized cylinder. As in the rinse/recharge phase, the water is diverted back and forth between the column pair on a one-minute cycle for a total of four minutes. A second set of ball valves is then actuated, and the columns receiving incoming water are switched to the treatment mode, and those in the treatment mode are switched to receive and discharge water. Thus, at any one time, one out of the four columns is receiving and discharging water, and one of the columns is receiving water recycled through the carbonator.

The PLB system recovers and reuses some of the CO_2 dissolved in the effluent. The amount of CO_2 generated is proportional to the incoming acidity, and in samples with high influent acidities, sufficient CO_2 can be generated so that no makeup CO_2 is required, thus further decreasing reagent costs. After discharge from the columns, the effluent is sent to a stripping tower, where air is passed counter-current to the water through a packed bed. The CO_2 is stripped from the effluent and recycled to the incoming water to increase the concentration of dissolved CO_2. Water exiting the stripping unit is routed first through a 7.9 m^3 diffused air aeration basin and then into four parallel settling tanks each with an effective volume of 15 m^3.

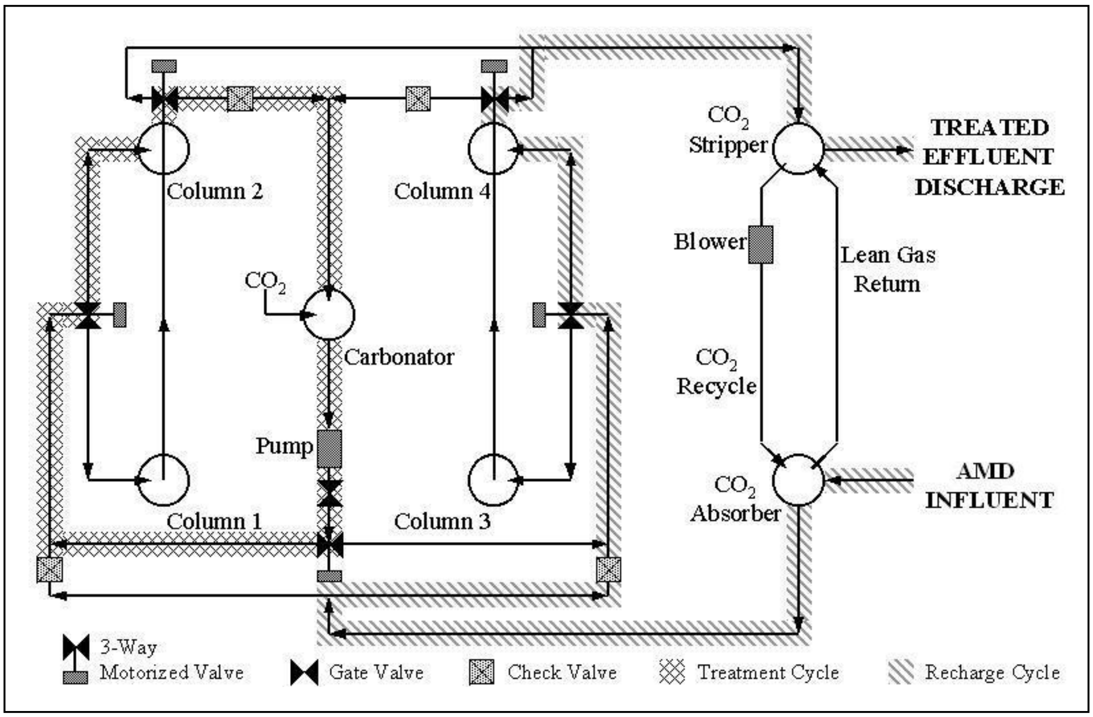


Figure 1. Pulsed limestone bed process schematic including a carbon dioxide recovery and recycle component.

Plant influent and effluent samples were analyzed approximately every three weeks from June 2000 through August 2001 for metals and total suspended solids by Geochemical Testing, in Somerset, Pennsylvania. The settleable solids content of treated water samples at the plant was measured using a volumetric procedure with 1.0 L Imhoff cones (American Public Health Assoc., 1995). Additional samples of plant feed, product water and waste sludge were transported to the Leetown Science Center in Kearneysville, West Virginia for further testing. Samples were kept in a walk-in cooler to minimize aging effects. Settling tests were done by adding one liter of well-mixed pulp to a stoppered graduated cylinder, inverting three times, and recording the level of the solid liquid interface as a function of time with a stopwatch. Flocculation tests were conducted with seven different formulations of long chain polymeric flocculants produced by Ciba Specialty Chemicals. Test procedures for settling tests and flocculant screening and dosage trials followed those given by the flocculant manufacturer (Ciba, 1998). Vacuum filtration tests were conducted using a 9 cm Buchner funnel, with various grades of Whatman filter paper. Vacuum was generated by a Gast diaphragm pump, monitored through a class “A” vacuum gauge and regulated with a bleed valve. Pulp and cake water content were measured by weighing before and after drying at 105 ºC overnight, to determine the water lost by difference. Pressure filtration testing was performed at a manufacturers’ operation for a test sample shipped directly from the Friendship Hill site. A whole rock analysis was conducted on a sample of the dried sludge using X-ray fluorescence methods by Bondar Clegg, Inc. in Sparks, Nevada. K Chem Lab, Inc., in Latrobe, Pennsylvania performed a Toxicity Characteristics Leaching Protocol (TCLP) analysis of the sludge for determination of its status as a hazardous or non-hazardous waste.

Results and Discussion

Plant tests

The AMD neutralization plant was run continuously, except for maintenance shutdowns, from June 2000 through August 2001. Despite an influent acidity of over 1000 mg/L and iron content of 200 mg/L, the pH of the plant effluent averaged 6.5, with a minimum of 5.8, and effluent alkalinity ranged from 50 to 300 mg/L, depending on plant operating conditions. Average values of the pertinent water quality parameters over the entire course of treatment are shown in Table 1. Metal removal was good for Al and Fe(III). However, except under high-flow conditions in the spring, a significant fraction of the Fe in the plant effluent was in the Fe(II) state. Some of the Fe(II) was removed by

oxidation to Fe(III) after pH adjustment in the pulsed limestone bed, but not all Fe(II) was removed because of limited aeration capacity. Manganese was not affected by the limestone treatment due to the elevated pH at which Mn undergoes hydrolysis and precipitation. These factors resulted in a measurable acidity in the process effluent at certain times of the year. However, overall acidity removal for the plant was over 95%.

Table 1. Average values for treatment plant influent and effluent composition. Standard deviation shown in parentheses.

Sample	pH	Acy	Alk	Al	Fe	Mn
Influent	2.7 (0.1)	1095 (121)	0	61 (8)	200 (39)	11 (1)
Effluent	6.5 (0.3)	35 (61)	85 (49)	3 (2)	39 (33)	10 (1)

Over the course of plant operation, 50 million liters of AMD were treated, and over 450 metric tons (MT) of metal hydroxide sludge removed. The cumulative sludge production rate is shown in Figure 2, which also shows the plant treatment flowrate. As would be expected, that the rate of sludge production was proportional to the plant treatment rate as was the limestone ($CaCO_3$) addition.

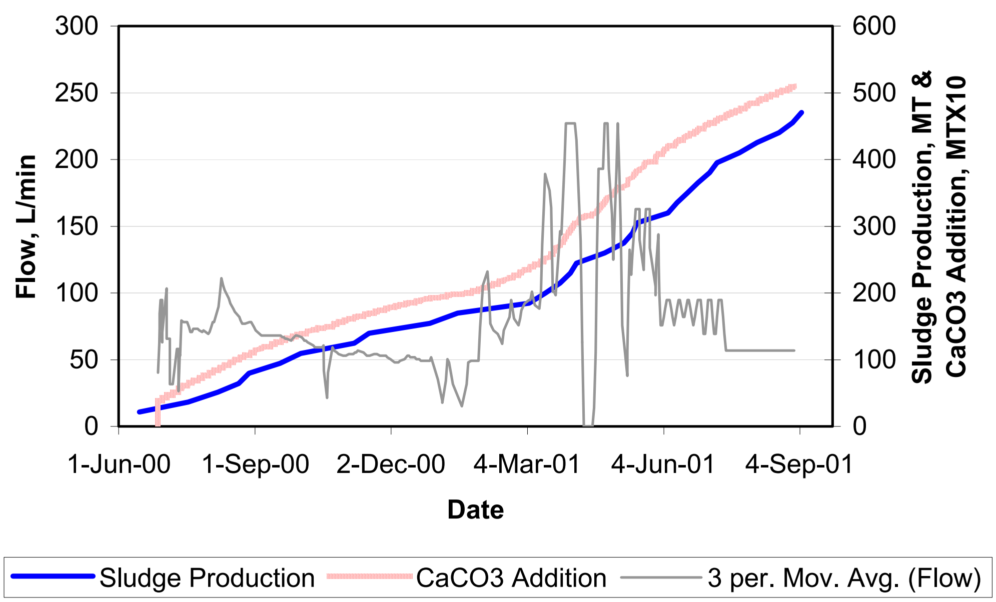


Figure 2. Sludge generation at the Friendship Hill site.

A sample of sludge generated at the Friendship Hill site was analyzed for elemental composition, by X-ray fluorescence (Table 2).

Table 2. Bulk X-Ray Fluorescence analysis of AMD Sludge. Composition reported in percent.

Sample	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	SO_3	LOI	Total
FRHI AMD sludge	6.82	18.66	33.15	0.04	0.43	5.89	7.19	28.73	101.18

The main components of the sludge are aluminum and iron hydroxide (oxyhydroxides), gypsum, and silica. The loss-on-ignition (LOI) figure shows bound waters of hydration, and possibly, unreacted carbonate from the limestone. Table 3 shows the results of the TCLP test. All metal concentrations in the TCLP leachate were below regulatory limits, thus the sludge would be classified as non-hazardous.

Table 3. Toxicity Characteristics Leaching Protocol (TCLP) test results.

Element	As	Ba	Cd	Cr	Pb	Hg	Se	Ag
Regulatory Limit	5.0	100	1.0	5.0	5.0	0.2	1.0	5.0
FRHI AMD Sludge	0.015	0.10	0.02	0.13	0.03	<0.0005	0.008	<0.01

Typical results of the Imhoff cone measurements (June 2001) showed settleable solids content of 28 to 29 mL/L sample in the limestone column effluent, and less than 0.5 mL/L in the clarifier overflow, indicating a removal efficiency of more than 98%. A second indicator of the solids separation efficiency, the total suspended solids (TSS) of the plant effluent, was measured approximately every three weeks during plant operation. The results showed a TSS of less than 30 mg/L during the spring and early summer months. However, during the fall and winter months the TSS rose to as high as 120 mg/L, probably due to incomplete removal of Fe(II), and subsequent precipitation of iron hydroxide solids. Improvement of the air-stripping step used should help to alleviate this problem. However, these results generally indicate good solids removal efficiency for sludge produced by the pulsed limestone bed neutralization process, with as little as two hours settling time, and without the addition of any settling aids.

The typical solids content of the settled sludge was 5 to 10%. Due to aesthetic concerns, the sludge was removed from the property and disposed of offsite by a contractor. The sludge was transported to a filtration/disposal site using a septic system-type pumping truck, with a capacity of 3500 gallons. The sludge disposal cost was $40.00/m^3$ (15¢/gallon). Thirty-three sludge pickups were made, at a total cost of $17,500. Since this was a substantial fraction of the total plant operating cost, an alternative to offsite disposal would be preferable. Possibilities included purchase of a mechanical filter for minimization of the sludge quantity or installation of a settling/ percolation basin on site, with subsequent solid disposal. Small-scale tests of these options were therefore conducted on site as well as at the laboratory at the Leetown Science Center.

Laboratory tests

Settling tests. Settling tests were performed on samples of treated water from the pulsed limestone bed process at the Friendship Hill site. For comparison to the pulsed limestone bed process, settling tests were also performed for Friendship Hill AMD treated with other neutralization agents. The reagents used were hydrated lime [$Ca(OH)_2$], sodium hydroxide [NaOH], and ammonium hydroxide [NH_4OH]. These reagents were added to 1 liter of AMD until a target pH of 7.5 was reached. The settling rate was recorded for each cylinder as well as final sludge volumes. The settling rates are shown in Figure 3. The limestone treated pulp settled most rapidly, but left a hazier supernatant, which made it difficult to follow the solid liquid interface. The $Ca(OH)_2$ pulp settled at an intermediate rate, and the NH_4OH and NaOH pulps were the slowest to settle, although the supernatant was the clearest in these samples. The supernatant clarity and sludge volume at 1 hour and at 20 hours are shown in Figures 4a and b. For the limestone pulp, the volume of sludge after 20 hours of settling was just 20 mL/L pulp, and indicates a clear advantage for limestone neutralization. The NH_4OH sludge was the most voluminous, at 120 mL/L. The NaOH sludge volume was slightly less, at 100 mL/L, and the $Ca(OH)_2$ sludge volume was 50 mL/L. Thus limestone neutralization generated 50% less sludge than $Ca(OH)_2$, and 70 to 80% less than NH_4OH or NaOH. The solids were filtered from the pulps, and dried and weighed. All of the solids had nearly the same weight, about 0.7 g dry solids per liter of water treated.

Polymeric flocculants are often used to increase supernatant clarity. A suite of seven flocculants including anionic, cationic and nonionic forms with varying molecular weights was tested on samples of treated water from the Friendship Hill site. A prescreening test was performed with all of the flocculants using an addition of 5 mg/L to one liter of Friendship Hill

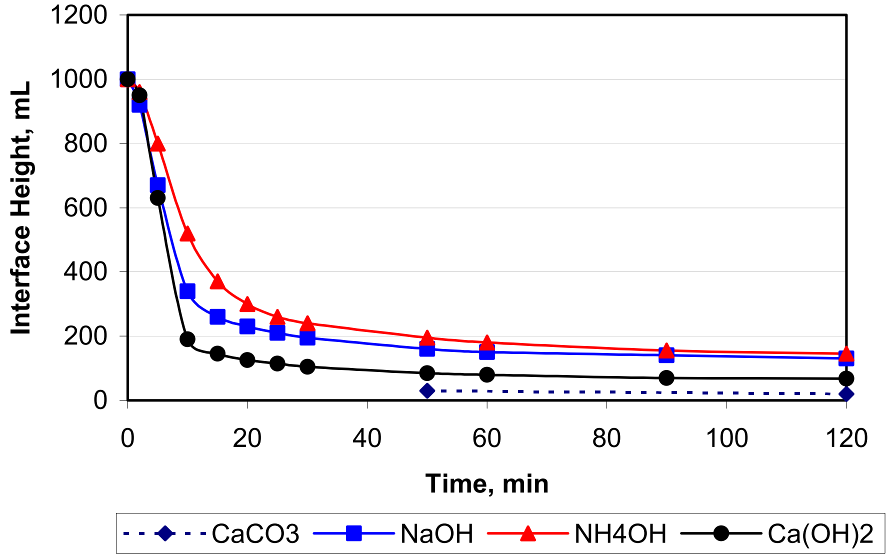
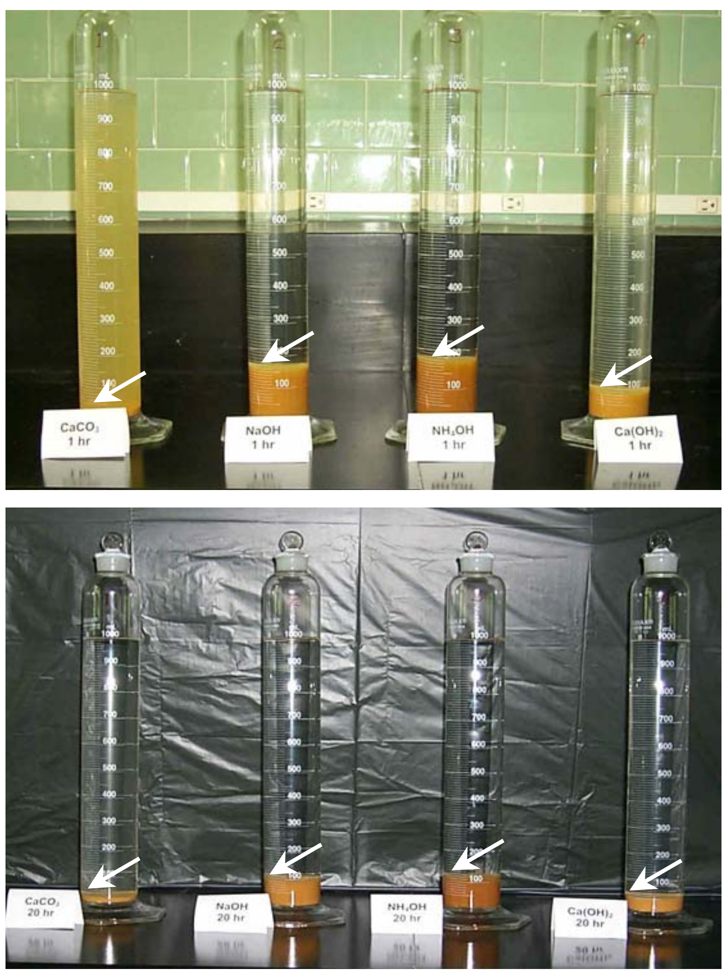


Figure 3. Settling curves for various neutralization reagents.



Figures 4a and b. Settling results after 1 and 20 hours.

water. The flocculant was prepared as a solution of 0.1% strength, and then 5 ml added with a syringe to the sample. The sample was then hand stirred with a spatula for approximately three minutes and allowed to settle. All of the flocculants improved the rapidity of the settling and the clarity of the supernatant. However, Percol E-10 performed better than most of the others, so it was selected for further testing. The effect of the flocculant dosage was tested at additions of 0, 5, 10 and 20 mg/L. All of the pulps with flocculant addition settled quickly, within 60 seconds, however, the volume of the settled pulp was almost doubled, from 20 to 40 mL/L. It was decided that the expense of the flocculant and the increased sludge disposal cost offset the greater clarity of the supernatant, so flocculant testing of the product water was not pursued at the Friendship Hill site.

Vacuum filtration tests. Another method of reducing the cost of sludge disposal would be to reduce the sludge volume by rejecting more water. Filtration is one possible method of doing so. A suite of vacuum filtration tests was undertaken in the laboratory to determine the optimum conditions for filtration. Filtration data can be analyzed based on the Poiseuille Equation (Dahlstrom, 1985; Perry and Chilton, 1973):

$$\frac{dV}{Adt} = \frac{P}{\mu\left(\frac{\alpha wV}{A} + r\right)} \qquad (1)$$

where V= Volume of filtrate, m^3

A=Area of filter, m^2

t=time, s

P=Pressure drop across filter, N/m^2 (Pa)

μ=fluid viscosity, $N\bullet s/m^2$

w=weight of dry cake solids, kg/m^3

α=specific resistance of the filter cake, m/kg

r=media and drainage resistance, m^{-1}

Rearranging,

$$\mu\alpha wV\frac{dV}{A^2}+\mu r\frac{dV}{A}=Pdt \qquad (2)$$

At constant pressure, and with constant temperature and feed conditions, we can integrate to get:

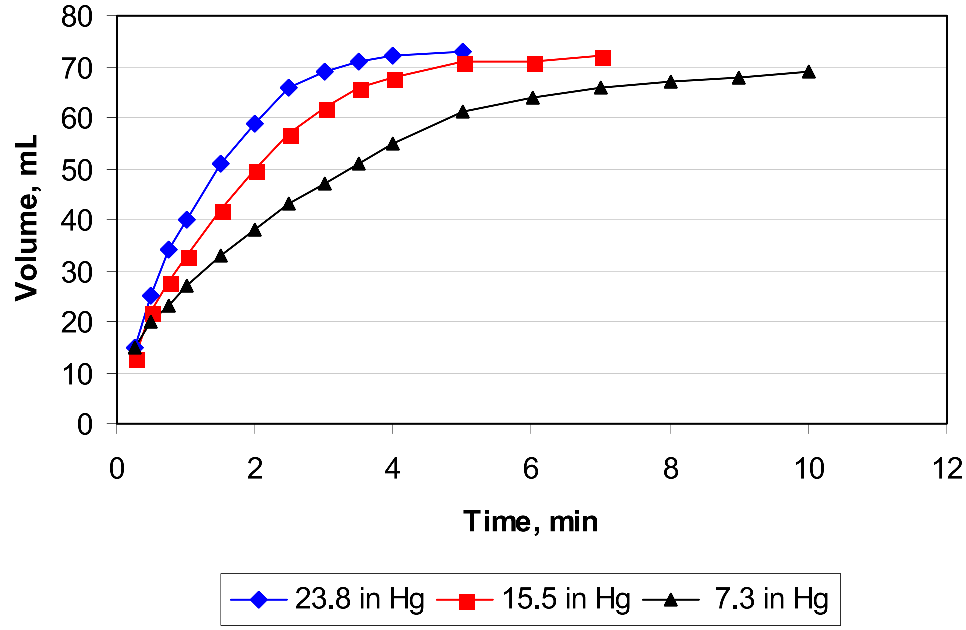
$$\frac{\mu\alpha wV}{2A^2}+\frac{\mu rV}{A}=Pt \qquad (3)$$

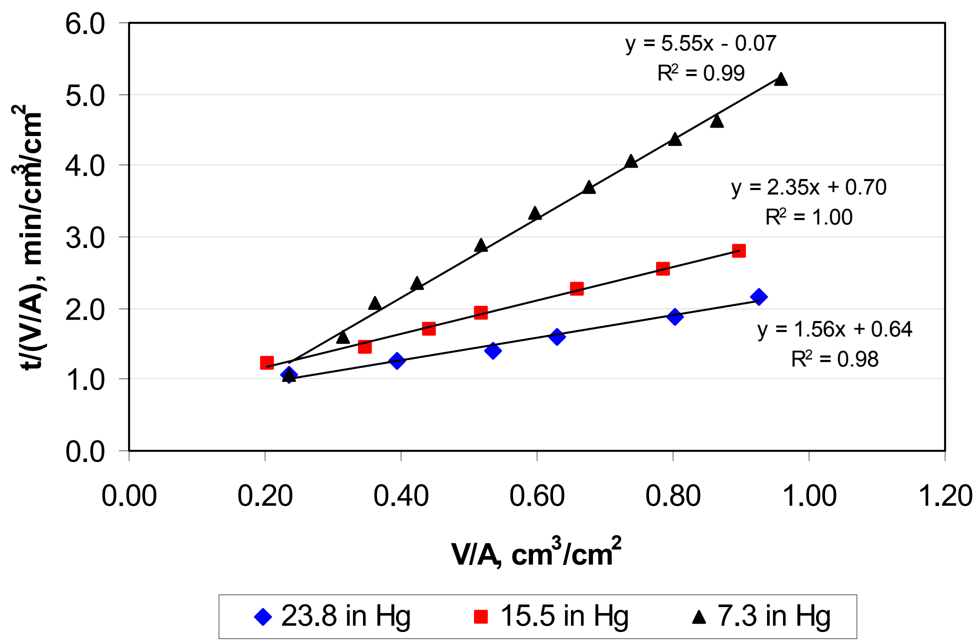
Dividing by V/A and P, and rearranging,

$$\frac{t}{V/A}=\frac{\mu\alpha wV}{2PA}+\frac{\mu r}{P} \qquad (4)$$

Thus, a plot of t/V/A versus V/A will give a straight line with a slope of $\mu\alpha w/2P$ and an intercept of $\mu r/P$. The specific resistance of the filter cake α and the drainage resistance r can be found from these values and compared for different experimental conditions.

The first parameter to be examined was the effect of the pressure differential across the filter. Figure 5a shows the rate of filtrate production as a function of time for the Friendship Hill test pulp at three different vacuum levels. It is clear that increased vacuum levels, corresponding to increasing pressure differentials across the filter paper, give increased filtration rates. The linearized plot in Figure 5b shows that the effect of the increased pressure drop was a decrease in slope for each of the tests in Figure 5a. When the specific resistance α was calculated for these tests, α was found to be relatively constant at 1.3 to 1.5 $\times 10^{12}$ m/kg, indicating that the increases in slope were offset by decreases in the associated pressure drop. Dempsey and Jeon (2001) measured the specific resistance to filtration of several AMD sludges from passive treatment operations and obtained values of 1 to 10 $\times 10^{10}$ m/kg. Much lower pressure differentials were used in their work, which may explain the lower values of α, since α is proportional to the applied pressure.



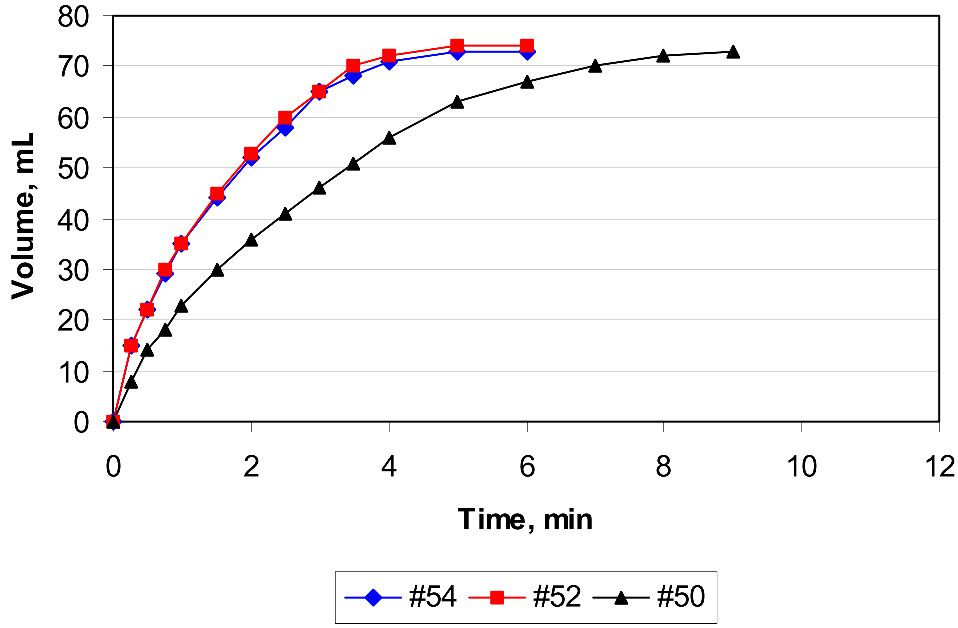


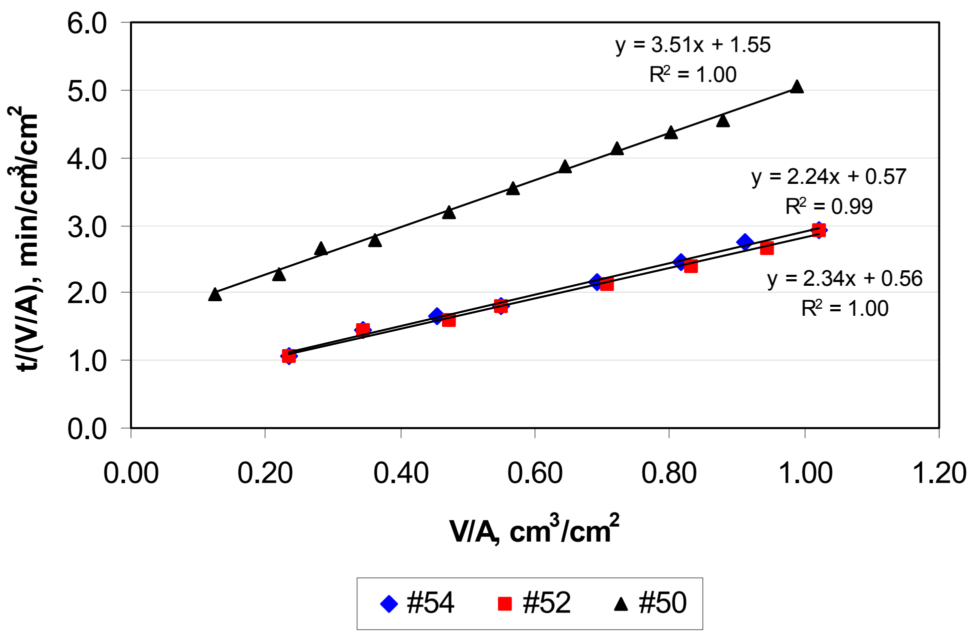
Figures 5a and b. Effect of vacuum level on filtration rate.

The compressibility coefficient s, a measure of the compressibility of the filter cake, can be determined from the slope of a plot on a logarithmic scale of α versus applied pressure. For the data plotted in Figure 5, s works out to about zero, since there was essentially no change in α as the pressure increased. This usually signifies an incompressible cake composed of particles such as sand or diatomaceous earth. Metal hydroxide cakes are more typically thought to be compressible, with the value of s approaching 1.0 (Dahlstrom, 1985). Dempsey and Jeon (2001) found that AMD sludges from passive treatment systems had coefficients of 0.3 to 0.4, while an active lime treated sludge had a coefficient of 0.86. It was expected that the pulsed limestone bed filter cake would exhibit some compressibility because of its composition, but this was not supported by the data. This may be an indication that the sludge produced by the pulsed limestone bed process is in fact less compressible than active lime-based sludges. From the plant operation perspective, this is a positive result, since it indicates that increasing pressure will cause an increase in filtration rate.

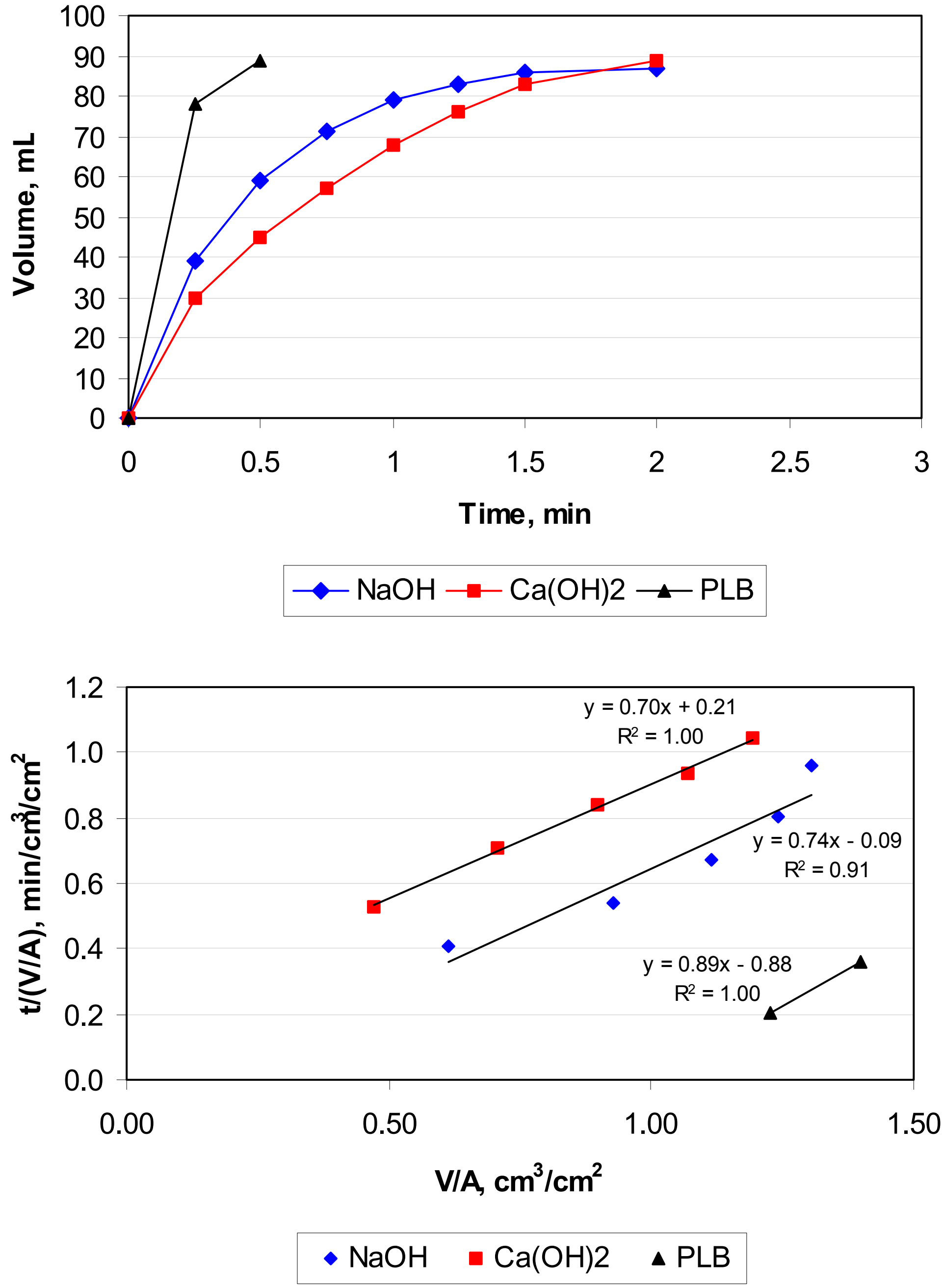
The effect of the filter media was tested next. Figures 6a and b show the effect of the filtration media on the rate of filtrate collection. Both the #54 and 52 papers offered minimal resistance to filtration. However, the #50 paper slowed the rate of filtrate collection, due to the additional resistance of the media. The linearized plot of the data is shown in Figure 6b. As shown earlier, the media resistance is proportional to the y- intercept of the plot. Therefore, the resistance of the #50 paper is almost three times that of the other papers. Although filtrate clarity was better with this grade of paper, the increased resistance to filtration would be a drawback. There would be an optimum tradeoff between filtrate clarity and collection rate, probably in this case, as demonstrated by the #52 paper. Most of the solids in the filtrate appeared in the first few seconds of the test, until the cake thickness had been built up enough to retain solids.

Tests were also run to compare the filterability of pulps generated with different neutralization reagents. Test results are shown in Figures 7a and b. Batches of 3.8 L (1 gallon) of Friendship Hill influent were neutralized with $Ca(OH)_2$ and NaOH. A similar volume of product water from the plant was also tested for comparison. After settling, the pulp volumes were 310 ml for the $Ca(OH)_2$, 365 ml for the NaOH and 95 ml for the plant product water. The solids content of these pulps were much lower than the pilot plant pulps tested earlier. The pilot plant pulp contained about 8% solids by weight, but solids contents were only 1.2, 0.9, and 2.8% for the $Ca(OH)_2$, NaOH, and product water samples, respectively. Filtration performance was tested for 100 mL aliquots of these settled solids. Due to the greater dilution of these samples, filtration was rapid, but this does not necessarily demonstrate better performance. The resistance to filtration coefficient α was calculated for each of these tests from the linearized plot in Figure 7b.





Figures 6a and b. Effect of filter media on filtration rate.



Figures 7a and b. Effect of neutralization reagent on filtration rate.

The coefficients were 3.1, 7.3 and 10.4 x10^{12} m/kg for the limestone, $Ca(OH)_2$ and NaOH cakes, respectively. It is clear that the NaOH and $Ca(OH)_2$ pulps offer greater resistance to filtration than the pulsed bed limestone pulp, in addition to the greater volume of pulp generated by neutralization. Also, the solids contents of the filter cakes were lower for the $Ca(OH)_2$ and

NaOH samples, at 9.2 and 6.1%, compared to 28% for the limestone sample. Therefore, the pulsed limestone bed process has several advantages: decreased sludge production rate, greater filtration rate, and increased solids content in the filter cake.

Pressure filtration tests. Because of the expense of continuous vacuum filtration units, many AMD treatment operations use pressurized plate and frame filters for dewatering of settled solids. A five-gallon sample of Friendship Hill pulp was sent to a manufacturer of plate and frame filters for testing in their laboratory. The solids content of the sample was 5%, and the press generated a filter cake with a solids content of 31%. This represents an eight-fold reduction in water content, which would lead to large savings in sludge disposal costs. The capital cost of the plate and frame filter sized for the Friendship Hill operation was about $35,000. If the solids were to be disposed off-site as before, the costs of a storage container such as a roll-off dumpster, and pickup and disposal would also need to be considered. However, the plate and frame filter offers the possibility of much lower maintenance costs than continuous vacuum equipment, and would be an attractive option for the Friendship Hill site, were processing to proceed for an extended period of time.

Settling/percolation tests. Although not currently considered at the Friendship Hill site, a settling/percolation pond is a possible, more economical option, if sufficient land were to become available. Small-scale percolation tests were performed at Friendship Hill using settled solids pulp added to duplicate 6-inch PVC columns with a sand bed. The results are shown in Figure 8. The poor reproducibility of the duplicates may have been related to disturbance of the sand bed when the sludge was added to the column. Even with the slower percolation rate data from column A, a 67% reduction in sludge volume was observed after 20 days of settling. This would be a low cost, low maintenance approach for dewatering on site, if a suitable location could be found.

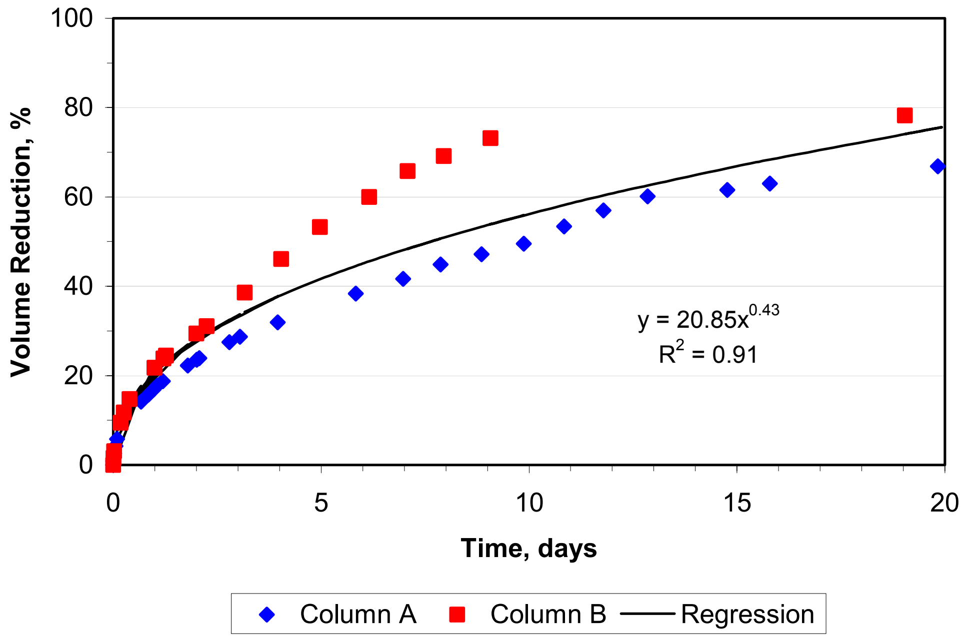


Figure 8. Percolation rate tests.

Conclusions

Although the pulsed limestone bed treatment plant at Friendship Hill performed well over the 14-month run, sludge disposal costs were elevated due to off-site disposal. Therefore, several alternate sludge treatment options were tested in the laboratory. Test results showed that the limestone sludge volume was much less than that found for equal volumes of influent treated with lime, ammonium hydroxide or sodium hydroxide. The limestone sludge also settled more rapidly, though with a greater haziness in the supernatant. The limestone sludge responded favorably to polymeric flocculants, but the increase in reagent cost, coupled with the decrease in settled pulp density was deemed too expensive. Vacuum filtration tests showed that the limestone-based solids were readily filtered, and showed increasing filtration rates as pressure increased. The effect of the filter media was found to be significant for the #50 paper, suggesting that a less retentive media would be better suited to the process. The filterability of limestone-based pulps was compared to pulps made with $Ca(OH)_2$ and NaOH. These alternate basic reagents had several drawbacks compared to the limestone pulp: greater sludge volume, greater specific resistance to filtration, and decreased solids content in the filter cake. For the settled limestone pulp, vacuum filtration increased the solids content of the cake from 8% to about 25%, thus rejecting 67% of the total sludge volume. Pressure filtration removed even more of the water, forming a cake with as much as 31% solids. This option would be attractive for this site due to lower maintenance and operating costs. Finally, settling/percolation rates of the limestone-based sludge were measured. Here, the solids content could be thickened to 25%, with a concurrent three times reduction in volume, with a very low operating cost. However, this option would require finding a suitable on-site location for the settling/percolation pond. Due to site constraints, the pressure filtration option, using a plate and frame filter press, is probably the best option, should treatment continue at this site.

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