MINE WATER TREATMENT USING MAGNETITE AND IRON FERRITES

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ABSTRACT

Iron ferrites and magnetite have been used for the removal of radioactive and heavy metals from liquid effluents. The mineral magnetite, or synthetically prepared iron ferrite ($FeO \bullet Fe_2O_3$), is typically utilized. Iron ferrite can be employed for mine water treatment in a batch mode by adding preformed ferrite to the solution or by preparing the ferrite in situ, thus using the existing iron in the effluent. Natural magnetite can also be used in a batch mode, or better, in a column mode. In the presence of an external magnetic field, enhanced capacity was observed when using a column of supported magnetite for removal of actinides and heavy metals from wastewater. The enhanced capacity is thought to be primarily due to magnetic separation of colloidal and submicron particles along with some complex and ion exchange sorption mechanisms. The loaded magnetite can easily be regenerated by removing the magnetic field and using a regenerating solution. This paper briefly reviews previous work on the use of iron oxides for wastewater treatment and presents new results on using magnetite and iron ferrites for the effective treatment of several different mine waters and for valuable metal recovery.

1. INTRODUCTION

Treatment of liquid wastes containing radioactive and hazardous metals has employed a variety of iron oxides utilizing adsorption, precipitation, and other chemical and physical techniques (King and Navratil, 1986; Driscoll, 1986; Freeman, 1988; Macasek and Navratil, 1992). For example, a radioactive wastewater precipitation process utilizes ferric hydroxide to remove radioactive contaminants such as americium, plutonium, and uranium (Boyd et al., 1983). Some adsorption processes for wastewater treatment have utilized ferrites and a variety of iron-containing minerals such as akaganeite, feroxyhyte, ferrihydrite, goethite, hematite, lepidocrocite, maghemite, and magnetite (Schwertmann and Cornell, 1991). Ferrite is a generic term for a class of magnetic iron oxide compounds that possess the property of spontaneous magnetization and are crystalline materials soluble only in strong acid (Reynolds, 1980). Iron atoms in iron ferrite ($FeO \bullet Fe_2O_3$) can be replaced by many other metal ions without seriously altering its spinel structure (Boyd et al., 1986).

Ferrites and natural magnetite were used in batch modes for actinide and heavy metal removal from wastewater (Boyd et al., 1986; Kochen and Navratil, 1987; Navratil, 1988; Navratil, 1989; Navratil, 1990). Supported magnetite was also used in a column mode, and in the presence of an external magnetic field, enhanced capacity was found for removal of plutonium and americium from wastewater (Kochen and Navratil, 1997; Navratil et al., 1995). These observations were explained by a nano-level high-gradient magnetic separation (HGMS) effect, as americium, plutonium, and other hydrolytic metals are known to form colloidal particles at elevated pH levels. Recent modeling work supports this assumption and shows that the smaller the magnetite particle, the larger the induced magnetic field around the particle from the external field (Ebner et al., 1999). Other recent studies have demonstrated the magnetic enhanced removal of arsenic, chromium, cobalt, iron, and uranium from simulated groundwater and wastewater (Cotten et al., 1999; Cotten et al., 1999a; Navratil, 2008).

The magnetic field-enhanced separation process differs from magnetic separation processes used in the processing of minerals and, more recently, for water treatment and environmental applications. Conventional magnetic separation processes use, for example, fine stainless steel wool to form a magnetic matrix within a flow field of a solution containing mineral particles to be separated. For this reason, in order for such processes to remove metal ions and nano-particles from solution, precipitating or flocculating agents must first be added to effect formation of large particles. In contrast, our process is unique because a highly porous adsorbent material not only acts as a magnetic matrix, it also contributes an adsorptive component to the system. This adsorptive component allows for the removal of ions and complex metal species from solution, while the relatively low-field magnetic matrix allows for the removal of nanoparticles through a HGMS effect.

This paper briefly reviews previous work on the use of iron oxides for wastewater treatment and the development and potential of the magnetic filtration/sorption process. New results on using magnetite and iron ferrites for the effective treatment of several different mine waters are also presented along with a discussion of the potential use of the process for valuable metal recovery from mine waters.

2. FERRITE PROPERTIES AND PREVIOUS STUDIES

In most conventional wastewater treatment processes metals are removed in the form of metal hydroxides because they have low solubilities. As noted in the introduction, ferric hydroxide is often added to scavenge a wide variety of heavy metal contaminants (Dzombak and Morel, 1990). However, the metal hydroxide solids can form gelatinous precipitates, which are difficult to filter. Consequently, filter aids must sometimes be added to facilitate the filtration process.

The use of iron ferrite and magnetite in wastewater treatment has a number of advantages over conventional flocculent precipitation techniques for metal ion removal (Boyd et al., 1986). Ferrite solids are crystalline materials, unlike hydroscopic metal hydroxide sludges, and can be more readily filtered; their ferromagnetic character permits use of magnetic separation of the solids from solution. A wide variety of metal ion impurities (e.g., arsenic, cadmium, cobalt, copper, iron, lead, manganese, mercury, molybdenum, nickel, silver, antimony, titanium, tungsten, vanadium, zinc, and zirconium) can be effectively removed in one treatment step. Their removal is not seriously affected by high salt concentrations (Boyd et al., 1986; Navratil, 1988). The ferrite method does not require expensive chemicals, and because iron is usually a constituent of waste solutions, its oxidation states can be adjusted by chemical or electrolytic means to form ferrite. Both in situ and preformed ferrite methods have been used in wastewater treatment applications (Boyd et al., 1986).

With the in situ method, ferrous iron is added to the waste solution (usually heated to 60–90°C), followed by the addition of base and subsequent oxidation (usually by aeration). Ferrite is formed within the waste solution itself. In this technique, metal ions undergo ferrite formation and can be incorporated into the lattice of ferrite crystals. In the preformed case, ferrite is prepared separately using the in situ procedures described and added in solid or slurry form to the waste solution. Magnetite, a naturally occurring ore prevalent in most parts of the world, can also be used in place of preformed ferrite. However, natural magnetite needs activation to have the same capacity as preformed ferrite (Kochen and Navratil, 1997).

Ferrite functions as an adsorption medium to remove contaminating species from solution. In addition to its chemical incorporation capabilities, in situ ferrite also functions in this manner. The adsorption mechanism of ferrite occurs through metal hydroxide species. In alkaline solution, most metal ions form insoluble species, which may be complexed with additional hydroxide ions, depending on the chemistry of the metal and the pH of the solution. Hydroxide ions are strongly adsorbed onto the surface of ferrite particles so that metal ions can bond to the ferrite surface through the hydroxide ions to which they are bound. Alkali, alkaline, and other metals that do not form insoluble hydroxides are not removed by ferrite, and metals that form soluble hydroxide complexes at high pH values, such as aluminum and zinc, can only be removed at moderate pHs (Boyd et al., 1986).

For column operations, crystalline ferrites and powdered magnetite must be supported with another material to achieve low backpressure and good water flow through the column. The first experiments of supported magnetite involved using a column containing a nonporous polyamine-epichlorohydrin resin bead coated with activated magnetite surrounded by an electromagnet of 0.3 tesla (Kochen and Navratil, 1997). In this work, the capacity of the magnetite-coated resin for both plutonium and americium removal from water increased by a factor of five compared with using unsupported magnetite particles in the absence of a magnetic field. These observations were explained by a nanolevel HGMS effect, as americium, plutonium, and other hydrolytic metals are known to form colloidal particles under alkaline conditions (Ebner et al., 1999). The pores of the magnetite-coated resin are large enough to permit the free displacement of the colloidal particles. When the field of the electromagnet is turned on, the magnetite particles are magnetically induced, creating a field that contributes to the net field sensed by the colloidal particles. When the magnetic force is sufficiently greater than the force associated with Brownian (thermal) motion, the magnetic force created by the field can be attractive and sufficiently large to allow the magnetite to sorb the colloidal particles, provided they have sufficient magnetic susceptibility. Recent modeling work has shown that the smaller the magnetite particle, the larger the induced magnetic field around the particle from the external field (Ebner et al., 1999). When the electromagnet is turned off, the nanoparticles are released and dispersed in solution by thermal motion (metal hydroxide complexes would still be adsorbed, however, and would need to be removed by an eluent solution).

Recent studies have demonstrated the magnetic-enhanced removal of arsenic, chromium, cobalt, iron, and uranium from simulated groundwater and wastewater (Cotten et al., 1999; Cotten et al., 1999a; Navratil, 2008). These results also indicate that natural components of groundwater, such as calcium and magnesium, do not interfere and are not significantly removed with the process. Several suitable support media, including glass beads and sand, were tested for mixing with natural magnetite to achieve satisfactory flow characteristics while retaining the magnetic field-enhanced sorption properties of the magnetite (Navratil, 2008).

3. EXPERIMENTAL

Iron ferrites were generally prepared in situ by adding solutions of ferric and ferrous ions (2:1 mole ratio, respectively) to an acidic mine water sample followed by fast addition of a sodium hydroxide solution to a pH above 8. Preformed ferrite was prepared in a manner similar to the in situ technique and added directly to the pH-adjusted mine water as a wet solid. Both methods were performed at room temperature, ~22-24C. Magnetite was supplied by Alfa Aesar as a 97% powder (minus 325 mesh) and activated prior to use by contacting with 1 M sodium hydroxide solution followed by water washing. The reagent solutions used were prepared from purified U_3O_8 acidified with reagent-grade hydrochloric or sulfuric acid, Alpha Aesar 98% anhydrous iron (III) chloride, Alpha Aesar 99% iron (II) chloride hydrate, Baker Scientific reagent-grade ferrous sulfate heptahydrate, Baker Scientific reagent-grade ferric sulfate, and Alpha Aesar sodium hydroxide (98%) pellets. Oglebay Norton silica sand was wet-screened to obtain a minus 10- by 14-mesh fraction for column support material. Deionized water was used, and all other materials were reagent-grade.

The column experiments utilized 10-mL glass columns (0.5-inch inside diameter) surrounded by four 1.5-inch-diameter NdFeB ring magnets supplied by Amazing Magnets of Irvine, California, USA. Typically, a mixed magnetite/sand ratio of 4:1 by weight was utilized.

The analysis of the solutions (filtered through Whatman 42 filter paper and acidified) and solids were performed by Hazen Research, Inc. Analytical Laboratories. The uranium was determined by fluorometric techniques, selenium was analyzed using an x-ray fluorescence technique, and the other metals were determined by spectroscopic analysis.

4. RESULTS AND DISCUSSION

Selenium Experiments

This work was undertaken because there are selenium removal problems with some mine waters, and selenium removal with ferrites and magnetite has not been reported in the scientific literature. In the study, in situ ferrite, preformed ferrite, and magnetite were added at 2 wt% to synthetic 10 mg/L Se (IV) solutions adjusted to pH 9-10 and mixed for 30 minutes. Preformed ferrite and magnetite experiments were made using sorbent that was washed with deionized water as well as with a dilute cuprous chloride solution. Results showed that each experiment removed over 99% of the selenium, and only the untreated preformed ferrite experiment had more than 0.05 mg/L Se remaining in the filtrate. The magnetite showed the best selenium removal, and both the in situ ferrite and preformed ferrite washed with CuCl had similar removal percentages (Table 1).

Table 1. Results of selenium removal from synthetic 10 mg/L Se feed

Conditions	Se Concentration after Treatment, mg/L	Se Removed, %	Fe Concentration after Treatment, g/L
In situ ferrite	0.040	99.6	<0.001
Preformed ferrite	0.089	99.1	<0.001
Preformed ferrite - CuCl treated	0.035	99.6	<0.001
Deionized water washed magnetite	0.023	99.8	<0.001
CuCl washed magnetite	0.012	99.9	<0.001

Uranium Studies

Magnetite, in situ iron ferrite, and preformed iron ferrite in batch modes and magnetite and preformed iron ferrite in column modes (with external magnetic enhancement) were investigated for the recovery of uranium from synthetic solutions. In the preformed ferrite experiments, 1- and 5-mL batches of ferrite were added to 100-mL batches of 30 ppm U_3O_8 solution with pH adjustment to 10. After 30 minutes of mixing at room temperature, the ferrite was observed to settle very fast. The decanted solution was filtered, acidified to pH 1, and submitted for analysis. The results showed that 1- and 5-mL batches of preformed ferrite removed 98 and 97% of the uranium, respectively.

For the in situ experiments, ferric and ferrous ions were added directly to uranium feed solutions. Both chloride and sulfate solutions were studied to measure the effects of anion, final pH, mixing time, and ferrite concentration. Following mixing, the ferrite was observed to settle very quickly as with preformed ferrite. The solutions were decanted, filtered through Whatman 42 filter paper, acidified to pH 1, and submitted for iron and uranium analysis (Table 2). Magnetite by itself was not as effective as in situ ferrite, which removed >98% U, and the presence of carbonate appears to further reduce the effectiveness of magnetite removal of uranium (Table 3).

Table 2. Results of batch in situ ferrite experiments using synthetic 10 mg/L U_3O_8 feed

Conditions	Mixing, min	Sulfate System				Chloride System			
		pH	U_3O_8, mg/L	U_3O_8 Removed, %	Fe, mg/L	pH	U_3O_8, mg/L	U_3O_8 Removed, %	Fe, mg/L
20 mL U_3O_8 Feed 1 mmol Fe	30	9	0.3	97	0.26	9	<0.2	>98	0.18
20 mL U_3O_8 Feed 1 mmol Fe	30	12	0.2	98	0.2	12	<0.2	>98	0.16
20 mL U_3O_8 Feed 0.1 mmol Fe	30	12	<0.2	>98	0.16	10	<0.2	>98	0.08
20 mL U_3O_8 Feed 0.1 mmol Fe	30	9	<0.2	>98	0.16	9	<0.2	>98	0.08
20 mL U_3O_8 Feed 0.1 mmol Fe	10	12	<0.2	>98	0.28	11	0.7	93	0.1
20 mL U_3O_8 Feed 0.1 mmol Fe	10	8	<0.2	>98	0.3	9	<0.2	>98	0.12
20 mL U_3O_8 Feed 0.05 mmol Fe	10	9	<0.2	>98	0.12	11	<0.2	>98	0.14

Table 3. Results of magnetite experiments using synthetic 10 mg/L U_3O_8 feed

Conditions	Magnetite, g	U_3O_8, mg/L	U_3O_8 Removed, %	Fe, mg/L
50 mL U_3O_8 Feed	0.0003	2.7	73	0.16
50 mL U_3O_8 Feed	0.0017	3.1	69	0.48
50 mL U_3O_8 Feed	0.007	2.6	74	0.2
50 mL U_3O_8 Feed	0.0141	2.5	75	0.24
50 mL U_3O_8 Feed	0.052	2.7	73	0.18
50 mL U_3O_8 Feed	0.999	2.7	73	0.16
50 mL U_3O_8 Feed 0.5 g Na_2CO_3	0.9989	3.4	66	0.2
50 mL U_3O_8 Feed 1.0 g Na_2CO_3	0.9999	3.1	69	0.26
50 mL U_3O_8 Feed 1 mmol Ferrite	-	<0.2	>98	0.14

Uranium breakthrough was studied using columns containing a mixture of magnetite and sand fully surrounded by four donut-shaped magnets. Using a 10 mg/L U_3O_8 feed, 58% uranium breakthrough occurred after passing 200-mL of feed. Two more similar column experiments were performed with the magnetite/sand mixture, at two different feed flow rates. The slower flow rate, 4 mL/min, provided average uranium breakthroughs of 30% and 50% after passing 500 and 1,000-mL of feed, respectively. The faster flow rate, 8 mL/min, had average breakthroughs of 75% and 85%, respectively. As a comparison, sand-supported preformed ferrite was studied in similar column experiments. Surprisingly, higher uranium breakthroughs (approximately 90%) were observed than in the two column experiments. The loaded uranium columns were eluted with sodium bicarbonate solution to evaluate the removal of uranium. Using 100-mL of bicarbonate solution, 70–80% of the uranium was eluted from the columns, and a second 100-mL batch of bicarbonate solution eluted an additional 20–25% of the uranium, demonstrating that the magnetite or preformed ferrite could be regenerated.

Mine Water Treatment with Potential Metal Recovery

Experiments were performed with five different mine waters using magnetite and in situ ferrites. Magnetite is an ideal sorbent if the metals removed from the water have enough value to be recovered and sold. Additionally, a magnetite system can be regenerated and reused. In situ ferrite precipitation is useful to tie up the metals in the matrix of the precipitate as magnetic ferrites for disposal. The precipitate formed is crystalline and easily filterable, and is more resistant to leaching than normal caustic precipitates.

The effect of pH as well as the effect of the amount of magnetite used in the treatment of both Mine Water A and Mine Water B was studied. Various amounts (0.05–0.5 g) of magnetite were used to treat 100-mL of each sample. Overall, it was found that the change in sorbent weight had little effect on sorption of the metals. Experiments were performed at pH values of 6, 8, and 10 to remove the major metal contaminants, copper, total iron, manganese, and zinc.

Complete removal of copper, iron and zinc occurred at pH 6–8, and manganese was effectively removed at pH 10 (Figures 1 and 2). Magnetite sorption was also performed on both Mine Water A and Mine Water B samples at the natural mine water pH (3.2 and 3.7, respectively) and at a pH of 1. Results showed that there was insignificant metal sorption occurring on the magnetite and actual leaching of iron from the magnetite at pH 1, confirming the ability to strip the loaded sorbent with pH 1–3 water to recover the metals and reuse the sorbent.

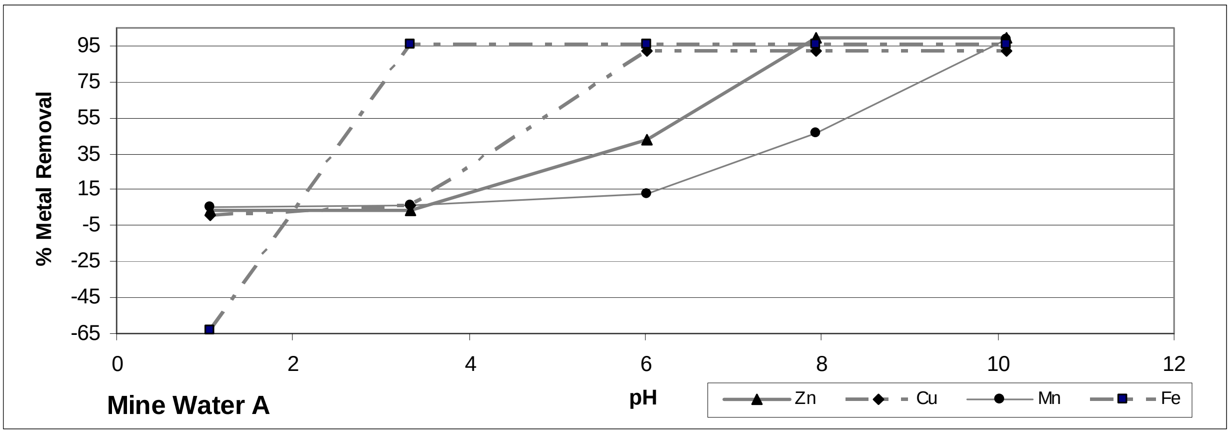

Figure 1. Effect of pH on metal removal from Mine Water A using magnetite

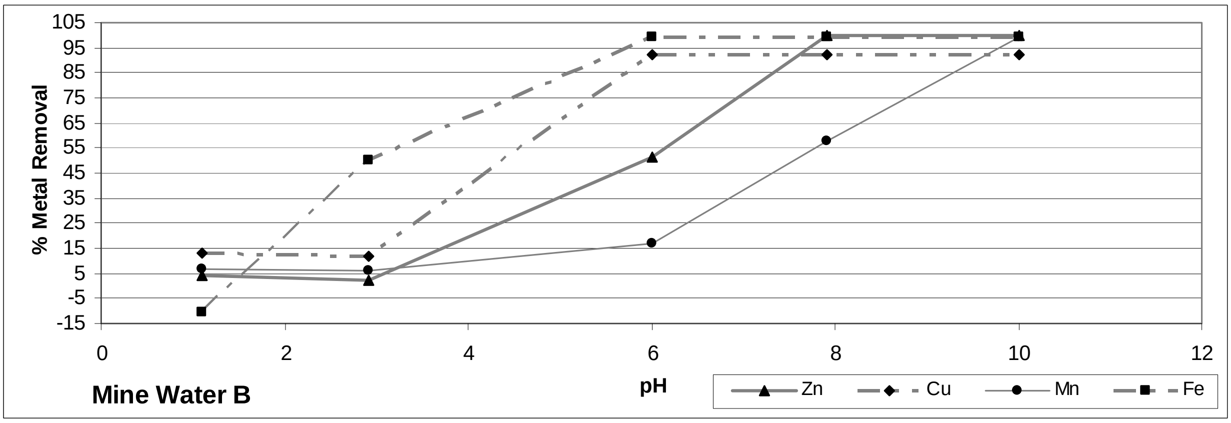

Figure 2. Effect of pH on metal removal from Mine Water B using magnetite

In situ ferrite was also tested on both mine waters; all the primary metals of concern were effectively precipitated at a pH greater than 9. Because magnetite was effective in selectively removing copper and zinc at pH 6–8, one proposed remediation scheme for the mine water would be to use a magnetite/sand column operation with the mine water adjusted to pH 7, followed by batch treatment of the column effluent using in situ ferrite for precipitation of other contaminants at pH 9.

Four samples of Mine Water C were tested using magnetite and in situ ferrite. The natural pH of the samples were 3.8–4.1, and the main contaminants found were 3 mg/L Fe, 14 mg/L Mn, and 60 mg/L Zn. After treatment of all four mine water samples using both magnetite and in situ ferrite at pH 10, the main contaminants were reduced to less than 1 mg/L Fe, less than 0.5 mg/L Mn, and less than 0.2 mg/L Zn. The water samples were also tested for metal adsorption using 0.5 g of magnetite at pH values of approximately 4, 6, 8, and 10. The analysis of the filtrate (Figure 3) showed that the optimal removal of zinc was achieved at a pH of 6 and manganese at pH 10. Further experiments were then run to determine the effect on metal adsorption when the amount of magnetite was varied at a constant pH of 10. Various amounts of magnetite (0.05 g, 0.1 g and 0.5 g) were tested. The analysis (Figure 4) showed that the removal percentages of iron, manganese and zinc were fairly well maintained for the various amounts of magnetite with minimal removal of calcium and magnesium; this correlated to previous experiments in which approximately 2% by weight of magnetite showed optimal results.

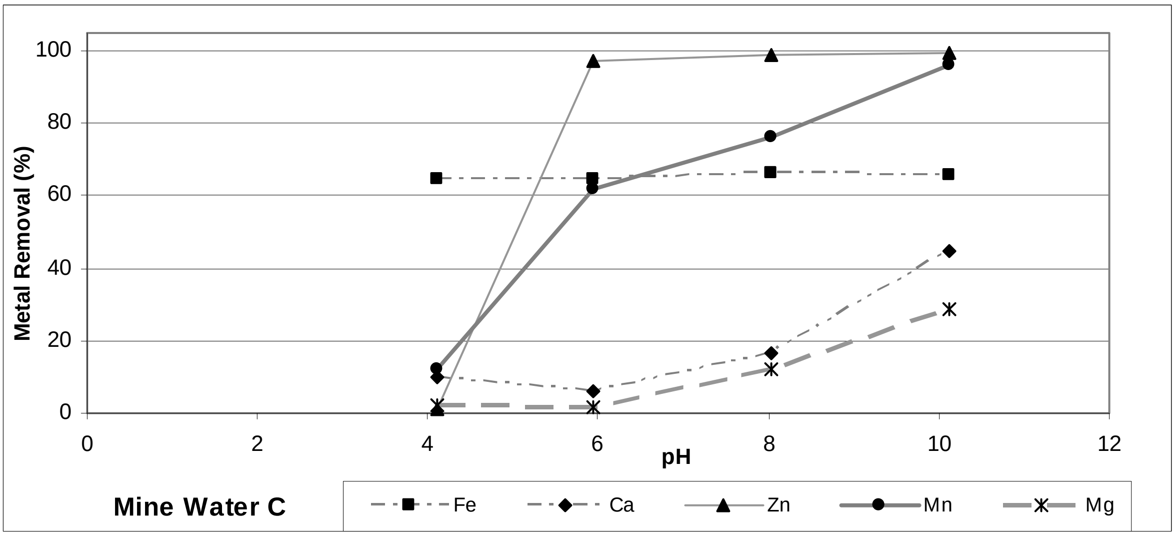

Figure 3. Effect of pH on metal removal from Mine Water C using magnetite

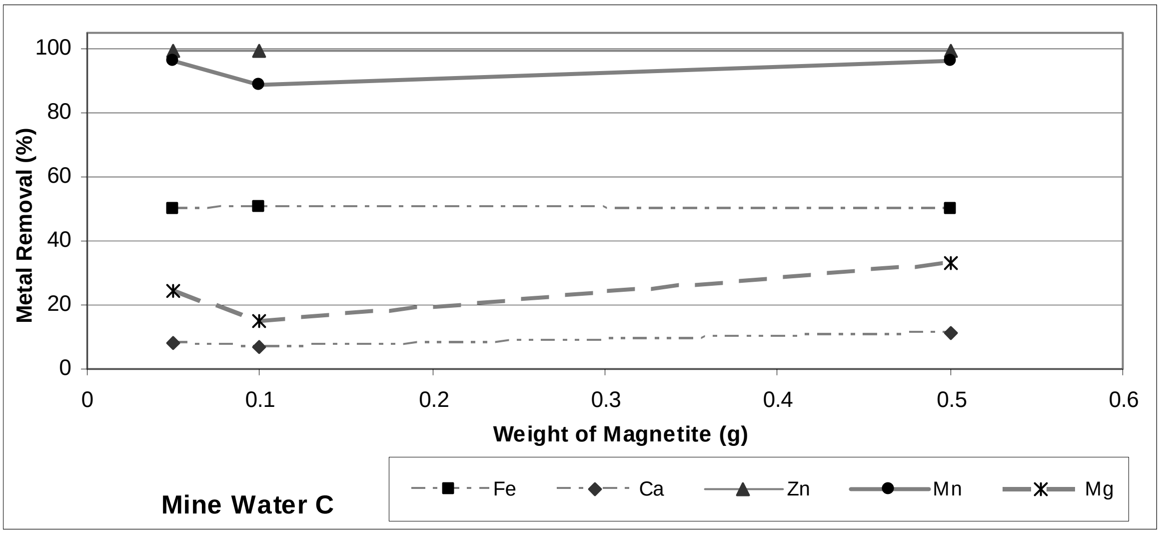

Figure 4. Effect of magnetite mass on metal removal from Mine Water C

The natural pH of the Mine Water D was 3.4, and samples were adjusted to pH values of 6, 8, and 10 and equilibrated with 0.5-g batches of magnetite, filtered, and analyzed. The analysis of the major contaminates in the original and adjusted samples following contact with the sorbent are shown in Table 4. The results show that magnetite was effective in mainly removing copper and iron at pH 6, zinc at pH 8, and manganese at a higher pH of 10. Thus one proposed remediation scheme for this mine water would be to utilize a magnetite/sand column operation with the mine water adjusted to pH 6–8 to selectively recover zinc and copper. Another recovery scheme would be to extract all the metal contaminants by operating a magnetite column at pH 9–10.

Table 4. Major contaminants in the untreated Mine Water D and magnetite treated water at various pH

Contaminants, mg/L	Untreated Feed pH 3.4	pH 3.4	pH 6	pH 8	pH 10
Copper	2.7	3.9	<0.5	<0.5	<0.5
Iron	124	63	<1	<1	3
Manganese	35	35	33	23	<0.5
Zinc	9.8	10	5.6	<0.2	<0.2

The natural pH of the Mine Water E was 6.7, and samples of the water were equilibrated with 0.5 g of magnetite at pH values of 6.7, 8.0, and 10.0. The analysis of the major contaminates in the original and pH-adjusted samples following contact with the sorbent are shown in Table 5. The results show that magnetite was effective in removing arsenic, cadmium, lead, and major amounts of zinc at pH 6.7 and manganese and the remainder of the zinc at a higher pH of 10.

Table 5. Major contaminants in the untreated Mine Water E and magnetite treated water at various pH

Contaminants, mg/L	Untreated Feed pH 6.7	pH 6.7	pH 8.0	pH 10.0
Arsenic	0.03	0.007	0.012	0.015
Cadmium	<1	<0.01	<0.01	0.2
Lead	<1	0.2	<0.2	<0.2
Manganese	2.9	3.1	4	<0.1
Zinc	4.9	0.18	0.18	<0.1

Mine Water F is a low-volume stream containing mainly zinc as the contaminant of concern. Most of the experiments for this water were performed on two separate samples to show reproducibility of the results. The natural pH of the water samples was 7.3, and the main contaminant found was 0.2 mg/L Zn. After treatment of both mine water samples (25-mL batches) at various pH values using 0.5 g magnetite, more than 80% of the zinc was removed above pH 8 in most cases (Table 6). These results are similar to those of other mine waters tested. The zinc can be recovered from the loaded sorbent by treatment with small volumes of pH 2–3 water. Thus one proposed remediation scheme for this water, provided the zinc could be economically recovered and sold, would be to use a magnetite/sand column for the water with minimal pH adjustment for removal of zinc followed by recovery of the zinc from the magnetite using pH 3 water.

Table 6. Zinc (mg/L) in magnetite-treated Mine Water F at various pH.

Sample Number	pH 6.97–7.15	pH 7.95–8.01	pH 8.85–8.75	pH 10.02–9.93
1	0.11	<0.02	0.05	0.17
2	0.05	0.04	0.03	0.03

5. CONCLUSIONS

Results of selenium removal using ferrites and magnetite showed that each experiment removed over 99% of the selenium, and the magnetite experiments showed the best removal of selenium. However, in the uranium studies, uranium recoveries greater than 98% were observed with iron ferrite compared to magnetite, and batch experiments out performed column operations; effective elution of the uranium with bicarbonate solution for reuse of the sorbent appears possible. The effect of pH and amount of magnetite on contaminant removal from various mine water samples was studied. Varying amounts of magnetite were used to treat some of the samples, and overall, it was found that the amount of sorbent had minimal effect on metal removal. Experiments were performed on some of the samples at various pH values to remove the major metal contaminants, copper, iron, manganese, and zinc. Generally, complete removal of copper, iron and zinc occurred at pH 6–8, and manganese was effectively removed at pH 10. Experiments at pH of 1–3 showed that there was insignificant metal sorption occurring on the magnetite, confirming the ability to strip the loaded sorbent with pH 1–3 water to recover the metals and reuse the sorbent. In situ ferrite was also tested on some of the mine waters, and all the primary metals of concern were effectively precipitated at a pH greater than 9. Since magnetite was effective in selectively removing copper and zinc at pH 6–8, one proposed remediation scheme for a contaminated mine water would be to utilize a magnetite/sand column operation with the mine water adjusted to pH ~7 to capture the commodity. This would be followed by a batch treatment of the column effluent for precipitation of the other contaminants at a pH greater than 9 using in situ ferrite.

6. ACKNOWLEDGMENTS

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