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Pyrolusite Process® to Remove Acid Mine Drainage Contaminants from Kimble Creek in Ohio: A Pilot Study

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Abstract

The Kimble Creek abandoned coal mine site, located on Wayne National Forest in southeastern Ohio, is among several abandoned coal mine sites that have been responsible for the acid mine drainage (AMD) polluting ground and surface water. Materials released by AMD include iron, aluminum, manganese, other hazardous substances, and acidity that are harmful to aquatic life. We tested at the Kimble Creek site the Pyrolusite Process®, a microbiological water treatment system that employs metal-oxidizing bacteria adsorbed on limestone rocks. The pilot treatment plant constructed was inoculated with a mixture (five strains) of metal-oxidizing bacteria. Water was examined at regular intervals for pH, concentrations of metals (Fe, Al, Mn, Mg, Zn, Ca), dissolved oxygen, temperature, conductivity, hardiness, sulfate and chloride contents, acidity, alkalinity, and dissolved solids. Results showed that the Prylosite Process consistently increased the pH of effluent water to above-neutral levels. The iron and aluminum contents, which were as high as 72 mg/L and 50 mg/L, respectively, were below detectable levels in the effluent water. Scanning electron microscope (SEM) and DNA analyses of rock samples showed growth and density increases of inoculated bacteria. The sludge contained iron (predominantly Fe^{+3}), aluminum, and small amounts of zinc, magnesium, and manganese. Based on the performance of the pilot system, a full-fledged system was constructed at the Kimble Creek site which is functioning as expected based on preliminary tests.

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INTRODUCTION

Abandoned coal mines produce acid mine drainage (AMD) that pollutes ground water supplies. The materials in AMD vary by site, but typically include heavy metals (such as iron, aluminum, and manganese), other hazardous substances, and acidic water that harms aquatic life. Improved economical methods are needed to remove these substances from water that otherwise could have detrimental effects on forest health and related wildlife.

The Kimble Creek abandoned coal mine is located in Lawrence County, within the Wayne National Forest in southeastern Ohio. The location, a moderately steep, forested, north-south trending hill, has three mining-related benches crossing the hillside. There are three known portals and another suspected portal along the lowermost of the three benches. All three portals drain into Kimble Creek, which ultimately flows into the Ohio River. Coal waste, known as gob piles, is located near or downhill from these portals and also contributes to the AMD. The AMD emerging at this site crosses the bench and flows downhill through gob, coalescing into a single flow path at the toe of the gob. Flow measurements have shown that water volume increases from the portal to the toe of the gob, indicating that subsurface flow through the gob is augmenting surface flow. Maximum flow measured at the toe was at 15 gallons per minute.

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The AMD at the site became evident after a 1995 storm when orange-tinted water was observed in Kimble Creek; farther downstream, a fish kill occurred in Pine Creek. The Forest Service and other Federal and State agencies tested water quality and concluded that it was related to AMD and included pollutants, or contaminants, as defined by Comprehensive Environmental Response, Compensation and Liability Act (also known as the Superfund law) section 101(14), low pH surface water discharge from an abandoned coal mine entrance, and low pH seepage from coal mine gob piles. The Pine Creek water samples also exhibited iron hydroxide precipitate, abnormal surface water chemistry, and lack of normal stream fauna. Investigations also found that surface water leaving the abandoned mine site contained concentrations of metals and net acidity known to be detrimental to aquatic life.

A preliminary assessment/site inspection (PA/SI) and an engineering evaluation/cost analysis (EE/CA) concluded that a 0.4-acre area within the underground mine is partially filled with approximately 168,000 gallons of acidic water. The water exiting the mine as surface flow through one or more portals is responsible for the observed AMD. The EE/CA surveyed 14 removal action options and investigated three options in detail: 1) mine dewatering with expanded flow diversion; 2) mine dewatering with limited flow diversion and bioaugmentation; and 3) microbiological treatment system for pollution removal, known as the Pyrolusite Process® (Vail and Riley 1995, 1997, 2000; Allegheny Mineral Abatement, no date).

In the Pyrolusite Process®, AMD-affected water passes through a limestone ‘bed’ on which suitable naturally occurring metal-oxidizing bacteria grow. This method described and patented by Vail and Riley (1995, 1997, 2000; Allegheny Mineral Abatement) appears to be very effective compared to several passive systems described for removal of iron, aluminum, and acidity from AMD (Faulkner and Skousen, 1993, 1994; Hedin et al. 1994a, b; Rose and Dietz 2002; Watzlaf et al. 2000; Ziemkiewicz et al. 1997). Similar methods of using microbial mitigation reactors have also been reported (Bechard et al. 1993, 1994; Cairns et al. 1988; Mills 1985).

Because of potential problems associated with options 1 and 2 that might have impeded success of the remediation project, we decided to investigate the feasibility of the third option through a pilot project before a full-scale system is employed for bioremediation of water at the Kimble Creek site.

In this paper we describe the results of the pilot study.

MATERIALS AND METHODS

The Pyrolusite Process® consists of a limestone "bed" inoculated with selected bacteria. These bacteria sustain growth in an oxygenated environment and are nonpathogenic to plants and animals. As the bacteria grow on rocks, they etch away the limestone, releasing calcium carbonate to neutralize the acidity of the AMD and simultaneously oxidize metals to remove them from water (Vail and Riley 1995, 1997, 2000).

Removal of manganese under these conditions is achieved at a pH 6 to 7, while standard chemical treatment would require the pH to be raised to 9.5. Iron, which is a major component of AMD and normally precipitates out as a voluminous hydroxide (yellowboy) at pH 4, is removed as a dense, crystalline, low-volume oxide. The removal of iron as oxide prevents the system from clogging and keeps water flowing unobstructed. This system also removes aluminum abiotically when the pH increases.

For systems such as these, limestone with the highest calcium content is preferred. The advantages of using limestone include low cost, ease of use, and formation of a dense, easily handled sludge. The disadvantages include slow reaction time, loss in efficiency of the system because of coating of the limestone particles with iron precipitates, difficulty in treating acid mine drainage with a very high ferrous-ferric ratio, and ineffectiveness in manganese removal. However, the utilization of microbes mitigate most of these disadvantages.

The underground treatment plant designed by Robert Kerber of the Wayne National Forest is shown in Figure 1. It had three waterproof compartments roughly 50 ft wide, 15 ft long, and about 5 ft deep. Each compartment contained a bed of limestone.

The influent water (a part of the total flow) entered from bottom of the first compartment, flowed into the second compartment from the top, entered the third compartment from bottom, and finally exited the plant from the top.

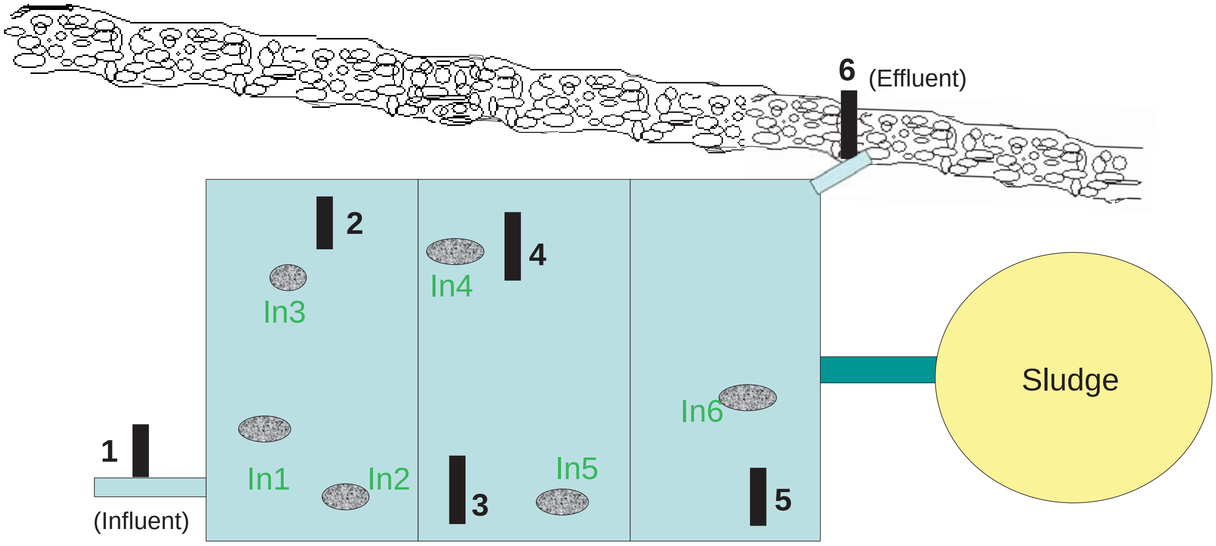


Figure 1.—Aerial view of pilot treatment plant layout and sample collection locations. The treatment plant consisted of 3 water-proof cells, influent water entering cell 1 and exiting from cell 3. Black rectangular icons (numbered 1 to 6) represent ports from where water and rock samples were obtained for analyses. Locations 1 and 6 represent the influent and effluent water, respectively. In1-6 (green) indicate locations of ports for inoculating the system with bacteria. The system was designed such that the sediment could be flushed out from the bottom of the plant to obtain the sludge.

The retention time was about 1,500 cubic ft limestone/gallon/min of flow and the plant was set for an average flow rate of 2 to 4 gallon per minute and a maximum of 15 gallons per minute. The plant was equipped with ports for obtaining water and rock samples (black rectanges 1-6, Fig. 1), as well as ports for inoculating the limestone bed with bacteria (In1-6, Fig. 1).

Design was completed in March 2003, construction was completed in May 2003, first inoculation was done in May 2003, and initial samples were collected in June 2003.

Significant changes in the plant design were made after the plant was in operation for 6 to 12 months. In the original design, water from the hillside portals was pooled at the top of the hill, flowed through an underground pipe, and entered the first compartment from the bottom. However, the oxygen content of the water was too low, which probably caused reduced oxidation of metals leading to a clogged system. To correct this, a manifold was added near the plant entrance to aerate the influent water. In the revised system, water flowed through a manifold and entered the first compartment from the top.

Suboptimal bacterial growth on the limestone surfaces led to another change. This was traced to an inadequate carbon source required by the bacteria.

Therefore, an upstream holding cell covered with vegetation was added to provide more nutrients essential for the bacteria. This design was not initially considered since the site was thickly vegetated. However, after poor performance of the system in early periods, it was added to improve growth of bacteria.

Inoculation

The limestone bed was inoculated with bacteria grown on rock surfaces as well as liquid medium through inoculation ports by the Allegheny Mineral Abatement, Inc. The liquid medium consisted of a mixture of five uncharacterized metal oxidizing bacteria described by Vail and Riley (2000).

Monitoring

Samples of influent water, effluent water, and water from internal points in the plant were collected monthly. A portion of the water sample was acidified with nitric acid immediately after collection for measuring certain characteristics. To measure the Fe^{+2} content, a portion of the sample was preserved in air-tight sample containers to limit spontaneous oxidation. Most chemical analyses were performed by the Ohio Environmental Protection Agency at the agency's Reynoldsburg, Ohio, laboratory. The analysis of iron content (Fe^{+2} and total) was performed by the Aqua Tech Environmental Laboratories, Inc., Marion, Ohio. Water samples were analyzed for aluminum, manganese, magnesium, pH, hardness, chloride, sulfate, alkalinity, and dissolved oxygen.

Rock samples from the limestone bed (5 rocks from each port) were also collected for evaluating presence and growth of bacteria. Bacteria were visualized through a scanning electron microscope (SEM) coupled to an x ray machine; x ray analysis used to identify elements present on the rock surface.

Bacterial growth on the rocks was estimated by determining the total DNA content. Dry rock surfaces were scraped to remove adhering material and subjected to DNA extraction. The material from each rock was suspended in 1 ml 50 mM Tris-HCl, pH 8.0, containing 10 mM EDTA. After disruption for 30 seconds using a Tissuemizer probe attached to a Dremel tool, sodium dodecyl sulfate and proteinase K were added to final concentrations of 0.5 percent and 100 µg/ml, respectively. The mixture was incubated at 37 °C for 1 hour. Sodium chloride (0.7 M final) and hexadecyltrimethylammonium bromide (CTAB) (1% final) were added and incubated at 65 ˚C for 10 minutes. The resulting mixture was extracted with an equal volume of a mixture of chloroform:isoamyl alcohol (24:1). The aqueous portion containing DNA was collected after brief centrifugation and the DNA concentration was determined using a Pharmacia (Basking Ridge, NJ) GeneQuant spectrophotometer.

Sludge Analysis

The plant bed was flushed and all the sediment removed after 8 months. The sludge was examined for the presence of metals and hazardous materials. Also, analysis was performed to measure the forms of iron (Fe^{+2} or Fe^{+3}).

RESULTS AND DISCUSSION

We looked at three main parameters to assess usefulness of the Pyrolusite Process® in improving the water quality at the Kimble Creek site: 1) pH and metal content of influent and effluent water; 2) growth of bacteria and metal deposition on rock surfaces; and 3) the sludge. Sludge was analyzed to determine whether bacteria were oxidizing iron hydroxides to convert into ferric oxides, the quantity of sludge generated, and to test whether the sludge is nontoxic/nonhazardous for disposal according to U.S. Environmental Protection Agency standards.

The original plan was to monitor the system for a 12- to 24-month period after it was in operation. However, in the first year the system did not function well because of the poor design as previously described. After modifications, the pilot study was extended for almost 3 years (until 2007).

One of the major modifications was the addition of a manifold to increase oxygen content of the water. It was essential because the low dissolved oxygen content in the water was not enough for the bacteria to carry out oxidation. This resulted in the accumulation of voluminous ferrous sulfate which was responsible for the poor flow and significant reduction in removal of iron (see Table 1). After the manifold was added, there was significant increase in the dissolved oxygen level which resulted in unobstructed flow through the system and removal of iron. The second major modification was that of adding an upstream holding cell covered with vegetation. This was constructed for dual purposes: to increase AMD collection system and bacterial nutrients, and to provide an oxygenated environment. These modifications resulted in significant improvement of the system's performance. Although the pilot study was extended for 3 years, we present in this report results of the initial period: 2004 to 2005.

Sample Collection

Fig. 1 shows the diagram of the treatment plant and ports from which samples were collected every month. Although the main goal was to test the quality of influent and effluent water, samples from within different compartments were also tested to get an idea about the functioning of the plant. Locations 1 and 6 represent the influent and effluent water, respectively. We arbitrarily chose locations 3 and 5 for routine analyses to understand functioning within different compartments, and later added location 4 to this list. Rock samples were collected from all six locations. Since there were some questions about whether adequate oxygen was available inside the plant for the growth and functioning of the bacteria, the dissolved oxygen (and water temperature) was also measured at all locations.

Water Analyses

The Pyrolusite Process® is designed to use bacteria to oxidize ferrous (Fe^{+2}) hydroxides to the crystalline ferric (Fe^{+3}) oxide form. To test whether oxidation was occurring at the rock surface, we analyzed water samples for Fe^{+2} as well as total iron (Fe^{+2} + Fe^{+3}). This is a difficult task since spontaneous air oxidation can occur during sample collection. However, airtight containers were used to limit such oxidation. We also used chemical stabilizers to arrest most of such oxidation for measuring other parameters.

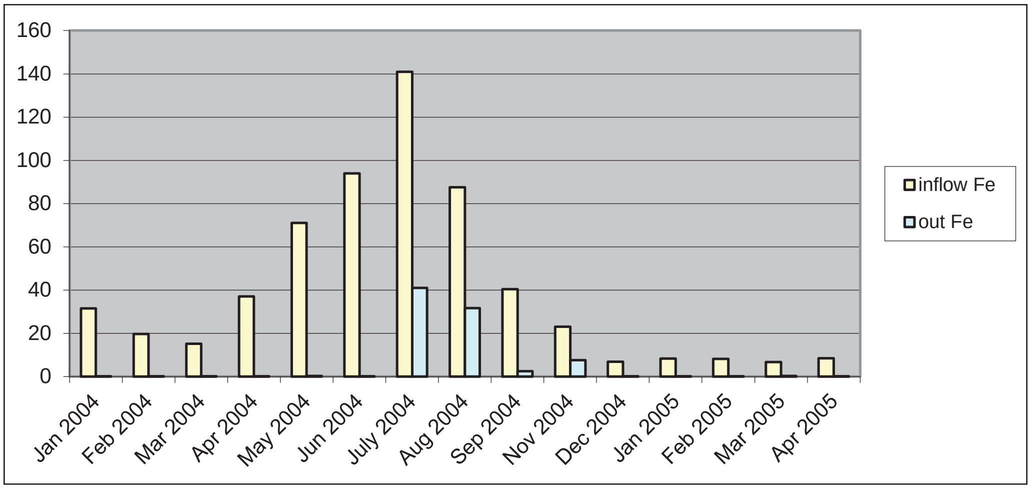


Figure 2.—Iron analysis for the period January 2004-April 2005. Although we monitored samples from all port locations, the figure shows data for the influent (yellow) and effluent (blue) water samples. Iron content is expressed as mg/L.

Analysis showed that while nearly 90 percent of Fe in the influent water was in the ferrous form (+2), by the time it reached location 3 nearly 75 percent was in the ferric (+3) form (data not shown). Samples taken at location 5 had on average less than 1 percent of Fe levels present in the influent water, and most of it was in the ferric form. These results proved that bacteria were indeed oxidizing iron.

Figure 2 shows the amount of iron removed from the water for the period January 2004 to April 2005; some of the earlier data (July 2003 to April 2004) is summarized in Table 1. Due to some initial problems (October to December 2003) with the system, iron was not completely removed (Table 1). We believe several factors were probably responsible. Bacteria had been just introduced and may not have had enough time to establish on the rocks. Also, during these months the amount of iron in the inflow water was unusually high compared to what had been observed earlier. However, the major cause was traced to two factors: inadequate dissolved oxygen essential for bacteria in the oxidation process, and inadequate nutrients necessary for sustained growth of bacteria. This was based on

Table 1.—Iron removal data for Kimble Creek

	Iron Content			pH	
Month	Influent	Effluent	% Removed	Influent	Effluent
July	59.8 mg/L	<0.05 mg/L	100	4.0	7.2
August	53.0	26.8	49	3.27	6.79
September	46.5	5.83	87	3.49	7.38
October	72.1	29.0	60	3.31	6.85
November	35.5	19.0	46	3.77	6.92
December	62.1	14.0	77	3.63	7.15
January	31.5	<0.05	100	4.94	7.49
February	19.5	<0.05	100	4 .81	7.62
March	15.2	<0.05	100	4.97	7.48
April	36.6	<0.05	100	4.91	7.30

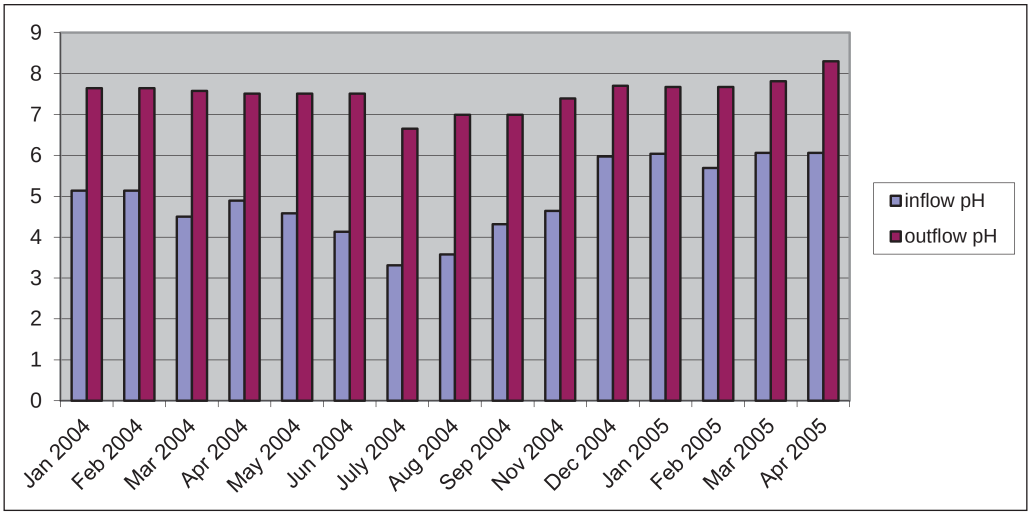


Figure 3.—pH measurements of influent (purple) and effluent (red) water samples.

actual measurement of dissolved oxygen in water samples and bacterial growth on the rocks. After the addition of the manifold and inserting an upstream holding cell, the plant was able to remove almost 100 percent of iron present in influent water.

The pH data followed a similar profile to iron in water samples. The data in Fig. 3 and Table 1 showing the consistently above-neutral pH of the effluent water indicated that the system was functioning efficiently as expected.

Data also indicate that there are seasonal changes in the quality of the influent water. In cold months, the pH was typically higher and amount of iron was low. It is possible that during these months, there is less solubilization of iron at the site of origination and, therefore, less iron in the AMD. This is supported by observed increases in the iron content as soon as the environmental temperature increased (April onwards). In any case, the system appeared to function well throughout the seasonal cycle after the first year.

We also measured dissolved oxygen, temperature, conductivity, concentrations of other metals (Al, Mn, Mg, Mn, Zn, Ca), hardness, sulfate and chloride content, acidity, alkalinity, and dissolved solids. We do not present all the data in this report. However, aluminum was the second most common contaminant observed in the AMD at the Kimble Creek site. The levels, which were as high as 50 to 100 mg/L during some months, were reduced to almost undetectable levels in the effluent samples. Another major pollutant, manganese, which was as high 1 to 1.5 mg/L , was also completely removed by the system. In summary, the effluent water contained no AMD contaminants at levels considered harmful.

Data also indicates that the acidity of the influent water was almost completely removed by the Pyrolusite Process® (Fig. 4). The effluent water consistently had a pH near or above neutral range.

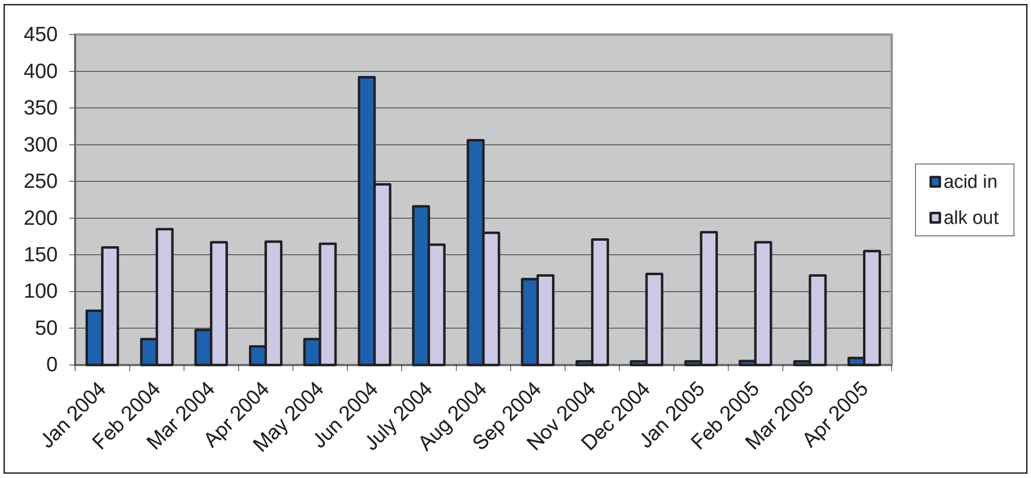


Figure 4.—Acidity (mg/L) and alkalinity (mg/L) of influent (dark blue) and effluent samples (light blue).

Rock Analyses

We assessed the presence and density of bacteria through a scanning electron microscope (SEM) and DNA measurements. The SEM analysis showed presence of bacteria on rock surfaces (Fig. 5). Monthly monitoring suggested that bacterial population on the rocks steadily increased and showed no major differences through seasonal changes.

We also empirically measured bacterial density by determining total DNA mass on the rock surface. This provided a comparative measurement to determine relative growth of bacteria. Results (data not shown) showed that there was a steady increase in DNA mass on the rock surfaces post-inoculation. This would be expected if inoculated bacteria colonized rocks and kept growing over time. However, since these were total DNA measurements, it was not possible to confirm whether the DNA measured is from the inoculated bacteria or from other bacteria or organisms that colonized rock surfaces during the same period.

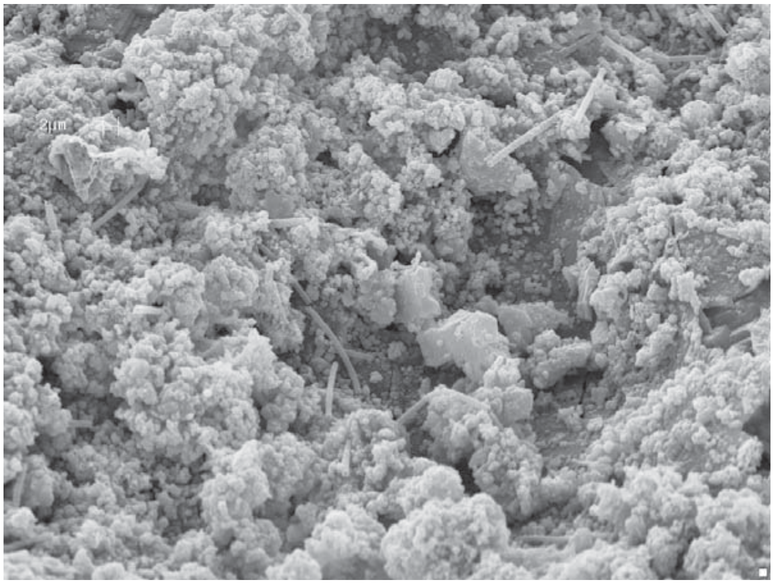
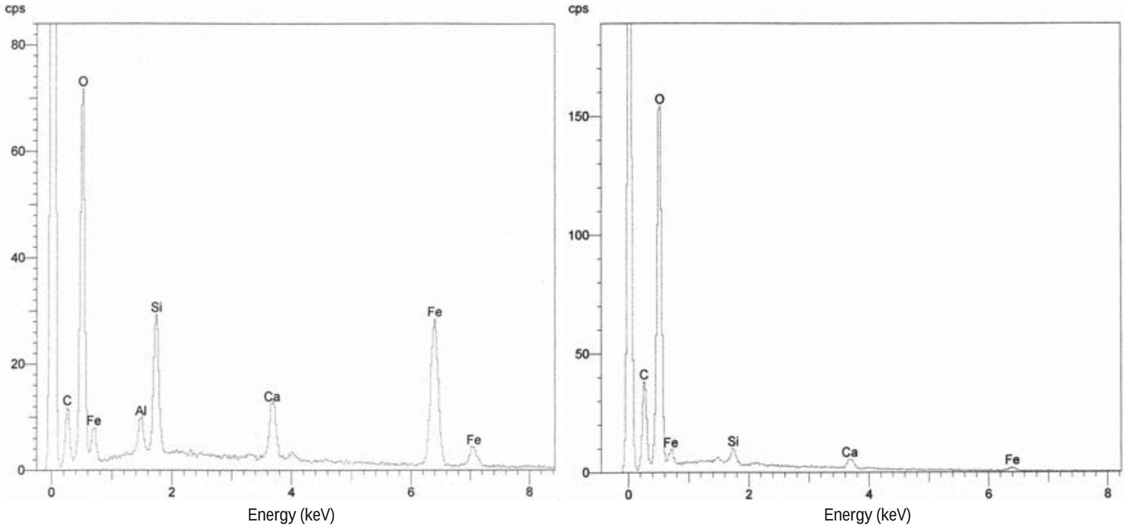


Figure 5.—Bacteria growth on rock surface, shown at 1,000× magnification in this image.



Location 2 **Location 5**

Figure 6.—Elemental analysis of rocks using a scanning electron microscope equipped with a x ray machine. At location 2, iron could be seen whereas by the time water reached location 5, it was hardly detectable. Similar results were observed with aluminum depositions.

We also subjected rock surfaces to elemental analysis using SEM coupled to a x ray machine (Fig. 6). Such analysis shows presence of elements including metals present on the rock surface. Since the probe analyzed only a tiny spot on the rock surface each time, the data gave only a general idea about the contents on the rock and could not be extrapolated to the entire rock surface. For each rock piece, we measured three or four suitable locations. The analysis gave a rough idea about metal deposition and a clue to whether extensive armorization was occurring on the rock surface. The latter phenomenon leads to clogging and slowing/stoppage of water flow. Fe on rocks in location 5 was significantly lower than in location 2 (Fig. 6). This is to be expected since most of the iron precipitated as soon as the pH increased in compartment 1, thereby leaving little iron moving into subsequent compartments of the treatment plant.

Sludge analysis

After 12 months of operation, the system was flushed and the sludge was analyzed. Many pollutant metals from AMD were present in the sludge, but the highest levels were of Fe, Al, and Mn. The solid part of the sludge was estimated to contain 178 g Fe per kg of sludge, 85.7 g Al per kg of sludge, 17.5 g Ca per kg of sludge, 0.85 g Mn per kg of sludge, 1.94 g of Mg per kilogram of sludge, and 0.59 g Zn per kilogram of sludge.

Since we wanted to learn more detail about the oxidation process in the Pyrolusite Process®, we tried to determine the nature of Fe (ferrous [Fe+2] and ferric [Fe+3] forms) in the sludge sediment. Although it is very difficult to stop air oxidation of Fe^{+2} during collection, it was minimized by using stabilizers. It is not easy to accurately determine relative amounts of Fe^{+2} in solid samples, however, a colorimetric method provided a means to approximate the percentage of Fe^{+2} and Fe^{+3} in the sample. Such analyses showed that 90 percent of iron was $Fe^{+3}$and only about 10 percent Fe^{+2}. Finally, the sludge sample was subjected to a toxicity characteristic leaching procedure (TCLP) digestion/analysis to test for hazardous material leaching. Hazardous material amounts were below detectable levels (data not shown) indicating that the material was nonhazardous as per U.S. Environmental Protection Agency regulations and could be disposed as solid waste.

CONCLUSIONS

The Pyrolusite Process® performed very effectively in removing the pollutants from the AMD at the Kimble Creek coal mine site. Results in 2006 to 2007 were similar to what was seen in the latter part of 2005 with minor seasonal changes. Results from this pilot study showed that this system could be employed to improve quality of water flowing into the Kimble Creek. A large-scale system was constructed in 2008 and water from all portals was directed through the system. The large-scale plant has been functioning well and has been effective in increasing the pH and removing all the pollutants contributed by the AMD.

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