Acidification of lead/zinc mine tailings and its effect on heavy metal mobility

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Abstract

The acid-forming potential of lead/zinc (Pb/Zn) mine tailings at Lechang City of Guangdong Province was studied using both net acid generation (NAG) and acid–base accounting (ABA) methods. The pyritic and total sulfur contents of the tailings were 12.6\% and 18.7\%, respectively. The mean acid neutralization capacity (ANC) was 63.5 kg H\textsubscript{2}SO\textsubscript{4}/t while three oxidized tailings samples had an ANC less than zero. The NAG and net acid production potential (NAPP) values were 220 and 326 kg H\textsubscript{2}SO\textsubscript{4}/t, and both the NAG and NAPP results indicated that the tailings had a high acid-forming potential. NAG was more accurate than NAPP in predicting acid-forming potential of the tailings due to uncompleted oxidization of pyritic sulfur. Analysis of samples from two profile tests indicated that acidification mainly occurred at the surface (0–20 cm) and had little effects at deep layer of the tailings. Total concentrations of Pb, Zn, Cu, and Cd were increased greatly with depth at the acidified tailings profile, while heavy metal concentrations at different depths of nonacidified tailings profile were similar. The results indicated that depletion of heavy metals at the acidified surface was due to acidification. The diethylenetriamine pentaacetic acid (DTPA)-extractable Pb, Zn, Cu, and Cd concentrations of acidified tailings surface (0–20 cm) were significantly higher than those of nonacidified tailings, which further revealed that acidification enhanced the mobility of heavy metals in the tailings.

Keywords: Mine tailings; Acid-forming potential; Net acid generation; Acid–base accounting; Heavy metal; China

1. Introduction

Mine tailings produced from the mining activities are the major solid wastes in China and are of environmental concern due to potential hazards of surface or groundwater pollution (Shu et al., 2000). Pyrite bearing mine tailings disposed of at neutral or slightly alkaline conditions can weather within months or few years to produce extreme acidity and lead to acid mine drainage (AMD; Robbed and Robison, 1995). AMD usually contains a high level of heavy metals besides having a low pH and significantly impacts water quality and natural ecosystems in southern China (Hu, 1998). It is also a serious environmental problem around the world (Dudka and Adriano, 1997; Wang, 1999; van Geen et al., 1999). Acidification is also a major constraint for mine tailings revegetation, which is a common and cost-effective method for stabilizing and controlling pollution derived from mine tailings (Bradshaw and Chadwick, 1980). The direct effect of acidity on plants is the high concentrations of hydrogen ions, which inactivate most enzyme systems, restrict respiration, and root uptake of mineral salts and water (Gemmell, 1977; Williamson et al., 1982). Under highly acidic conditions, metal ions including Pb, Zn, Cu, Cd, Fe, Mn, and Al will be released from tailings in levels toxic to plants. Ecological surveys and greenhouse experiments demonstrated that acidification of pyrite in lead/zinc (Pb/Zn) mine tailings in southern China is the most important constraint on plant establishment (Shu, 1997; Wong et al., 1998; Ye et al., 2000). Therefore, accurate prediction of acid forming is necessary for the mine waste disposal management and revegetation strategy (Ritye, 1989). During the past 30 years, various techniques have been developed to predict acid-forming potential of mine waste materials, and the prediction tech-
techniques can basically be categorized into three groups: (1) geological assessment; (2) geochemical static tests; and (3) geochemical kinetic tests. Compared with geological assessment and geochemical kinetic tests, the static test is a simple, rapid, and relatively inexpensive test to qualitatively predict the acid-forming potential of a mine waste material. There are two common approaches to static testing, which included acid–base accounting (ABA) procedure and the hydrogen peroxide direct oxidation procedure: net acid generation (NAG) test (Ferguson and Erickson, 1988; Paktunc, 1999; Jennings et al., 2000).

The Pb/Zn mine tailings in southern China commonly contain high concentrations of pyrite. Oxidation of pyrite resulted in extreme acidity and salinity and the increase of soluble heavy metals. As a consequence, the tailings are almost completely deprived of vegetation. Therefore, the major objectives of the present study were (1) to evaluate the acid-forming potential of Pb/Zn mine tailings at Lechang, Guangdong Province using both the ABA and NAG methods and (2) to investigate the effect of acidification on mobility of heavy metals of the tailings. It was expected that results generated from the present experiment could be useful for the management and revegetation of the tailings.

2. Materials and methods

2.1. Site description

The Lechang Pb/Zn mine is about 4 km east of Lechang City with an area of about 1.5 km². The climate is humid subtropical with an annual rainfall of about 1500 mm, mainly from summer thunderstorms. The mine deposit is located in the east of the mine site. The minerals mainly consist of pyrite, sphalerite, galena, and magnetite and to a lesser extent calcite, muscovite, and quartz. Mining is carried out in a conventional underground way. Mine tailings produced from milling process were discharged into tailings pond as slurry. Approximately 25,000 t of waste rocks and 30,000 tailings are generated annually with a dump area about 8300 and 60,000 m², respectively. Tailings were generally low in nitrogen (N), phosphate (P), and organic matter contents but rather high metal contents. The total Pb, Zn, Cu, Cd, and Mn concentrations (tailings/DTPA, 1:2 w/v) were determined by AAS (Baker et al., 1982).

2.2. Sampling and chemical analysis

Eighteen surface tailings samples were collected randomly at 0–20 cm of tailings ponds, and another 24 samples (three samples at each layer) from two vertical profiles were collected at depths of 0–10, 10–20, 20–50, and 50–100 cm for investigating the changes in oxidation status of the tailings and heavy metal (Pb, Zn, Cu, and Cd) concentrations at different depth. All the samples were air-dried and ground to pass through a 1-mm sieve for determining paste pH and electrical conductivity (EC) using pH meter and EC meter accordingly. Total S (PE CHNS autoanalyzer) and pyritic S contents: Samples were washed with 1 N HCl and digested by 6 N HNO₃ and 70% HClO₄, then iron was determined by atomic absorption spectrometry (AAS; Costigan, 1979; Costigan et al., 1981). Total metal (subsamples digested with concentrated HNO₃/concentrated HClO₄, 5:1 v/v) and DTPA-extractable Pb, Zn Cu, and Cd contents (tailings/DTPA, 1:2 w/v) were determined by AAS (Baker et al., 1982).

2.3. Acid neutralization capacity (ANC), net acid production potential (NAPP), and NAG procedures

ANC: One gram of tailings was added to a 100 ml beaker containing 25 ml of 0.2 mol/l HCl acid and warmed at 90°C for 3 h. After cooling, excess acid was determined by titrating with 0.2 mol/l NaOH, and then the ANC of the samples was calculated in terms of kg H₂SO₄/t; NAPP was then calculated by subtracting the ANC from the calculated maximum potential acidity (MPA) using the following equation (Sobek et al., 1978):

\[ \text{NAPP} \ (\text{kg H}_2\text{SO}_4/\text{t}) = \text{MPA} - \text{ANC} \]

Where MPA = pyritic sulfur content (%) × 30.6.

NAG capacity: 2.5 g of pulverized sample were added to 250 ml of 15% H₂O₂; the mixture was placed inside a fume hood for 24 h, then boiled for 1 h. After cooling to room temperature, the final NAG-pH was measured and then the acidity of the solution was determined by titrating with 0.1 mol/l NaOH solution to pH 7. The NAG was calculated in terms of kg H₂SO₄/t (Finkelman and Giffin, 1986).

3. Results and discussion

3.1. Acid-forming potential of Lechang tailings

Table 1 shows the results of NAG and ABA, including paste pH and EC, ANC, NAPP (based on the pyritic S content), NAG, final NAG-pH, and total and pyrite sulfur contents.

3.1.1. Paste pH and EC

Paste pH of the tailings varied from 1.69 to 7.40 with an average of 5.38, while paste EC ranged from 2.09 to 8.37 dS/m with an average of 4.24 dS/m. The relationship between paste pH and EC is further illustrated in Fig. 1. pH values were negatively correlated with EC (P<.01), indicating that under acidic conditions, the tailings matrix...
was easily dissolved resulting in a higher salt content in the tailings.

It is well known that acidity affects plant growth directly and indirectly. Normal plant growth can be achieved in the pH range of 5–7, but pH values below 3 and above 9 impose adverse effects on plant growth and establishment. Plant species also differ considerably in their ability to tolerate salt content in root medium. In general, all species survive in EC range of 0–2 dS/m and sensitive species are affected by EC of 4–8 dS/m, while only tolerant species can achieve satisfactory growth when EC is greater than 8 dS/m (Gemmell, 1977; Williamson et al., 1982). The lowest pH and highest EC value recorded in the sample collected from Lechang tailings were 1.69 and 13.1 dS/m, indicated that acidity and salinity of the tailings would restrict plant growth and establishment. Therefore, limestone or other alkalinity material should be added to adjust pH and EC conditions of the tailings before planting.

### 3.1.2. Total and pyritic sulfur

Total S contents of the tailings were high, ranging from 9.88% to 26.6%, with an average of 18.7%. Pyritic S contents of the tailings ranged from 4.07% to 20.1%, with an average of 12.6%. Pyritic S contents accounted for 30.4–99.3% of total S contents (with an average of 65.7%), which were varied between samples. In general, the percentage of pyritic S content of total S content was positively \( P < .01 \) correlated with the paste pH (Fig. 2), which indicated that pyritic S of tailings had been oxidized under lower pH. Therefore, the acidic samples often had relatively low contents of pyritic S.

### 3.1.3. ANC of the tailings

The ANC values of the tailings varied from -8.09 to 159 kg \( \text{H}_2\text{SO}_4/\text{t} \), with an average of 63.54 kg \( \text{H}_2\text{SO}_4/\text{t} \). ANC is a measure of the ability of the materials to neutralize acid generated from sulfide oxidation. The inherent base content of mine waste materials includes carbonates, exchangeable cations on clays and silicate minerals.

#### Table 1

<table>
<thead>
<tr>
<th>Sample</th>
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<th>EC (dS/m)</th>
<th>ANC (kg ( \text{H}_2\text{SO}_4/\text{t} ))</th>
<th>NAG (kg ( \text{H}_2\text{SO}_4/\text{t} ))</th>
<th>NAPP (kg ( \text{H}_2\text{SO}_4/\text{t} ))</th>
<th>Total S (%)</th>
<th>Pyritic S (%)</th>
<th>NAG-pH (%)</th>
<th>PS/TS (%)</th>
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<td>67.1</td>
<td>209</td>
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<td>18.3</td>
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<td>2.21</td>
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<tr>
<td>Mean</td>
<td>5.38</td>
<td>4.24</td>
<td>63.5</td>
<td>220</td>
<td>326</td>
<td>18.7</td>
<td>12.7</td>
<td>1.97</td>
<td>65.7</td>
</tr>
</tbody>
</table>

PS/TS: the percentage of pyritic S of total S.

\[ y = -0.9076x + 9.1358 \]
\[ R^2 = 0.8711 \]

Fig. 1. The relationship between pH and EC of Lechang Pb/Zn mine tailings.

Fig. 2. The relationship between percentage of pyritic S of total S and pH of the tailings.
Carbonate minerals, such as calcite (CaCO$_3$) and dolomite [CaMg(CO$_3$)$_2$], are usually the major minerals responsible for the neutralization capacity. It was noticed that the acidic samples had lower or negative ANC values; for example, the pH values of Samples 2, 4, and 6 were 1.88, 1.69, and 1.77, respectively, while the ANC values were $-3.18$, $-6.98$, and $-8.09$ kg H$_2$SO$_4$/t accordingly. The negative ANC values indicated the highly acidic nature of the tailings once they were oxidized.

### 3.1.4. NAPP and NAG

ABA is routinely applied to the analysis of mine waste materials to assess the ability of materials to generate acid upon weathering, and the calculated result of acidification potential is expressed as NAPP. Theoretically, a sample with NAPP > 0 has a MPA greater than the inherent ANC and therefore has a potential to generate acid, whereas a sample with NAPP < 0 has an ANC greater than or equal to the MPA and has the capacity to neutralize acidity generated by the contained sulfides (Finkelman and Giffin, 1986; Jennings et al., 2000). NAPP of Lechang tailings varied from 104 to 594 kg H$_2$SO$_4$/t, with an average of 326 kg H$_2$SO$_4$/t. Both the ABA and NAG tests indicated all the samples were acid forming. Fig. 3 shows the relationship between the calculated NAPP values and the experimental NAG values for the tailings samples. Regression analysis indicated that NAG had a linear relationship with NAPP ($r^2=0.5318$, $P<.01$). However, NAG and NAPP tests yielded different amounts of acid generation. Generally, NAG values were about 1/3 less than NAPP values. Considering that NAG is the actual acid generated from the reactive sulfur while NAPP is a theoretical prediction of the net acid from total sulfur, the calculated NAPP may overestimate the acid generation potential of the tailings due to uncompleted oxidation of pyritic S (Ferguson and Erickson, 1988; Ritcy, 1989). Paktunc (1999) also pointed out that ABA test might result in overestimation or underestimation of acid-forming potential of samples with complex mineral compositions due to the limitation in testing ANC of complex samples. Furthermore, various factors influenced the oxidation of pyrite, such as size, morphology, and type of the pyrite present. The morphology and particle size have an important influence on reactivity, with particles less than 0.25 µm being oxidized readily and greater than 50 µm being fairly stable. The reactivity is also related to the morphology of the pyrite, with frambooidal pyrite (composed of particles less than 0.5 µm) being decomposed rapidly and tabular-euhedral (composed of crystals or euhedra of cubical or triangular sharp, 5–10 µm in diameter) being fairly inert (Pugh et al., 1984; Caruccio et al., 1988). The present study also revealed the uncompleted oxidation of pyrite in the tailings. Percentage of pyrite S vs. total S content of the four strongly acidified samples (Samples 2, 4, 5, and 6) were used to assess the extent of sulfide oxidation. The results indicated that pyrite S accounted for about 40% of the total S contents, and less than 60% of the pyrite S oxidation existed under the field condition at Lechang mine tailings.

<table>
<thead>
<tr>
<th>Sample</th>
<th>pH</th>
<th>EC</th>
<th>ANC</th>
<th>NAG</th>
<th>NAPP</th>
<th>Total S</th>
<th>Pyrite S</th>
<th>NAG-pH</th>
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<td>181e</td>
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</tbody>
</table>

Different letters indicate a significant difference at 5% level according to LSD test (A1, A2, A3, and A4: Samples of 0–10, 10–20, 20–50, and 50–100 cm collected from profile of Site A; B1, B2, B3, and B4: Samples of 0–10, 10–20, 20–50, and 50–100 cm collected from profile of Site B).
3.2. Acid formation properties of the samples at different depths

Acid-forming condition of the samples obtained from the two vertical profiles, A and B at depths of 0–10, 10–20, 20–50, and 50–100 cm, is listed in Table 2. Site A represented the acid-forming surface while Site B the non-acid-forming surface. In Site A, it was noticed that pH values increased with depth. pHe values at 0–10 cm was 1.78, while at 10–20, 20–50, and 50–100 cm were 5.35, 6.20, and 6.86, respectively. EC values were in line with pH. The highest EC value (9.12 dS/m) was obtained at the top 10 cm and descended with depth. The low pH and high EC values indicated that the tailings surface had been acidified. ANC, NAG, NAPP, and total and pyritic S all increased with depth. In the nonacidic profile of Site B, all the parameters at different depths were similar and stable. Results presented here were in line with other studies. The lowest pH values generally occurred at 10–22 cm depth in the coal waste of UK, whereas in South Africa, acidification occurred at surface 0–30 cm depth due to the fact that pyrite oxidation occurs mainly in the surface layers where moisture and oxygen are present (Doubleday, 1972). Morrell et al. (1996) also showed that acidification occurred only at 0–20 cm surface of an abandoned base-metal tailings at Te Aroha, New Zealand.

3.3. Distributions of heavy metals in tailings profile

The distributions of total and extractable heavy metals in tailing profiles are listed in Table 3. In general, the tailings contained high concentrations of Pb and Zn and low concentrations of Cu and Cd. In the acidified profile of Site A, total Pb, Zn, Cu, and Cd concentrations of the surface layer (0–10 cm) and subsurface layer (10–20 cm) were significantly lower than those of deep layers (20–50 and 50–100 cm), while DTPA-extractable metals significantly decreased with depth. In the nonacidified profile of Site B, both total and DTPA-extractable concentrations of Pb, Zn, Cu, and Cd were similar among different depths. It was also found that the extractable concentrations of Pb, Zn, Cu, and Cd in 0–10 cm layer of Site A were significantly higher than those of site B, while total metal concentrations in 0–10 cm layer of Site A were significantly less than those of Site B. The results indicated that acidification accelerated metal solubility, which resulted in depletion of heavy metals in the surface tailings, and acidic reaction might be the most important single factor enhancing soil metal mobility (Morrell et al., 1996; Dudka and Adriano, 1997; Alastuey et al., 1999). Field observation at Lechang indicated that oxidation of sulfides led to formation of extensive white sulfate crust during the dry season from October to February due to the intense evaporation and upward movement of metals by capillary action. However, during the rainy season from March to September, crust completely disappeared because of water erosion. This also contributed to the depletion of heavy metals from the acidic tailings surface.

4. Conclusion

The Lechang Pb/Zn mine tailings contained high contents of total and pyritic S and relatively lower levels of ANC. Both the NAG and ABA tests indicated that the tailings had high acid-forming potential. However, the ABA test is based on the total pyritic sulfur content, which may overestimate the amount of acid generation due to the uncompleted acidification of pyritic sulfur. Many factors are associated with the oxidation of pyritic sulfur such as morphology, oxygen, moisture, structure, and particle size. The present study indicated that only about 60% pyrite S of the surface 20 cm tailings was oxidized at the field conditions. Therefore, the NAG test, which is based on the direct reaction of ANC components with acid produced from pyritic S oxidized by H₂O₂, is more accurate than ABA test in predicting acid-forming potential of Pb/Zn mine tailings. EC values were negatively correlated with paste pH and indicated that the acid released would accelerate dissolution of the solid matrix, resulting in the increase of both cations and anions in solution. The profile test demonstrated that acidification is mainly occurred at the surface 0–20 cm of the tailings and has little effects at lower depth. The results suggested that the Lechang tailings had high acid-forming potential, and the acid tailings would prevent colonization of plant due to toxicities of acidity, salinity, and heavy metals. Therefore, applying alkalinity materials such as limestone or covering the surface with soil and/or placing a barrier layer between the surface and soil cover would be necessary for controlling the acidification of tailings and revegetation.

Acknowledgments

The authors would like to thank the Lechang Lead/Zinc Mine for collecting samples. Financial support from the...
Natural Science Foundation of China (No. 39770154) and the Research Grants Council of University Grants Committee of Hong Kong is gratefully acknowledged.

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