Leaching of Metal Ions and Suspended Solids from Slag Corroded by Acid-base Solutions: An Experimental Study

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ABSTRACT

This study focused on the ion release and microstructure of slag during its degradation following erosion by different pH solutions. It focused on controlling factors such as slag particle size range, pH value of the solution, and soaking time. The surface microstructure and particle size distribution of slag with the particle size of 0.075–5.0 mm, the mineral composition of suspended pollutants larger than 0.45 µm, and the phenomenon of nano-scale ion release were examined. When slag was soaked in solutions with different pH values for 30 days, the pH value of leachate tended to be neutral, the release amount of Ca, Mg, Fe, and Cd ions increased and the release rate gradually decreased. The dissolution process of slag in the alkaline solution was slower than that in acid, but suspension and gels formed more easily in an alkaline environment. Nitric acid accelerated the chemical reaction of akermanite, gehlenite, and hawleyite, and released Ca, Mg, and Cd ions. There were clear damage cracks and various irregular pores on the slag surface. Under the attack of alkali solution, the weight of akermanite in slag increased, the Mg ion content in solution decreased, and the suspended solids of calcite and portlandite increased. At pH 12, unlike at pH 3, there were no large surface cracks in the slag and the interface damage was small. Compared with pH 7, there were more irregular substances, such as flakes and spheres. The particle size of slag was mainly 0.1–0.5 mm, the content before and after leaching was 52.80%–55.87% and 55.00%–58.27%, and the slag was in a poor grading state. The findings of this study act as an important reference for understanding the influence of slag leaching on water and soil pollution.

INTRODUCTION

At present, thousands of slag dumps exist globally, such as those left by small historical iron furnaces in some parts of the United States (Piatak et al. 2015). Slag pollution caused by mining and smelting activities has also become a problem that cannot be ignored in China. Ferrous slag is used in the generation of several materials, but the various sulfides, intermetallic compounds, silicates, calcium, iron, magnesium, aluminum, cadmium, and other trace elements generated after soaking of slag can cause serious environmental pollution.

Water from slag contains different concentrations of ions, and the highest concentration of each ion is located at different depths of an aquifer (Bayless et al. 1998). Where groundwater reaches the surface, slag produces mineral precipitation and releases a large quantity of trace elements (cadmium, chromium, copper, lead, nickel, and zinc) with high mobility (Roadcap et al. 2005). Leachate produced by ferrous slag soaking contains a large number of trace elements, which have harmful effects on surface water and

groundwater. After an artificial leaching experiment, the concentration of some metal ions in Fe slag was found to exceed the standard (Piatak & Seal 2012, USEPA 2009a, b). Fällman & Hartlén (1994) compared the pH value and redox potential of slag leachate in laboratory tests and field tests. Sloto & Reif (2011) found that the composition of groundwater and surface water was similar to that of leachate extract, indicating the feasibility of indoor leaching tests. The acidic neutralization ability of slag could be used as a soil conditioner to treat acidic wastewater or acidic sulfate soil (Álvarez-Valero et al. 2007, Wendling et al. 2012, 2013). Research has also shown that the reaction products of alkali-activated slag are mainly C-S-H and C-(N)-A-S-H gels (Hong & Glasser 2002, Escalante-García et al. 2003, Skibsted & Andersen 2012, Myers et al. 2013).

Some scholars have focused on the effects of pH value, liquid-solid ratio, temperature, slag fine particle ratio, and mineral phase on ion dissolution kinetics in slag. For example, Chaurand et al. (2006) used static leaching tests (pH value controlled at 5) to study the release of Cr and V from slag, and

Ning et al. (2016) discussed the extraction mechanism of Cr and V from slag. Barna et al. (2004) showed that the release of ions from slag is controlled by the pH value of leachate. The solubility of Pb ion at pH 1.5 and 1.7 was simulated (Bosso & Enzweiler 2007), and the problem of high release of Pb ion was further explored (De Angelis & Medici 2012). Different liquid-solid ratios and temperatures had different effects on metal release from slag (De Windt et al. 2011, Nikolić et al. 2016). Fine fractions from slag sample crushing usually promote leaching of Cu, Co, and Zn (Vítková et al. 2011). The mobility of heavy metals in slag depends on the morphology of the ore-bearing stage (Chaurand et al. 2007). The mineral phase of slag is also easily dissolved in rainwater. A large amount of As, Pb, and Cu can be rapidly distributed into the environment near slag fields (Ettler et al. 2009), thus increasing the research demand on slag mineralogy (Ettler et al. 2012). However, to our knowledge, there are few comparative studies on the release characteristic of various icons of the same kind of slag in acid-base solutions with different concentration gradients and the deterioration of slag.

Therefore, this paper is focused on the quantification of the environmental aspects of slag waste through the study of the release characteristics and microscopic changes of ions during slag soaking. This study aimed to evaluate the release of potentially harmful trace elements in slag, further describe the potential environmental impact laws of slag, and provide reference data for use in preventing pollution problems caused by slag accumulation and leaching. The surface microstructure and particle size distribution of slag with a particle size of 0.075–5.0 mm, the mineral composition of suspended pollutants larger than 0.45 μm, and the phenomenon of nano-scale ion release were the main research objects. The findings of this study can serve as an important reference for understanding the influence of slag leaching on water and soil pollution.

MATERIALS AND METHODS

Test Materials

The slag used in the test was taken from a ferrous slag yard (China Baowu Steel Group Co. Ltd., Qingshan District,

Wuhan, China). The samples did not show excessive aging or weathering. Slag was divided into three different particle sizes: 0.075–0.3, 0.3–1.6, and 1.6–5.0 mm. The main chemical compositions of slag are shown in Table 1, and the main mineral compositions of slag are shown in Table 2.

The soaking solution used in the test was prepared with HNO_3, NaOH, and demineralized water. The initial pH values of the solution were 3, 5, 7, 9, and 12.

Test Methods

Leaching tests: When the test temperature was controlled at $25 \pm 2°C$, every 100 g of ferrous slag with different particle sizes (D = 0.075-0.3, 0.3-1.6 and 1.6-5.0 mm) was submerged in 300 mL of solution with different pH values (pH 3, 5, 7, 9 and 12), and the leaching solution was taken out at increasing periods (T = 1, 3, 5, 10, 20 and 30 days). For the leaching solution, approximately 50 mL was placed into a centrifuge tube. Then centrifuge was rotated for 1 h (rotating speed: 3000 rpm, radius: 13.5 cm, centrifugal force: 1349 g) to obtain supernatant (Du et al. 2005). Finally, the pH value, metal ion concentration, and suspended solids (SS) quality of the leaching solution were tested. The PHS-3E pH Meter (Shanghai INESA Scientific Instrument Co., Ltd, China) has an accuracy of ± 0.01 units. The concentration of metal ions was determined with an AA320 atomic absorption spectrophotometer (Shanghai INESA Analytical Instruments Co., Ltd). Before the SS in the solution was recorded, it was dried to constant weight at 50°C.

X-ray diffraction (XRD) tests: After 30 days of leaching, for slag of 0.075-0.3 mm, three groups of slag samples (soaked in solutions with pH 3, 7, and 12) were ground through a No. 350 sieve, and the two groups of precipitates (pH 3 and 12) obtained by centrifugation in the leaching tests were filtered through a 0.45-μm membrane. The powdery slag and SS samples were air-dried to constant weight. Mineralogical analyses of the sample were conducted using an XRD analyzer (PANalytical B.V., The Netherlands).

Scanning electron microscopy (SEM) tests: After leaching solutions with different pH values for 30 days, three groups of 0.075–0.3mm blocky slag samples (soaked in solutions

Table 1: Main chemical composition of slag.

Main chemical compositions	SiO_2	Al_2O_3	CaO	Fe_3O_4	MgO	AlN	CdS
W_t / [%]	28.74	14.27	36.07	0.34	9.74	0.34	0.53

Table 2: Main mineral compositions of slag.

Main mineral compositions	Akermanite	Gehlenite	Yavapaiite	Gypsum
W_t / [%]	30.6	36.2	25.8	7.3

with pH 3, 7, and 12) were magnified and observed with an S-3000N SEM (Kabushiki Kaisha Hitachi Seisakusho, Japan).

Particle size distribution (PSD) analysis tests: To explore the change of particle size of slag in different acid and alkali solutions in the actual environment, the weight of slag in seven particle size ranges (>5, 1–5, 0.5–1, 0.25–0.5, 0.1–0.25, 0.075–0.1 and <0.075 mm) was tested before and after leaching. First, 500 g subsamples were taken from each of the nine groups of slag before soaking, and 5-mm and 1-mm sieves were used to screen out slag with a particle size larger than 1 mm for weighing. The slag particles with a diameter of less than 1 mm were then subjected to PSD analysis tests. The PSD was tested with Mastersizer 3000 laser diffraction particle size analyzer (Malvern Instruments Ltd., UK). Finally, the slag was submerged in the solution (pH 3, 7, and 12) for 30 days in a liquid-solid ratio of 3:1, and the PSD analysis method was applied again as described above. Every three groups of slag were soaked in a solution, and the weight of the three groups of slag was calculated and summed to obtain grading curves for the slag particles.

RESULTS AND DISCUSSION

Leaching of Slag

The slag leachate in this test contains a large amount of Ca, Mg, Fe, Cd, and trace heavy metals. The concentration of ions has far exceeded the threshold of China's national standard - "Identification standards for hazardous wastes – Identification for extraction toxicity" (GB 5085.3-2007). These ions enter the surrounding soil and groundwater, which will seriously damage the soil environment and lead to reduced water quality as a result of water hardening and water pollution (Xue et al. 2013).

The curves of Ca, Mg, Fe, Cd concentration, and pH value of the solution are shown in Fig. 1 and Fig. 2 (a). With the increase of dissolution time, the different acid-base solutions tend to become neutral. After 30 days of leaching, the pH values of solutions with initial pH 3 and 5 increased by 2.28-3.22 and 1.1-1.43, while those with initial pH 7, 9, and 12 decreased by 0.24-0.82, 0.82-1.24, and 1.21-2.25, thus the pH values of solutions with initial pH 3 and 12 changed the most. The release amount of the four cations gradually increased and the release rate decreased, but the release behavior results varied among the four cations.

The release amount of iron ions in ferrous slag was large, for example, slag with a particle size of 0.3-1.6 mm released up to 9.779 mg.L^{-1} of iron ions in the initial pH 3 solution. The particle size of slag had little influence on the dissolution behavior of Cd, but at pH 12, the release amount of Cd accounted for 3.02%-5.17% of the amount at pH 3. With the increase of the particle size range, the dissolved amount of Ca decreased by 39.09%-60.43%. The dissolved amount of Mg was strongly affected by the change of particle size, showing a trend of increasing first by 1.71%-49.77% and then decreasing by 0.95%–78.26%. Slag with the same particle size range was leached for 30 days, and the leaching amount of Ca and Mg at pH 12 accounted for 1.73%-9.55% and 0.08%-0.26% of that at pH 3, respectively. The characteristics of ion leaching in slag show that the dissolution process of Ca, Mg, and Cd in the alkaline solution was very slow, and the dissolution rate and amount were less than those in an acidic solution.

The variation curves of suspended solids' weight during the leaching of slag with different particle sizes are shown in

Table 3: Mineral composition of slag and suspended solids.

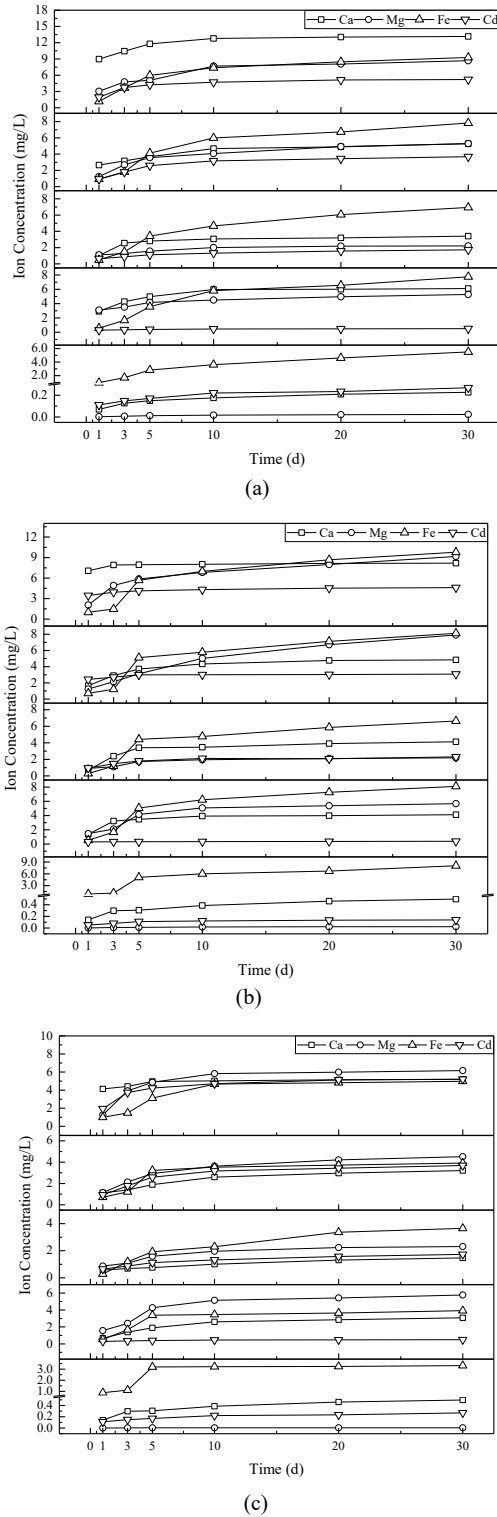| W$_t$ / [%]	Phase Slag				suspended solids (SS)	
	Before leaching	pH=3 T=30d	pH=7 T=30d	pH=12 T=30d	pH=3 T=30d	pH=12 T=30d
Akermanite	30.6	14.7	50.7	34.6	4.7	—
Gehlenite	36.2	17.6	42.2	29.8	6.8	—
Yavapaiite	25.8	—	—	—	—	—
Gypsum	7.3	—	2.5	—	80.6	—
Rustumite	—	67.7	—	—	—	—
Hawleyite	—	—	0.4	0.2	—	—
Hydrotalcite	—	—	4.2	—	—	—
Calcite	—	—	—	35.4	5.7	76.9
Quartz	—	—	—	—	2.2	—
Portlandite	—	—	—	—	—	23.1

Fig. 1: Curves of Ca, Mg, Fe and Cd concentrations in the solution over
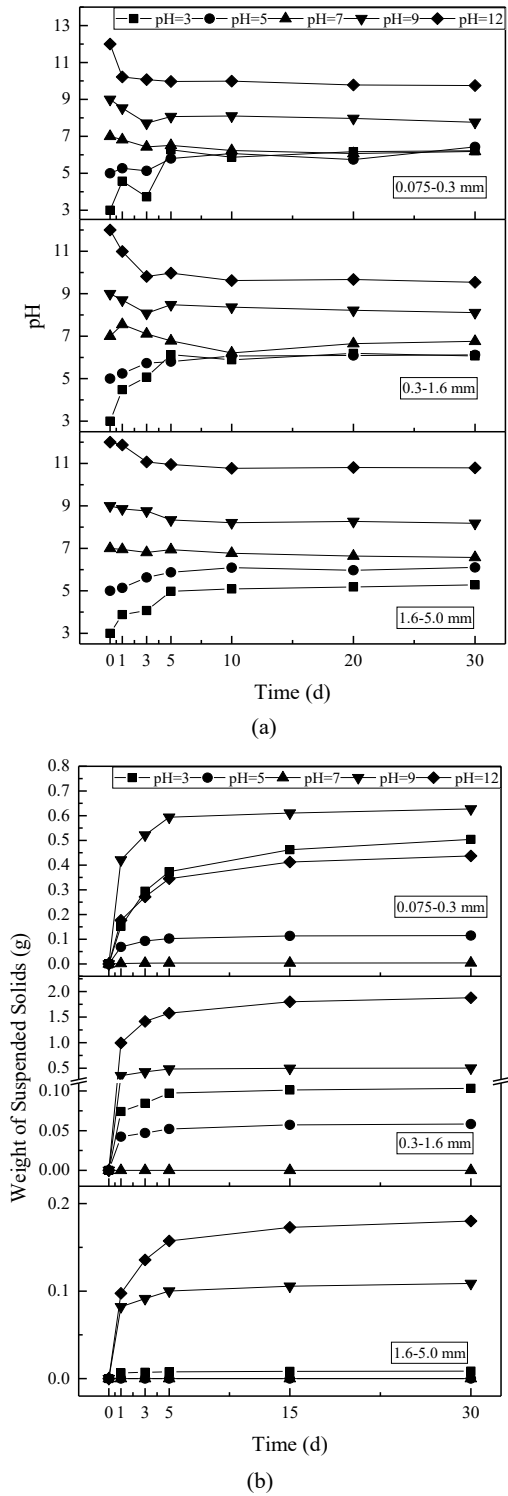time: (a) D=0.075-0.3mm, (b) D=0.3-1.6mm,
(c) D=1.6-5.0mm.

Fig. 2: Variation curves of solution pH value and suspended solids weight
during leaching of slag with different particle sizes:
(a) pH, (b) suspended solids weight.

Fig. 2(b). At pH 3, 5, 7 and 9, the weight of SS decreased in the following order: $W_{0.075-0.3\ mm} > W_{0.3-1.6\ mm} > W_{1.6-5.0\ mm}$. This shows that the particle size was negatively correlated with the total mass of SS in the solution. However, at pH 12, the weight of SS decreased in the following order: $W_{0.3-1.6\ mm} > W_{0.075-0.3\ mm} > W_{1.6-5.0\ mm}$. This indicates that when the particle size was between 0.3 and 1.6 mm, the quantity of SS leached from slag was greatly affected by the pH, not the particle size. Considering that the testing effect of 1.6–5.0 mm slag was unclear, the 0.075–0.3 mm slag was selected for XRD and SEM tests.

There are two possible explanations for these findings. On the one hand, under acidic conditions, all calcium compounds were separated and decalcified, resulting in structural changes in which the surface of slag formed a porous gel-like cover. The acid reacted with the slag, and the products were soluble calcium salt and poorly cohesive silica-rich gel (Bernal et al. 2012). The calcium salt and gel left the surface of slag to become SS, and various metal ions were also released into the solution. On the other hand, in the alkaline activation process of slag, a hydrated calcium silicate of the C-S-H gel-type was formed as the main reaction product, which has a low C/S ratio (Puertas et al. 2000). Many metal ions reacted with anions in the solution to generate precipitates that are in SS form in the solution.

Mineral Composition

According to XRD test results in Figs. 3, 4, and Table 3, after leaching in the solution with pH 3 for 30 days, the weight (W_t) of akermanite ($Ca_2MgSi_2O_7$) in slag decreased by 15.9% and increased by 20.1% and 4.0% at pH 7 and 12. The weight of gehlenite ($Ca_2Al_2SiO_7$) decreased by 18.6% and 6.4% at pH 3 and 12 and increased by 6.0% at pH 7. The XRD results showed that nitric acid at pH 3 expedited the corrosion of akermanite and gehlenite in slag, which led to the increase of

Ca ion and Mg ion concentrations in the solution. Rustumite [$Ca_{10}(SiO_4)(Si_2O_7)_2(OH)_2$] was produced by the reaction of Ca ions with silicate in slag, and the weight of rustumite in the slag was 67.7%. Ca ions reacted with sulfate in solution to form gypsum ($CaSO_4.2H_2O$), with a weight of 80.6 %.

At pH 12, the weight of akermanite in slag increased, whereas that of gehlenite decreased, indicating that Ca ions were alkaline solutions, and calcium silicate hydrate (C-S-H) and calcium-sodium aluminosilicate hydrate [C-(N)-A-S-H] gels are also formed on the surface of slag (Myers et al. 2013). In the current study, hawleyite (CdS) reacted more obviously when released from slag as a whole, but almost no Mg ions were released into an alkaline solution. Calcite ($CaCO_3$) and portlandite [$Ca(OH)_2$] suspensions are more easily formed in the acidic solution at pH 3 than in an alkaline environment, releasing a small amount of H_2S gas and Cd ions. Yavapiaite [$KFe(SO_4)_2$] was soluble in water and released Fe ions in solutions with different pH values.
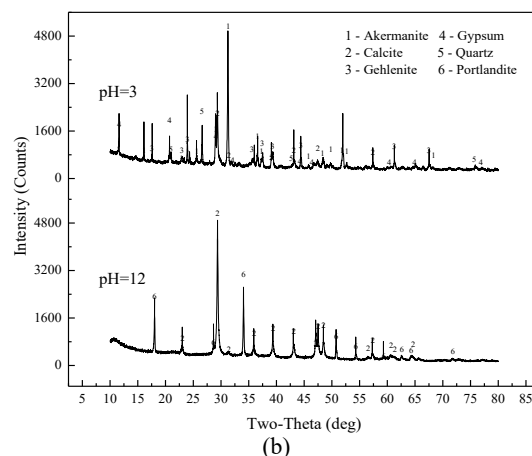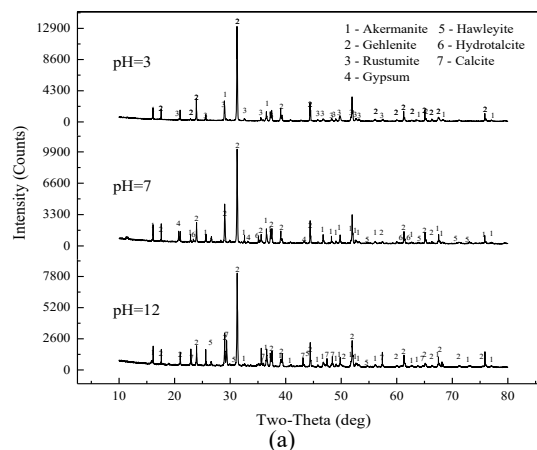
Fig. 4: XRD curves of slag and suspended solids after leaching in different pH solutions for 30 days: (a) slag, (b) suspended solids.

Fig. 3: XRD curve of slag before leaching.

Surface Microstructure

The surface microstructure of slag during leaching with an acid-base solution is shown in Fig. 5. The slag surface before soaking is smooth and complete, with few pores and a relatively dense microstructure. After soaking in different pH solutions for 30 days, the slag surface was eroded and destroyed. The cracks, dispersed fine particles and pores between fine particles increased substantially.

The SEM figures clearly show that the microstructure of the slag surface changed considerably after soaking in solutions with different pH values. For 0.075-0.3mm blocky slag samples, the HNO_3 solution was more destructive than the demineralized water and alkali solution. The slag was corroded most seriously at pH 3 and there were apparent damage cracks and various irregular pores on the surface. The cracks were 589-651 μm long and 70-79 μm wide, which was probably due to the formation of soluble calcium salts in the nitric acid solution by akermanite and gehlenite. At pH 7, dispersed particles and apparent irregular pores could be seen on the slag surface. At pH 12, the surface cracks of slag were not prominent compared with those at pH 3. Moreover, the interface damage was less and there were no obvious wide cracks. Compared with pH 7, although there were irregular pores on the surface of slag, after soaking in alkali solution, the unconsolidated tiny particles on the surface of slag were less attached, but more irregular materials such as flakes and spheres had agglomerated. The XRD test results indicate that this phenomenon was caused by the formation of C-S-H and C-(N)-A-S-H gels from the alkali-activated slag.

PSD of Slag

The curve and the discrimination index of particle size gradation after leaching in different pH solutions for 30 days are shown in Figs 6 and 7. The particle size of slag was mainly concentrated in the range of 0.1-0.5 mm, and the contents before and after leaching were 52.80%-55.87% and 55.00%-58.27%, respectively. The weight of slag larger than 0.1 mm after soaking was reduced by 5.03%-37.35%, whereas the slag smaller than 0.5 mm was increased by 3.67%-30.20%. D_{10}, D_{30}, and D_{60} (particle size less than 10%, 30%, and 60% of the total mass in the slag grading curve) all showed a decreasing trend, among which D_{10} changed most substantially. This indicates that the smaller the particle size, the larger the specific surface area, and the more serious the erosion by a chemical solution. At pH 3 and 12, D_{10} decreased by 21.33% and 14.93%, respectively. Together with the results of the leaching and XRD tests, this indicates that these decreases occurred as the result of slag being eroded and releasing ions in the solution at pH 12, and gels were then formed in the alkaline environment.

According to ASTM D 2487 (2017), before and after leaching, 88.07%-90.07% and 86.33%-87.07% of slag were
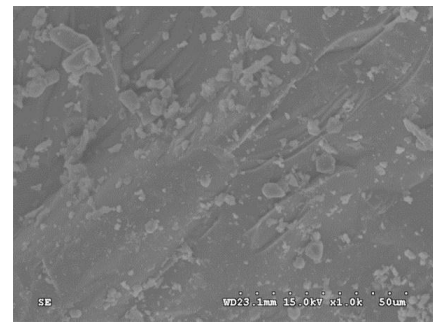
(a)
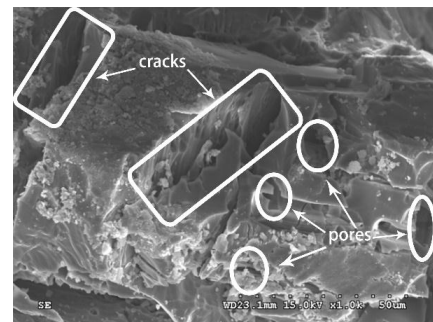
(b)
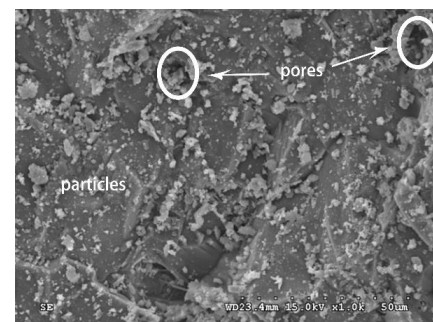
(c)
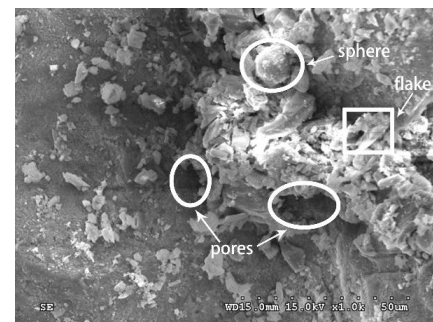
(d)

Fig. 5: Surface microstructure of slag: (a) Before leaching, (b) pH=3, T=30d, (c) pH=7, T=30d, (d) pH=12, T=30d

retained on the 0.075-mm sieve, and 93.47%–94.47% and 95.73%–96.53% passed through the 5-mm sieve. In the leaching process of different pH solutions, the coefficient of curvature (C_c) increased from 0.716–0.788 to 0.819–0.877 but remained below 1. This indicates that the slag with different particle sizes decreased after leaching, but the continuity was always poor. The coefficient of nonuniformity (C_u) increased from 4.560–4.925 to 5.034–5.527. This shows that the slag before soaking was uniform, the gradation is poor, and the particle size of slag after leaching is gradually uneven. In short, according to the changes of C_c and C_u, the conditions $C_u < 6$ and $C_c < 1$ were always true, indicating that the slag before and after leaching is poorly graded slag.

CONCLUSION

A series of experiments were carried out on the influence of different factors on slag leaching, such as slag particle size range, pH value of the solution, and soaking time. This paper mainly studied the change of slag particle size at the millimeter level (0.075-5.0 mm), the leaching of suspended solids at the micron level (larger than 0.45 μm) and the metal ions release behavior at the nanometer level. The following conclusions were drawn:

1. With the increase of dissolution time, the pH of slag leachate tended to be neutral. After 30 days of leaching, the pH values of solutions with initial pH 3 and 5 increased by 2.28–3.22 and 1.1–1.43, while those with initial pH 7, 9, and 12 decreased by 0.24–0.82, 0.82–1.24, and 1.21–2.25. Therefore, the pH values of solutions with initial pH 3 and 12 changed most substantially. The dissolution process of Ca, Mg, and Cd in alkaline solution was very slow, and the dissolution rate and amount were lower than those in acidic solution. At pH 12, the dissolved amount of Ca and Mg accounted for 1.73–9.55% and 0.08–0.26% of that at pH 3, respectively.

2. Nitric acid at pH 3 expedited the corrosion of akermanite and gehlenite in slag, which led to the increase of Ca and Mg ion concentrations in the solution, and Ca ion reacted with soluble salt to form a rustumite and gypsum suspension. Hawleyite released a small amount of H_2S gas and Cd ions in an acidic solution. At pH 12, the concentration of Ca ions in the solution formed calcite and portlandite suspended solids, whereas there were too few Mg ions to form precipitates.

3. After soaking in solutions with different pH values for 30 days, the slag surface was eroded and destroyed, cracks dispersed fine particles, and pores between fine particles increased substantially. Slag corrosion was most serious at pH 3 and there were clear cracks on the surface, which were 589–651 μm long and 70-79 μm wide. At pH 12, there were no large cracks on the slag surface, but there were more irregular substances such as flakes and spheres.

4. Before and after slag leaching, the particle size was mainly concentrated in the range of 0.1-0.5 mm. The weight of slag larger than 0.1 mm after soaking was reduced by 5.03%-37.35%, whereas the slag smaller than 0.5 mm was increased by 3.67-30.20%. The C_c increased from 0.716-0.788 to 0.819-0.877, and the C_u increased from 4.560-4.925 to 5.034-5.527, but the conditions $C_u < 6$ and $C_c < 1$ were always true, indicating that the slag had always been in a poor grading state.

ACKNOWLEDGMENTS

Fig. 6: Grading curve of slag particle after leaching in different pH solutions for 30 days.

Fig. 7: Discrimination index of particle size gradation after leaching in different pH solutions for 30 days.

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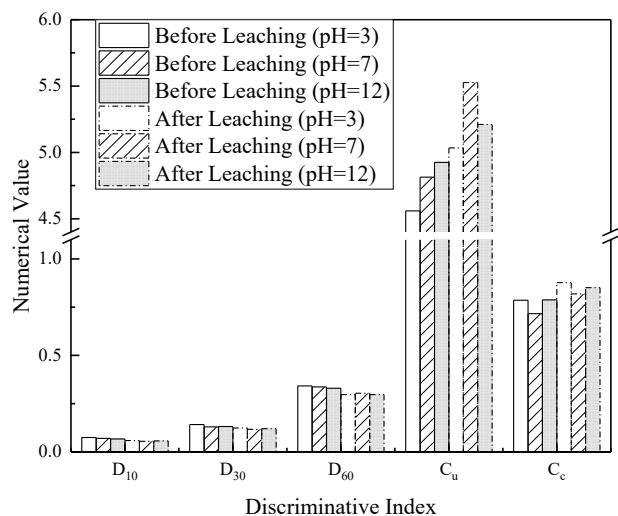Bosso, S.T. and Enzweiler, J. 2007. Bioaccessible lead in soils, slag, and mine wastes from an abandoned mining district in Brazil. Environ. Geochem. Health, 30(3): 219-229.

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