Leaching behaviour of zinc from a smithsonite ore in sodium hydroxide solutions

Ilhan Ehsani, Ayse Ucyildiz, Abdullah Obut

Hacettepe University, Mining Engineering Department, 06800 Beytepe, Ankara, Turkey

Corresponding author: ilhan.ehsani@hacettepe.edu.tr (Ilhan Ehsani)

Abstract: In this study, the leaching behaviour of zinc from a smithsonite ore sample (23.43% Zn) having goethite and calcite as main gangue minerals was investigated in sodium hydroxide solutions using X-ray diffraction and chemical analyses. Within the studied NaOH concentration range (1-4 mole/dm³), higher leaching ratio values for Zn were obtained by leaching at 298 K because of the decreased stability of soluble zincate species in solution at higher temperatures. When the concentration of NaOH solution was increased from 1 to 3 mole/dm³ at 298 K, leaching ratio value of Zn increased from 3.8 to 70.1%. At 4 mole/dm³ NaOH concentration, Zn leaching ratio value (70.9%) levelled off due to the formation of solid hydrated calcium zincate phase in leaching solution. On the other hand, very low leaching ratio values of Zn were obtained by leaching in 1 mole/dm³ NaOH solution at 298 K (3.8%) and 363 K (1.4%) due to the formation of solid zinc hydroxide and solid zinc oxide phases, respectively. It was observed that increasing leaching time from 1800 to 14400 s at 3 mole/dm³ NaOH concentration, favoured the formation of hydrated calcium zincate phase and so decreased the leaching ratio of Zn to a lower value (60.6%). The effect of solid/liquid ratio was also investigated and it was found that when solid/liquid ratio was decreased, Zn leaching ratio values decreased at constant OH/Zn mole ratio and increased at constant NaOH concentration conditions. Besides, for some of the experiments, leaching ratio values of lead were also given.

Keywords: alkaline leaching, calcium zincate, nonsulfide zinc ore, smithsonite, zinc carbonate

1. Introduction

Zinc is an industrially important bluish-white, lustrous, low boiling point metal, and often used for galvanization and alloying. It has a valence of +2 and may form complexes with ammonia, halide ions, etc. (Graf, 2005; ILZSG, 2017). Zinc is found mainly in two different types of ores, namely the sulfide and the nonsulfide ores. In sulfide ores, sphalerite (ZnS) is the main ore mineral and is easily concentrated by conventional flotation method. Most of the world zinc production comes from the sulfide deposits. However, because of the steadily decreasing sulfide ore reserves and the increasing demand for zinc, in recent years, the scientific and economic interest for the processing of nonsulfide ores are increasing (Qin et al., 2007; Liu et al., 2011; Irannajad et al., 2013; Abkhoshk et al., 2014; Feng et al., 2015; Rao et al., 2015; Zhang et al., 2016; Ghasemi and Azizi, 2017). In nonsulfide ores, the economically important zinc minerals may be smithsonite (ZnCO₃), hemimorphite (Zn₅Si₄O₁₂(OH)₂·H₂O), hydrozincite (Zn₅(CO₃)₂(OH)₆), willemite (Zn₅SiO₄) and sauconite (Na₈.5Zn₅(Si,Al)₈O₁₀(OH)₂·4H₂O) (Hitzman et al., 2003), and due to their lower grades and comparatively complex nature, these ores cannot be effectively beneficiated (Hosseini and Forssberg, 2009; Ejtemaei et al., 2014). As a result, leaching becomes a good option for the extraction of zinc from these zinc ores.

Other than acidic leaching systems using sulfuric (Frenay, 1985; Espiari et al., 2006; Abdel-Aal et al., 2016; Ghasemi and Azizi, 2017), hydrochloric (Dhawan et al., 2011; Ghasemi and Azizi, 2017), nitric (Ghasemi and Azizi, 2017), citric (Irannajad et al., 2013; Ghasemi and Azizi, 2017), boric (Abali et al.,
and 0.15
the sample, whose amount was calculated according to the selected solid/liquid
was transferred into the aqueous solution of sodium hydroxide
cerussite in lower amounts.
calcite
ore sample collectively indicated that the sample
heating rate of 2°/60 s
(XRD) pattern (Rigaku
ignition value of the sample were determined as 1.42% and 24.37%, respectivel
Ba, Cu, Ag, Ni, Co and As were less than 0.1%. On the other hand, the moisture content and loss on
was finer than 38 µm.
sieving showed that cumulative 80% passing size of the sample was 124.5 µm and 52.5% of
closed circuited ball mill with a sieve.
In the experimental studies, Merck grade reagents, classical laboratory equipments, deionized water,
was finer than 38 µm. The sample also contained other than the main chemical components (Table 1), i.e. 1.92% Pb, 0.26% K, 0.30% Mg, 0.111% Cd and 0.11% TiO₂, and the contents of S, Mn, P₂O₅, Na, Cr₂O₃, Ba, Cu, Ag, Ni, Co and As were less than 0.1%. On the other hand, the moisture content and loss on
ignition value of the sample were determined as 1.42% and 24.37%, respectively. The X-ray diffraction
(XRD) pattern (Rigaku Dmax-2200, operated at 40 mA/40 kV using CuKα radiation at scanning speed
of 2°/60 s), the thermal (TG/DTA) curves (Setaram Labsys, obtained under static air atmosphere at a
heating rate of 0.167 K/s) and the infrared (FTIR) spectrum (Perkin Elmer, KBr pellet method) of the
ore sample collectively indicated that the sample was mainly composed of smithsonite, goethite and
calcite, and it also contained quartz, clay minerals (kaolinite, trace illite), hemimorphite, hematite and
berussite in lower amounts.

2. Materials and methods

In the experimental studies, Merck grade reagents, classical laboratory equipments, deionized water,
and a ground smithsonite ore sample were used. The representative ore sample taken from the
nonsulfide zinc ore deposit in Kayseri was first crushed with a jaw crusher then it was ground using
closed circuited ball mill with a sieve. The particle size distribution of the sample determined by wet
sieving showed that cumulative 80% passing size of the sample was 124.5 µm and 52.5% of the sample
was finer than 38 µm. The sample also contained other than the main chemical components (Table 1),
i.e. 1.92% Pb, 0.26% K, 0.30% Mg, 0.111% Cd and 0.11% TiO₂, and the contents of S, Mn, P₂O₅, Na, Cr₂O₃, Ba, Cu, Ag, Ni, Co and As were less than 0.1%. On the other hand, the moisture content and loss on
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calcite, and it also contained quartz, clay minerals (kaolinite, trace illite), hemimorphite, hematite and
cerussite in lower amounts.

<table>
<thead>
<tr>
<th>Component</th>
<th>Value (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn</td>
<td>23.43</td>
</tr>
<tr>
<td>Fe</td>
<td>18.54</td>
</tr>
<tr>
<td>CaO</td>
<td>7.37</td>
</tr>
<tr>
<td>SiO₂</td>
<td>6.75</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>3.16</td>
</tr>
</tbody>
</table>

The leaching experiments were conducted in a magnetically stirred Pyrex reactor (0.6 dm³) equipped
with a reflux condenser inside a temperature controlled water bath. In a representative experiment, an
aqueous solution of sodium hydroxide (NaOH) at predetermined concentration (1, 2, 3 and 4 mole/dm³)
was transferred into the reactor, brought to the selected temperature (298, 323, 343 and 363 K) and then
the sample, whose amount was calculated according to the selected solid/liquid (S/L) ratio (0.075, 0.10
and 0.15 kg dry ore/dm³ solution), was added to the solution. The leaching experiment was continued
for the selected leaching times (1800, 3600, 7200 and 14400 s) under constant magnetic stirring. The studied concentrations and temperatures of NaOH solutions, S/L ratio values and leaching times were selected according to the preliminary tests and previous literature studies. Following leaching, the undissolved leach residues were separated by filtration, water-washed, dried, weighed and stored in sealed bottles for further analyses. Finally, leaching ratio values (%) of zinc and lead were calculated according to Eq. 1. Because it is very important to conduct the experiments under the same conditions in zinc-sodium hydroxide-water system, i.e. small changes in experimental conditions cause different results (McBride et al., 2003; Moezzi et al., 2011; Mukhopadhyay et al., 2015; Top and Cetinkaya, 2015), some of the randomly selected leaching experiments were repeated three times and the experimental errors were found to be always less than ±3.5%.

\[
Leaching \ ratio \ (%) = \frac{\text{(amount of Zn in the ore sample)} - \text{(amount of Zn in the leach residue)}}{\text{(amount of Zn in the ore sample)}} \times 100
\]  

(1)

3. Results and discussion

The dissolution of smithsonite, which is insoluble in water, in NaOH solution can be described by Eq. 2. As the most important parameter on leaching of Zn, in accordance with Eq. 2, the effect of NaOH concentration was investigated in the first place at values of 1, 2, 3 and 4 mole/dm³ (Table 2) for constant leaching time of 3600 s and a S/L ratio of 0.15 kg/dm³. In these experiments, the leaching temperature was also kept constant at 298 K, because lower leaching ratio values of Zn were obtained at higher temperatures in the studied NaOH concentration range. As an example, the leaching ratio values of Zn in 3 mole/dm³ NaOH solution at 323, 343 and 363 K were 62.7, 52.5 and 40.5%, respectively, probably due to the decreased stability of soluble zincate (Zn(OH)₂⁻) species at higher temperatures, which caused solid zinc oxide formation according to Eq. 3 (Debiemme-Chouvy and Vedel, 1991; Uekawa et al., 2004; Li et al., 2007; Moezzi et al., 2011).

\[
\text{ZnCO}_3 + 4\text{OH}^- \rightarrow \text{Zn(OH)}_2^{2-} + \text{CO}_3^{2-}
\]

(2)

\[
\text{Zn(OH)}_2^{2-} \rightarrow \text{ZnO} + 2\text{OH}^- + \text{H}_2\text{O}
\]

(3)

<table>
<thead>
<tr>
<th>NaOH concentration (mole/dm³)</th>
<th>Leaching Ratio (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Zn</td>
</tr>
<tr>
<td>1</td>
<td>3.8</td>
</tr>
<tr>
<td>2</td>
<td>46.0</td>
</tr>
<tr>
<td>3</td>
<td>70.1</td>
</tr>
<tr>
<td>4</td>
<td>70.9</td>
</tr>
</tbody>
</table>

Table 2. Effects of NaOH concentration on leaching of Zn and Pb

As shown in Table 2, the leaching ratio of Zn increased about eighteen times when the concentration of NaOH increased from 1 to 3 mole/dm³, and over 3 mole/dm³ NaOH, it was levelled off and maximum leaching ratio of 70.9% was obtained by leaching in 4 mole/dm³ NaOH solution at 298 K. The leaching ratio values of Zn obtained from various smithsonite bearing nonsulfide ores in the related literature were very different due to the differences in both mineralogy and chemical compositions of the ores used, and the experimental conditions applied during leaching. For example, Mujahed (1966) reported Zn leaching ratio value of 90.5% from a -700 µm Develi (Turkey) oxidized ore containing 14.8% Zn by leaching in 5.25 mole/dm³ NaOH solution (temperature: 363 K, S/L ratio: 0.15 kg/dm³, time: 14400 s). Frenay (1985) obtained Zn leaching ratio values between ~22-36% and ~66-100% from ~400 µm Belgian oxidized ores having Zn grades ranging between 15.6-44.4% by leaching in 1 and 6 mole/dm³ NaOH solutions, respectively (temperature: 293 K, S/L ratio: 0.05 kg/dm³, time: 3600 s). Zhao and Stanforth (2000) reported Zn leaching ratio values of about 12, 23, 40, 61 and 85% from a -150 µm oxide ore having 20.66% Zn by leaching in 1, 2, 3, 4 and 8 mole/dm³ NaOH solutions, respectively (temperature: 373 K, S/L ratio: 0.14 kg/dm³, time: 7200 s). Liu et al. (2011) reported Zn leaching ratio value of 85.14% from a -200 µm Mengzi (China) oxidized ore containing 17.3% Zn by leaching in 6 mole/dm³ NaOH solution (temperature: 363 K, S/L ratio: 1:5 wt.:vol., time: 7200 s). Zhang et al. (2013) reported Zn leaching ratio of 81% from a -150 µm Lanping (China) oxide ore with 10.15% Zn by leaching...
in a 20% NaOH solution solution (temperature: 373 K, S/L ratio: 1:80, time: 14400 s). Ghasemi and Azizi (2018) obtained Zn leaching ratio values of 18, 45, 54 and 67% from a -150 µm smithsonite containing Goshfil (Iran) mine tailings having ~4.75% Zn by leaching in 0.5, 1, 2 and 4 mole/dm³ NaOH solutions, respectively (temperature: 343 K, S/L ratio: 0.05 kg/dm³, time: 3600 s). On the other hand, the leaching ratio values of Pb (Table 2), which were generally lower than Zn leaching ratio values, continually increased, but the increase in leaching ratio value slowed down over 3 mole/dm³ NaOH concentration.

According to Table 2, the most efficient NaOH concentration was selected as 3 mole/dm³ considering the obtainment of higher leaching ratio value for Zn and lower concentration of used NaOH solution. Then, XRD pattern of the leach residue obtained after leaching of the sample in 3 mole/dm³ NaOH solution was determined (Fig. 1). The peaks belonging to smithsonite (JCPDS File No: 8-0449) in the XRD pattern of sample (Fig. 1) observed at 2θ = 25.01°, 32.49°, 38.61°, 42.75°, 46.57°, 51.29° and 53.63° disappeared in the pattern of leach residues obtained after leaching in both 3 and 4 mole/dm³ NaOH solutions, showing the dissolution of smithsonite in the ore sample. The XRD peaks of other main components, i.e. goethite and calcite, in the ore sample did not affect much as smithsonite peaks by NaOH leaching and stayed practically unchanged.

![XRD patterns](Fig. 1. XRD patterns of the sample and leach residues obtained after leaching at indicated NaOH concentrations (for inset, please see the text))

The dissolution of smithsonite in the ore sample was also followed with the help of FTIR and TG/DTA analyses. The FTIR spectra and TG/DTA curves of the sample and leach residue obtained after leaching in 3 mole/dm³ NaOH solution were given in Fig. 2. The characteristic absorption band for planar bending of carbonate group of smithsonite in the sample at 744 cm⁻¹ (Fig. 2a) disappeared and was not observed in the spectra of residue, indicating again the dissolution of smithsonite in the sample by NaOH leaching. For the leach residue, the band for out-of-plane bending of carbonate group at 871 cm⁻¹ in the spectra of sample observed at higher, i.e. 876 cm⁻¹, and the broad band for asymmetric stretching of carbonate group at 1428 cm⁻¹ in the spectra of sample observed at lower, i.e. 1424 cm⁻¹, wavenumbers. Both observations may indicate that smithsonite in the sample was dissolved and calcite in the sample remained as an undissolved phase (Weir and Lippincott, 1961). The main difference in DTA curves of the sample and the residue (Fig. 2b) was the disappearance of endothermic peak at 630.8 K, which was attributed to decomposition of smithsonite phase according to Eq. 4 (Zhang et al., 2013). As their presence were substantiated by XRD analyses, the endotherms at 566.7 and 1080.4 K were attributed the decomposition of goethite (Prasad et al., 2006) and calcite (Smykatz-Kloss et al., 2003) phases in the sample according to Eqs. 5 and 6, respectively. The presence of these peaks in DTA curves of both the sample and the residue showed that goethite and calcite phases were resistant to leaching in NaOH solution under the applied experimental conditions. On the other hand, the formation of double peak at the calcite decomposition region in DTA curve of the residue should be studied in future by detailed thermal studies. With the dissolution of smithsonite, the weight loss value at about, for
example, 750 K decreased greatly for the leach residue. As will be mentioned in the next paragraph, the very small intensity endothermic peak near 440 K indicated the presence of hydrated calcium zincate phase in the leach residue.

\[
\begin{align*}
ZnCO_3 &\rightarrow ZnO + CO_2 \uparrow \\
2aFeOOH &\rightarrow aFe_2O_3 + H_2O \uparrow \\
CaCO_3 &\rightarrow CaO + CO_2 \uparrow
\end{align*}
\]

7.44) contained approximately 0.5, 1.5 and 2 times stoichiometric NaOH for dissolving ZnCO\(_3\) (OH/Zn mole ratio: 1.86), 3 mole/dm\(^3\) (OH/Zn mole ratio: 4) and 4 mole/dm\(^3\) (OH/Zn mole ratio: 5.58) according to Eq. 2.

The XRD pattern of leach residue obtained after leaching in 4 mole/dm\(^3\) NaOH solution was also substantiated the presence of CaZn\(_2\)(OH)\(_6\)·2H\(_2\)O phase in this residue (Wang et al., 2008; Hao et al., 2014). One separate leaching experiment was also conducted in highly concentrated 8 mole/dm\(^3\) NaOH solution (OH/Zn mole ratio: 14.89, according to Eq. 2 stoichiometric OH/Zn mole ratio: 4) at 298 K to observe the change in Zn leaching ratio value and the presence of XRD peaks of hydrated calcium zincate in the pattern of leach residue. Both the increase in leaching ratio of Zn, which was 78.5% (58.2% for Pb), and the presence of very low intensity XRD peaks of hydrated calcium zincate phase in the leach residue obtained after leaching in 8 mole/dm\(^3\) NaOH solution (pattern was not given) indicate the decomposition (or nonformation) of hydrated calcium zincate phase in highly concentrated alkaline solutions, as also observed by Wang (1990).

As mentioned previously, the dissolution of smithsonite, which is insoluble in water, in NaOH solution can be described by Eq. 2. The 2 mole/dm\(^3\) NaOH solution (OH/Zn mole ratio: 3.72) approximately contained the stoichiometric amount of NaOH (OH/Zn mole ratio: 4) required to dissolve ZnCO\(_3\) in the used ore sample according to Eq. 2. Then, NaOH solutions of 1 mole/dm\(^3\) (OH/Zn mole ratio: 1.86), 3 mole/dm\(^3\) (OH/Zn mole ratio: 5.58) and 4 mole/dm\(^3\) (OH/Zn mole ratio: 7.44) contained approximately 0.5, 1.5 and 2 times stoichiometric NaOH for dissolving ZnCO\(_3\) according to Eq. 2. Besides, for 1 mole/dm\(^3\) NaOH solution, the general reaction given in Eq. 7 could also occur, where the reaction product zinc hydroxide is a water insoluble compound. Although the amount of NaOH in 1 mole/dm\(^3\) leaching solution was approximately equal to the half of NaOH required to dissolve Zn stoichiometrically in the ore sample, the very low leaching ratio value for Zn (3.8%) obtained after leaching in 1 mole/dm\(^3\) NaOH solution at 298 K may be the result of Eq. 7. In order to check this statement, the leach residue obtained after leaching in 1 mole/dm\(^3\) NaOH solution for leaching time of
3600 s and S/L ratio of 0.15 kg/dm³ at 298 K was dried under room conditions to prevent the possible thermal transformation of zinc hydroxide to zinc oxide under the applied drying conditions (Nicholas et al., 2012; Top and Cetinkaya, 2015), and then XRD pattern of this residue was obtained (Fig. 3, pattern 298 K). Fig. 3 (pattern 298 K) showed that part of smithsonite phase in the sample was converted into solid zinc hydroxide (JCPDS File No: 38-0385) in 1 mole/dm³ NaOH solution, probably following a dissolution and reprecipitation path (Usui, 2009; Giannakoudakis et al., 2015), resulting very low Zn leaching ratio value. The XRD peaks belonging to zinc hydroxide phase were not observed (Fig. 3, pattern 363 K) in the pattern of room temperature-dried leach residue obtained after leaching in 1 mole/dm³ NaOH solution at 363 K, for which again very low leaching value for Zn (1.4%) was obtained.

The effects of leaching time were studied at 3 mole/dm³ NaOH concentration for temperature and S/L ratio values of 298 K and 0.15 kg/dm³, respectively. The Table 3 showed that Zn dissolution was very fast and 70.8% of Zn in the sample was leached just in 1800 s. When the leaching time increased over 7200 s, the leaching ratio value of Zn started to decrease and reached 60.6% after 14400 s, mainly due to the increased formation of hydrated calcium zincate phase (Wang, 1990) in the solution (Fig. 4). On the other hand, the leaching ratio values of Pb, again being lower than the leaching ratio values of Zn, practically stayed unchanged in the studied range of leaching times.

The effects of solid/liquid ratio were studied at 2 mole/dm³ NaOH concentration for leaching temperature and leaching time of 298 K and 3600 s (Table 4), respectively, where an average Zn leaching ratio value (46.0%) was obtained and by this way higher and lower leaching ratio values could easily be seen. The possible formation of different zinc containing phases as observed before also led the authors to choose approximately stoichiometric 2 mole/dm³ NaOH solution concentration. When the S/L ratio was decreased keeping OH/Zn mole ratio value constant at about 3.72, the leaching ratio values of Zn were also reduced due to the decrease in concentration of NaOH solution (for S/L ratio values of 0.075, 0.10 and 0.15 kg/dm³, the approximate solution concentrations were 1, 1.35 and 2 mole/dm³ NaOH, respectively). When the S/L ratio was decreased keeping NaOH solution concentration constant at 2 mole/dm³ NaOH, the leaching ratio values of Zn increased due to the increase in OH/Zn mole ratio values in the solution (for S/L ratio values of 0.075, 0.10 and 0.15 kg/dm³, the corresponding OH/Zn mole ratio values were 7.44, 5.58 and 3.72, respectively), as expected. The
leaching ratio values of Pb were affected less by the changes in the S/L ratio values when compared to the corresponding leaching ratio values of zinc.

Table 3. Effects of leaching time on leaching of Zn and Pb

<table>
<thead>
<tr>
<th>Leaching time (s)</th>
<th>Leaching Ratio (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Zn</td>
</tr>
<tr>
<td>1800</td>
<td>70.8</td>
</tr>
<tr>
<td>3600</td>
<td>70.1</td>
</tr>
<tr>
<td>7200</td>
<td>70.7</td>
</tr>
<tr>
<td>14400</td>
<td>60.6</td>
</tr>
</tbody>
</table>

Table 4. Effects of solid/liquid ratio on leaching of Zn and Pb

<table>
<thead>
<tr>
<th>Solid/Liquid ratio (kg/dm³)</th>
<th>Leaching Ratio (%)</th>
<th>At constant NaOH concentration</th>
<th>At constant OH/Zn mole ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Zn</td>
<td>Pb</td>
<td>Zn</td>
</tr>
<tr>
<td>0.075</td>
<td>71.7</td>
<td>48.7</td>
<td>31.0</td>
</tr>
<tr>
<td>0.100</td>
<td>68.4</td>
<td>44.8</td>
<td>40.1</td>
</tr>
<tr>
<td>0.150</td>
<td>46.0</td>
<td>37.5</td>
<td>46.0</td>
</tr>
</tbody>
</table>

4. Conclusions

The leaching behaviour of Zn from a Turkish smithsonite ore sample containing 23.43% Zn was investigated in sodium hydroxide solutions at different temperatures, leaching times and solid/liquid ratio values using XRD and chemical analyses. Between the studied 1-4 mole/dm³ NaOH concentration range, most of the leaching experiments were conducted at 298 K, because higher Zn leaching ratio values were obtained at 298 K due to the lowered stability of soluble zincate species at higher temperatures, i.e. 363 K. The increase in NaOH concentration from 1 to 3 mole/dm³ greatly increased the leaching ratio of Zn (from 3.8 to 70.1%) at 298 K, and for 4 mole/dm³ NaOH concentration, the leaching ratio of Zn (70.9%) levelled off because of the formation of hydrated calcium zincate in the leaching solution. When the leaching process was conducted at 1 mole/dm³ NaOH concentration, very low leaching ratio values of Zn were obtained by the formation of solid zinc hydroxide at 298 K and solid zinc oxide at 363 K. With the increase of leaching time at 3 mole/dm³ NaOH concentration, leaching ratio value of Zn decreased because of the increase in the amount of formed solid hydrated calcium zincate phase in the solution. The solid/liquid ratio parameter affected significantly leaching of zinc. It was observed that when solid/liquid ratio was decreased, at constant OH/Zn mole ratio the leaching ratio values of Zn decreased and at constant NaOH concentration the leaching ratio values of Zn increased. For some of the leaching experiments, the leaching ratio values of Pb were also determined and it was observed that the leaching ratio values of Pb were generally lower than the corresponding values for Zn. As conclusion, due to the hydrolysis of zinc and the possibility of formation of different zinc compounds during alkaline leaching of nonsulfide zinc ores having different gangue components, each parameter affecting leaching must be carefully tested in detail.
Acknowledgments

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