Abstract

The process tailings of base metal industries contain considerable amounts of acid generating pyrite and sulfides of other minerals. The recovery of sulfide minerals from the tailings not only will have environmental advantage but will also result in conservation of mineral wealth. For any such recovery activity, it is essential to characterize the tailings for physical, chemical and mineralogical characteristics. In the present study, typical lead-zinc tailings sample from the operating plant at Rampura-Agucha mines of Hindustan Zinc Limited was collected and characterized for size distribution, size by size chemical composition and mineral phase analysis through X-ray diffraction method. The size-by-size metal contents indicated increasing levels of silver and pyrite with decreasing particle size and the lead and zinc bearing minerals are segregated at relatively coarser size fractions. Microscopic studies of the coarser fractions have indicated that the lead and zinc minerals are in unliberated form. The XRD study has indicated that quartz is the major mineral phase followed by pyrite, sphalerite, galena and silver. The overall tailings sample was found to contain 19.41% of pyrite, 2.02% of zinc, 0.69% lead, and 26.94 ppm of silver contents.

INTRODUCTION

Lead and zinc are important non-ferrous minerals. In India major lead-zinc deposits are located in Rajasthan state. Among the reserves in Rajasthan State, the deposit at Rampura-Agucha in Bhilwara district is a promising one. The deposit has an ore reserve of 61.1 million tons, with an average grade of 1.50% of lead and 4.5% of zinc (Bull, 1981). In general lead-zinc ores are concentrated adopting grinding followed by sequential flotation technique in which lead minerals are floated in the first stage further followed by flotation of zinc minerals (Gokhale et al., 1983). During flotation, pyrite is rejected into the tailings by adding suitable depressants like sodium sulfide and cyanides. In this process some portions of sulphide minerals of lead, zinc and precious metals like silver and cadmium are also lost into the tailings as metal losses. The sulphides of these minerals entering the tailings generate acids, which leach away the heavy metals and further contaminate ground water. It is estimated that for every 141,374 tons of zinc and 45,655 tons of the lead metals recovery, the tailings generated would be around 6.935 million tons. Having realized the importance of mineral conservation and environmental point of view, a test work has been conducted to characterize the lead-zinc process plant tailings of Rampura-Agucha Mines to obtain information on mineralogy, size distribution and size-by-size metal composition.

Characteristics like particle size distribution, size-by-size metal composition and mineralogical data are a reliable guide that will give some idea on the performance of different mineral processing
techniques. Though the report on the characterization studies of a lead-zinc ore is available (Okafor et.al., 1997; Udaya Bhaskar et.al., 2004), till date information on the mineralogy, particle size distribution and size-by-size metal composition of lead-zinc process plant tailings of Rampura-Agucha mines is limited. Thus, the present study was performed to characterize the lead-zinc tailings sample of Rampura-Agucha Mines prior to its beneficiation.

SAMPLE COLLECTION

The lead-zinc process plant tailing was collected from the operating plant at Rampura-Agucha Mines of Hindustan Zinc Limited. The sample collection point in the operating plant is shown in Figure 1. During the flotation process, due to fluctuations in the feed composition, the reagents dosage may act either as insufficient or excessive in concentration, which results in losses of valuable lead, zinc and silver bearing minerals along pyrite to the tailings. Thus, the flotation tailing slurry sample of 1 tonne solids was collected. The slurry collected was filtered and dried in an electric oven. The dried solids were thoroughly mixed and representative samples were prepared using standard sampling methods.

RESULTS AND DISCUSSIONS

The lead and zinc tailings sample from Rampura – Agucha mines of Rajasthan state was studied for size by size mineralogical distribution and mineral phase identification using the following techniques:

1. Particle size analysis by Fritz wet sieve analysis method.
2. X-Ray diffraction analysis (XRD) by Bruker X-Ray diffractometer.

Particle Size Analyses

Representative sample around 100gms is collected with the help of riffle samplers from randomly selected bags. The size analysis of the sample was carried out using Fritz wet sieve shaker under standard set of sieves. The size distribution and size-by-size metal composition of the feed sample is presented in Table 1. To know the reproducibility and accuracy of sampling procedure and size analysis, a set of two more samples are drawn and size analysis is carried as mentioned earlier. The data indicate that sampling procedure and size analysis is reproducible.
### Table 1: Size Distribution and Size-By-Size Metal Composition of Feed Sample

<table>
<thead>
<tr>
<th>Size Microns</th>
<th>wt %</th>
<th>Chemical analysis (%)</th>
<th>Cum. metal (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Lead (wt %)</td>
<td>Zinc (wt %)</td>
</tr>
<tr>
<td>212</td>
<td>6.7</td>
<td>0.54</td>
<td>1.75</td>
</tr>
<tr>
<td>150</td>
<td>17.7</td>
<td>0.66</td>
<td>1.99</td>
</tr>
<tr>
<td>106</td>
<td>19.15</td>
<td>0.75</td>
<td>2.35</td>
</tr>
<tr>
<td>75</td>
<td>18.25</td>
<td>0.72</td>
<td>2.36</td>
</tr>
<tr>
<td>45</td>
<td>18.3</td>
<td>0.60</td>
<td>2.10</td>
</tr>
<tr>
<td>38</td>
<td>2.95</td>
<td>0.48</td>
<td>1.60</td>
</tr>
<tr>
<td>25</td>
<td>5</td>
<td>0.44</td>
<td>1.40</td>
</tr>
<tr>
<td>-25</td>
<td>11.95</td>
<td>0.96</td>
<td>1.39</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Lead (wt %)</td>
<td>Zinc (wt %)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.54</td>
<td>1.75</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.63</td>
<td>1.92</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.68</td>
<td>2.11</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.69</td>
<td>2.18</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.67</td>
<td>2.17</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.66</td>
<td>2.15</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.65</td>
<td>2.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.69</td>
<td>2.02</td>
</tr>
</tbody>
</table>

It can be observed from the table that the weight percent report of the material above 45µm is 80.1% with a corresponding lead and zinc content of 0.67% and 2.17% respectively. The table also indicates that all the fractions above 45µm contain lead and zinc content between 0.54% to 0.67% and 1.75% to 2.17% respectively. This indicates that the lead and zinc minerals are segregated at coarser fractions above 45 microns. It can be observed from the table that the size fraction below 25 microns contains maximum lead around 0.96%. It can be also noticed that the pyrite which is a major sulfur bearing mineral in the ore is found be more at fines below 45 microns. The size fractions below 45 microns contain pyrite content between 15.47% to 19.41%. However, silver is found to increase with decreasing particle size indicating the presence of liberated silver mineral fractions at finer sizes. The overall feed sample contained 0.69% of lead, 2.02% of Zinc, and 19.41% of pyrite and 26.94 ppm of silver contents. The detailed information of the weight percent report at each sieve size is presented in Figure.2 and size by size metal composition of the feed sample is presented in Figure.3
Silver (ppm)
Pyrite (%)
Zinc (%)
Lead (%)

Size by Size Lead
The size-by-size lead distribution at each particle size and size wise cumulative data on the feed sample is presented in Figure 4. The figure indicates a general trend of increase in lead content as the particle size increases from 25 microns to 106 microns and further there is decrease in lead content as particle size increases above 106 microns. The cumulative data indicates that the fractions above 45 microns contain lead between 0.67 to 0.69 % indicating segregation of lead at coarser fractions above 45 microns.

Size by Size Zinc
Figure 5 represent the size-by-size zinc content and its cumulative data on the feed sample. It can be observed that figure that there is an increase in zinc content with increase in size between 25 microns to 106 microns. However with further increase in size above 106 microns and decrease in size below 45 microns the zinc content is found decreasing. A maximum zinc content of 2.36% is reported at 75
microns. The cumulative data indicates that the fraction above 38 microns and below 150 microns contains zinc between 2.11 to 2.18 indicating segregation of zinc within this size range.

![Graph showing size-by-size zinc content and size-wise cumulative data on the feed sample.]

**Fig. 5: Size by Size Zinc Content and Size Wise Cumulative Data on the Feed Sample**

**Size by Size Pyrite**

The size-by-size pyrite content and size wise cumulative data on the feed sample is presented in Figure 6. It indicates that the pyrite content gradually increases with decrease in particle size from 150 microns to 25 microns. A maximum pyrite content of 44.94% is reported at 25 microns fractions. It can be noticed that the pyrite, which is major sulfur bearing mineral in the ore, is found to be more at fines below 45 microns. A mere concentration of the feed material in an enhanced gravity concentrator would result in recovery of pyrite, which are segregated at much finer sizes.

![Graph showing size-by-size pyrite content and size-wise cumulative data on the feed sample.]

**Fig. 6: Size by Size Pyrite Content and Size Wise Cumulative Data on the Feed Sample**

**Size by Size Silver**

Figure 7 represents the distribution of silver content (in ppm) at different size fraction and size wise cumulative data on the feed sample. It can be observed from the figure that the silver content (ppm) increases with decrease in size below 150 microns. A maximum silver content is reported at 25
microns indicating segregation of silver at fine sizes. It can be noticed from the cumulative data that as the particle size decreases there is increase in the silver content. For instance, the silver content at 150 microns is 17.72 % and as the particle size decreases below 150 microns, a general trend of increase in silver content is observed. The concentration of feed material in any of the enhanced gravity technique would recover the silver, which is segregated at much finer fractions.

![Size by Size Silver Content and Size Wise Cumulative Data on the Feed Sample](image)

**Fig. 7: Size by Size Silver Content and Size Wise Cumulative Data on the Feed Sample**

**Mineralogical Studies**

The X-ray diffraction studies were carried with the help of Bruker X-ray diffractometer (Model No.D-8) using filtered Cu-Kα radiation operated at a voltage of 40KV and 40mA current. The powdered feed sample was packed into a sample container and the surface is exposed to X-rays. The XRD patterns were recorded from 10.0 to 90.0 degrees with a step size of 0.020 degrees per second. The data obtained on the d-values and the corresponding intensities were compared with the standard powder diffraction files (Mineral powder diffraction search file, data book, 1980) published by Joint committee on powder diffraction standards.

The XRD pattern obtained is presented in Figure 8. Further, the characteristic d-values used for confirming the presence of individual minerals are presented in Table 2. The table confirms the presence of the following minerals in the feed sample:

- Galena (Pbs)
- Sphalerite (ZnS)
- Pyrite (FeS₂)
- Silver (Ag)
- Quartz

<table>
<thead>
<tr>
<th>Minerals</th>
<th>XRD (Ref) File No</th>
<th>XRD Peaks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Galena</td>
<td>5-592</td>
<td>2.97 (100)</td>
</tr>
<tr>
<td>Sphalerite</td>
<td>5-566</td>
<td>3.12 (100)</td>
</tr>
<tr>
<td>Pyrite</td>
<td>6-710</td>
<td>1.63 (100)</td>
</tr>
<tr>
<td>Silver</td>
<td>4-783</td>
<td>2.36 (100)</td>
</tr>
<tr>
<td>Quartz</td>
<td>5-490</td>
<td>3.34 (100)</td>
</tr>
</tbody>
</table>

Table 2: the Minerals Identified in the XRD Analysis of the Feed Sample

CONCLUSION

The characterization of lead-zinc tailings of Rampura-Agucha Mines was carried out to obtain information on the mineralogy, particle size distribution and size-by-size metal composition prior to its beneficiation. The studies have indicated that about 93 % of the material passes through 212 microns and 19.9 % of the material passes through 45μm. The size-by-size metal contents indicated increasing levels of silver and pyrite with decreasing particle size and the lead and zinc bearing minerals are segregated at relatively coarser size fractions above 45 microns. The XRD study has indicated that zinc, lead, iron-bearing minerals present in the sample are sphalerite, galena and pyrite form respectively. Silver is present in native form. Quartz is the siliceous gangue mineral present. The overall tailings sample was found to contain 19.41 % of pyrite, 2.02 % of zinc, 0.69 % lead, and 26.94 ppm of silver contents. From the feed characterization studies it can be concluded that concentration of the tailings using classification followed by enhanced gravity techniques would result in recovery of pyrite and silver values. The studies also indicate that the recovery of lead and zinc from the tailings may possibly be achieved only after grinding for further liberation.

REFERENCES