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Characterization of Rampura-Agucha Lead-Zinc Ore using Electron Probe Micro Analyzer (EPMA) in View of Beneficiation

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Abstract

Beneficiation test work on a Pb-Zn sample from Rampura-Agucha mines was taken up with a view to (i) explore the possibility of reducing the Zn losses in tailings from the present level of 2.4% to as low as possible, (ii) assess the reason for low grade Zn and Pb concentrates presently produced in the plant and (iii) evaluate the reason for very low Pb recovery values. This paper highlights the significant role played by Electron Probe Micro Analyzer (EPMA) in understanding the textural and compositional features of various minerals prior to the beneficiation test work and also in suggesting necessary alterations in the process parameters whilst mineral processing studies are underway.

INTRODUCTION

A lead-zinc sample (designated as North ore) from Rampura-Agucha mines, Bhilwara district, Rajasthan, was taken up in the Ore Dressing Division of the Indian Bureau of Mines at Nagpur for beneficiation test work with a view to (i) explore the possibility of reducing the Zn losses in tailings from the present level of 2.4% to as low as possible, (ii) assess the reason for low grade Zn and Pb concentrates presently produced in the plant and (iii) evaluate the reason for very low Pb recovery. This North ore is said to constitute up to 30% of the total Pb-Zn reserves in Rampura-Agucha mines. In this paper, the significant role played by Electron Probe Micro Analyzer (EPMA) in assessing the above objectives by the way of understanding textural (geometrical) and compositional features of various minerals in the sample prior to the beneficiation test work and also in suggesting necessary alterations in the process parameters whilst mineral processing studies are underway are presented and highlighted.

SAMPLE CHARACTERIZATION

The as received sample consisted of fines with substantial quantity of hard and compact lumps ranging from 50-75 mm in size. A few lumps were in the size of 100-150 mm. The mineralogy (by the combined microscopic and XRD studies) and chemical analysis of the representative portion of the as received sample is given in **Table 1.**

Sphalerite, galena, pyrite and pyrrhotite constitute main sulphide minerals whereas quartz and graphite are the major gangue minerals in the sample **(Table 1)**. All the Pb and Zn values are attributed to the presence of respective sulphide minerals viz., galena and sphalerite only.

ELECTRON PROBE MICRO ANALYZER (EPMA) STUDIES

Extensive flotation test work was carried out on this sample to establish and optimize various parameters for flotation in order to reduce the tailing losses to minimum possible extent and improvement of the grade and recovery. The tailing losses in respect of both lead and zinc were found to be quite high in spite of adequate dosages of collector and stages of flotation. Therefore, advanced physico-chemical characterization studies of the original sample and various ore dressing products were carried out by an Electron Probe Micro Analyzer (EPMA) of CAMECA SX100 model, recently installed in our laboratory, to find out the reason.

Table 1: Approximate Mineral Distribution and Chemical Analysis of as Received Sample

Mineral	Apprx %	Element/oxide	Assay %
Sulphides		Pb	2.81
Sphalerite	25	Zn	14.64
Pyrite	6-7	Cu	Traces
Pyrrhotite	4	S (Total)	12.30
Galena	2-3	Fe	8.98
Marcasite	<1	SiO_2	34.79
LÖllingite	Traces	Al_2O_3	9.59
Arsenopyrite	Traces	CaO	0.92
Tetrahedrite	Traces	MgO	8.43
Carbonates		Gr. C	4.11
Calcite	1-2		
Smithsonite	Traces	Trace Elements	ppm
Cerussite	Traces	Ag	93
Dolomite	Traces	Mn	817
Oxides/Hydroxides		Ni	145
Goethite/limonite	<1	Co	64
Rutile	Traces	As	495
Magnetite	Traces	Cd	465
Silicates & Other gangue			
Quartz	25-30		
Mica: Muscovite Biotite	 8-10 5-7		
Graphite	4-5		
Clay (kaolinite)	3-4		
Feldspar	2-3		
Amphibole (mostly tremolite)	2-3		
Sillimanite, Garnet	<1		
Chlorite, Pyroxene, Tourmaline, Zircon	Traces		

EPMA technique may be defined as a non-destructive analytical technique with a capacity to reveal rapidly and accurately elemental distribution (qualitative) and concentration (quantitative) on a micrometer (1-2 micron) scale with a sensitivity at level of ppm. This instrument also has high imaging facility with high magnifications of up to more than 50,000 times.

Low Grade Zinc Concentrate & Zinc Losses in Tailings

EPMA studies reveal that there is a variable Fe replacement in the individual sphalerite grain lattice structure ranging from 6.37-10.81 wt% (**Table 2**). Correspondingly, the maximum Zn content in the analyzed sphalerites do not exceed 59.61 wt%. Hence, the entire sphalerite in the sample is interpreted to be marmatite. This information was utilized to modify the flotation parameters (especially pH) to obtain two types of zinc concentrates (I and II). and reduction of the Zn tailing losses from 2.4% to

1.78% .A good grade zinc (lower degree substitution of zinc by iron) floats at the initial float (zinc concentrate I) and thereafter inferior grade zinc (higher degree substitution of zinc by iron) floats along with pyritic and siliceous/graphitic gangue (zinc concentrate II).

Table 2: Chemical Composition (Wt%) of Individual Sphalerite Grains in Various Products of the Sample

Elements	1	2	3
S	33.25	33.83	33.70
Mn	0.69	0.22	0.15
Fe	6.37	10.81	10.26
Co	0.00	0.00	0.01
Ni	0.00	0.00	0.00
Cu	0.00	0.00	0.00
Zn	59.61	55.36	56.56
As	0.00	0.00	0.03
Ag	0.01	0.00	0.03
Cd	0.20	0.19	0.10
Sn	0.00	0.00	0.00
Sb	0.00	0.00	0.00
Au	0.00	0.00	0.01
Hg	0.00	0.03	0.06
Pb	0.16	0.11	0.00
Bi	0.09	0.00	0.02
Total	100.40	100.55	101.03

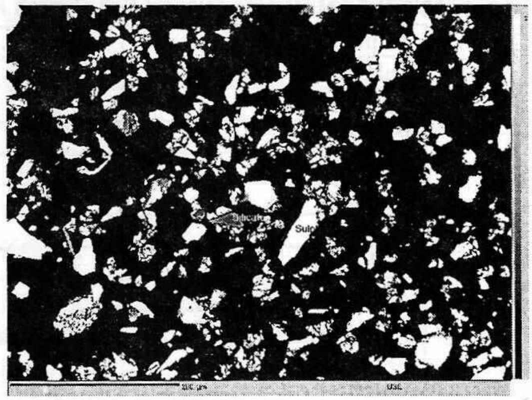


Fig. 1: (Zinc Conc I) Back Scattered Electron (BSE) Image by EPMA of Zinc Concentrate Showing the Overall Free Degree of Liberation of Sulphide Grains (White). Note That the Silicate Gangue (Gray) in the Product is in Minor Proportion

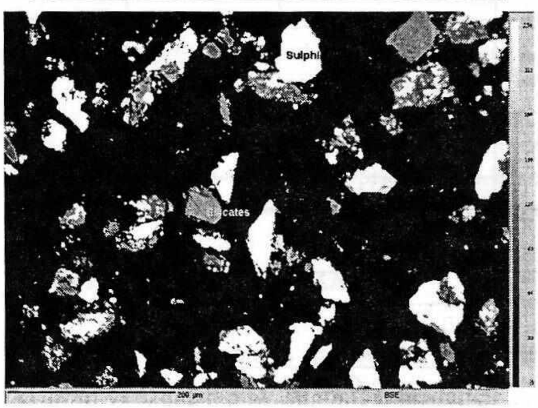


Fig. 2: (Zn Conc II) Back Scattered Electron (BSE) Image Showing the Intimate Interlocking/ Association of Most of the Sulphide Grains (White) with the Silicate Gangue (Gray). Note the Increase in Silicate Content Compared to That in Fig. 1

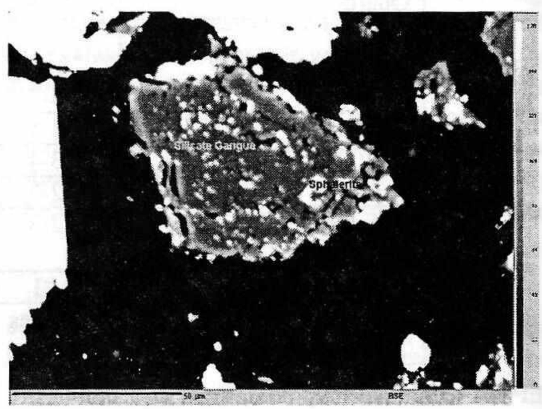


Fig. 3: (Zinc Conc II) Back Scattered Electron (BSE) Image by EPMA of Zinc Concentrate Showing That Extremely Fine Inclusions (5 to 15 Microns) of Sphalerite (White) are Present Within Silicate Grains (Gray)

The zinc concentrate I is characterized by low silicate gangue (**Fig.1**) and also less Fe substitution in sphalerite. It assay 54.15% Zn, 0.87% Pb, 8.48% Fe, 2.66% SiO_2 and 1.35 % Gr. C with 59.6% zinc distribution (wt% yield 16.5) and meets the requirement for metallurgy. Apart from higher substitution of zinc by iron in sphalerite mineral, the zinc concentrate II also contains higher siliceous and graphitic impurities, iron sulphides, galena and these occur as finely interlocked phases (**Fig. 2**) or intimately associated with sphalerite (**Fig. 3**) and thereby diluting the grade. The zinc concentrate II assayed 46.43% Zn, 3.43% Pb, 11.25% Fe, 13.85% SiO_2 and 4.13 % Gr. C with 17.6% zinc distribution (wt% yield 5.7) and is of inferior grade.

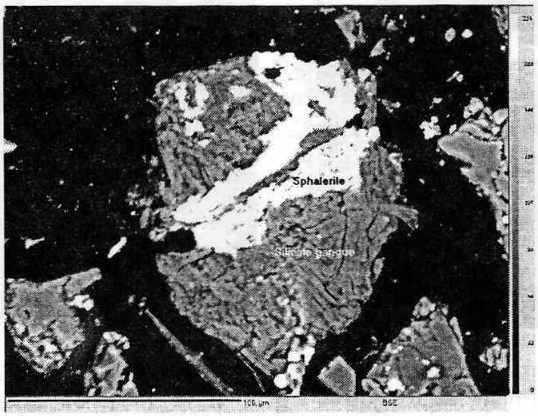
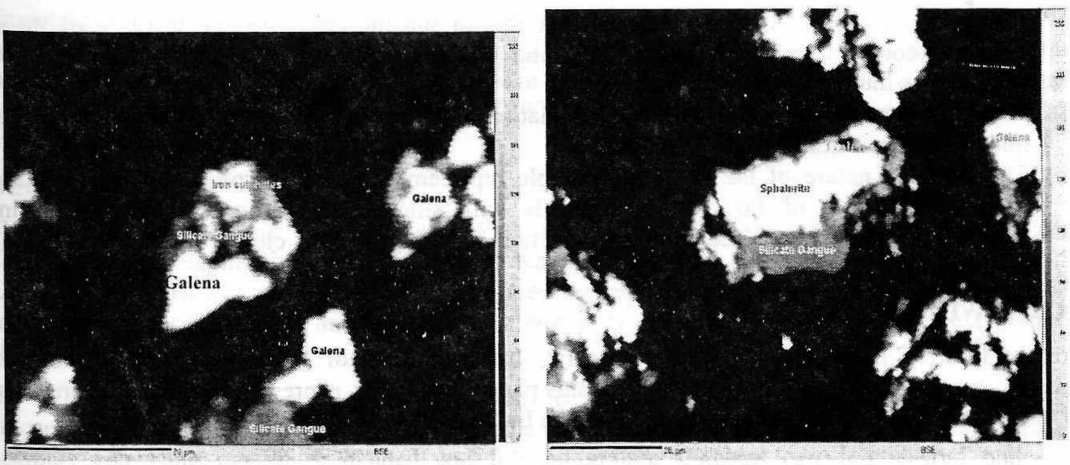


Fig. 4: (Zinc Tails) Back Scattered Electron (BSE) Image Showing the Distribution of Sphalerite (White) (I) Along the Cleavage/Fracture Planes of the Silicate Gangue Minerals (Gray) and (II) Also as Fine to Extremely Fine Inclusions in the Silicate Gangue

The composite of zinc concentrate I and II assay 52.17% Zn and 1.64% Pb with overall zinc distribution of 77.2% (wt% yield 22.2). The lower alkaline pH 8.4-8.5 in zinc flotation could help to float higher degree of substituted zinc by iron in sphalerite resulting in reduction of tailing losses to 1.78%. However, clean zinc concentrates of higher grade and recovery could not be achieved due to the complex nature of the sample. EPMA examination of rougher tailings revealed that the distribution of sphalerite along with cleavage/fracture planes of the silicate gangue minerals and also as fine inclusions in the latter (**Fig. 4**) is responsible for the loss of zinc values.

Low Grade Lead Concentrate & Lead Losses in Tailings

The best lead concentrate obtained during the process assayed 41.68% Pb, 15.61% Zn, 7.11% Fe, 10.47% SiO_2 and 4.02% Gr. C with 41.2% Pb distribution (wt% yield 2.9). Further improvement of its



A B

Fig. 5: (A & B) (Pb Conc) Back Scattered Electron (BSE) Image Showing Fine Interlocking of Galena, Sphalerite and Iron Sulphides (White) with the Silicates at About 15-20 Microns Size

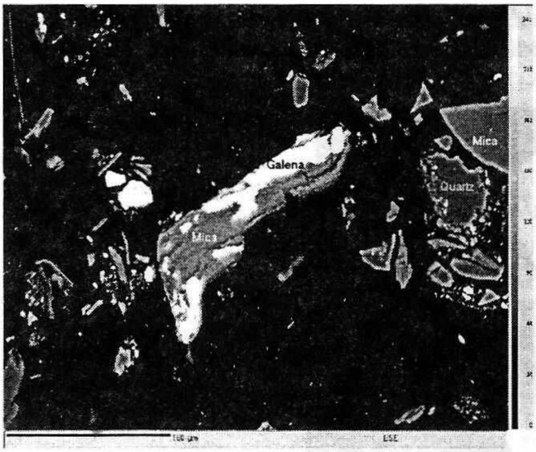


Fig. 6: (Rougher Tails) Back Scattered Electron (BSE) Image Showing Intimate Association of Galena (White) As Inclusions in Mica and Other Silicate Gangue Minerals (Gray)

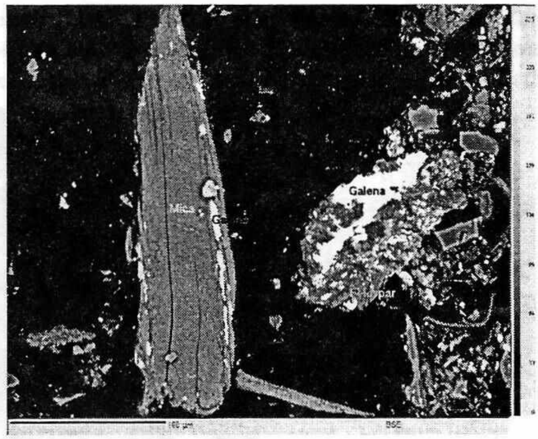


Fig. 7: (Rougher Tails) Back Scattered Electron (BSE) Image Showing Distribution of Galena (White) Along the Cleavage Planes of Mica (Gray) and As Patchy and Disseminated Inclusions in Other Silicates Viz., Feldspar (Dark Gray)

grade could not be possible because of extremely fine interlocking and/ or inclusions of galena with silicates, sphalerite and iron sulphide even at about 15-20 microns size (**Fig. 5 A & B**). Extremely fine interlocking and/ or inclusions of galena with silicates is the main factor that contributed to the lead loss in tailings (**Figs 6 & 7**).

Other Trace Element Substitutions

EPMA studies also reveal that (i) Mn (up to 0.72%), Cd (up to 0.36%), Hg (up to 0.26%) and also Ag (up to 0.03%) are the important trace elements substituting in the sphalerite lattice (ii) lÖllingite (FeAsS) is the main carrier of As in the sample and (iii) pyrrhotite in the sample is not strictly "ideal' and shows slight transition to pyrite

CONCLUSIONS

(i) The lower grade of the zinc concentrate is due to substitution of (a) Fe in place of Zn in sphalerite composition in sizeable amounts and (b) fine association of silicate/graphite and iron sulphides as interlocked phases.

(ii) Higher lead losses are due to intimate association of galena as fine inclusions (<25 microns) in mica and other silicates such as feldspar.

(iii) The complex nature of the ore (e.g. variable replacement of Fe in sphalerite structure) and textural behaviour of Pb and Zn minerals with silicates and other sulphides (e.g. fine inclusions/interlockings) preclude the production of high grade and clean concentrate with high recoveries.

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