Acid Leaching Studies on Sphalerite Ore of Kohistan Hazara for the Recovery and Separation of Lead and Zinc

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Summary: Investigations were carried out for the recovery and separation of Lead and Zinc from the Sphalerite Ore of Kohistan-Hazara by acid leaching. Parameters like acid concentration, temperature and time of leaching were optimized for the maximum recovery of Lead and Zinc from the Ore. It was found that a recovery of 90% of Zinc and 70% of Lead could be obtained if the observed optimum conditions for the parameters studied are followed:

Introduction

The Lead Zinc mineralization of galenosphalerite from Besham area of Kohistan situated at a distance of 150 Km from Abbottabad was first reported by Ashraf *et al.*, [1]. Geologically the area is complex having igneous and metamorphic rocks. The igneous rocks consist of granite while the metamorphic rocks include schists and marbles. Two major areas have been identified for Lead-Zinc mineralisation in Kohistan area. These are Lahor and Pazang situated on the right and left bank of Indus river containing roughly 5 million tones of mineable grade sphalerite ore. Lahor granite is brownish in colour while graphite granite exist in Pazang area. The sphalerite varies from 5.7% but in some samples if goes up to 10% of the rock. Other associated metallic minerals are pyrite, Chalcopyrite and pyrrlhotite [2]. Precious metals are generally associated with such mineralisation and hence are of considerable economic significance. At Pazang the mineralisation is associated with a very distinctive rock type which weathers to give a nobby and pitted appearance. The mineralization at Lahor area is associated with the Carbonate rocks, barite and layered silica rich rocks [3].

Extensive studies have been made in the past for the beneficiation of sphalerite by bacterial leaching process [4-7]. Other Leaching techniques were also applied for the exploitation of sulfide minerals [8-10]. Studies were also made for the flotation of sphalerite from galena using sodium carboxymethylcellulose as a depressant [11]. The kinetics of pressure leaching of sphalerite has been reported by some workers [12]. The Geology, petrography and mesh size liberation studies undertaken by some authors indicate that the material of 100-150 mesh size is suitable for the upgradation of sphalerite ore of Kohistan Hazara [13]. The present studies are aimed at the separation as well as recovery of Lead and Zinc by acid leaching of sphalerite ore of Kohistan Hazara for their utilization in Paints and Pigments making.

Results and Discussion

Chemical composition

Samples collected from 15 different points of the deposits in Besham-Kohistan area were chemically analyzed by conventional as well as instrumental techniques [14]. The composition of major elements and those of associated trace metals are shown in Table-1 and 2 respectively along with their arithmetic mean and standard deviations. The average content of zinc as its oxide is 1.77% and Lead is 9.79% as PbO. he minor elements detected have the average content of 0.18% Cu, 0.01% Ag and 0.9% Mn with traces of Co, Ni, Cr and Au.

Effect of hydrochloric acid concentration on the recovery of Pb and Zn

A representative sample from the bulk (15) samples having 150 mesh size was treated with hydrochloric acid. 10 gm powdered ore was digested with various concentrations of the acid and its was observed that maximum recovery of Lead and Zinc occur at a concentration of 50 mls of 2.5 M HCl (Table-3). The results obtained were encouraging as 90% recovery of Zinc and 70% of Lead occur at 2.5 M concentration of the hydrochloric acid.

Table-1: Chemical composition of the Sphalerite Ore (Major elements) expressed in percent.

Sample No.	Loss on Ign.	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	Na_2O	K_2O	PbO	ZnO	MnO	Total
1.	9.20	38.54	8.09	12.11	8.48	2.82	0.12	0.08	16.53	2.15	1.57	99.68
2.	2.04	37.69	8.68	20.84	2.09	1.42	0.50	0.12	18.93	3.84	3.54	99.69
3.	10.27	41.91	11.40	15.54	4.19	0.58	0.31	0.11	13.26	0.68	0.79	99.04
4.	4.37	22.44	14.40	27.28	6.64	1.92	0.08	0.02	13.57	6.58	1.79	98.75
5.	6.19	39.21	5.62	17.47	8.74	2.53	0.05	0.01	18.42	0.18	1.21	99.63
6.	11.95	30.08	7.09	10.79	13.39	0.64	0.15	0.10	24.47	0.08	1.01	98.96
7.	2.19	56.26	11.36	13.97	1.94	0.38	0.63	0.25	10.58	1.47	0.13	99.56
8.	4.79	60.22	9.90	91.07	1.95	0.51	0.53	0.18	9.86	0.93	0.13	99.97
9.	8.19	60.23	9.90	16.07	1.95	0.91	0.73	0.35	0.63	Nil	0.09	99.05
10.	7.86	33.47	15.49	26.91	2.76	1.82	0.42	0.13	6.64	3.02	1.40	99.92
11.	3.39	80.71	2.85	6.14	1.95	0.84	0.21	0.03	0.95	0.78	0.09	100.21
12.	5.75	60.33	9.95	18.40	0.83	0.71	0.48	0.21	1.41	1.42	0.19	99.66
13.	5.43	47.67	5.61	34.17	0.16	1.42	0.32	0.10	1.92	1.59	1.19	99.58
14.	5.550	55.84	11.36	13.26	0.83	0.91	0.72	0.28	0.98	1.72	0.19	99.64
15.	14.50	60.25	7.12	4.35	2.79	0.58	0.63	0.31	8.80	0.38	0.11	99.82
Mean	7.44	49.32	9.25	21.89	3.91	1.20	0.39	0.15	9.80	1.77	0.89	99.54
Sted.Dev.	±3.83	±15.35	±3.36	±20.72	±3.74	±0.76	±0.23	±0.10	±7.69	±1.74	±0.95	±0.41

Table-2: Composition of the Sphalerite (Trace elements) expressed in percent.

Sample No.	Co	Cu	Ni	Cr	Au	Ag
1.	N.D.	0.48	N.D.	N.D.	N.D.	0.05
2.	"	0.09	"	"	"	0.01
3.	"	0.03	"	"	"	N.D.
4.	"	0.23	"	"	"	0.07
5.	"	0.23	"	"	"	0.03
6.	"	0.12	"	"	"	N.D.
7.	"	0.23	"	"	"	0.01
8.	"	0.66	"	"	"	N.D.
9.	"	0.14	"	"	"	"
10.	"	N.D	"	"	"	"
11.	"	"	"	"	"	"
12.	"	0.35	"	"	"	"
13.	"	0.21	"	"	"	"
14.	"	0.35	"	"	"	"
15.	"	0.11	"	"	"	"
Mean	N.D.	0.175	N.D	N.D.	N.D.	0.02
Std.Dev.	N.D.	±0.14	N.D.	N.D.	N.D.	±0.02

N.D = Not detected.

Table-3: Effect of hydrchloric acid concentration on the recovery of Lea and Zinc from Sphalerite

S. No.	wt. of sample	Conc. of HCl (M)	Temp (C°)	Time (hrs)	% recovery of Pb	% recovery of Zn
1.	10 gm	0.5	80	12-14	20	20
2.	"	1.0	"	"	30	45
3.	"	1.5	"	"	50	55
4.	"	2.0	"	"	75	60
5.	"	2.5	"	"	90	70
6.	"	3.0	"	"	70	60
7.	"	3.5	"	"	60	50
8.	"	4.0	"	"	55	45
9.	"	4.5	"	"	20	40
10.	"	5.0	"	"	10	35

Effect of leaching time on the recovery of Pb and Zn

Different time intervals for the digestion of 10 gm Ore with 50 mls of 2.5M HCl were studied and it was found that maximum recovery of Lead and Zinc occur by treating the rock with the acid for a period of 12-14 hrs as shown in Table-4.

Effect of temperature on the recovery of Pb and Zn

The effect of temperature on the overall recovery of Lead and Zinc has also been studied 10 gm ore was treated with 50 mls of 2.5 M HCl for a period of 12-14 hrs at different temperature and it was found that maximum recovery of Pb and Zn occur at a temperature of 80°C (Table-5) which becomes constant with further increase of temperature.

Composition of leach and residue after acid leaching

After treating the Ore at the optimized acid concentration, time and temperature, the detailed chemical analyses of the leach and residue were undertaken. The results are shown in Table-6

Table-4: Effect of time on the recovery of Zinc and Lead from Sphalerite.

S. No.	wt. of sample	Conc. of HCl (M)	Temp (C°)	Time (hrs)	% recovery of Pb	% recovery of Zn
1.	10 gm	2.5	80	2	10	20
2.	"	"	"	4	20	35
3.	"	"	"	6	35	48
4.	"	"	"	8	43	60
5.	"	"	"	10	58	73
6.	"	"	"	12	70	90
7.	"	"	"	14	70	90
8.	"	"	"	16	63	85
9.	"	"	"	18	56	80
10.	"	"	"	20	51	73

Table-5: Effect of temperature on the recovery of Lead and Zinc from Sphalerite

S. No.	wt. of sample	Conc. of HCl (M)	Temp (C°)	Time (hrs)	% recovery of Pb	% recovery of Zn
1.	10 gm	2.5	R.T	12-14	12	10
2.	"	"	30	"	20	15
3.	"	"	40	"	25	25
4.	"	"	50	"	35	35
5.	"	"	60	"	50	50
6.	"	"	70	"	60	65
7.	"	"	80	"	70	90
8.	"	"	90	"	70	90
9.	"	"	100	"	70	90
10.	"	"	120	"	70	90

R.T = Room Temperature

Table-6: Analysis report of leach and residue in percent (Results calculated on the basis of ore weight)

	Leach	Residue
SiO_2	-	80.20
Fe_2O_3	9.22	1.30
Al_2O_3	20.68	2.15
CaO	18.20	1.85
MgO	2.43	0.40
Na_2O	0.56	0.03
K_2O	0.16	0.005
PbO	8.80	2.15
ZnO	1.62	0.10
MnO	0.80	0.04

indicating a recovery of 6.80% Lead and 1.62% Zinc in the leached solution and 2.15% Pb and 0.10% Zn as their oxides in the insoluble matter left as residue. The silica content has been found to be 80% in the residue whereas the leached solution has no silica content. Similarly iron and aluminum contents in the leached solution are 9.2 and 20.7% respectively while in the residue these have been found to be 1.3 and 2.15% respectively. The calcium and magnesium contents are also higher in the leach as compared to the residue. The alkali metals (Na, K) are also higher in the leached mass as compared to the insoluble residue.

Experimental

The representative sample obtained as a result of mixing all the 15 samples collected from the area was ground, homogenized and passed through 150 mesh screen. Conventional volumetric and gravimetric methods were applied for the determination of silica, iron, aluminum, calcium and magnesium [14]. The alkali metals (Na, K) were determined with a Flame photometer (Corning Model-104). U.V. Spectrophotometer (U-2000, Hitachi-Japan), was used for the determination of Lead and Zinc applying spectrophotometric techniques. The trace elements like Co, Cu, Ni, Au and Ag, were determined by a Flameless Atomic Absorption Spectrophotometer (Z-2000, Hitachi Japan).

Procedure

10 gm of the representative sample was taken in a beaker and digested with 50 ml of 2.5 HCl solution at 80°C on a hot plate with constant stirring for a period of 12-14 hours. The solution is then filtered. The filtrate having Iron and aluminum were precipitated as their hydroxides with ammonia and ammonium chloride and are separated out. The filtrate after iron and aluminum separation were digested with ammonium oxalate to precipitate the calcium and magnesium as their oxalates and are separated out.

The lead and Zinc remaining in the solution as their chlorides were treated with ammonium sulphate. The solution containing Lead and Zinc as their sulphates are placed on a hot plate at a temperature of 60-80°C whereby lead is crystallized as Lead sulphate which is separated from the Zinc sulphate. The solution containing Zinc sulphate is again placed on the hot plate at 100-120°C when Zinc sulphate is crystallized out.

Conclusion

The present studies reveal that sphalerite ore of Kohistan-Hazara could be gainfully utilized for the recovery and separation of Lead and Zinc by acid leaching with 2.5 M HCl solution. The purity of both the products have been found to be 98% with a recovery of 90% of Zinc and 70% of Lead.

The Lead and Zinc or their compounds have wide application in making pigments for paints and a driers. Leaded Zinc Oxides are oxides of Zinc produced from Ores of Zinc which contain in association a certain proportion of Lead sulfide Ores, that impart to the paint film certain desirable weathering characteristics of basic lead sulphate. The zinc pigment group consists of two major compounds known as Zinc Oxide and Zinc sulfide and are used in paints as exterior finish coat and white enamels. Besides this other valuable chemicals of Zinc and Lead like Zinc chromate, Zinc sulphate,

Lead chromate and Lead sulphate could also be prepared from the ore.

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