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Optimization of Acidic Ferric Chloride Leaching of a Copper Concentrate

M. R. Patil[1], S. J. G. Krishn[2], P. Reddy[3], B. P. Ravi[*4], C. Rudrappa[5] & P. S. Kumar[6]
[1-6] VSKUPG Centre, Nandihalli, Karnataka, India 583119

Abstract: *The hydrometallurgical process for extracting copper offers an ecologically and economically viable way to extract metal locally independent of smelters. Optimization of Bench scale acidic ferric chloride leaching of copper concentrate obtained from flotation tests on a low grade copper ore from Mine dumps of Chitradurga, Karnataka have been carried out by statistically designed experiments. Factors like size, leaching time, Ferric chloride concentration and reactant weight ratio, have been varied. About 90% of copper was leached out from copper concentrates assaying 23.00% Cu, 6.68%SiO_2, 2.60% $Al_2O_3$4.20% CaO, 3.20% MgO, 4.31% Alkalis, 26.46% Fe (T), 24.90% S(T) and 0.45% Zn under optimum conditions of -100 mesh, 6 hours leach time, 6 M $FeCl_3$ Concentration, pH 2, 2.70 $FeCl_3/CuFeS_2$ weight ratio, 95^o C and 200 rpm agitation.*

Key words*;* chalcopyrite, flotation, leaching.

1. Introduction

India is yet to be self sufficient in copper and has to import it. Hence beneficiation, utilization of low grade copper from copper mines needs a relook, though due to open market global principle domestic copper market is under recession cycle. The review of literature on geology of copper indicated that small copper resources in Karnataka like Chitradurga, Kalyadi, Kallur, Machanur and Thinitini, were mined, concentrated locally and extracted in Indian pyro-metallurgical smelters[1]. The increase in demand for copper led to exploitation of small and low grade copper deposits located away from pyro-metallurgical smelters like small copper resources in Karnataka. The problems associated with such small deposits are fall in tenor, low capacity, increased production costs, dependence on smelters and pollution problems of conventional smelters. Hydrometallurgical process offers scope to extract metal economically, ecologically and independent of smelters using ammonia solvent with oxygen as oxidant or acidic ferric chloride as oxidant leachant. Pike et.al. (1930), Haver et.al (1974, 1975), Nagaraj et.al. (1978), Illagovan et.al.(1985) and Ravi et.al.(1987) opted for the acidic ferric chloride leaching of chalcopyrite concentrates due to fast reactivity, ease of regeneration, high solubility, stability of reactants - products and low costs

2. Experimental

2.1. Material

Flotation tests on a low grade copper ore from Mine dumps of Chitradurga, Karnataka were carried out. Copper concentrates assaying 23.00% Cu, 6.68%SiO_2, 2.60% $Al_2O_3$4.20% CaO, 3.20% MgO, 4.31% Alkalis, 26.46% Fe (T), 24.90% S(T) and 0.45% Zn were produced during bulk locked flotation tests which formed the raw material for leaching tests. Dilute HCl was used to adjust pH. Commercial grade $FeCl_3$ >95%, was used as lechant.

2.2. Methods

Weighed amounts of chalcopyrite concentrates of stipulated size is added after the solution reached the designated temperature. Evaporation losses are minimized by placing the funnel over the flask. After stipulated time the contents of flask are filtered and samples are withdrawn for analysis. Factors like size, leaching time, leaching temperature, Ferric chloride concentration, pH, reactant weight ratio, reaction temperature, and agitation rpm have been varied.

3. Results and discussion

3.1. Factorial experiment

Since acidic $FeCl_3$ leaching is a heterogeneous reaction, experiments were carried out to study effect of factors like particle size, leaching time, weight ratio of reactants, ferric chloride concentration were varied at two levels to study the effect of factors, their interactions and factors significantly affecting the copper extraction. The results were analyzed by Yate's algorithm method. The significance of factors was established. A regression equation relating factors to % Cu extraction was obtained. The optimum conditions were arrived by method of steepest ascent. The temperature, rpm of stirrer were kept constant at 97^{o}C and 200 rpm respectively based on the previous leaching amenability tests Krishna et.al. (2016).The conditions and levels of 2^4 Factorial experiments is given in Table 1. The result of 2^4 Factorial experiment varying size, reactant weight ratio, concentration of $FeCl_3$ and leaching time, is given in Table 2.

Table1; Conditions, levels of 2^4 factorial experiment

Conditions :97°C, pH 2, 200 rpm

Factors	Notation	Levels			Step
		Base 0	High +	Low -	
Time hrs	a	3	4	2	1
$FeCl_3/CuFeS_2$	b	2.275	2.730	1.820	0.455
$FeCl_3$ Conc M	c	2.5	3	2	0.5
Size %-400#	d	-200# / 60%-400 #	-300# / 80%-400 #	-100# / 40%-400 #	20% -400 #

Table 2; Results and Yate's analysisis of 2^4 factorial experiment

Symbol 1	%Cu Ex 2	3	Coeff 4	DF 5	Variance 6	FCal 7	Sig 8
1	28.61	932.74	58.300	-	-	-	-
a	44.05	64.54	4.035	1	260.34	24.95	**S**
b	39.27	229.10	14.320	1	3280.43	314.40	**MS**
ab	44.94	9.32	-0.595	1	5.43	0.52	NS
c	51.67	194.34	12.145	1	2360.50	226.10	MS
ac	56.89	12.22	0.765	1	9.33	0.89	NS
bc	72.78	-53.66	-3.355	1	179.96	17.24	S
abc	80.88	5.80	0.365	1	2.10	0.20	NS
d	35.69	114.56	7.160	1	820.25	78.61	**MS**
ad	46.89	34.90	2.175	1	75.69	7.25	NS
bd	63.17	-29.52	-1.845	1	54.46	5.21	NS
abd	76.58	4.46	0.280	1	1.24	0.12	NS
cd	57.25	-36.36	-2.225	1	82.63	7.92	NS
acd	72.10	-11.32	-0.710	1	8.00	1.50	NS
bcd	80.98	24.56	1.535	1	37.90	3.64	NS
abcd	91.09	19.50	-1.215	1	2.93	0.25	NS

MS = most significant, NS = not significant, S= significant, The effects of interactions of 3 variables or more are neglected, F ratio for 1 x 15 degree of freedom at 95% confidence limit was16.5 and F cal ratio more than 16.5 is considered as significant. The variance due to experimental error is assumed to be average variance of interaction of 3 factors or more per degree of freedom.

The results indicate the following;

1. $FeCl_3$ and $CuFeS_2$ reactant weight ratio was the most significant factor which increases the % Cu extraction.
2. $FeCl_3$ concentration was the next most significant factor.
3. Interaction factors of leaching time and $FeCl_3$ and $CuFeS_2$ reactant weight ratio was third most significant factor which increases % Cu extraction with an increase in time and reactant weight ratio.
4. Size of the concentrate was the next most significant factor which increases the % Cu extraction with increase in fineness.
5. Leaching time was also the significant factor which increases the % Cu extraction.
6. The regression model obtained was:
7. Model % Cu extracted $y = 58.30 + 4.035\ a + 14.32\ b + 12.145\ c + 0.765\ d - 0.595\ ab$, where a, b, c and d are time, reactant ratio, concentration of lechant and size .

3.1.1. Effect of $FeCl_3$ concentration and $FeCl_3$ and $CuFeS_2$ weight ratio: The results indicate that the copper extraction increases significantly with increase in $FeCl_3$ and $CuFeS_2$ weight ratio and then attains saturation with values exceeding 3. This is attributed to reaction (1) with $FeCl_3$ and $CuFeS_2$ weight ratio of 2.67.

$$CuFeS_2 + 3\ FeCl_3 = CuCl + 4FeCl_2 + 2S \quad (1)$$

However leaching of $CuFeS_2$ further continues with additional $FeCl_3$ as attributed to reaction (2) with $FeCl_3$ and $CuFeS_2$ weight ratio of 3.54

$$CuFeS_2 + 4\ FeCl_3 = CuCl_2 + 5FeCl_2 + 2S \quad (2)$$

But the $CuCl_2$ along with acidic ferric chloride further reacts with $CuFeS_2$ as attributed to reaction (3).

$$CuFeS_2 + CuCl_2 + 2\ FeCl_3 = 2CuCl + 3FeCl_2 + 2S \quad (3)$$

Incidentally the combination of equations (2) and (3) gives equation (1). Haver *et. al.* (1974 and 1975) employed low $FeCl_3$ concentration and high retention time for leaching while Nagaraj *et.al*. (1978), Illagovan *et.al*.(1985) opted for high $FeCl_3$ concentration and low retention time for leaching. The mechanism of leaching is based on initial mass transfer and by diffusion at later stages of leaching.

3.1.2. Effect of size: As leaching is a heterogeneous reaction, larger particle size – finer particle size enhances the kinetics of dissolution. The leaching tests were done at 40%, 60% and 100% minus 400 mesh. Leaching at maximum fineness size of 100% -270 mesh has been chosen for final test work, but a regrinding of a concentrate and dewatering is needed which is a cost intensive opreation. Nagaraj *et.al.* (1978) and Illangovan *et.al.* (1985) opted for 80% -270 mesh size for leaching of chalcopyrite concentrates. Hence, for subsequent tests the as received flotation concentrate without grinding is chosen.

3.1.3. Effect of time: The leaching time was varied from 2to 4 hours. The extraction of copper increases with time up to 4 hours. The kinetics may be slow after 4 hours leaching perhaps due to diffusion kinetics through the S layer over chalcopyrite nucleus (Krishna et.al. (2016)). Haver et. al. (1974 and1975) employed low $FeCl_3$ concentration and high retention time for leaching [> 6 hours] while Nagaraj et.al. (1978), Illagovan et.al. (1985) opted for high $FeCl_3$ concentration and low retention time for leaching.

3.2 Test under optimum conditions

The optimum conditions were arrived by method of steepest ascent. The tests were conducted under optimum conditions of 4 hours leaching time, 97°C, 200 rpm, 4M $FeCl_3$ Concentration, $FeCl_3/CuFeS_2$ molar weight ratio of 3.62, and pH of 2 on ungrounded copper flotation concentrates as well as ground concentrates. . The results are given in Table 3.

Table 3; Optimization by method of steepest ascent

Conditions

Factors	Not.	Levels			New Step	Limit level
		Base 0	Step	Coefficient		
Time hrs	a	3	1	4.035	0.4	6.0
$FeCl_3/CuFeS_2$	b	2.275	0.455	14.320	0.65	3.54
$FeCl_3$ Con M	c	2.5	0.5	12.145	0.62	6
Size %-400#	d	60% -400 #	20%- 400 #	0.765	14%	80%

Results;

Trial No	a hrs	b $FeCl_3/CuFeS_2$	c $FeCl_3$ Concn M	d Size %- 400#	% Cu Extracted
1	3.4	2.925	3.12	60	88.0
2	3.8	3.575	3.74	66	92.1
3	4.2	3.575	4.32	80	93.7
4	4.0	3.575	4.00	73	93.1
5	6.0	2.700	6.00	60	92.0
6	**6.0**	**2.700**	**6.00**	**40**	**90.0**

~ 90% of copper was leached out from copper concentrates assaying 23.00% Cu, 6.68%SiO_2, 2.60% $Al_2O_3$4.20% CaO, 3.20% MgO, 4.31% Alkalis, 26.46% Fe (T), 24.90% S(T) and 0.45% Zn at ungrounded concentrate 100% - 100 mesh, 6 hours leach time, 6 M $FeCl_3$Concentration, pH 2, 2.7 $FeCl_3/CuFeS_2$ weight ratio, 95^o C and 200 rpm agitation by scaling up the weight of $CuFeS_2$ from 30 to 500 gms. The results are a combination of previous works of Haver *et. al.* (1974 and 1975) employing low $FeCl_3$ concentration and high retention time for leaching while, Nagaraj *et.al*. (1978), Illagovan *et.al*.(1985) opting for high $FeCl_3$ concentration and low retention time for leaching.

4. Conclusions

Optimization of Bench scale acidic ferric chloride leaching of copper concentrate obtained from flotation tests on a low grade copper ore from Mine dumps of Chitradurga, Karnataka have been carried out by statistically designed experiments. Factors like size, leaching time, Ferric chloride concentration and reactant weight ratio, have been varied. About 90% of copper was leached out from copper concentrates assaying 23.00% Cu, 6.68%SiO_2, 2.60% $Al_2O_3$4.20% CaO, 3.20% MgO, 4.31% Alkalis, 26.46% Fe (T), 24.90% S(T) and 0.45% Zn under optimum conditions of -100 mesh, 6 hours leach time, 6 M $FeCl_3$ Concentration, pH 2, 2.70 $FeCl_3/CuFeS_2$ weight ratio, 95^o C and 200 rpm agitation.

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