

Beneficiation of refractory Au ore from G.R.Halli, Karnataka

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Abstract - A sulphidic carbonaceous refractory gold ore from HGML mine dumps, G.R.Halli, Chitradurga district, Karnataka was subjected to beneficiation for developing an exploratory flowsheet. The sample analysed 4.73ppm Au, 229 ppm Ag, 1.05% Pb, 1.74% As, 7.27% S(total), 12.24% Fe(total), 56.51% SiO₂, and 0.4% fixed carbon. The sample contained 50-60% quartz, 10-15% ankerite, 10-15% pyrite, 1.0% galena, 1% graphite, 3-4% arsenopyrite and 5-7% sericite. The exploratory flowsheet consisted of bulk flotation at 72 mesh, 6.5 pH using Potassium Amyl Xanthate, MIBC, CuSO₄ and H₂SO₄ as reagents, dead roasting of the flotation concentrate followed by acid leaching, cyanidation (extraction of precious metals from acid leach residue). The process recovered about 95% Au ,d 90% Ag and 80% Pb.

Key Words: Au ore, Argentiferrous galena, flotation, tabling, preconcentration, CIP, Roast - leach, CIL

1. INTRODUCTION

India is yet to be self sufficient in gold and has to import it. Hence beneficiation, utilization of low grade gold from Au mines needs a relook, though due to open market global principle domestic market is under recession cycle. Though a lot of work has been carried out on beneficiation of low grade Au from India in general and Karnataka in particular, [5-10], still a relook is studying the amenability of old ore mine dumps is needed in changed economic environmental circumstances, as an import substitute and to extract lean precious metal resources. A refractory gold ore sample from G.R.Halli Mines, Chitradurga District, Karnataka was collected for evolving an exploratory flow sheet which is viable and adaptable in the erstwhile 500 tpd carbon in pulp plant of CGU – HGML. It was reported by HGML that the reserves were about 1.0 million tons and gold recoveries in CGU carbon in pulp plant were 40% only. The review of literature ^[2-17] on treatment of refractory gold ores centered on (1) chlorination / hypochlorite aided carbon in pulp / carbon in leach, of ore or, Au-Ag enriched concentrates (2) conventional method of roasting and leaching of Au-Ag enriched preconcentrates (3) acidic oxidative autoclave leaching method followed by cyanidation (4) bio-oxidation pre-treatment followed by cyanidation

2. MATERIAL AND METHODS

2.1. Ore sample

The as received sample was subjected to standard sample preparation techniques to obtain stock sample for test work.

2.2. Reagents

Commercial grade (>90% assay / purity) potassium amyl xanthate (KAX) methyl isobutyl carbinol (MIBC) CuSO₄, CaO, H₂SO₄, HCl, NaCl, NaCN, NaOH and 6 x 16 mesh coconut shell activated charcoal were used.

2.3. Equipment

Denver 300 x 125 mm ball mill with 18.2 kg 25 mm steel balls, Denver Laboratory flotation machine, Elico pH meter, 300 x 300 mm wide mouth ceramic jar mill, Remi Teflon coated pyro-magnetic stirrer, and laboratory electrical muffle furnace were used

2.4. Methods

The sample was subjected to ;

1. Characterization studies comprising of physico-chemical analysis, mineralogical studies, diagnostic amenability tests and grindability tests as per standard methods.

2. Physical processing methods to concentrate Au, Ag values by tabling and generic bulk sulphide flotation tests varying mesh of grind.

3 Chemical processing methods to extract the values of Au, Ag and Pb, of preconcentrate by Carbon inpulp [CIP], carbon in leach [CIL], Dead roasting, hot acid leaching etc. to extract the precious metal values.

3. RESULTS AND DISCUSSION

The experimental comprises of characterization studies followed by the effect of preconcentration by tabling and flotataion, and extraction of metals by chemical processing methods like roasting, acid leaching, CIP/CIL etc.

3.1 Characterization studies

The representative samples were subjected to physicochemical analysis, mineralogical studies, diagnostic amenability tests and grindability tests. The physico-chemical analysis indicated that the as received sample was dark grey coloured hard and compact lumps of ~3.0 specific gravity and analysed 4.13 ppm Au, 228.6 ppm Ag, 110 ppm Sb, 1.74% As, 7.27% S, 1.05% Pb, 0.14 Zn, 56.51% SiO₂, 12.24% Fe, 4.85% Al₂O₃, 2.80% CaO, 1.24% TiO₂, 0.55% Alkalies and 0.4% fixed carbon. The standard Denver grindability test indicated that the sample belongs to medium-medium hard category. Medium-coarse granular (0.5 – 1 mm) quartz (50 – 55%) occurs as aggregates forming the main matrix.



Medium to coarse (0.5 - 1.2 mm) sub euhedral granular pyrite (8 - 10%) is intermixed and replaced by other sulphides occurs as aggregate of sulphides, besides occurring as coarse grains enclosing fine silicates and galena. Subhedral elongated medium grains (0.5 - 1.0 mm) of arsenopyrite (3 - 4%) is associated with pyrite and some times has fine inclusions of carbonaceous matter, galena, silicates and pyrite. Medium to fine granular (0.05 - 0.5 mm)sub-euhedral galena occurs with silicates and as network in fractured pyrite. Fine-medium clusters of ankerite and siderite (10 - 15%) are intermixed with quartz. Most of sulphides are fairly liberated at -72 mesh. Preliminary diagnostic leach tests at -150 mesh grind categorized 30% for free milling type Au, ~55% for Au associated with carbonates, sulphides and carbonaceous type and rest encapsulated. The tailings analysed 0.52 ppm Au. Sink and float tests yielded a heavy fraction rich in Au, Ag and Pb with 75% Au distribution. The above diagnostic characterization tests indicated that the sample from G R halli was a refractory carbonaceous, medium grained sulphidic argentiferrous galena bearing gold and silver sample responding to concentration of values at liberation size of -72 mesh similar to previous findings^[8-10]. The 2kg sample was stage ground to varying MOG of -52/-72/-100/ -150 mesh [D₈₀ 200/150/90/60 microns respectively]at 67%S in 300 mm x125 mm ball mill 18 kg ball charge of 30, 25 and 20mm dia at stage grinding time of 3 minutes each in 3, 5, 7 and 9 stages respectively and size analysis of MOG is given in Fig1.

Fig 1 : Size Analysis of Rol Mill Grindshilly

4.2. Effect of MOG on tabling

Tabling tests were carried out varying the mesh of grind [MOG] from -52, -72, -100 and -150 mesh using MPE Model Diagonal deck lab table . The test conditions and results are given in Table 1. The tabling test varying MOG indicates that: (1)The sample is amenable to gravity concentration by tabling.(2) Increasing the grind size decreases the wt% yield of concentrate (3) Optimum mesh of grind was found to be -72 #, D₈₀ 150 microns as concentrate assaying 20.8 ppm Au, 1025 ppm Ag was obtained with 80.3% Au and 70.7 % Ag distribution respectively at wt.% yield of 14.7. The results are concordant with DAT tests and finding of previous works using gravity concentration on Au ores [$^{5-10}$]

Table- 1; Effect of MOG

Results;

MOG #	Products	Wt.%	Assay	' ppm	% D	istn
D ₈₀ µ	Products	WL.%	Au	Ag	Au	Ag
-52#	Table conc.	18.3	14.9	667	65.0	55.5
200 µ	Table tails	81.7	1.80	120	35.0	44.5
	Head	100.0	4.20	220	100.0	100.0
-72#	Table conc.	14.7	20.80	1075	80.3	73.7
-72# 150 μ	Table tails	85.3	0.88	66	19.7	26.3
	Head	100.0	3.81	215	100.0	100
-100#	Table conc.	13.0	23.38	1052	76.0	68.4
	Table tails	87.0	1.03	73	24.0	31.6
90 µ	Head	100.0	4.00	200	100.0	100.0
1504	Table conc.	12.0	24.73	1200	67.0	60.0
-150# 60 μ	Table tails	88.0	1.66	109	33.0	40.0
ου μ	Head	100.0	4.43	240	100.0	100.0

4.3. Effect of Mesh of grind [MOG] on bulk flotation;

The characterization studies, tabling tests and review of literature $^{[4-10]}$ indicated that Au bearing sullphides can be bulk floated from associated non sulphide – arsenide gangue for pre-concentration. Bulk flotation tests were conducted varying MOG from - 52 to 150 mesh at 6.5 pH, 40% solids, using 0.8 kg/t H₂SO₄, 1.0 Kg/t CuSO₄, 0.18% kg/t KAX and 0.18 kg/t of MIBC to concentrate Au and Ag bearing sulphides. The test conditions and results is given in Table 2.

Table- 2 Effect of MOG on bulk flotation

Conditions;

2 batches of 2 kg each sample stage ground to -52, -72, -100 and -150 mesh $[D_{80}\,;\,200/\,150/90/60\,$ microns respectively],

		kg/t		
	H ₂ SO ₄	0.7+0.1+0.1	2'	-
	CuSO ₄	1.2+0+0	5'	-
1500	KAX	0.1+0.05+ 0.03	1'	-
	MIBC	0.1+0.05+ 0.03	1'	3'
	1500	CuSO ₄ 1500 KAX	CuSO ₄ 1.2+0+0 1500 KAX 0.1+0.05+0.03	CuSO4 1.2+0+0 5' 1500 KAX 0.1+0.05+0.03 1'

Results;

esults;							
MOG #	Products	Wt.%	Assay	' ppm	% D	% Distn	
D80µ	Products	W L. %0	Au	Ag	Au	Ag	
-52#	BF conc.	36.4	10.52	559	87.0	78.3	
200 μ	BF tails	63.6	0.9	89	13.0	21.7	
	Head	100.0	4.40	260	100.0	100.0	
72.11	BF conc.	23.6	17.6	950	95.9	87.2	
-72# 150 μ	BF tails	76.4	0.23	43	4.1	12.8	
150 µ	Head	100.0	4.33	257	100.0	100.0	
100#	BF conc.	14.0	28.87	1511	94.0	84.6	
-100#	BF tails	86.0	0.30	45	6.0	15.4	
90 µ	Head Cal	100.0	4.30	250	100.0	100.0	
-150#	BF conc.	13.0	31.12	1624	93.0	82.8	
60 µ	BF tails	87.0	0.35	52	7.0	17.8	
	Head	100.0	4.35	255	100.0	100.0	

The bulk flotation tests varying mesh of grind from -52, -72, -100 and -150 mesh D_{80} ; 200/ 150/90/60 microns respectively using 0.8 kg/t H₂SO₄, 1.0 Kg/t CuSO₄, 0.18% kg/t KAX and 0.18 kg/t of MIBC indicated that: (1)The sample is amenable Bulk flotation and is more effective than tabling for precious metal preconcentration.(2) Increasing the grind

size increases the wt% yield of concentrate. (3)Optimum mesh of grind was found to be -72 #, D_{80} 150 microns as concentrate assaying 17.6 ppm Au, 950 ppm Ag was obtained with 95.9% Au and 85.2 % Ag distribution respectively at wt.% yield of 23.6.(4)The wt.% yield is more than tabling probably due flotation of graphitic carbon activated gangue and efficiency of concentration of slimes by flotation vis-à-vis tabling.(5)A combination of tabling followed by flotation of table rejects may increase the metal recovery as practiced in some Au plants. ^[4 & 6] The results are concordant with DAT tests and finding of previous works using bulk concentration on Au ores. ^[5-10]

4.4. Preconcentration test comprising of tabling followed by bulk flotation tests

Since both tabling and bulk flotation tests had an optimum mesh of grind of -72 mesh, a beneficiation test comprising of tabling at -72 mesh, D_{80} 150 μ natural thickening of table tails and re-circulating cleaner tails, rougher bulk flotation at 6.5 pH, 40% solids, using 0.8 kg/t H₂SO₄, 1.0 Kg/t CuSO₄, 0.18% kg/t KAX and 0.18 kg/t of MIBC followed by two stages of cleaning to enrich the grade and reciruclating back the I and II cleaner tails for dewatering along with table tails for next stage flotation. The test conditions and results of equilibrium cycle locked table & bulk flotation is given in Table 3. The tabling and bulk flotation indicated that a concentrate assaying 15.02 ppm Au, 811 ppm Ag, 3.67 % Pb, 28.20% S, 6.82% As and 0.50 % Zn is obtained at 96.6%Au, 92.6% Ag and 90% Pb at wt.% vield of 24.5. Au, Ag and Pb may be extracted from the above pre concentrate. The sample is amenable to beneficiation as pre concentrates with over 90% metal recovery is obtained.

Table- 3; Tabling and bulk flotation of table tails

Conditions for each cycle;

 $\begin{array}{ll} \mbox{2Batches of 2 kg each sample stage ground to -72\#, D_{80}; 150 μ Lab model Deister tabling - Feed 25%S 1 lpm slurry, Feed water4lpm + 4lpm dressing 2 water + 2 lpm dressing 2 water, Tilt 2.5°, amplitude= 8mm, $$frequency 300. $ frequency 300. $ \end{array}$

Floation conditions; feed; Naturally thickened table tails and flotation cleaner tails

Stage	Cell kg	% S	rpm	Reagent	Dosage kg/t	СТ	FT
RBF1, 2 & 3	2kg	40	1500	H ₂ SO ₄ CuSO ₄ KAX MIBC	$\begin{array}{c} 0.7 + 0.1 + 0.1 \\ 1.2 + 0 + 0 \\ 0.1 + 0.05 + 0.03 \\ 0.1 + 0.05 + 0.03 \end{array}$	2' 5' 1' 1'	- - - 3'
ICl BF	1	30	1200	-	-	-	3
II Cl BF	0.5	15	1200	-	-	-	2

Results

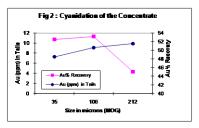
Products	Wt.%	Assay	/ ppm	% Distn		
Floutets	WU.70	Au	Ag	Au	Ag	
Table concentrate	14.7	20.80	1075	80.3	73.7	
II Cl Bulk float	9.8	6.34	415	16.3	18.9	
Final Bulk tails	75.5	0.17	21	3.4	7.4	
Head	100.0	3.81	215	100.0	100	
Composite concentrate	24.5	15.02	811	96.6	92.6	

4.5 Classical CIP / CIL/ Roast - CIP method

Based on HGML plant data, characterization tests and review of literature^{1-6, 12,13,} preliminary CIP / CIL tests were conducted on ore samples varying MOG (-100 / -150 / -200

mesh), NaCN concentration (0.05 / 0.10 / 0.2 / 0.3%), time (12 / 24 / 48 hrs) and carbon load (17 / 34 / 51 Kg/t) at 11.0 pH using 12 kg/t CaO. Only 61% Au could be extracted under intensive CIL conditions at -200 mesh size, 48 hrs time, 0.3% NaCN concentration, 51 kg/t activated carbon, while CIP tests yielded only 35% Au extraction. The bulk flotation concentrate was leached with 18 kg/t NaCN, 12 kg/t CaO, 50% solids, 11 pH, for 24 hrs, varying size (- 72 mesh -150 mesh, -400 mesh). The results are shown in Fig.2 which indicates only 53% Au, 60% Ag extraction.

A cyanidation test was conducted on dead roasted concentrate at -150 mesh size, 11 pH, 0.5% NaCN concentration, 0.2% NaOH concentration for 24 hrs, which yielded only 70% Au and 94% Ag extraction. Intensive cyanidation of dead roasted ground concentrate improved Au recovery to 70% and Ag recovery to 90% confirming that unlocking of mineral lattice will open precious metals for better cyanidation ²⁻⁶. The Au recovery is lower than anticipated. Mineralogical studies indicated that presence of thick metal oxide coatings over grains might have caused surface closure for reaction preventing cyanidation



4.6 .Pre concentrate, roast- acid leach- CIP

The Au recovery is lower than anticipated in roasting and CIP of concentrates. Mineralogical studies indicated that presence of thick metal oxide coatings over grains might have caused surface closure for reaction preventing cyanidation. Hence a test was conducted by roast - acid leach and cyanidation on pre concentrates to over come the problem of metal oxide coating impeding cyanidation and extraction of Au and Ag. The test conditions and results are given in Table 4. The float, roast - acid leach - cyanidation method yielded 94.9% Au, 87% Ag, extraction and tails assayed 0.27 ppm Au, 43 ppm Ag and 0.27% Pb. The reagent consumption was 1.2 kg/t CuSO₄, 0.18 kg/t KAX 0.16 Kg/t MIBC, for flotation 300 kg/t H₂SO₄, 53 kg/t HCl for acid leaching, 0.6 kg/t NaCN, 12 Kg/t NaOH, 4 kg/t CaO for cyanidation. The roast acid leach cyanidation of concentrate enhanced the Au, Ag recovery to 90% (table 3) besides yielding Pb as by product. The results agree well with diagnostic leach test and previous work [1-3, 4-17]. The expensive eco-sensitive classical roast acid leach step may be replaced either by costly eco-friendly acid oxidative autoclave leaching method ^[12] or by eco friendly state of art bio-oxidation method ^{[11&15].} Incidentally, Raichur *et.al* ^[11] while working on auriferous sulphide concentrates from the same area have extracted over 90% precious metals by bacterial oxidation treatment of concentrates prior to cyanidation.

Table-4: Bulk flotation, roasting, acid leaching & cyanidation

Conditions;

 $\label{eq:Pre-concentration:Locked bulk flotation at -72 mesh, 6 pH with 2 cleanings in counter current circuit at 40% S using 1.2 kg/t CuSO _4, 1.0 kg/t H_2SO _4, 0.18 kg/t KAX and 0.18 kg/t MIBC$

Roasting and acid leaching:Dead roasting concentrate (950°C, 3 hrs) leached with H_2SO_4 and HCl at 95°C, 150 mesh, 50% S, 3-6 hrs filtered and neutralized with hot NaOH.

Cyanidation: Acid leach residue, cyanided with 0.1% NaCN for 24 hrs at 50% Solids, 11 pH,-150#

Results	
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Products	Wt.%	Assay	v ppm	% Distn	
Products	W L. %	Au	Ag	Au	Ag
CN-liquor	4.2	97.86	5328	94.9	87.0
CN residue	4.2	1.04	10	1.0	0.2
Acid Leach liquor	11.7	BDL	BDL	-	-
Calcine	15.9	26.12	1410	95.9	87.2
Lss On Calcination	7.7	-	-	-	-
Conc (BF)	23.6	17.6	950	95.9	87.2
Bulk Tails	76.4	0.23	43	4.1	12.8

4.7 Final confirmative test;

A final test modifying the above roast acid leach method of Au-Ag concentrates comprised of (a) pre-concentration of Au-Ag-Pb values by tabling followed by bulk flotation of table tails, at72 mesh (b) acid leaching of dead roasted concentrate at –150 mesh, 90°C at 50% solids with H₂SO₄ and NaCl. (c) Cyanidation of neutralized acid leach roast residue to extract Au and Ag. The H₂SO₄ may be regenerated from roaster gases. The reagent consumption was 1.2 kg/t CuSO₄, 0.18 kg/t KAX, 0.18 kg/t MIBC, 300 kg/t H₂SO₄, 132 kg/t NaCl, 0.6 kg/t NaCN, 4 kg/t CaO 12 kg/t NaOH, 50 kg/t activated carbon. The results are given in table 5. The above process extracted about 95.4% Au, 92.4% Ag besides ~80% Pb as by product

Table-4: Concentration, roasting, acid leaching & cyanidation

 Conditions:

Pre-concentration:Locked bulk flotation at –72 mesh, 6 pH with 2 cleanings in counter current circuit at 40% S using 1.2 kg/t CuSO₄,1.0 kg/t H₂SO₄, 0.18

kg/t KAX and 0.18 kg/t MIBC **Roasting and acid leaching**:Dead roasting concentrate (950°C, 3 hrs) leached with H_2SO_4 and HCl at 95°C ,150 mesh, 50% S, 3-6 hrs filtered and neutralized with hot NaOH.

 $Cyanidation: \mbox{ Acid leach residue, cyanided with 0.1% NaCN for 24 hrs at 50% Solids, 11 pH,-150#$

Results;

Products	Wt.% Assay ppm		% Distn		
Products	WL.%	Au	Ag	Au	Ag
CN-liquor	4.5	80.71	4407	95.4	92.4
CN residue	4.5	1.04	10	1.2	0.2
Acid Leach liquor	14.4	BDL	BDL	-	-
Calcine	18.9	19.47	1052	96.6	92.6
Lss On Calcination	5.6	-	-	-	-
Head	100.0	3.81	215	100.0	100.0
Final Tails	80.0	0.22	20	4.6	7.6

4. CONCLUSIONS

A lead bearing sulphidic carbonaceous refractory Au-Ag ore was subjected to an exploratory flow sheet processing comprising of pre concentration of Au-Ag values by tabling and bulk flotation, dead roast acid leaching of concentrates followed by extraction by cyanidation yielded over 92% precious metal recovery and 80% Pb as by-product. The process appears to be viable and adaptable in the nearby closed CIP plant. Further, there is a possibility of increasing the economic viability by replacing the classical, expensive eco-sensitive roast – acid leach step by bacterial – oxidation method. Detailed work in this regard is recommended followed by feasibility study.

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