FLOTATION OF COPPER MINERALS FROM NORTH WAZIRISTAN COPPER ORE, ON PILOT-SCALE

M. Mansoor Khan,
Mazhar Rafiq and
Ehteshamullah Khan*

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ABSTRACT

Flotation process parameters were studied to concentrate the copper content i.e. Chalcopyrite of North Waziristan copper-ore on Pilot-scale to obtain a copper concentrate suitable for further metallurgical treatment. The important flotation parameters, e.g. type and dosage of collector, dosage of depressent, dispersent and frother and conditioning time for collector were examined. During stepwise optimization of flotation parameters, the copper content was upgraded from 0.9% to 20% in roughing stage, and to as high as 22% in a single-stage cleaning, with recoveries of over 83%. A flowsheet depicting different products of flotation, for an industrial concentrator, has also been suggested.

INTRODUCTION

In North-Waziristan, at places like Shinkai and Degan, huge deposits of copper are available. The inferred ore-reservers are 122.71 million tons,having a copper content varying from 0.3865% to as high as 2% (FATA DC, 1985); (Wang Zhitian, 1996). The purpose of this paper is to study concentrateion of the North Waziristan copper ore (Shinkai area), having copper content 0.9% to a level suitable for metallurgical treatment. The western part of Waziristan constitutes a complex igneous belt. The belt extends Northeast-Southwest and consist of ultramafic masses, consisting of harzburgite, pyroxenites, periododites and dunites, generally serpentinized. The intrusives comprise diorites, quartz-diorites, micro-quartz diorites, granodiorites, dolerites and gabbros.

Volcanics include fine-grained porphyritic pillow basalts and andesites, with subordinate breccias and minor dacite, rhyodacites, tuffs and agglomerates (Badshah 1983).

To reconfirm the previously carried out studies (Khan, 1994), laboratory-scale petrographic studies of the ore showed that the sulphide mineralization is present in the form of the stock work-internally brecciated. The suphide minerals are disseminated and are in skeletal form. The pillow breccia is chloritized and contains minerals like hornblende and pyroxene. The ore-minerals are mostly cube-shaped, sporadically distributed throughout the rock but also found in the form of veinlets. Chalcopyrite exists as a major mineral, with malachite, azurite, tennantite as minor valuable minerals. Pyrite, pitchlimonite, bronchantite, graphite, chamosite, nimite and quartz exist as gangue minerals (Rafiq Mazhar, 1999).

PILOT-PLANT FLOTATION STUDIES

The aim of the present research is to develop an economically viable commerical process to utilize the 122 million tons of copper ore of the North Waziristan area.Pilot-scale flotation studies were carried out to upgrade its copper-content by physical concentration method to a level which is suitable for metallurgical treatment to produce blister copper. Flotation-process parameters, such as collector type and dosage, dispersent, pulp density, etc, were investigated.

FLOTATION TEST-PROCEDURE AT PILOT SCALE

Minerals

The copper ore was from the Shinkai area (North Waziristan) and assayed at 0.9% Cu, 0.0134% Zn, 0.0126% Pb, 0.0110% Co, 0.0067% Ni, 1.740 ppm Ag, and 0.22 ppm Au. Quartz was the main gangue mineral.

Communition

For each test-run of the flotation-process, approximately 100 kgs of copper ore was reduced in size to 80% passing 4mm. The crushed material was wet-ground for 20 min in a closed-circuit ballmill classifier, at a solid-liquid ratio of 1:2.3 (i.e. 30% solids by weight), to 80% passing 75mm. The coarse particles were difficult to float, so the grind was chosen to be fine i.e. 75mm (Rafiq Mazhar, 1999). The fine-

* Faculty members, Department of Mining Engg., NWFP University of Engieering & Technology, Pakistan.

Table 1. Pilot-Scale Flotation Process Parameters

Test	Pulp Density Weight/Volume gms/ton	Collector Dosage gms/ton	Frother Dosage	Pulp pH	Depressant Dosage gms/ton	Sulphidizer Dosage gms/ton	Dispersent Dosage gms/ton	Reagent Conditioning Time (Min)	
1-5	30	50~250	75	11	0	0	0	10	Bulk Roughing - I
6-10	30	50-250	75	11	0	0	0	10	
11-15	30	50-250	75	11	0	0	0	10	
16-20	30	200	75	10~12	0	0	0	10	
21-25	30	200	75	11.5	10~30	0	0	10	
26-30	30	200	75	11.5	25	10~60	0	10	
31-33	30	200	25~70	11.5	25	50	0	10	
34-37	15~35	200	46	11.5	25	50	0	10	
38-41	30	200	46	11.5	25	50	0	10~18	
42-46	15	40	40	8~12	0	0	0	10	Bulk Roughing - II
47-50	15~30	40	40	10	0	0	0	10	
51-55	25	0~90	40	10	0	0	0	10	
56-59	25	10~90	40	10	0	0	0	10	
60-63	25	60	0~60	10	0	0	0	10	
64-67	25	60	100	10	0	0	0	7~16	
68-71	20	40	35	10~11.67	0	0	0	8	Cleaning - I
72-76	20	40	35	10	0	0	0~180	8	
77-80	20	10~40	35	9.5	0	0	150	8	
81-83	20	10~30	35	9.5	0	0	150	8	
84-87	10~19	20	35	9.5	0	0	150	8	
88-91	15	20	35	9.5	0	0	150	5~14	

size grinding also promotes the self-induced flotability of the particles (Senior G.D. and Trahar W.J., 1976).

Flotation Testing

The product of the ball-mill was conditioned and subjected to flotation in a bank of 25 and 28 litres flotation-cells. All the reagents were added in the conditioning stage, except caustic soda (NaOH) which was added during the grinding stage as pH regulator. The conditioned pulp was subject to two roughing-stage flotations and one cleaning-stage flotation. The concentrates from the Bulk Roughing-I and Bulk Roughing-II were combined and cleaned, to obtain the final concentrate. For Bulk Roughing-I, the parameters investigated were type and dosage of collector, depressant dosage, sulphidizer dosage, pine-oil dosage, dispersent dosage and conditioning time. In any stage of flotation . i.e. roughing or cleaning, while studying the effect of variation of any parameter on the grade and recovery of copper, the rest of the parameters were kept constant. (Table 1). Normal tap-water was used for conducting all the tests. All the reagents were of technical grade, except sodium cyanide (NaCN), caustic soda (NaOH) and sodium sulphide (Na_2S) which were of commercial grade. Chemical analyses were carried out with X-Ray Spectrometer System (Jeol, Model JSX-603).

FLOTATION TESTS FOR BULK ROUGHING-I

In the flotation method of concentrating minerals, fast floating minerals are recovered in bulk-roughing stages, in order to obtain maximum recovery of valuable minerals (Wills B.A, 1985). The parameters investigated during this stage were type and dosage of collector, pH value, depressant dosage, sulphidizer dosage, pulp density, and pine-oil dosage. Generally, higher dosages of reagents are used during this stage of flotation.

Details of Tests

Fifteen tests were carried out to evaluate the flotation- response , using different dosages and. type of collector. Five tests were conducted each to investigate the effect of varying pH and depressent dosage on the grade and recovery of copper. The effects of sulfidizer was investigated by using dosage in the range of 10- 60 gms/ton. Three tests were conducted to investigate the effect of pine oil (frother) by varying its dosage between 25 and 70 gms/ton. The effect of variation in pulp-density was investigated by varying it between 15 and 35%. Similarly, a few tests were conducted to investigate the effect of conditioning--time on the grade and recovery of copper. All the products, including tailings, recovered during. flotation test work, were dried and analyzed for copper. From the results, grade and recovery curves were drawn. The concentrate from the Bulk Roughing-I was then routed to the cleaning stage.

DISCUSSION OF RESULTS

Collector Type and Dosage

The curves in Figure 1 (Tests 1-5) show that, with an increase in NaPX (Sodium Propyl Xanthate) there is a corresponding increase in the grade/ recovery of copper (Ralston V.E., 1991, Senior, 1991, Keble 1982, Woods, 1971, Ross, 1981, Swat 1991). Beyond the dosage of 200 gms/ ton, there is a decrease in grade and a slight decrease in recovery. This may be due to the non-specific adsorption of the collector by the gangue particles (Woods, 1971; Ross,1991) and, possibly, due to the development of collector multilayers on the particles, reducing the proportion of hydrocarbon radicals oriented into the bulk solution (Smart 1991; Fuerstenau and Palmer 1976; Smith and Akhtar, 1976), thus reducing the grade of the concentrate; the critical dosage of NaPX observed at this stage was 200 gms/ton. The curves in Figure 2 (Tests 6-10) show a similar behaviour of NaEX (Sodium Ethyl Xanthate) to NaPX. By using NaEX, the grade/recovery is almost the same upto 150 gms/ton but, later, the recovery has not improved as in case of NaPX. This may be due to the fact that hydrophobic action of Xanthate collectors decreases with the, decrease in molecular weight of the alkyl group (Wills,1985); (Majima and Takeda, 1968); (Kelly and Spottiswood, 1982). The curves in Figure 3 (Tests 11-15) show the effects of KPX (Potassium Propyl Xanthate) on the grade and recovery of copper. The curves reveal a similar behaviour of KPX to that of NaPX. By using collector KPX, there is not much effect on the grade/recovery of copper

Flotation Process Parameters Investigated for bulk Roughing

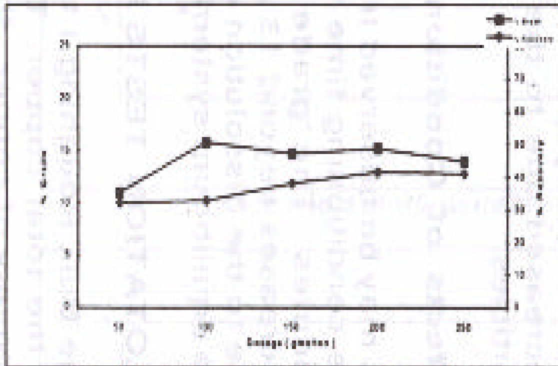

Figure. 1 Effect of Collector (NaPX) on Grade and Recovery of Copper

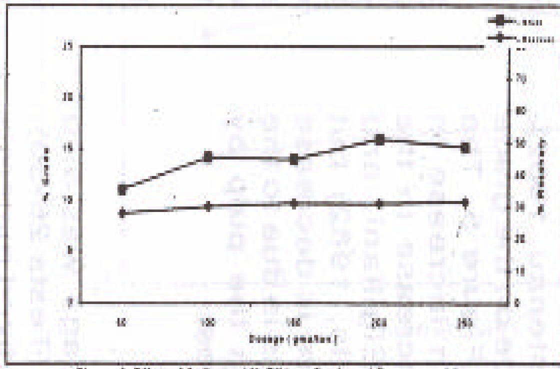

Figure. 2 Effect of Collector (NaEX) on Grade and Recovery of Copper

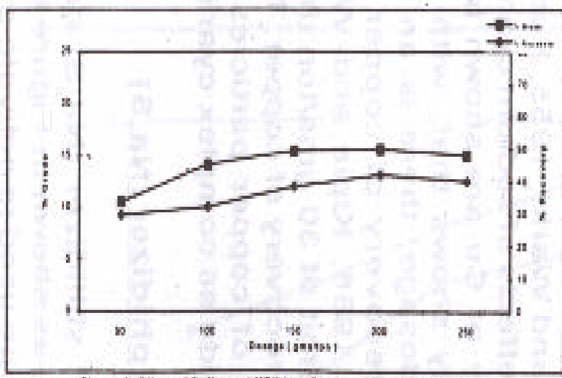

Figure. 3 Effect of Collector (KPX) on Grade and Recovery of Copper

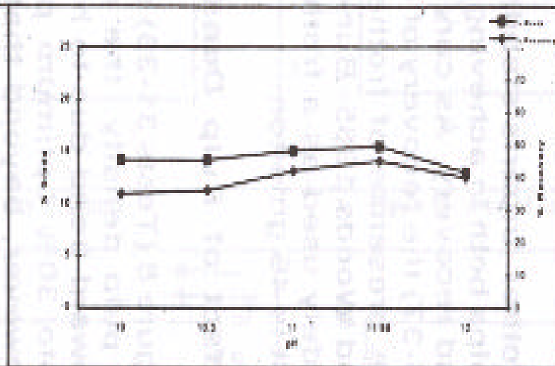

Figure. 4 Effect of pH on Grade and Recovery of Copper

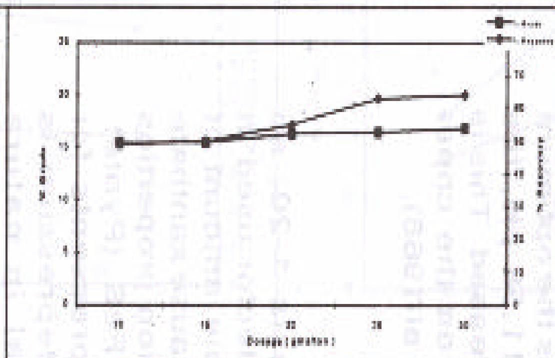

Figure. 5 Effect of Depressant (NaCN) on Grade and Recovery of Copper

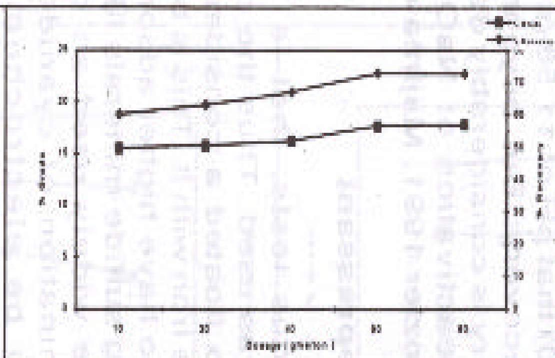

Figure. 6 Effect of Sulphidizer (Na_2S) on Grade and Recovery of Copper

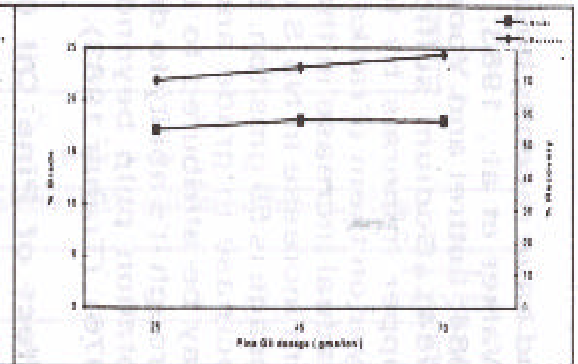

Figure. 7 Effect of Frother (Pine Oil) on Grade and Recovery of Copper

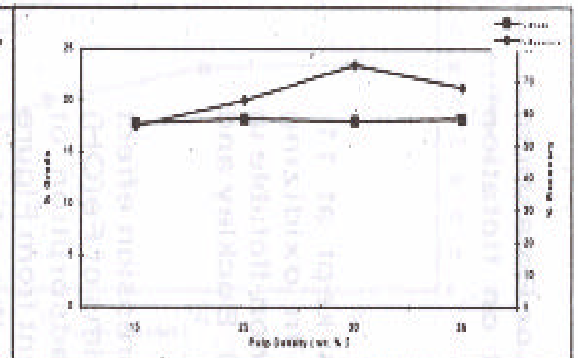

Figure. 8 Effect of Pulp density on Grade and Recovery of Copper

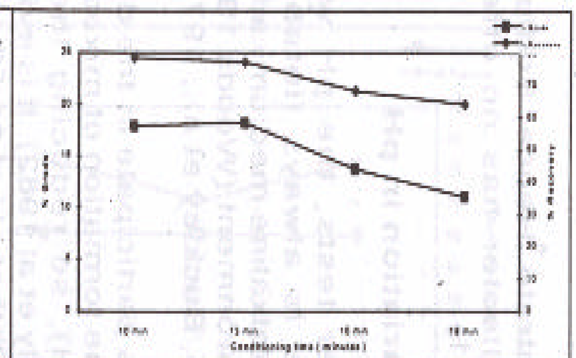

Figure. 9 Effect of Conditioning time of Collector (NaPX) on Grade and Recovery of Copper

in the concentrate. Hence, Sodium or Potassium cation in collector has no effect on flotation (Crozier, 1991).

Effects of Variation in pH

In the earlier tests, the pH was kept at 11. Chalcopyrite is always flotable in oxidizing environment (alkaline medium) and non-flotable in reducing environment (Woods 1950; Buckley and Woods, 1985, Buckley et al., 1976).

Hydroxyl ions participate in the depression effect on pyrite by the formation of mixed films of Fe(OH) and FeO(OH), so reducing the adsorption of Xanthate (Kelly et al 1982). It is evident from Figure 4 (Tests 16-20) that pH of 11.58 gives the optimum grade and recovery. But beyond 11.58 pH, the grade/recovery is considerably decreased. This is due to the deactivation of NaOH on the coper minerals. (Crozier 1991; Majima et. al 1968).

Effect of Depressant

In the previous tests, that is Tests 1-20, no depressent was used. Thus the collector used in the tests also floated a considerable amount of FeS_2, and free Iron with it. This is because xanthate collectors also have higher adsorption properties for Fe-bearing sulfide minerals like FeS_2 (Pyrite). Cyanides are widely used as depressents for pyrite. Examination of cyanide-depression is indicated to be electrochemical in nature (Sutherland and Wark., 1955, Fuerstenau, 1962). The positive effects of sodium cyanide on the grade and recovery of Cu are shown in Figure 5. The figure clearly shows that, with an increase in depressent dosage, there is an increase in the grade and recovery of copper (Elgillani, and Fuerstenau, 1968, Kipie and Wells, 1982) but beyond the limit of 30 gms/ton there is decrease in grade and recovery of copper. This is due to the deactivation of copper particles in the pulp by sodium cyanide as complex cyanides.

Effect of Sulphidizer (Na_2S)

The effect of sulfidizer (Na_2S) dosage was also investigated, as shown in Figure 6 (Tests 26-30). Sulphidizers are used to activate oxidized minerals and have recently been shown to induce flotation. (Walker et al., 1983; Yoon, 1981, Walker et al. 1984; Luttrel and Yoon 1984; Heyes and Trahar., 1984). Sodium sulfide activates the oxidized copper minerals by forming a psuedo-sulphide layer on them (Franks, 1975). The curves show a gradual increase in the grade/recovery of copper with increase in Na_2S dosage. The most effective dosage is 50 gms/ton. Beyond that, there is a slight decrease in grade and recovery of copper. This may be attributed to depressing action of Na_2S, through it's ability to decrease the potential of the flotation pulp beyond critical amount (Woods, 1976); (Trahar 1983).

Effect of Pine Oil (Frother)

Frother imparts stability to the mineral froth and helps both in acheving higher-grade concentrates and recovery. As can be seen in Figure 7 (Tests 34-37) the recovery of chalcopyrite is enhanced in the presence of frother (Woods, 1950; Buckley. and Woods 1985; Buckley et. al, 1976). Pine oil is widely used as a frother; the best dosage found out is 46 gms/ton.

Effect of Pulp Density

Figure 8 (Tests 31-33) shows that, with an increase in pulp density, the recovery/grade shows an upward trend due to hindered settling conditions upto 30% optimum pulp density (Wills 1985). However, beyond that, the grade has markedly decreased due to the entrainment of fine slime particles.

Effects of Conditioning Time

As may be observed from Figure 9 (Tests 38-41), the conditioning time varies between 10 and 13 minutes. The grade and recovery markedly decreases beyond 13 minutes conditioning time, due to the dissolution of Copper Xanthate ions in the equilibrium system. (Khan et. al. 1984).

FLOTATION TESTS FOR BULK ROUGHING-II

The Bulk Roughing-I tailings still contained 30% of the total copper. Therefore, to enhance the recovery of copper, the Bulk Roughing-I tailings

Flotation Process Parameters Investigated for bulk Roughing II

Flotation Process Parameters Investigated for bulk Roughing II

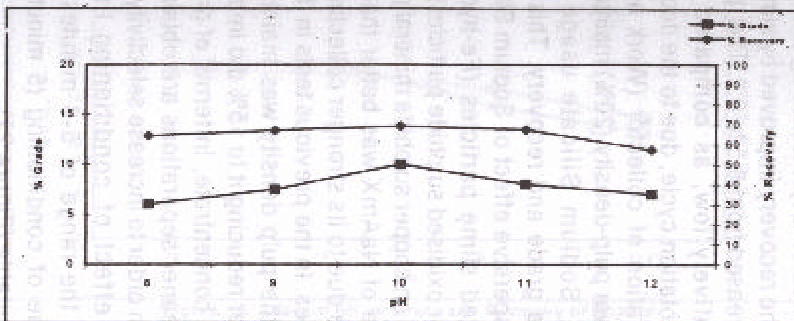

Figure.10 Effect of pH on Grade and Recovery of Copper

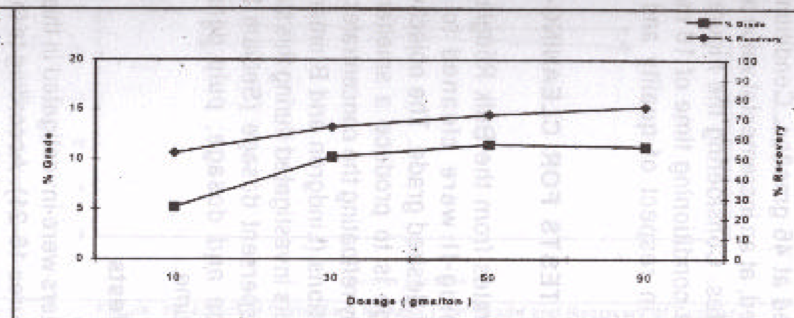

Figure.13 Effect of Collector (NaEX) on Grade and Recovery of Copper

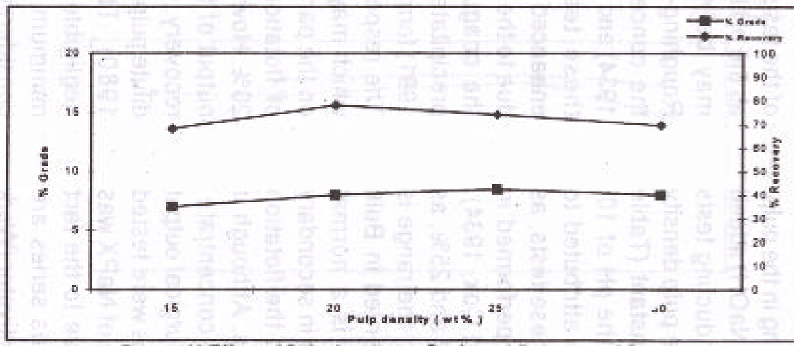

Figure. 11 Effect of Pulp density on Grade and Recovery of Copper

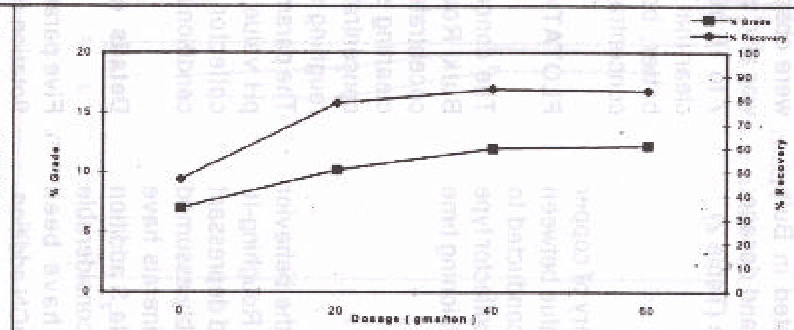

Figure. 14 Effect of Frother (Pine Oil) on Grade and Recovery of Copper

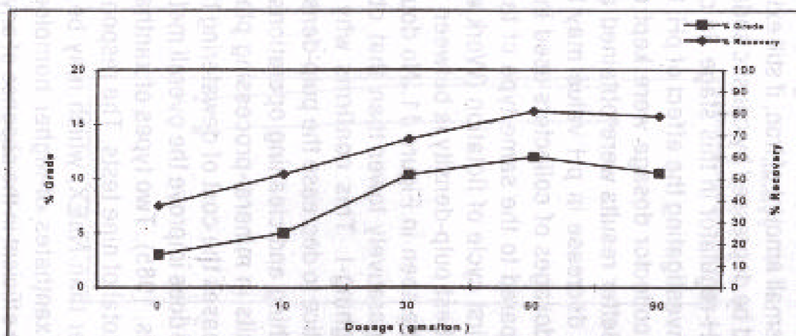

Figure. 12 Effect of Collector (NaPX) Grade and Recovery on Copper

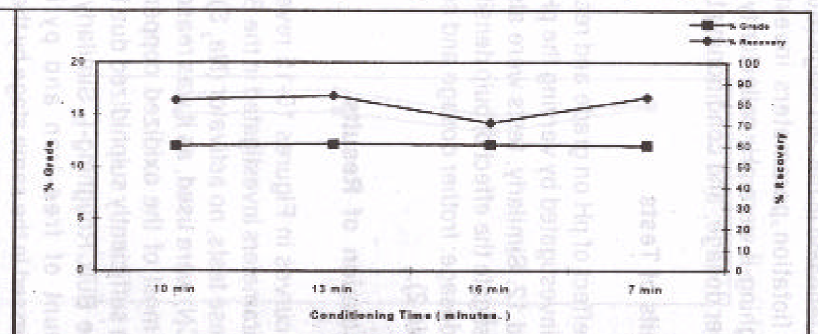

Figure. 15 Effect of Conditioning time on Grade and Recovery of Copper

were refloated in the 2nd stage (Bulk Roughing-II). The flotation parameters investigated in Bulk Roughing-II were pH; collector type and dosage; frother dosage; and conditioning time (Table 2).

Details of Tests

The effect of pH on grade and recovery of copper was investigated by varying the pH value between 8 and 12. Similarly, tests were also conducted to investigate the effect of pulp density; collector type and dosage; frother dosage and conditioning time (Table 2).

Discussion of Results

The curves in Figures 10-15 reveal the behavior of parameters investigated in the Bulk Roughing-II. In these tests, no activator (Na_2S) and depressant (NaCN) were used, as it was reasonably assumed that most of the oxidized copper minerals have been sufficiently sulphidized due to Na_2S addition in the Bulk Roughing-I. Similarly a considerable amount of free Iron and pyrite have been depressed in the same stage by the NaCN addition. Any small amount of Iron, if still existing in the pulp, could be depressed by caustic soda (NaOH) added as pH-regulator in this stage. In conducting tests for investigating the effect of pH, the pulp density and collector dosage were kept constant (Table 2). Better results were obtained at the pH of 10. This decrease in pH value may be attributed to low dosages of collectors used in these tests, as compared to the same type of test performed in the first cycle of flotation (Work and Cox, 1934). The best pulp-density is between 22 and 25%, as can be seen in Figure 11. No doubt, the range is comparatively lower than that observed in Bulk Roughing-I. This confirms why it is a normal practice to decrease the pulp-density in secondary roughing and cleaning operations of the flotation circuits in mineral-processing plants. Although it increases the cost of dewatering the concentrate, but it does improve the overall metallurgical output (Wills, 1985). Two types of xanthates were tested in a total of nine tests. The response of NaPX was better than NaEX, which may be due to the fact that xanthates of higher homologous series are more efficient in the absence of any activator (Wark and Cox, 1937). Of the total number of tests conducted, the best results for pine-oil (frother) were observed at 46 gms/ton. Conditioning-time was also varied, at an incremental value of 3, from 7 to 16 minutes. Considering the Figure 15, it is clear that the conditioning time of 10 minutes is better, both in respect of quality and cost of concentrate.

FLOTATION TESTS FOR CLEANING-I

The concentrates from the Bulk Roughing-I and Bulk Roughing-II were cleaned to get the cocentrate of desired grade. The objective of the cleaning stage is to produce a smelter's grade concentrate by refloating the concentrates from the roughing sections (Lindgren and Broman, 1976). The parameters investigated during this stage were pH value; dispersent dosage (Sodium Silicate); collector type and dosage; pulp density and conditioning time.

Details of Tests

Five parameters were investigated in this cycle of flotation (Figures 16-21). According to the results of the tests, the recovery improved by almost 40% as the pH increased from 8.5 to 9.2. This. pH value may be relatively low, as compared to Bulk Roughing-I flotation cycle, due to the decrease in the concentration of collector (Work and Cox, 1934) and lower pulp-density (20%) maintained for these tests. Sodium Silicate usage further enhanced the grade and recovery. This may be due to the dispersive effect of Sodium Silicate on the coagulated slime particles (Fe-hydroxides precipitates or oxidised sulphide particles) (Smart, 1991) formed on copper sulphide mineral particles. The response of NaAmX was better than NaEX, which may be due to its stronger collecting action on the particles. In the previous tests in this cycle of flotation, the pulp density was maintained at 20%. However reducing it to 15% did improve the output of the concentrate, in terms of grade and recovery. Cleaner separations are obtained with dilute pulps, in order to increase selectivity (Poling, 1980). The effect of conditioning time was negligible in the range of 5-8 minutes. Hence minimum time of conditioning (5 minutes) was considered suitable (Figure 21).

Flotation Process Parameters Investigated for Cleaning I

Flotation Process Parameters Investigated for Cleaning I

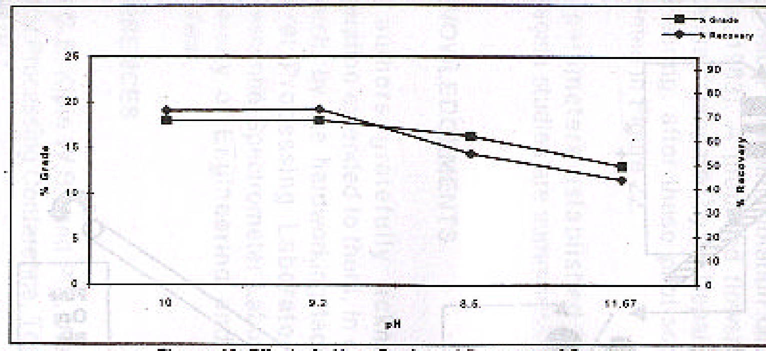

Figure. 16 Effect of pH on Grade and Recovery of Copper

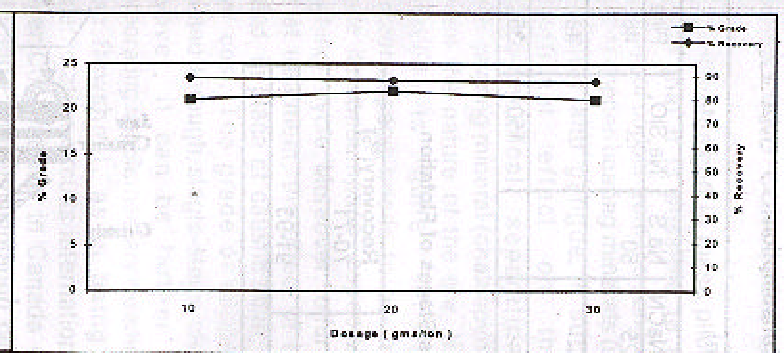

Figure. 19 Effect of Collector (NaAmX) on Grade and Recovery of Copper

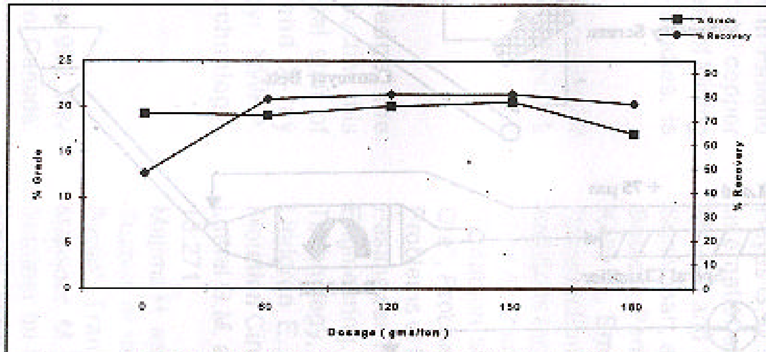

Figure.17 Effect of depressent on Grade and Recovery of Copper

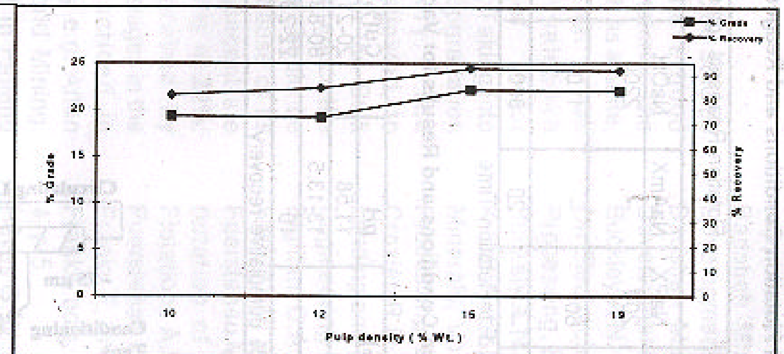

Figure. 20 Effect of pulp density on Grade and Recovery of Copper

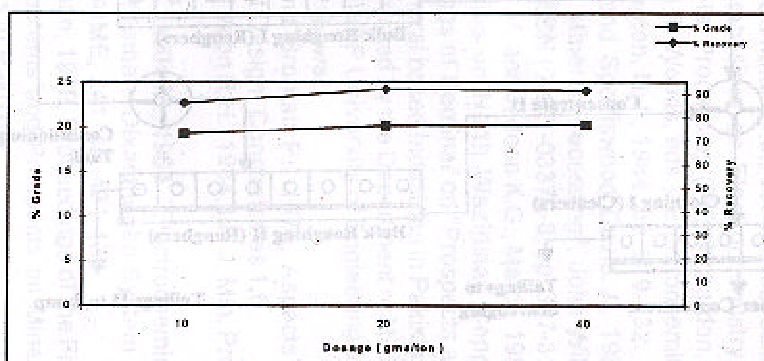

Figure. 18 Effect of Collector Dosage (NaEX) on Grade and Recovery of Copper

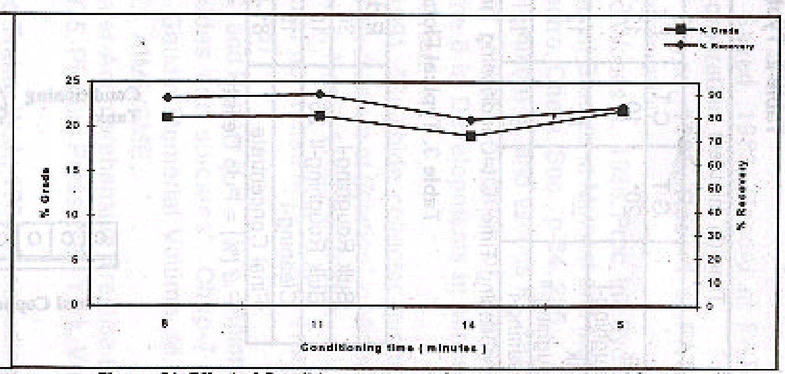

Figure. 21 Effect of Conditioning time on Grade and Recovery of Copper

Table 2. Typical Flotation Conditions and Reagent Consumption

Time (in minutes)				Flotation Reagents (gms/ton)						
	G.T.	C.T	F.t	NaPX	NaAmX	NaOH	NaCN	Na_2S	Na_2SiO_3	Pine Oil
Bulk Roughing-I	20	10	12-15	200	--	2200	25	50	--	46
Bulk Roughing-II	--	8	12	60	--	1100	--	--	--	46
Cleaning-I	--	5	12	--	20	800	--	--	150	35

G.t=Grinding Time, C.t=Conditioning Time, F.t=Flotation Time

Table 3. Typical Flotation Conditions and Results for Various Stages of Flotation

	P.d (%)	pH	Cu(%)	Recovery(%)
Bulk Roughing-I	30	11.58	20-21	70-71
Bulk Roughing-II 25	10	13-13.5	80-83	
Cleaning-I	15	10	22-23	91-93
Final Concentrate	81-83% cumulative recovery			

P.d (%) = Pulp Density

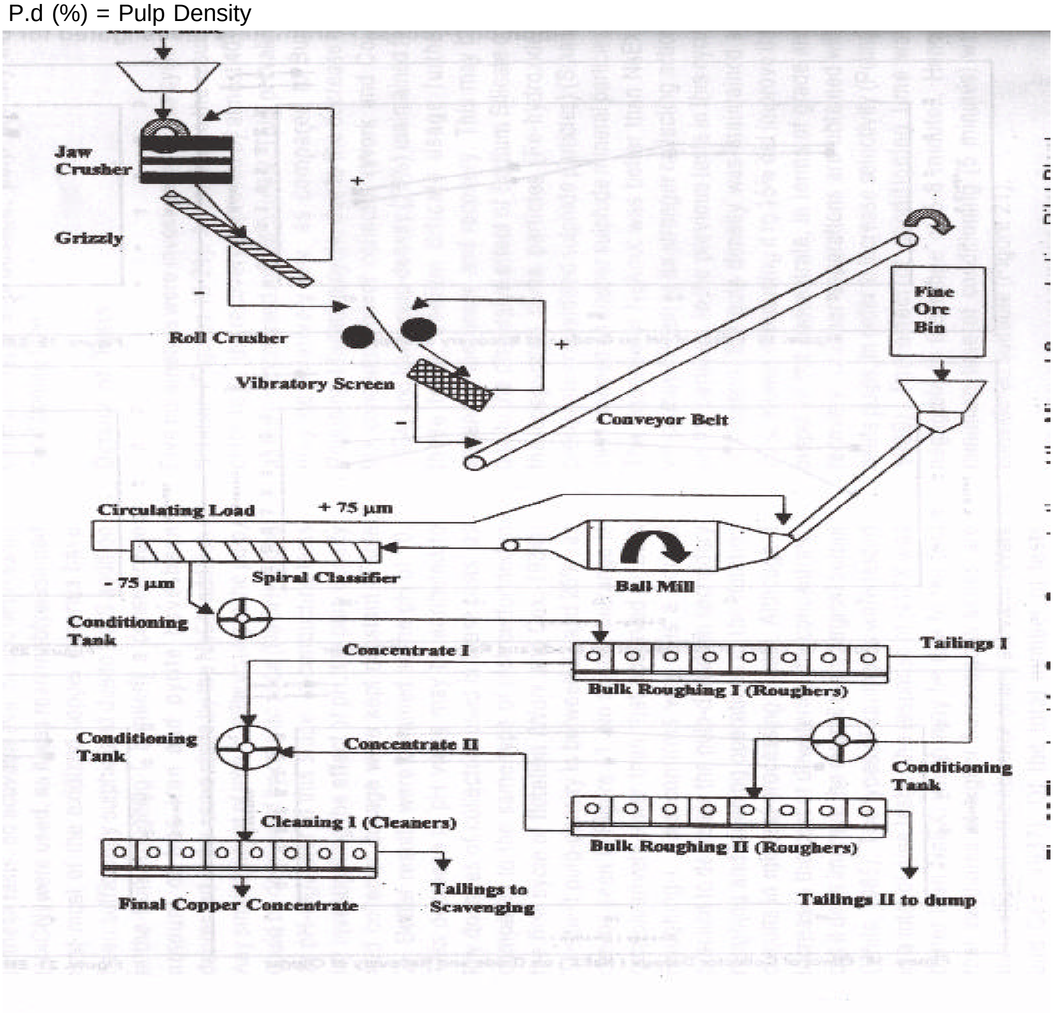

Fig. 22 Flowsheet for Copper Concentration at the Mineral Concentration Pilot Plant

SUMMARY AND CONCLUSIONS

The results obtained in these pilot-scale studies indicate that it is possible to achieve good separation of copper from the gangue material. The nature of copper-bearing minerals (such as azurite, malachite) and gangue is such that it has insignificant effect on the selective physico-chemical separation of major copper-bearing mineral (chalcopyrite). Taking into account the nature of the ore, the upgradation achieved is quite satisfactory. The collector-dosage needs to be investigated, to reduce its consumption, especially in the Bulk Roughing-I cycle. Moreover, to further reduce the cost of treatment, the effect of lime should be studied, in place of caustic soda, because of its lower cost. The grade of copper-concentrate obtained through single-stage cleaning is 22%. However, it can be further improved by incorporating supplementary cleaning-stages in the basic flowsheet, as is being practiced in copperflotation circuits internationally e.g. Afton Copper Concentrator in Canada (World Mining, 1978) and Pyhasalmi flotation circuit in Finland (Wills, 1983). A proposed flowsheet of copper concentrator prepared by the Department of Mining Engineering, after these pilot-scale studies, is presented in Figure 22.

The parameters established as a result of pilot-scale studies are summarised in Tables 2 & 3.

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