

BENEFICIATION OF PRECIOUS METALS BEARING IRON ORES OF CHILGHAZI, PAKISTAN

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الخلاصة :

تحتوي خامات حديد شيلغازي على كميات كبيرة من معادن النحاس ، والذهب ، والفضة ، والبلاتين . ولقد أوضحت الدراسات أن لكل من تمعدن الحديد والمعادن النفيسة أطوار خاصة ونشأة متميزة ، فمعدن الكالكوبيريت (كبريتيد النحاس والحديد) وهو المصدر الرئيسي للنحاس - في الخام المدروس - يتواجد كنسيج في أكسيد الحديد المغناطيسي (الماجنيتيت) . ولأن المعادن النفيسة توجد أساسا مصاحبة لمعدن الكالكوبيريت على هيئة إحلال له ؛ فإنه من الممكن الاستفادة بالخام لإنتاج الحديد بواسطة عملية فصل مغناطيسي بسيطة يتم خلالها أيضا تركيز كميات مناسبة من المعادن النفيسة في الشوائب المتبقية عن عملية الفصل المغناطيسي ، وهذه يمكن زيادة تركيزها بواسطة عملية التعويم أو بنزع الأملاح المعدنية منها (تصويل المعدن الخام) . ولقد وُجد أن عملية تعويم معدن الكالكوبيريت كانت أيضا ملائمة لاستخلاص المعادن النفيسة وتنظيف الركاز ، كذلك فقد تمت عملية تركيز شديدة عند نقطة التعادل في الترقيم الهيدروجيني باستخدام الزنثات في عملية غسل تراوح فيها الرقم الهيدروجيني من (٩) الى (١٠) ، وبذلك أمكن الحصول على ركازة بها (٢٢٪) نحاس و (٤٥,٨) جزء من المليون من الفضة و (٥,٣) جزء من المليون من ذهب و (٢١,٨) جزء من المليون من البلاتين .

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ABSTRACT

The iron ore of Chilghazi contains significant quantities of copper, gold, silver, and platinum. The mineralization of iron and precious metals show separate phases and paragenesis. Chalcopyrite, which is the main copper mineral, occurs as matrix mineral in magnetite; the precious metals were found to be associated mainly with chalcopyrite by substitution. The ore can be utilized for the production of iron after low intensity magnetic separation. Appreciable contents of precious metals, enriched in the gangue during magnetic separation, can be further concentrated by flotation and leaching. The flotation of chalcopyrite was also found suitable for the recovery of precious metals. More vigorous concentration at neutral pH, and cleaning at pH 9 to 10 using xanthate, gave a product containing 22% Cu, 45.8 ppm Ag, 5.3 ppm Au, and 21.8 ppm Pt.

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INTRODUCTION

Sinjirani volcanic rocks of andesitic composition, contain iron ore horizons in the Chilghazi area 51 km northeast of Dalbandin in Chagi District, Pakistan. The ore consists of magnetite, pyrite, chalcopyrite, epidote, quartz, calcite, and pyrophyllite minerals. The percentage of magnetite varies from 60 to 80% in solid bands with Fe (total) ranging from 32 to 55%. Total reserves for the solid bands were estimated to be 2.5 million tons of proved and 3.4 million tons of probable ore, with an average grade of about 45% Fe.

The ore, which is reported [1] to occur as a magmatic injection, has been shown to bear appreciable amounts of gold, silver, copper, and platinum. The objective of the present work is to find a suitable method of beneficiation, after determination of its chemical assay, mineralogical composition, texture, and physical characteristics. The grade and recovery of magnetite as well as those of chalcopyrite and precious metals concentrates under optimum conditions are studied at different stages.

ORE SAMPLE PREPARATION

The weights of the representative ore samples, received from the exploratory mines, were about 2 kg for samples number 1 and 2 and 10 kg for bulk sample number 3. The sieve analysis of the bulk ore showed that 93.33% material was retained on a 3.00 mm sieve.

The ore was subjected to crushing in Jaw and Roll crushers. A sample weighing 0.5 kg was ground in a Teema mill to about minus 180 microns size for mineral separation, mineralogical, and chemical studies. The product was sampled for chemical assay and further crushed to about 50 microns in a Teema mill.

MINERALOGY

The Chilghazi iron ore contains magnetite with lesser amounts of sulfide ore minerals and silicate gangue. The microscopic examination of rock samples showed coarse-grained gangue consisting of altered ferromagnesian minerals, quartz, epidote, andesine, and coarse-grained ore minerals including magnetite, hematite, pyrite, and pyrrhotite, (rarely

galena and sphalerite). The rocks appear to be altered by martization, sericitization, and propylitization. The characteristics of the individual ore minerals as found by ore-microscopy and by observation in stereomicroscope are as follows.

Magnetite

The magnetite occurs as dark brown mineral with a wide variety of texture ranging from fine-grained dendritic to spherulitic and granoblastic to massive with veining of gangue. The gangue in the magnetite, which generally consists of a mixture of ferromagnesian (grey) and felsic (black) minerals, is present as an interstitial matrix of 20–200 microns size and as inclusions (2–10 microns size). Within the recrystallized coarse-grained massive magnetite, the inclusions and interstitial gangue are reduced, probably due to expulsion out of grain boundaries. Hematite/martite is formed at the expense of magnetite as fine-grained or replacement martite texture. The grain size of magnetite is generally 100–200 microns with inclusion of 2–10 microns consisting of mafic and felsic gangue. The inclusion rarely contain sulfide minerals.

Chalcopyrite

The grain size of chalcopyrite is extremely variable ranging from 5 to 400 microns. It is generally present as flakes of 100–200 microns and as disseminations of 5 to 10 micron size. It is recognized by its deep yellow color and reflectance.

Pyrite/Pyrrhotite

Pyrite is an important gangue mineral present as disseminations (10–50 microns) commonly filling veins in the rocks. It is generally found as small anhedral crystals but sometimes shows a cubic shape. Its color varies from yellow to light yellow. At a few places it appears to have an alteration relationship with pyrrhotite which occurs as anhedral crystals having a light brown to bronze color.

CHEMICAL ANALYSIS

The results of chemical analysis of the ore samples are given in Table 1. Major elements were determined by standard chemical methods of mineral analysis [2, 3]. For the determination of Cu, Au, and Ag, a

Table 1. Results of Chemical Analysis of Chilghazi Ore.

Constituents	Samples		
	1	2	3
Fe%	43.80	41.50	49.10
SiO ₂ %	14.20	13.80	13.20
Al ₂ O ₃ %	13.50	09.50	10.10
TiO ₂ %	00.80	00.70	00.60
CaO%	03.90	07.50	05.50
MgO%	02.10	01.70	01.10
P ₂ O ₅ %	00.50	00.30	00.50
Cu%	00.49	00.42	00.90
Au (ppm)	00.10	00.25	00.19
Ag (ppm)	02.00	03.10	01.50
Pt (ppm)	01.50	01.70	01.50

10 gm sample was taken in a teflon beaker, 20 ml hydrofluoric and 10 ml perchloric acid was added and the beaker heated to dryness on a hot plate. 10 ml nitric acid and 30 ml concentrated hydrochloric acid was added and the sample heated to near dryness without baking. The solution was diluted with 20% hydrochloric acid in a 100 ml flask, and analyzed for copper, silver, and gold using an atomic absorption spectrometer (Hitachi Model z-8000). Platinum was determined using the method of Stanton and Ramankutti [4]. A 25 g sample was mixed with 100 ml bromine solution and kept overnight. Excess bromine was boiled off and the material diluted to 250 ml with water. A portion of clear solution was taken after settling and subjected to solvent extraction with Methyl Isobutyl Ketone (MIBK) to remove Au and Te. A 0.25 ml solution of SnCl₂ (50%) was added and extraction carried out for Pt with MIBK. The extract was washed with 25 ml of 2M HBr and platinum analyzed using the atomic absorption technique.

GRINDABILITY STUDIES

The ore was passed through a jaw crusher for crushing and the product fed through a set of rollers. The roll product was ground in the ball mill for various time periods. The grinding was done in the dry condition and the results of the sieve analyses for different grinding stages are plotted in Figure 1. Grinding of a batch of 400 gram ore for 10 minutes in a ball mill was found to be optimum, yielding approximately 80% material passing 120 microns. This material was found suitable for subsequent beneficiation tests.

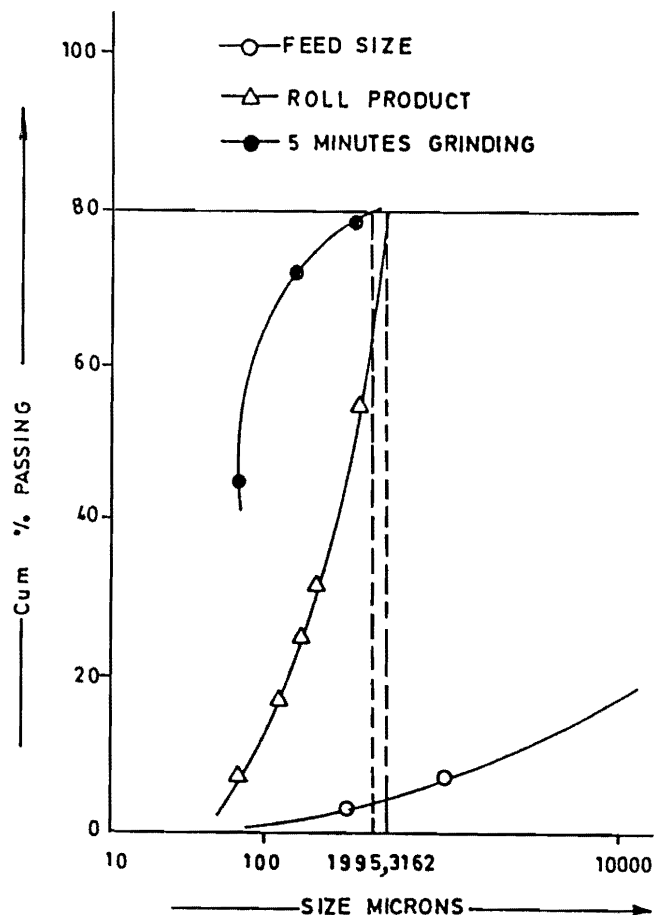


Figure 1. Particle Size Distribution on Crushing and Grinding.

ORE BENEFICIATION STUDIES

Magnetic Separation

Beneficiation tests in different trials have shown that Chilghazi iron ore can be beneficiated by magnetic separation. Despite the partial martization of the magnetite, the nature and texture of the ore responded favorably to magnetic separation. The behavior of the bulk ore No. 3 was similar to a certain extent to magnetite ore in concentration circuits. The samples No. 1 and 2, yielded slightly smaller recovery due to some alteration in magnetite grains (Table 2).

Table 2. Results of Magnetic Separation.

Samples	Assay of cleaner concentrate		Recovery %	
	Fe%	SiO ₂ %	Fe%	SiO ₂ %
1	61.53	5.5	89.6	16.8
2	66.80	2.5	86.1	21.5
3	65.1	3.5	96.9	16.8

The ore invariably showed micro inclusions of gangue minerals in magnetite grains. The size of micro inclusions (10–20 microns) was below the practical limit of mineral beneficiation. The matrix gangue mineral was found to be separable from the bulk magnetite by low intensity magnetic separation method and can be an economic by-product containing appreciable amounts of copper, gold, silver, and platinum.

Davis Tube Tests

Different batches of the bulk ore (sample 3) were ground and passed through Davis tubes. The results, listed in Table 3, show that at coarse mesh size, the weight percent recovery of magnetite particles in the Davis tube is low (65.09%). This is due to the presence of gangue-dominated locked particles, as can be seen under the stereomicroscope. On further grinding the weight percentages of the magnetic fraction are increased due to the liberation of magnetite and magnetite-dominated locked particles (Table 3).

The product obtained by magnetic separation were chemically analyzed. The chemical composition and recovery of different precious metals on magnetic concentrate and non-magnetic fractions are given in Table 4. It can be noted that the recovery percentages of Cu and precious metals are higher at minus 125 micron size, which could be a suitable

Table 3. Results of Davis Tube Test of Sample 3.

Sieve Sizes (Microns)	Magnetics (%)	Non-Magnetics (%)
1000	65.09	34.91
420	90.49	9.51
251	84.21	15.79
177	76.46	24.54
125	74.00	26.00
105	86.38	13.62
75	83.23	16.77

grind size for rougher concentration. The enrichment for this size fraction is of the order of three times in both Cu and Pt and slightly greater for the other metals. Although 7.0 ppm Ag and 7.0 ppm Pt were present in the 105 micron size non-magnetic fraction, the recovery was lower. It is, therefore, suggested to produce rougher concentrate at coarser mesh (150–125 microns size) and reclean the non-magnetics to obtain high grade concentrates with better recoveries of precious metals by magnetic separation. The deviation in the total sums of recoveries from 100% may be due to losses of precious metals in magnetic separation and also to analytical errors, as discussed under sampling.

Flotation Experiments

A Denver flotation machine model D.12, with impeller speed set at 1100 to 1200 rpm, was used for

Table 4. Composition and Recovery of Copper and Precious Metals in Magnetic and Non-Magnetic Fractions in Chilghazi Ore Sample No. 3.

	Weight (percent)	Silver		Gold		Platinum		Copper	
		ppm	Recovery (percent)	ppm	Recovery (percent)	ppm	Recovery (percent)	ppm	Recovery (percent)
Head Sample	100.00	01.50	100.00	00.19	100.00	01.50	100.00	00.90	100.00
Sieve fraction (251 microns)									
Magnetic	84.20	00.90	50.50	00.13	57.60	00.90	50.30	00.47	43.90
Non-Magnetic	15.80	03.80	40.00	0.45	42.40	04.20	49.70	03.20	56.60
Sieve fraction (125 microns)									
Magnetic	74.00	00.40	08.50	00.05	17.90	00.50	24.00	00.30	25.70
Non-Magnetic	26.00	05.50	95.30	00.70	82.10	04.50	76.00	02.60	66.40
Sieve fraction (105 microns)									
Magnetic	86.40	00.70	40.30	00.10	56.00	00.60	28.40	00.76	72.80
Non-Magnetic	13.60	07.00	63.50	00.50	44.00	07.00	71.60	02.10	31.70

flotation tests. The feed to flotation was the non-magnetic fraction obtained after the magnetic separation of the magnetite from the deslimed ore in the low intensity magnetic separator. The grind size 80% passing 125 microns, yielded 26% non-magnetic and 74% magnetic fraction in magnetic separator (Table 4). The pH during rougher concentration was neutral but it was changed to the basic range with the addition of the lime in varying amounts for cleaner concentration. The feed was conditioned for two minutes with potassium xanthate as collector and flotation was commenced after adding one drop of the frother. The froth was removed for ten minutes as a concentrate. The pH of the pulp was adjusted between 9 to 10 with the addition of the lime after roughing and more concentrate was obtained by scraping for an additional 5 minutes without further addition of any reagent such as xanthate. Ordinary tap water was used in these tests. The concentrate and tailings were decanted and dried in the oven at 105°C.

The pH regulators were used to modify the mineral surface and create a favorable condition in the pulp for successful flotation. First flotation at neutral pH was undertaken in order to recover the maximum amounts of sulfide and associated precious metals. Cleaning of the concentrate was later carried out at higher pH using indigenous low grade lime (CaO), so as to obtain maximum recovery of chalcopyrite. Au and Ag values and to lesser extent Pt values tended to report in the copper concentrate. The results of flotation are summarized in Table 5.

The heap sample No. 3 weighing about one kg ore was assayed as 0.9% Cu, 1.5 ppm Ag, 0.19 ppm Au, and 1.5 ppm Pt. The magnetic separation of this sample yielded about 0.25 kg of non-magnetic fraction, containing 2.6% Cu, 5.5 ppm Ag, 0.7 ppm Au,

and 4.5 ppm Pt. The cleaner concentrate obtained after flotation of the magnetic fraction assayed 22.0% Cu, 45.8 ppm Ag, 5.3 ppm Au and 21.8 ppm Pt. These results are encouraging as the grades of copper and the precious metals were obtained in a single cleaning. It is evident from the chemical analyses of the products that the precious metals have accumulated in the sulfide concentrate. These metals appear to be associated with sulfide minerals probably as a result of substitution. The fact that most of the silver and gold values can be recovered by flotation with xanthate support this observation. However, the platinum, which appears to have different paragenesis, showed lower recovery in flotation experiments.

The recoveries obtained for Cu, Ag, Au, and Pt in the concentrate were 86.0, 84.6, 76.9, and 49.2% respectively. The overall feed recoveries on the feed basis for both magnetic and flotation processes with one cleaning stage were 64.6% for Cu, 80.7% for Ag, 73.7% for Au, and 38.4% for Pt.

In conclusion it may be observed that the cleaning operation, especially with such rougher concentrate, produces a concentrate of reasonable grade with good recoveries. The concentrate produced is a mixture of low and high grade ore particles. Grinding of the cleaner concentrate is, therefore, essential to liberate chalcopyrite from gangue and regrinding of the cleaner concentrate to 80% 105 micron size is indicated by microscopic particle counting.

Leaching

In earlier years of the gold cyanidation process, it was a common practice to grind the ore with little or no concentration before cyanidation. With the advent of flotation and the fact that gold either free or finely disseminated in sulfide could be floated,

Table 5. Distribution of Copper, Gold, Silver, and Platinum in Different Flotation Products of Non-Magnetic Fraction of Ore Sample No. 3.

	Weight (grams)	Copper		Silver		Gold		Platinum	
		ppm	Recovery (percent)	ppm	Recovery (percent)	ppm	Recovery (percent)	ppm	Recovery (percent)
Ore	961.00	0.9	—	1.5	—	0.19	—	1.5	—
Head (Non-Magnetic)	250.00	2.6	100.00	5.5	100.00	0.70	100.00	4.5	100.00
Cleaner Concentration	25.40	22.00	86.00	45.8	84.60	5.30	76.90	21.80	49.20
Recovery Ore Basis	—	—	64.60	—	80.70	—	73.70	—	38.40
1st Tail	193.00	00.35	10.40	00.40	5.60	00.16	17.60	02.40	41.10
2nd Tail	10.30	01.30	2.10	05.00	3.70	00.50	2.90	03.50	3.20

Table 6. Effect of Sulfuric Acid Concentration on Leaching.

Sample Number	Concentration of Acid (100 ml)		Copper		Silver		Gold		Platinum	
			ppm	Recovery (percent)	ppm	Recovery (percent)	ppm	Recovery (percent)	ppm	Recovery (percent)
1	1.00%	100 m	0.65	72.00	00.00	00.00	00.05	26.00	0.60	40.00
2	2.50%	100 m	0.75	83.00	0.2	13.00	0.06	31.00	0.20	13.00
3	5.00%	100 m	0.90	100.00	0.8	53.00	0.02	10.00	0.30	20.00
4	7.50%	100 m	0.80	89.00	1.2	80.00	0.02	10.00	0.40	27.00
5	10.00%	100 m	0.88	98.00	1.2	80.00	0.03	16.00	0.80	53.00
6	Total extraction method		0.90	100.00	1.5	100.00	0.19	100.00	1.5	100.00

Ore size: 150 microns; Time of stirring: 30 minutes; Final volume made: 500 ml

many possibilities opened up for the treatment and concentration of gold bearing ores. It, also, opened up the possibilities for ores not amenable to direct cyanidation. The flotation concentrate from Chilghazi ore can be utilized for the extraction of gold by cyanidation or alternatively it may be treated in a smelter (if large scale operation is feasible). Experiments using 5 g of ore and one liter of 0.05% cyanide solution on agitation resulted in a recovery of 75% of gold in solution.

Experiments on extraction of copper by leaching from Chilghazi ore using sulfuric acid were, also, conducted. The results of these experiments listed in Table 6. The original ore containing 0.9% Cu was treated with sulfuric acid of different concentration as well as nitric acid. After stirring, the material was kept for 24 hours and then decanted and filtered.

It can be seen from these experiments that while copper and silver values can be recovered by sulfuric

acid leaching, most of the precious metals remain undissolved, making their recovery difficult. Sulfuric acid leaching is generally recommended for oxidized copper ores. However, nitric acid addition is required to oxidize the sulfide minerals.

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