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Flotation Study on Scheelite Ore of Chitral, Khyber Pakhtoonkhwa, Pakistan

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Abstract: The beneficiation of a siliceous scheelite ore of Chitral, Khyber Pakhtoonkhwa, Pakistan, was investigated by flotation process to determine the optimum conditions for obtaining maximum grade and recovery of the scheelite concentrate. The variables studied were including pH and pulp density maintained during conditioning, type and quantity of reagents added, conditioning time and froth collecting time. The results achieved were plotted against the grade and recovery of the rougher concentrate. Several methods of cleaning rougher concentrate were attempted. An ore initially containing 0.34% WO_3 was upgraded into a final concentrate assaying 64.66% WO_3 with 70.36% recovery. The tungsten concentrate obtained meets the specifications required to produce ferrotungsten and tungsten chemicals.

Keywords: tungsten ore, scheelite mineral, froth flotation industrial application

Introduction

Tungsten is a shiny white, and highly refractory metal having highest melting point (3422 °C) of all metals. It has good corrosion resistance, good thermal and electrical conductivity. It has excellent high temperature mechanical properties and above 1650 °C it has the highest tensile strength of all metals. It also has the lowest expansion coefficient (4.6 ppm/ °C) of all metals at 25 °C. With a density of 19.25 g/cm^3, tungsten is also among the heaviest metals. Because of its distinct properties it has many commercial, industrial, and military applications (Shedd, 2011).

Tungsten never occurs as a free element in nature. It occurs only in the form of combined state with other elements. The abundance of tungsten in the earth's crust is thought to be 1.5 parts per million (Foster, 1988). It is relatively a rare element. Although, more than twenty tungsten bearing minerals are known, but only two of them are important for industrial use, namely wolframite (Fe, Mn) WO_4 and scheelite $CaWO_4$. All tungsten deposits are of magmatic or hydrothermal origin (Blackburn and Denner, 1988).

Economically important tungsten ore deposits have been found at Mineki Gol and adjoining area, near Garam Chasma, about 60 km to the north of Chitral city, in Khyber Pakhtoonkhwa province of Pakistan. The deposits contain mainly scheelite mineral, which occurs predominantly in quartz rich metasedimentary rocks (Leake *et al,*.1989). The deposit consists of a number of ore bodies. The main rocks of Mineki Gol area are characterised by quartz, mica schist, marble, tourmaline, calcsilicate quartzite, phyllite and leucogranite. The indicated reserves of tungsten ore of Chitral have been estimated to be 1.8 million tonnes (Khan *et al.,* 2003). Beneficiation of a Chitral scheelite ore has been studied at Mineral Processing Laboratory, PCSIR, Lahore, Pakistan. The investigation was conducted in the field of flotation in order to establish the effect of certain variables on grade and recovery of rougher concentrate according to the traditional single-factor experimentation. The results of this investigation are recorded and a flowsheet has been developed including two stage grindings and three froth flotation operations. This paper presents results on the optimisation of flotation parameters for the preparation of metallurgical grade tungsten concentrate.

Materials and Methods

Sample preparation. The samples of tungsten ore were collected from various localities of Mineki Gol area. The representative sample of the ore was crushed through jaw crusher followed by rolls crusher to a product of 5-6 mm. Coning-quartering and riffling techniques of sampling were done to prepare head sample for chemical and mineralogical studies, while the remaining ore was used for processing studies. Head sample was pulverised

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to pass through 200 mesh sieve (74 µm) with a disc pulveriser (Denver, USA).

Chemical analysis. The chemical analysis of head sample and processed products was performed in accordance with ASTM methods. Tungsten was determined by spectrophotometer (Model: Spectronic-20, England). Sodium and potassium were determined by flame photometer (Model: PFP7, Jenway, England), while other constituents were estimated by atomic absorption spectrophotometer (Model: Z-8000, Hitachi, Japan). Silica was estimated gravimetrically.

Mineralogy. The pulverised ore sample and tungsten concentrate obtained were run on X-ray diffractometer (Model: D-5000, Siemens, Germany) for phase determination. X-ray diffraction was done with a 0.02° step size per second. The scan angle was ranged from 0-60° to get the peaks of different minerals present in it. The spectrum obtained was matched with standard data to identify the mineral phases.

Degree of liberation. The degree of liberation of scheelite mineral was measured by grain mounts. The roll crushed ore was ground in a rod mill for 15 min and separated into different size fractions using ASTM sieves. The grain mount of each fraction was prepared by putting some sample in one drop of glycerin on a glass slide and covering with a cover slip. These were analysed under the optical microscope using cross nickel at a magnification of 40×. The number of liberated and locked mineral grains in each sieve fraction were noted and the % age liberation of scheelite was calculated.

Flotation tests. The flotation tests were conducted in a flotation machine (Model: D-12, Denver, USA). The ore was carefully stage crushed through 4-mesh sieve and then split into approximately 1 kg samples. Each sample was wet ground in a rod mill (Denver, USA) with 1:1 solid to liquid ratio. The grinding time was adjusted according to the required size of feed. The ground ore was transferred to the stainless steel flotation cells of 4 L capacity and diluted with water to maintain the desired pulp density. The agitation of the flotation cell was controlled with knob. The pH of pulp was adjusted by means of sodium carbonate modifier and measurement of pH was made by means of Hanna digital pH meter. In all tests a graphite float was made to remove it from ore at natural pH of the pulp, prior to rougher scheelite flotation. The pulp was conditioned with reagents at room temperature (~25 °C) for several series of scheelite floats. Air was admitted in the test cells and the froth was collected until mineralisation ceased (barren).

Set of conditions. The study was completed under different sets of conditions for the optimisation of flotation parameters. The sets of conditions were as follows: grind size from 60 to ~100 % minus 200 mesh, pH of pulp from 8-11, pulp density from 20-40 % solids, agita- tion of the pulp from 800-1200 rpm, sodium oleate 200-1000 g/t) as collector, sodium silicate (600-1200 g/t) as depressant and polyglycol (20-60 g/t) as frother. The conditioning time was 2-10 min and the froth was collected for 5-25 min. In all tests a scavenging flotation was conducted and scavenger concentrate was mixed along with rougher concentrate. The rougher concentrates were reground and two cleaning flotation operations were carried out with addition of flotation reagents. Chemical analyses of the dried products were made by the colorimetric method. Finally, locked cycle tests were performed by circulating the cleaner tailings, according to their grades, at appropriate point of addition.

Results and Discussion

Chemical analysis. The complete chemical analysis of the representative sample of ore and final concentrate is presented in Table 1, which indicates the presence of 0.34 % WO_3 content in the tungsten ore of Chitral area of Pakistan. The obtained grade is sufficient to exploit the ore on commercial scale to produce tungsten concentrate (Pandey *et al.,* 2001; Srivastava and Pathak, 2000). However, the presence of silica, aluminum oxide, iron oxide, calcium oxide and graphite appeared to be the main impurities. In order to remove these impurities, froth flotation method was applied. The chemical analysis of the final tungsten concentrate as shown in Table 1, confirms the presence of 64.66% WO_3, 15.33%

Table 1. Chemical analysis of tungsten ore and final scheelite concentrate

Constituents	Ore (%)	Concentrate (%)
WO_3	0.34	64.66
SiO_2	65.53	11.48
Al_2O_3	11.67	2.54
Fe_2O_3	5.65	2.43
CaO	10.43	15.33
MgO	0.54	0.13
Na_2O	0.96	1.06
K_2O	0.38	0.05
C	2.12	0.10

CaO and 11.48% SiO_2 as major constituents with minor amount of iron oxide and aluminum oxide as impurities. Thus flotation process has been proved successful to reduce the impurities level of the ore significantly. The final tungsten concentrate obtained meets the specifications required to produce tungsten metal, ferrotungsten and tungsten based chemicals (Guar, 2006).

Mineralogy. The X-ray diffractrograms of the ore and final concentrate presenting the list of the significant minerals recognised are shown in Fig. 1- 2, respectively. X-ray diffractrogram of the ore (Fig.1) confirms the presence of reasonable amount of scheelite mineral. The gangue minerals identified comprised of mainly calcite, quartz, epidote, magnetite, muscovite, hydrobiotite, sapphirine and graphite. XRD of final tungsten concentrate (Fig. 2) shows the presence of significant amount of scheelite ($CaWO_3$) as major peaks correspond to standard peaks of scheelite.

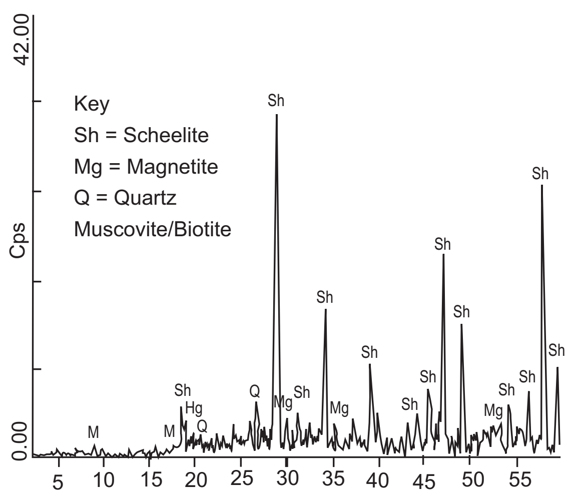

Fig. 1. XRD pattern of ore.

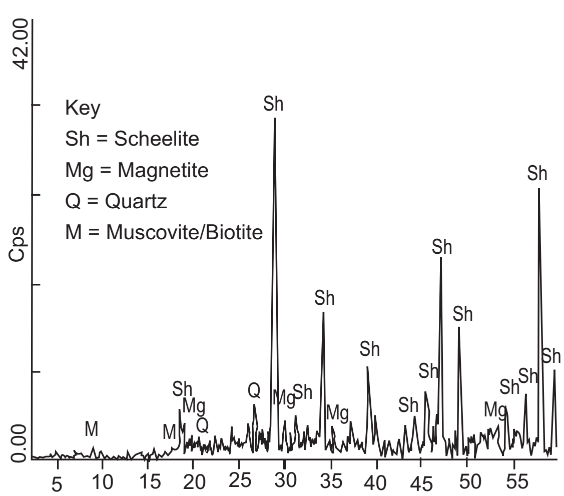

Fig. 2. XRD pattern of concentrate.

Degree of liberation. About 50 particles were counted for each slide. The data expressing the liberation summary of the size fractions is shown in Table 2. It shows that liberation increases with a reduction in particle size and about 94 % scheelite grains are liberated, when the ore is ground to minus 200 mesh (74 µm). The degree of liberation of economic mineral in new ore is determined to estimate the required grind size. Practically, ore was ground to a size, where the economic factor of the process is maximised and particle size is never reduced to achieve 100 % liberation (King, 1994). Therefore, in order to have a sufficient proportion of liberated scheelite grains with minimum level of locked particles, this ore should be ground at least 80% minus 200 mesh size.

Table 2. Mesh of liberation of scheelite mineral in Chitral tungsten ore

Size fraction (ASTM)	Free scheelite grains (%)	Locked scheelite grains (%)	Free gangue grains(%)	Scheelite liberation (%)
-50+70	3.63	8.39	87.98	32.20
-70+100	4.67	4.38	90.95	51.57
-100+150	4.93	2.20	92.77	69.10
-150+200	5.56	1.01	93.43	84.63
-200+250	5.61	0.37	94.02	93.81
-250+300	5.65	0.25	94.10	95.78
-300	5.66	0.18	94.36	96.87

Effect of grind size. In first series of tests, only grind size was varied while keeping other variables constant at arbitrarily chosen values. The metallurgical data regarding particle size *versus* grade and recovery (Fig. 3) shows that, both grade and recovery of rougher concentrate were improved by decreasing the particle size of the ore and maximum recovery was achieved at feed size of 80% passing 200 mesh (74 µm). Recent trend in plant practices is to grind the ore as coarsely as possible without sacrificing rougher recovery. The rougher concentrate then requires regrinding for adequate mineral liberation prior to cleaner flotation (Adams, 1986). Thus, this feed size was considered as optimum and selected for onward investigation.

Effect of pulp density. The effect of pulp density during conditioning (Fig. 4) shows that an increase in pulp density from 20% to 35% first increases and then decreases the grade of the rougher concentrate but improves the recovery gradually up to a certain point. These effects were most pronounced when the pulp was

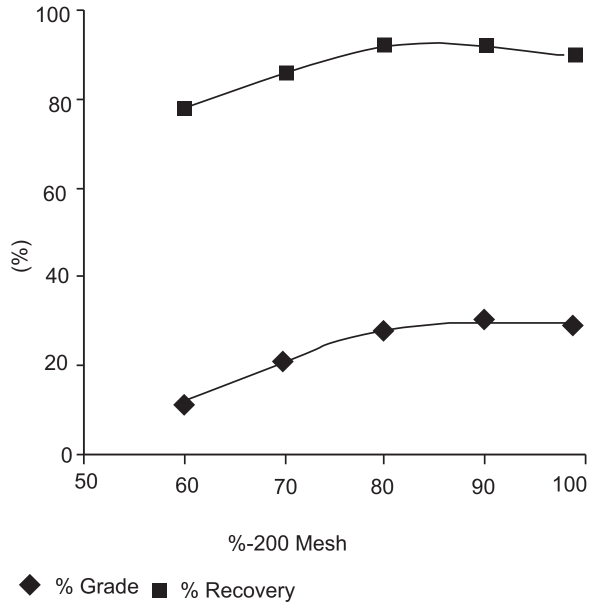

Fig.3. Effect of grind size on grade and recovery of rougher concentrate. Conditions**:** pH of pulp: 10.5, pulp density: 25%, impeller speed: 1000 rpm, sodium oleate: 500 g/t, polypropylene glycol: 50 g/t, sodium silicate: 1000 g/t, conditioning time: 5 min and flotation time: 15 min.

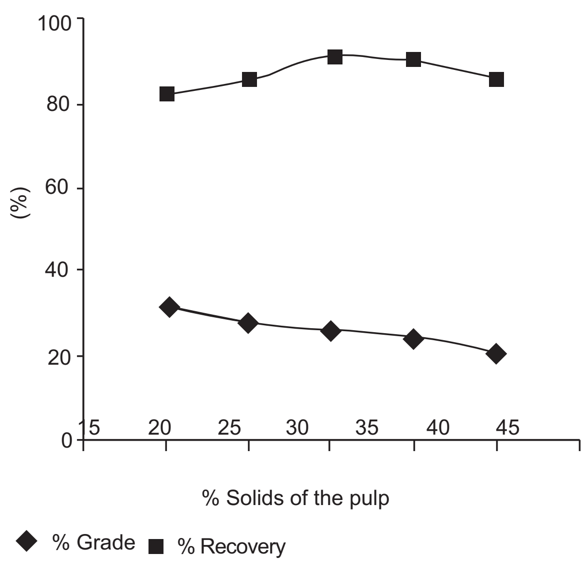

Fig. 4. Effect of pulp density on grade and recovery of rougher concentrate. Conditions**:** Grind size: 80%-200 mesh, pH of pulp: 10.5, impeller speed: 1000 rpm, sodium oleate: 500 g/t, polypropylene glycol: 50 g/t, sodium silicate: 1000 g/t, conditioning time: 5 min and flotation time: 15 min.

conditioned at 30% solids. The high pulp density inhibits proper air dispersion and good bubble formation thereby adversely affecting the recovery. As at rougher stage emphasis is given on higher recovery rather than on high grade of the concentrate, therefore, this value was selected for the remaining study. Generally, higher (upper limit) pulp densities are acceptable for heavier ores and our results also confirmed the same (Weiss, 1985).

Effect of pH of pulp. Figure 5 indicates the effect of pH variation of the pulp during conditioning on the grade and recovery of the concentrate. It is evident from this figure that under constant conditions of feed size and % solids, both the grade and recovery increase with increase in pH from 8 to 10 and then decrease. It was observed that the maximum grade and recovery was obtained at pH 10. One can conclude from these results that pH is critical when floating scheelite ore. A small variation in pH shows a significant change in grade and recovery. This effect is attributed to the maximum stability of collector-mineral complex at specific pH (Wills, 1992).

Effect of agitation speed. The results regarding the effect of agitation speed (aeration) on grade and recovery of rougher concentrate (Fig. 6) show that an impeller

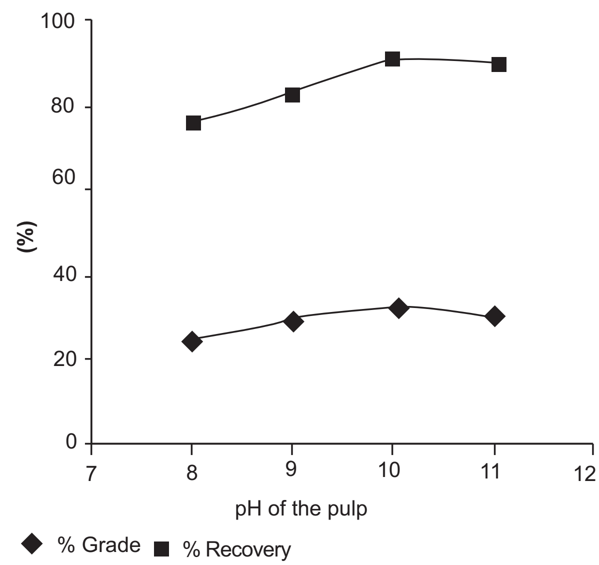

Fig. 5. Effect of pH on grade and recovery of rougher concentrate. Conditions: Grind size: 80%-200 mesh, pulp density: 30%, impeller speed: 1000 rpm, sodium oleate: 500 g/t, polypropylene glycol: 50 g/t, sodium silicate: 1000 g/t, conditioning time: 5 min and flotation time: 15 min

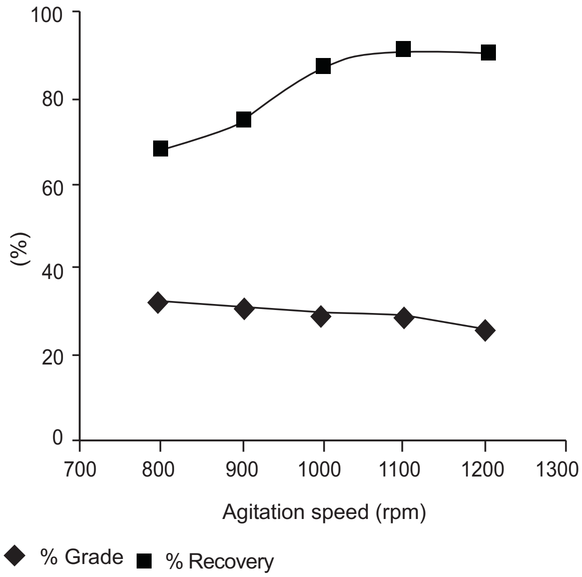

Fig. 6. Effect of agitation speed (aeration) on grade and recovery of rougher concentrate. Conditions: Grind size: 80%-200 mesh, pulp density: 30%, sodium oleate: 500 g/t, polypropylene glycol: 50 g/t, sodium silicate: 1000 g/t, conditioning time: 5 min and flotation time: 15 min.

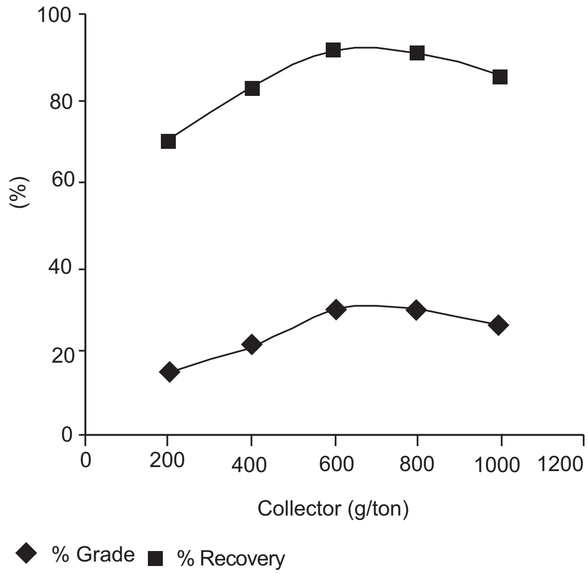

Fig. 7. Effect of collector on grade recovery of rougher concentrate. Conditions**:** Grind size: 80%-200 mesh, pulp density: 30%, pH of pulp: 10, impeller speed: 1100 rpm, polypropylene glycol: 50 g/t, sodium silicate: 1000 g/t, conditioning time: 5 min and flotation time: 15 min.

speed of around 1100 rpm gave better metallurgical performance at rougher stage. At lower speed, the recovery decreases but grade is improved slightly. It is desirable to have a higher recovery at rougher stage rather than grade so this value was selected. While, at cleaning stage, a lower aeration speed of 900 rpm was found to improve the grade. This is due to the fact that after regrinding, the particle size is further reduced which favours lower impeller speed.

Effect of collector. Sodium oleate and sodium petroleum sulphonate were attempted as scheelite collector. Sodium oleate produces better metallurgical results in terms of grade and recovery and hence, utilised for scheelite flotation. It is obvious from Fig. 7 that an increase in the quantity of collector (sodium oleate) from 200 g/t to 600 g/t proportionally increases the grade and recovery. Further increase in dose of sodium oleate (after starvation level) shows an adverse effect on grade and recovery. It is due to over oiling effect of the collector that reduces the selectivity by the development of multilayers on the mineral particles (Crozier, 1992).

Effect of frother. Polyglycol, heavy fuel oil, and pine oil were tried as frother. Polyglycol yields comparatively higher results as a frother because of its high wetting properties (Wills, 1992). It is apparent from the results obtained using different quantities of frother (Fig. 8) that a dosage of 50 g/t of frother gives a reasonable grade and recovery and addition greater than this value shows no significant improvement in this regard. It has also been observed that a slightly increased rougher concentrate grade could be obtained at essentially the same recovery by spraying the froth with water.

Effect of depressant. The effects of sodium silicate and sodium polyphosphate on the floatability of the scheelite mineral with sodium oleate as collector were investigated. It was found that the sodium silicate (Na_2SiO_3) was more effective gangue regulator for the flotation of scheelite (Rao, 1996). The results obtained using different quantities of depressant (Fig. 9) indicate that with increase in amount of the depressant, the grade was improved first, but after 1200 g/ton, the grade improved a little but the recovery decreased. It can be explained that sodium silicate efficiently depresses the quartz and silicates by using moderate amounts of it as a gangue depressant and slime dispersant. Excessive amount of depressant depresses the middling particles thus decreases the recovery (Martins and Amarante, 2013). It was found that addition of small amount of

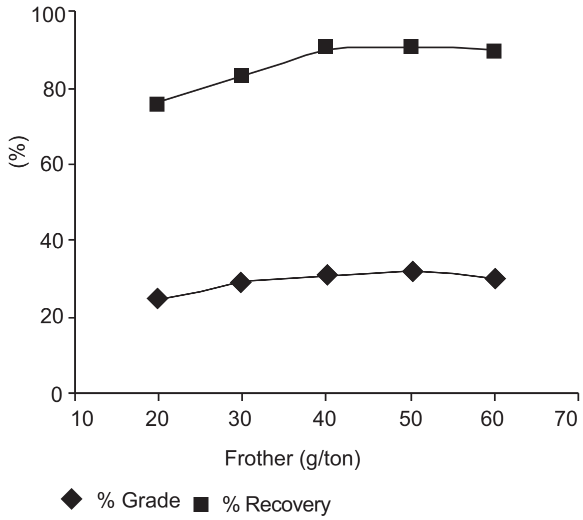

Fig. 8. Effect of frother on grade and recovery of rougher concentrate. Conditions: Grind size: 80%-200 mesh, pH of pulp: 10, pulp density: 30%, impeller speed: 1100 rpm, sodium oleate: 600 g/t, sodium silicate: 1000 g/t, conditioning time: 5 min and flotation time: 15 min.

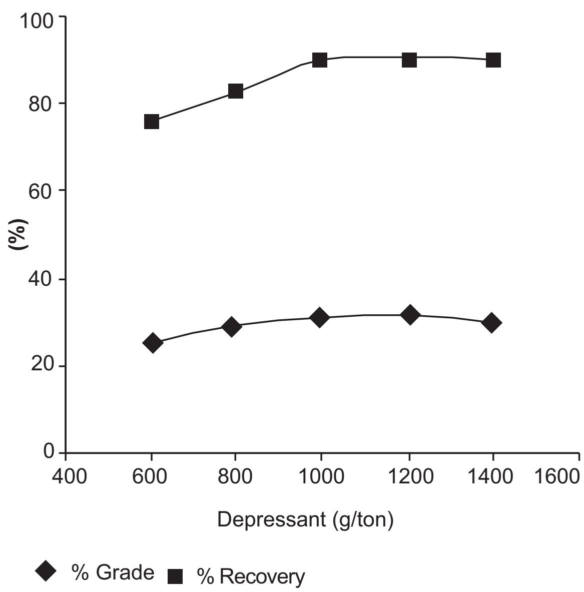

Fig. 9. Effect of depressant on grade and recovery of rougher concentrate. Conditions**:** Grind size: 80%-200 mesh, pH of pulp: 10, pulp density: 30%, impeller speed: 1100 rpm, sodium oleate: 600 g/t, polypropylene glycol: 50 g/t, conditioning time: 5 min and flotation time: 15 min.

quebracho (20 g/ton) in the cleaner circuits improves the metallurgy by depressing the calcite.

Effect of conditioning time. It appears from the results for different time intervals shown in Fig. 10 that a conditioning time of 6 min is sufficient for contact with mineral particles under the conditions existing in the flotation cell, for an optimum grade and recovery. Conditioning period greater than 6 min caused rapid decrease in grade but recovery remained relatively constant. It seems that prolonged conditioning time peels off the collector coating on mineral particles resulting in lower flotation grade and recovery.

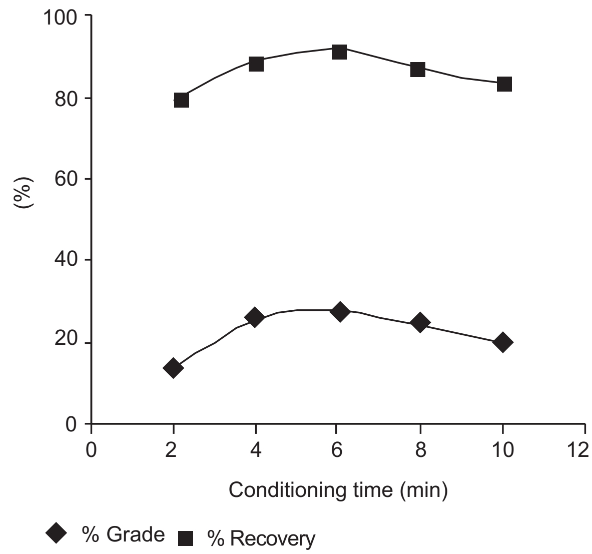

Fig. 10. Effect of conditioning time on grade and recovery of rougher concentrate. Conditions**:** Grind size: 80%-200 mesh, pH of pulp: 10, pulp density: 30%, impeller speed: 1100 rpm, sodium oleate: 600 g/t, polypropylene glycol: 50 g/t and sodium silicate: 1200 g/t, and flotation time: 15 min.

Effect of flotation time. It has been found from the results (Fig. 11) of different froth collecting time that a flotation time of 20 min is enough to completely barren the froth and further time has no significant influence on the resulting metallurgy.

Effect of cleaning and re-cleaning. The optimised conditions of flotation parameters including type and quantities of reagents as well as their order of addition are presented in Table 3. The cleaning of rougher concentrate by flotation was found to be extremely difficult because of the tenacious collector coating on both scheelite and middling material. In order to overcome this problem, the rougher concentrate was

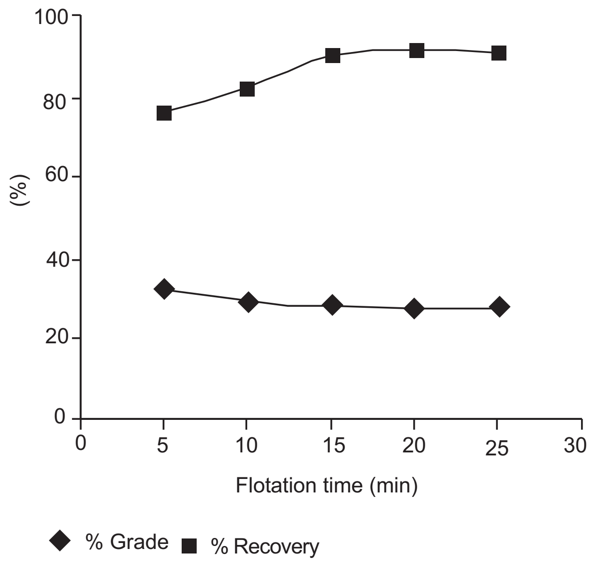

Fig. 11. Effect of flotation time on grade and recovery of rougher concentrate. Conditions: Grind size: 80%-200 mesh, pH of pulp: 10, pulp density: 30%, impeller speed: 1100 rpm, sodium oleate: 600 g/t, polypropylene glycol: 50 g/t and sodium silicate: 1200 g/t and conditioning time: 6 min.

Table 3. Optimum conditions of scheelite flotation

Parameters	Optimum conditions		
	Roughing	Cleaning	Re-cleaning
Feed size	~ 80%-200 #	~ 99% -200 #	~ 99% - 200 #
Pulp density	30 % solids	20% solids	20% solids
Agitation speed (Aeration)	1100 rpm	900 rpm	900 rpm
Pulp pH	~10	~10	~10
Collector (Sodium oleate)	600 g/t	500 g/t	Nil
Frother (Polypropylene glycol)	50 g/t	50 g/t	Nil
Depressant (Sodium silicate)	1200 g/t	400 g/t	Nil
Depressant (Quebracho)	Nil	20 g/t	Nil
Conditioning time	6 min	6 min	6 min
Flotation time	20 min	20 min	20 min

heated to destroy the coating of fatty acid collector. After that it was re-ground and cleaned by two cleaning operations to get the final concentrate. One cleaning operation on the rougher concentrate using additional quantity of reagents followed by another recleaning, without use of any additional reagents, make it possible to give a high-quality tungsten concentrate. Two stage grinding, first at coarse size and second at fine size make the process more economical by reducing the cost of grinding. It also helps in the achievement of higher grade final concentrate.

Metallurgical balance sheet. The metallurgical balance of the typical tests showing product distribution, grade of intermediate and final products is given in Table 4, which indicates that scheelite ore of Chitral containing 0.34% WO_3, can be upgraded up to 28.53% WO_3 at rougher flotation stage with 91.46% recovery after removal of graphite. Rougher concentrates were cleaned to 54.69% WO_3 with 82.03% recovery using additional quantity of sodium oleate as collector, sodium silicate and quebracho as regulators for the gangue minerals in the cleaner circuits. The cleaned concentrate was upgraded to 64.66% WO_3 during a recleaner float without using additional reagents. It is also important to note from Table 4 that two cleanings of the rougher concentrate and circulating of middling (cleaning tailings) back to subsequent cycle test have ensured a final high-grade concentrate containing 64.66% WO_3 content with 70.36% recovery.

Table 4. Metallurgical balance for scheelite flotation

Flotation product	Weight %	Grade %	Recovery %
Re-cleaner concentrate	0.37	64.66	70.36
Re-cleaner tailings	0.14	28.34	11.67
Cleaner concentrate	0.51	54.69	82.03
Cleaner tailings	0.58	5.53	9.43
Rougher concentrate	1.09	28.53	91.46
Graphite concentrate	2.14	0.00	0.00
Rougher tailings	96.77	0.03	8.54
Head sample	100.0	0.34	100.0

The flow-sheet developed on the basis of lock cycle for beneficiation of Chitral scheelite ore showing the regrinding of rougher concentrate and intermediate products (cleaning tailings) is given in Fig. 12.

Conclusion

It is concluded from the reported results that careful pH control around 10 must be maintained during conditioning in a scheelite flotation. It has been proved that the conditioning at high pulp densities of 30% or for periods greater than 6 min does not seem to offer any advantages. In fact, extended conditioning periods have proved deleterious to grade of concentrate. The most satisfactory reagent combination used was found

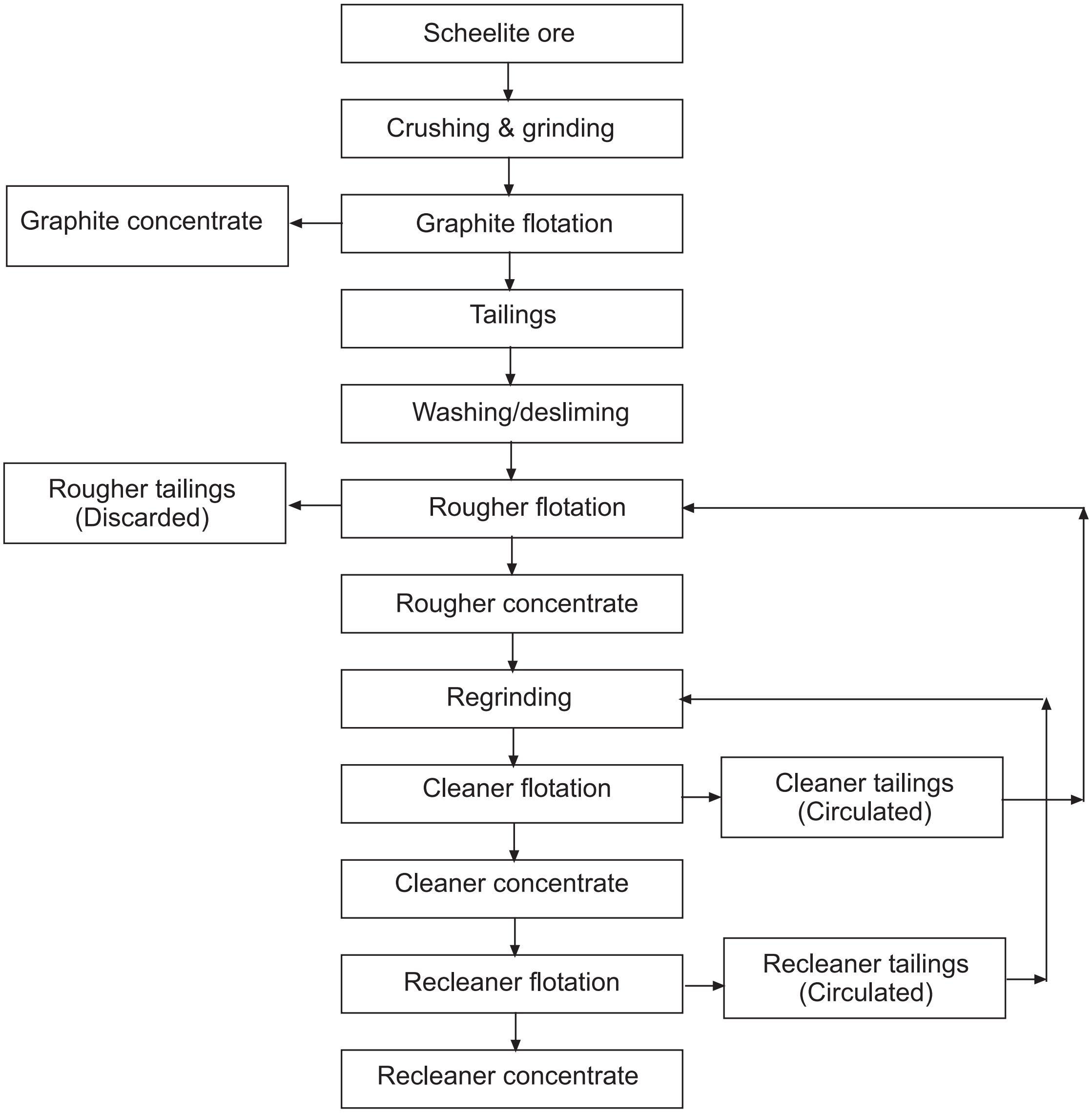

Fig. 12. Flow-sheet developed for beneficiation of scheelite ore of Chitral.

to be 600 g/t sodium oleate, 1200 g/t sodium silicate 50 g/t of polyglycol wetting agent. The laboratory scale flotation study on scheelite ore of Chitral area has established that it could be beneficiated into the metallurgical grade concentrate assaying 64.66% WO_3 with an acceptable recovery of 70.36%.

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