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Beneficiation Studies on Low-Grade Tungsten Ore of Chitral, Khyber Pukhtunkhwa, Pakistan

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Abstract. A bench-scale investigation was carried out for the beneficiation of tungsten ore of Chitral, Khyber Pukhtunkhwa, Pakistan. The ore initially containing 0.55% WO_3 was upgraded by applying a combination of physical beneficiation techniques i.e gravity concentration and magnetic separation. Diagonal Deck Concentration Table was used for gravity concentration studies. A series of tests was performed and the effect of various process variables such as feed rate, wash water flow rate, deck inclination angle and stroke length was studied. The experiments were conducted according to the traditional single-factor experimentation in order to get the real optimum conditions of processing. At optimum conditions, a tungsten concentrate having a grade of 30.85% WO_3 with 75.72% recovery was obtained. This concentrate was further upgraded by magnetic separation to 34.11% WO_3 with more than 75.72% recovery.

Keywords: Tungsten ore, gravity concentration, magnetic separation, scheelite concentrate

Introduction

Tungsten is a very important metal both strategically and industrially due to its high density (19.3 g/cm^3) and high temperature properties (m.p. 3410 °C). It is used in many diverse applications such as hard component in cutting tools and wear resistant materials, an alloy constituent in high speed tool and die steels, in filaments and electrodes as cathode in electronic devices, as a major constituent in the production of armor piercing ammunition and a catalyst in chemical industry (Shedd, 2011).

High price and demand for acceptable tungsten concentrate offer a very attractive incentive for the exploration of tungsten ores. China is the leading producer of tungsten concentrate. China contributes more than three-fourth of world production of tungsten concentrate. With most of the remaining demand fulfilled by Austria, Brazil, Canada, USA, Portugal, Russia, Sweden, France, Japan, South Korea and Thailand (Shedd, 2001). Pakistan has limited deposits of tungsten at Mineki Gole and Besti Gole in Garam Chasma area about 30 km from north of Chitral city. Around 50 thousand tonnes of tungsten ore deposits have been estimated recently which occur in 300 km long belt main scheelite mineralization occurs, in the Mineki Gole and adjoining area, consisting of mica schist, carbonates, clinozoisite, calc-silicate quartzite, tourmaline, phyllite and feldspar rocks (Khan *et al.,* 2003).

Most of the ore deposits usually contains tungsten in the range of 0.3-1.5% WO_3, therefore, concentration of the metal is necessary (Srivastava and Pathak, 2000). The main ore minerals of tungsten are wolframite (Fe, Mn) WO_4 with specific gravity of 7.1-7.9 and scheelite $CaWO_4$ with specific gravity of 5.9-6.1 (Blackburn and Denner, 1988). The beneficiation flow-sheet is followed depending upon the nature of mineralization in the ore body and the size of liberation of the tungsten minerals (Weiss, 1985). Because of the high specific gravity of the tungsten minerals, they are amenable to gravity concentration methods. Scheelite ores can be concentrated by gravimetric methods often combined with froth flotation, while wolframite ores can be concentrated by gravity separation sometimes in combination with magnetic separation (Weiss, 1985).

The main purpose of this investigation was developing a suitable beneficiation process for the preparation of the desired grade concentrate of tungsten from the indigenous ore. The ore originating from Mineki Gole (Chitral) area of Northern Pakistan, containing scheelite as valuable mineral was subjected to various mineral processing techniques. A suitable concentration process has been developed based on extensive bench-scale experimental work. The process includes two step

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gravity concentration i.e. one rougher and one cleaning operation followed by a final cleaning by low intensity magnetic separation.

Materials and Methods

Sample preparation. A mixed sample of the ore, obtained from different blocks of the deposit, consisted of lumps ranging in size 100-250 mm. Representative sample of the scheelite ore was crushed through primary jaw crusher and secondary rolls crusher. Coning-quartering and riffling techniques of sampling were applied to prepare the head sample for chemical analysis and X-ray diffraction studies, while the remaining ore was used for further processing.

Chemical analysis. Analysis of the head sample and the processed products was performed in accordance with ASTM methods.

Mineralogy. In co-operation with The College of Earth and Environmental Sciences, University of the Punjab, Lahore, the characteristics of Chitral scheelite ore were defined by petrography and X-Ray diffraction (XRD); the latter was carried out using Diffractometer D-5000 (Siemens, Germany) to identify various types of mineral constituents present in the ore.

Gravity concentration tests. Diagonal deck concentration table (Deister Concentrator Company, USA) having a flat deck (55 cm × 110 cm) moving with a horizontal motion of 300 cycles/ min was used for gravity concentration tests. A series of experiments was carried out by varying feed rate of scheelite ore 10 -30 kg/h, wash water flow rate 5- 9 L/min, deck inclination angle 4-7° and stroke length 7-10 mm.

Magnetic separation tests. Tungsten concentrate obtained from gravity concentration operation (shaking table) was subjected to magnetic separation. Wet Low-Intensity Magnetic Separator (Sala, Sweden) was used for the separation of magnetite from the scheelite concentrate. The field intensities were varied from 0.08 to 0.14 Tesla to upgrade the concentrate.

Flow-sheet developed for the beneficiation of Chitral scheelite ore is given in Fig. 1. The optimized conditions and the metallurgical balance are presented in Table 1 and 2, respectively.

Results and Discussion

Chemical analysis of the representative sample presented in Table 3 shows that the WO_3 content in the ore was 0.55%. The obtained grade is sufficient to exploit the ore on commercial scale for producing tungsten concentrate (Pandey *et al.,* 2001). However, the presence of silica, alumina, calcium oxide and iron oxide appeared to be the main impurities that are required to be removed.

The X-ray diffraction analysis (Fig. 2) shows that major peaks 3.1 (1) 4.76 (0.55), 3.072 (0.3) confirm the presence of considerable amount of scheelite ($CaWO_4$) mineral. It was further identified by JCP.CAT search/match programme of X-ray Diffractometer D-5000 (Siemens, Germany) that the other peaks corresponded to the gangue minerals which mostly comprised of calcite ($CaCO_3$), epidote Ca_2, Al_3 (SiO_4) (Si_2O_7) (O, OH), magnetite (Fe_3O_4), hydrobiotite K (Mg, Fe)$_3$ (Al, Si_3O_{10}) (OH, F)$_2$, muscovite

Table 1. Optimized conditions of gravity concentration tests

Operation (parameter)	Optimum value
Gravity concentration-I	
Feed size	80% - 100#
Feed rate	25 kg/h
Water flow rate	7 L/min
Stroke length	8 mm
Deck inclination angle	5 °
Gravity concentration-II	
Feed size	95% - 100#
Feed rate	25 kg/h
Water flow rate	7 L/min
Stroke length	8 mm
Deck inclination angle	5 °

Table 2. Metallurgical balance for beneficiation of Tungsten ore

Products	Weight (%)	Grade of WO_3 (%)	Recovery of WO_3 (%)
(Non-magnetic concentrate)	(1.22)	(34.13)	(75.72)
Magnetic concentrate	0.13	0.0	0.0
(Gravity concentrate-I & II)	(1.35)	(30.85)	(75.72)
Gravity middling-II	53.15	0.236	22.82
Gravity tailings-II	25.0	0.01	0.45
(Gravity concentrate-II)	0.85	30.17	46.63
(Gravity middling-I)	(79.0)	(0.49)	(69.90)
Gravity tailings-I	20.50	0.027	1.01
(Gravity concentrate-I)	(0.50)	(32.0)	(29.09)
Calculated head sample	100.0	0.55	100.0

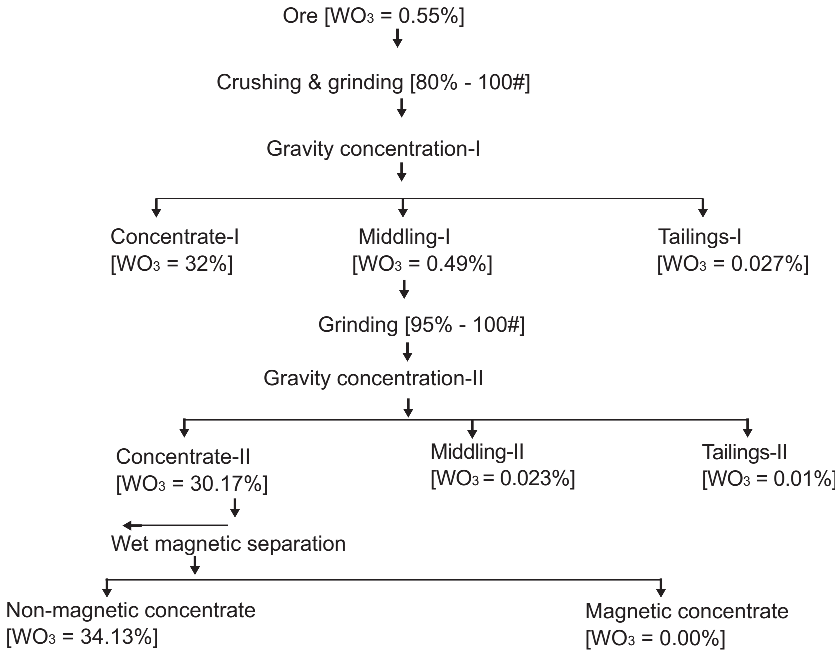


Fig. 1. Flow-sheet for beneficiation of Chitral Tungsten ore

K, Al_2 (Al, Si_3O_{10}) (OH, F), sapphirine $Mg_{3.5}$, $Al_9Si_{1.5}$ O_{20} and predominant quantities of quartz (SiO_2).

Since gravity methods work by virtue of differences in specific gravity of minerals, the greater the difference in specific gravity between the valuable gangue minerals, the more easily they can be separated. Generally, a difference of more than 2.0 is required for reasonable separation (Wills, 1992). The scheelite mineral has specific gravity of 5.9 to 6.1 and associated gangue minerals have specific gravity ranging from 2.6 to 4.3 except magnetite. Due to this significant difference in specific gravity, it was decided to upgrade this ore by gravity concentration technique which has the potential to reduce them effectively. Magnetite having specific gravity (5.0 to 5.5) close to scheelite is recovered along with scheelite concentrate and is separated by magnetic separation.

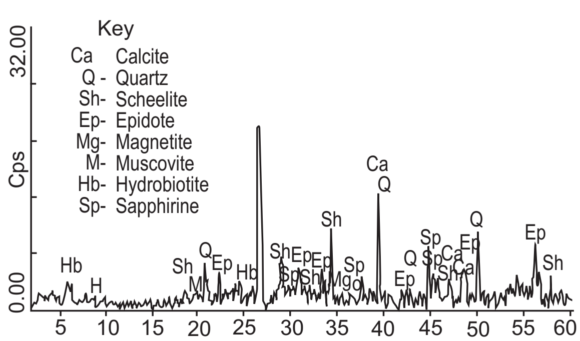


Fig. 2. XRD of Chitral scheelite ore.

The ore was ground to a size at which the maximum mineral grains liberated at the coarsest possible size, avoiding over-grinding which can lead to slimming problems (Wills, 1992). The microscopic study showed that scheelite was finely disseminated in the ore body. The grain size of scheelite ranged from 50-300 μm; however, the average grain size was 150 μm. Since on grinding scheelite tends to slime badly, being the most friable mineral, the ore was crushed and ground carefully in a rod mill in stages and fines were removed after each stage in order to minimize tungsten loses in slimes. Figure 3 shows that the grade and recovery of scheelite is improved by decreasing the particle size and maximum recovery is obtained at feed size of 80% passing 100 mesh (150 μm). This was considered as optimum feed size and selected for the further study. It is notable that although the grade improves but recovery decreases on further grinding of ore. It may be due to the reason that excessive grinding generates slimes that lower the recovery.

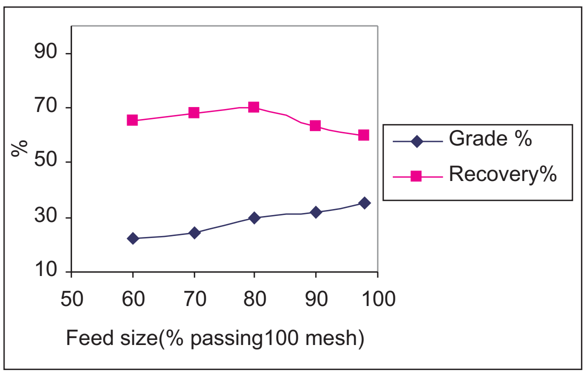
The ore feed rate of 25 kg/h gave a sharp separation of concentrate with optimum recovery (Fig. 4) under an arbitrarily fixed deck inclination angle of 6°, wash water flow rate of 6 L/min and stroke length of 9 mm. It is clear from Fig. 4 that the grade and recovery dropped with an increase in feed rate above 25 kg/h due to the fact that the larger amount of feed hinders the proper stratification of the feed over the deck, leading to low grade and recovery.

The effect of wash water flow rate on the grade and recovery of scheelite, presented in Fig. 5 was studied at the fixed feed rate of 25 kg/h, deck inclination angle of 6° and stroke length of 9 mm. It is evident from the figure that the wash water flow rate of 7 L/min gave the optimum result. Above this rate, grade and recovery dropped down since fine scheelite particles got transferred to middling due to relatively fast flow of water.

Figure 6 shows the effect of varying the deck inclination angle from 4 ° to 7 ° to the horizontal. It may be seen that the grade and the recovery increased up to deck inclination angle of 5° and then the grade of the scheelite concentrate improved while the recovery lowered down; this is due to the fact that further increase in angle imparted to the partially liberated scheelite particles the tendency to go to the middling. Hence, deck inclination angle of 5° was selected for further study.

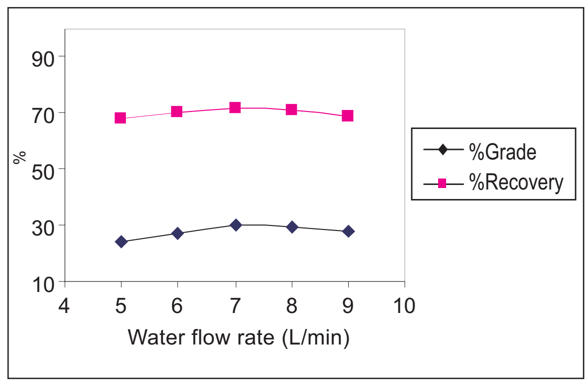
Figure 7 shows the effect of the stroke length on the recovery of scheelite at constant feed rate of 25 kg/h, wash water flow rate of 7 L/min and deck inclination angle of 5°. Generally, long stroke is suitable for coarse feed and reverse is true for the fine feed (Jain, 1986). It is seen from the figure that short stroke length of 8 mm produced better results, which is obviously due to the fine size of the feed.

The description of metallurgical balance given in Table 2 indicates that scheelite ore containing 0.55% WO_3 can be upgraded to 30.85% WO_3 with 75.72%



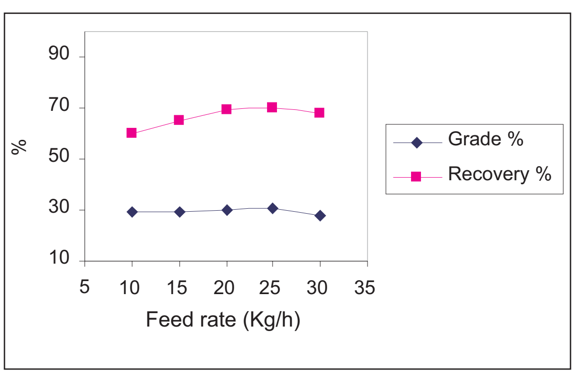
Water flow rate, 6 L/min; deck inclination angle, 6°; stroke length, 9 mm

Fig. 3. Effect of feed size on the grade and recovery of scheelite concentrate.



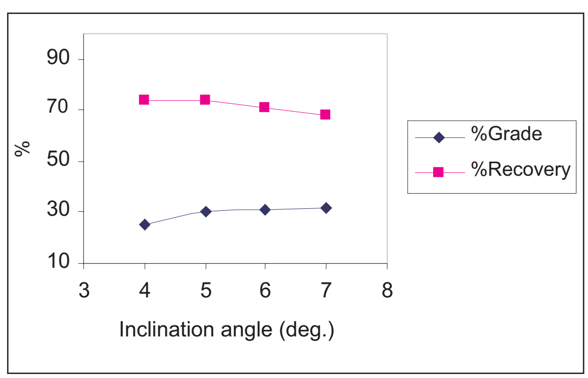
Feed rate, 25 kg/h; deck inclination angle, 6°; stroke length, 9 mm

Fig. 5. Effect of wash water flow rate on the grade and recovery of scheelite concentrate.



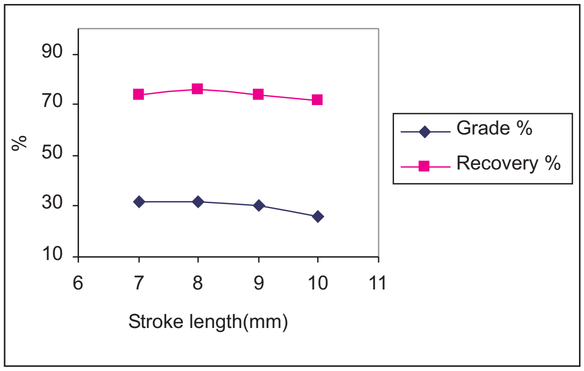
Water flow rate, 6 L/min; deck inclination angle, 6°; stroke length, 9 mm

Fig. 4. Effect of feed rate on the grade and recovery of scheelite concentrate.



Feed rate, 25 kg/h; water flow rate, 7 L/min; stroke length, 9 mm

Fig. 6. Effect of deck inclination angle on the grade and recovery of scheelite concentrate.



Feed rate, 25 kg/h; water flow rate, 7 L/min; deck inclination angle, 5°.

Fig. 7. Effect of stroke length on the grade and recovery of scheelite concentrate.

recovery at optimum conditions by gravity concentration. This recovery has been obtained, as mentioned in Table 1 at a feed size of 80% passing 100 mesh, feed rate of 25 kg/h, wash water flow rate of 7 L/min, deck inclination angle of 5° and stroke length of 8 mm. The middling assaying 0.49% WO_3, was subjected to re-grinding in a rod mill (178 x 356 mm) for 30 min for getting a grind size 95% passing 100 mesh followed by another gravity concentration operation. All the conditions of the second gravity concentration operation were the same except the grind size as presented in Table 1. With this operation, a tungsten concentrate assaying 30.17% WO_3 was obtained from the middling. It is also important to note in Table 2 that one cleaning of the rougher concentrate has ensured a final concentrate grade of 30.85% WO_3 with 75.72% recovery, prior to further up gradation by the magnetic separation.

The results obtained at different magnetic field intensities, using wet magnetic separation technique, are shown in Fig. 8. It is obvious from these results that there was a gradual decrease in iron content of scheelite concentrate with increase in the field intensity up to Tesla 0.12, but after that, further increase in field intensity did not improve the grade. This is due to the fact that, as the field intensity increases, more and more magnetite particles are attracted by the magnet and fall in the magnetic portion till the field intensity of 0.12 Tesla when almost all the magnetite particles are removed from the scheelite concentrate. Hence, this field intensity was selected for the cleaning of tabling concentrate. It has been found that with the wet magnetic separation, better results are produced as compared to the dry magnetic separation. It may be that during the wet separation, some of the fine scheelite particles that are carried to the magnetic portion, are washed down by water in the non magnetic portion.

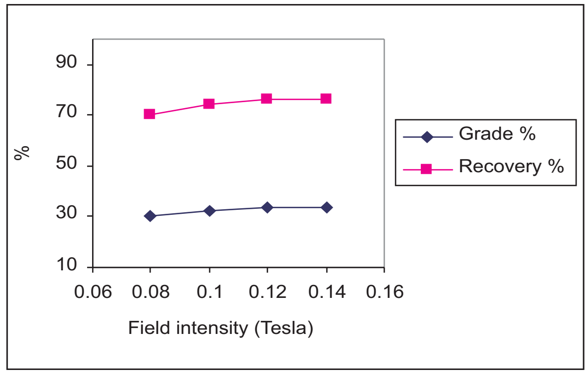


Fig. 8. Effect of magnetic field intensity on the grade and recovery of scheelite concentrate.

Results of the chemical analysis of the final concentrate obtained by gravity concentration followed by magnetic separation of the scheelite ore of Chitral area of Pakistan are presented in Table 3. It is obvious that the WO_3 content of the investigated ore increased from 0.55% to 34.13%. The grade of the final concentrate is quite suitable for the preparation of tungsten metal powder or tungsten based products by the hydrometallurgical route (Guar, 2006).Pandey *et al.* (2001) prepared a pre-concentrate with 13.06% WO_3 content from an ore containing 0.1-0.16% WO_3 by physical processing. It was then treated by chemical processing with the soda ash roast leaching and the alkali pressure leaching to obtain the tungsten values.

Most tungsten concentrates are processed chemically to ammonium paratungstate (APT). It is the intermediate

Table 3. Chemical Analysis of Tungsten ore and final scheelite concentrate

Constituents	Ore (%)	Concentrate (%)
WO_3	0.55	34.13
SiO_2	75.37	47.45
Fe_2O_3	4.45	0.71
Al_2O_3	8.75	5.33
CaO	6.75	8.36
MgO	0.16	0.02
Na_2O	1.07	0.96
K_2O	0.28	0.05
LOI	2.38	1.89

product for the preparation of either pure tungsten metal or tungsten based materials. (Li, 2004; Premchand, 1996). In order to produce APT, concentrate may be decomposed by acid leaching or by the autoclave-soda digestion process. Even, the low-grade concentrates (5-20% WO_3) can be leached for tungsten values with high recovery. Zhao *et al.* (1996) decomposed a low-grade tungsten ore using sodium carbonate and sodium hydroxide through mechanical activation, achieving, tungsten recovery up to 99%. Razavizadeh and Langroudi (2006) leached out low and high-grade scheelite concentrate with sulphuric acid. The resulting tungstic acid was reacted with ammonia to produce ammonium para-tungstate. The APT obtained from either of the process was eventually calcined to tungsten (VI) oxide which was then reduced with hydrogen to produce the powdered tungsten metal. The tungsten powder can be fused to form any number of things, from wire to other shapes and tungsten carbide (WC). High-grade tungsten concentrates (>65% WO_3) can be smelted directly with charcoal or coke in an electric arc furnace to produce ferrotungsten (Guar, 2006).

Conclusion

The results of the experimental work indicate that indigenous scheelite ore of Chitral area containing 0.55% WO_3 could be beneficiated by gravity concentration and magnetic separation technique to a concentrate assaying 34.13% WO_3 with 75.72% recovery at a feed size of 80 % passing 100 mesh, feed rate of 25 kg/h, wash water flow rate of 7 L/min, deck inclination angle of 5°, stroke length of 8 mm and field intensity of Tesla 0.12. The produced concentrate can be utilized for the production of tungsten metal and other value-added tungsten based products. However, froth flotation beneficiation studies are suggested for further up-gradation of this concentrate to yield high grade tungsten concentrate.

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