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Flotation Studies on Low-Grade Phosphate Rock of Tarnawai, District Abbottabad, KPK Province, Pakistan

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Abstract. A low-grade siliceous sedimentary phosphate rock assaying 22.05% P_2O_5 was upgraded by double float (direct and reverse) technology. The rock contains collophane (carbonate fluorapatite) as the valuable phosphate mineral along with siliceous, carbonaceous and clay minerals. A process was developed which yielded a high-grade phosphate concentrate with improved recovery rate. The developed process consists of three parts. The first part includes wet grinding of ore to liberation point followed by separation of fines (slimes) by cone classifier. The finely ground deslimed ore was floated by direct anionic flotation using oleic acid collector to get rougher phosphate concentrate. It was cleaned once using additional quantity of reagents. In the second part, the fines generated during grinding operation were floated by column flotation to recover phosphate values. The cleaned concentrate and column concentrate were mixed together and washed thoroughly with hot water to remove the attached reagents. The final part comprises of cationic flotation of combined phosphate concentrate with fatty amine collector to float away quartz and silicates and to leave behind phosphate values. The grade of final phosphate concentrate was found to be 32.85% P_2O_5 with an overall recovery of 88.14%. The concentrate obtained meets the specifications of fertilizer and acid grade.

Keywords: low-grade phosphate rock, carbonate fluorapatite, desliming, flotation,

Introduction

Phosphorous occurs only to the extent of 0.12 % in the lithosphere. It is the 11th most abundant element in the earth crust occurring as phosphate (Blackburn, 1988). It is considered as one of the major nutrients among the sixteen essential nutrients that plants need for growth and reproduction. Phosphorous particularly plays an important role in root development, synthesis of protein, plant structure, seed yield and genetic transfer, photosynthesis and respiration, energy storage and transformation, cell division and enlargement, tissue development and maintenance (Armstrong, 1991). It is a vital structural component of nucleic acids and nucleotides. It is essential for the formation of skeletal bones and teeth in animals. The creatine phosphates are involved in muscle contraction. Phosphates also play an important role in buffering the blood at proper pH value (Gieseke, 1985).

Phosphate rock deposits of the world are the only significant global resources of phosphorus. They are the basic raw material for production of phosphate fertilizers, animal feed, phosphoric acid and other industrial chemicals. About 90% of phosphate rock mined is used for production of and animal feed and the remaining for industrial chemicals (Jasinski, 2000). Phosphate rock deposits are widely distributed throughout the world, but the major economic ore bodies are located in the United States, Morocco, China, Russia, south Africa, Jordan, and Australia (Van Kauwenbergh, 2010). Pakistan has limited reserves of phosphate rock located in the north-east of the country. The largest ore body occurs in Hazara division in an area of about 155 km^2. The prospecting deposits are located near the villages of Kakul, Mirpur, Lagarban, Tarnawai, Kalu-di-Bandi, Dalola, Bagla gali, Bataknala, Barkot and Havelian, (Ahmad and Siddiqi, 1992). Extensive geological surveys have proved potential deposits of 26 million tons of rock phosphate which are exploitable. There exist two major types of rock phosphate, namely dolomitic containing low to medium P_2O_5 and siliceous containing medium to high P_2O_5. The reserves of dolomitic rock phosphate are estimated to be 14 million tons, while that of siliceous rock phosphate are 12 million tons (Mehmood *et al*., 2010). Lagarban and Tarnawai phosphate rock deposits have been reported

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to have re-coverable reserves of 7.5 and 4.4 metric tons respectively (Appleton and Northolt, 2002).

Low-grade phosphate ores are processed to reduce the content of gangue minerals such as silicates, carbonates and clays to meet the requirements of the phosphate industry (Zafar *et al*., 1995a). These are subjected to beneficiation processes such as dseliming, washing, sizing, selective flocculation, gravity concentration, heavy media separation, magnetic separation, froth flotation, leaching, calcinations etc. However, most of the world's marketable phosphate is produced by froth flotation process. It has been successfully applied to both sedimentary and igneous phosphates with siliceous gangue (Riaz *et al*., 2001). But the processing of sedimentary phosphate rock containing calcareous impurities such as calcite or dolomite in addition to siliceous gangue has numerous problems (Fang and Jun, 2011; Al-Fariss *et al*., 1991). Two-stage reverse anionic flotation has been appeared to be a promising separation technique for the removal of carbonates and silicates from sedimentary rock phosphate (Mohammadkhani *et al*. 2011; Abdel-Khalek, 2000). Column flotation has been applied successfully to a wide variety of phosphate ore types ranging from igneous to sedimentary (Abdel-Khalek *et al*., 2000). Calcination is the other technique used for up gradation of sedimentary phosphates with carbonate gangue but it involves high fuel consumption and lower acidulation reactivity of the calcined products (Abouzeid, 2008; Zafar *et al*., 1995b,). Leaching of carbonate ores with organic acids has been tried by many researchers (Gharabaghi *et al*., 2010; Zafar, 1993).

The present study is aimed at development of a suitable process for the up-gradation of indigenous low-grade phosphate rock of Tarnawai area. For this purpose, the froth flotation technology was applied for the possible removal of gangue minerals and subsequent up-gradation of phosphate rock. The comprehensive account of experimentations are obtained.

Materials and Methods

Sample preparation. A representative sample of phosphate rock weighing about 100 Kg was collected from the mines of village Tarnawai, near Qalandarabad city, district Abbottabad, Khyber Pukhtoon Khawa (KPK) province, Pakistan. The bulk sample of rock was consisted of lumps having size in the range of 10-25 cm. It was packed in two plastic bags and brought to Mineral Processing Research Centre (MPRC), PCSIR Laboratories Complex, Ferozepur road, Lahore for chemical evaluation and beneficiation studies. First of all it was subjected to size reduction process including crushing and grinding. Primary crushing was carried out using jaw crusher and secondary crushing by roll crusher. A head sample weighing about 1 Kg was obtained by riffling of the crushed ore. It was ground in disc pulverizer (Denver, USA) to get a product of minus 200 mesh size for chemical analysis (ASTM E-877). The rest of crushed ore was split up into 1 Kg samples manually for beneficiation tests.

Chemical analysis. The chemical analysis of representative sample of phosphate rock, processing products and final concentrate was performed according to ASTM as well as conventional methods. The finely ground ore was dried in electric oven at 105°C for one hour to determine moisture. Sample solution was made by HCl acid digestion method. It was used to determine all the constituents except sodium and potassium which were determined from solution prepared by treatment of another sample with HF. Silica and alumina were determined by gravimetric methods (ASTM E-247). The phosphorous was determined by colourimetric method using spectronic-20 (Bausch & Lamb) by developing phosphovanado-molybdate complex with ammonium molybdate and ammonium vanadate and measuring absorbance at 470 nm (Jeffery, 1989). The phosphorous content of the processed and products was also determined colourimetery. Iron was determined by oxidation-reduction titration using standard solution of potassium dichromate (ASTM E-246). Calcium and magnesium were estimated by atomic absorption spectrophotometer (Model: Z-8000, Hitachi, Japan). Alkali metals such as sodium and potassium were determined by flame photometer (Model: PFP7, Jenway, England). The chemical analysis of representative sample of phosphate rock and the final flotation concentrate is given in Table 1.

Flotation experiments. Anionic and cationic flotation tests were conducted in a laboratory size flotation machine (Model: D-12, Denver, USA). While column flotation tests were carried out in locally designed and fabricated column flotation cell by conventional methods (Wills, 1992). Flotation feeds were prepared by wet grinding of crushed rock in ball mill (Denver, USA) at solid to liquid ratio of 1:1. The ground ore was transferred to cone classifier to remove the fines (slimes) produced during grinding step. The ore was screened into two size fractions by cone classifier, a coarser flotation feed and fine column flotation feed. The fines separated were collected in a conditioning tank. The deslimed ore was transferred into stainless flotation cell of one liter capacity where flotation reagents were added. The pulp was conditioned at high solids of 65-70% for 5-6 min at a pH of 9.0-9.5 using oleic acid as collector and polypropylene glycol as frother. Sodium silicate was added as depressant and sodium carbonate as pH modifier prior to an anionic froth flotation. It was followed by pulp dilution to 20-40% solids with water. After optimizing the grind size of ore, the rougher flotation tests were performed to optimize pulp density, agitation speed, pH of pulp, quantities of reagents, conditioning time and flotation time. The traditional approach of changing one variable at a time was adopted (Day, 2002). The results obtained of these tests are presented in Fig. from 1-9. The rougher phosphate concentrate was cleaned once in cleaning flotation operation after regrinding in ball mill and using additional quantizes of reagents to get cleaner concentrate.

Slimes collected during desliming step were conditioned in a conditioning tank with same flotation reagents that were used in anionic flotation (Hussin and Ahmad, 2007). The conditioned slimes were transferred into column feed tank from where they were sent to column flotation cell. The compressed air from blower was admitted through the valve with controlled flow rate and distributed in cell through the sparger fixed at the base of cell. The slurry flow rate from the feed tank was controlled with valve to obtain smooth feed in the cell and for optimum residence time. The concentrate floated on the top of column cell was collected separately in the tray. It was mixed with cleaner concentrate.

The mixed concentrate was washed with hot water to detach the reagents and subjected to the reverse flotation using fatty amine as collector and diesel oil as frother to remove any remaining silica into the froth product at slightly acidic pH typically 6.5-7.0. Acetic acid was used as depressant to minimize the float of phosphate values in the froth. The various parameters of direct, reverse and column flotation were optimized for preparing high-grade phosphate concentrate. The optimum conditions of different stages of flotation are presented in Table 2, while metallurgical balance of the typical test is recorded in Table-3 separately. A process flow sheet developed on the basis of this research work is shown in Fig. 10.

Results and Discussion

Chemical analysis. The chemical analysis (Table 1) reveals that P_2O_5 content in the representative sample of the rock is 22.05%. This shows that rock is low to medium grade and cannot be used as such for industrial application until it is beneficiated to a commercial grade concentrate. However, obtained grade of rock is quite sufficient for up-gradation and commercial exploitation (Rao *et al*.,1992). Phosphate rock of a lower grade containing as low as 24% P_2O_5 and as high as 23% SiO_2 can be used for the production of elemental phosphorous. For the manufacture of single super phosphate the phosphate rock should have a minimum of 31% P_2O_5 content and SiO_2 up to 8% can be tolerated. The phosphate rock having minimum 32% P_2O_5 and maximum 5% SiO_2 is suitable for the production of phosphoric acid which is the starting material for the phosphate based chemicals (Hijran, *et al*. 2015; Ishaque and Ahmad, 1987).

At present granulated single super phosphate (GSSP) fertilizer is being prepared locally using a combination of indigenous medium-grade phosphate rock and imported high-grade rock (Zafar, 1993). Since high-grade rock phosphate is being imported mainly from Jordan and Morocco for manufacture of GSSP, because of non-availability of local high-grade rock phosphate. It is therefore essential to develop an effective process to upgrade indigenous low to medium grade sedimentary phosphates with high silica and carbonate gangue minerals to substitute imported high-grade ore. Table-1 shows that the major impurities found in the rock are silica, iron oxide, aluminum oxide, calcium

Table 1. Chemical composition of phosphate rock before (raw) and after beneficiation process (concentrate).

Constituents	Composition of raw phosphate rock (w/w%)	Composition of processed phosphate (w/w%)
Moisture @105°C	0.14	0.12
Loss on ignition (LOI)	8.36	4.87
Silica (SiO_2)	20.86	4.96
Phosphorous oxide (P_2O_5)	22.05	32.85
Aluminum oxide (Al_2O_3)	1.32	0.84
Iron oxide (Fe_2O_3)	4.23	0.16
Calcium oxide (CaO)	38.57	54.12
Magnesium oxide (MgO)	1.75	0.25
Sodium oxide (Na_2O)	0.53	0.06
Potassium oxide (K_2O)	0.94	0.08

oxide and magnesium oxide. The production of phosphate concentrate with quality according to the requirements of fertilizer and chemical grade demands the removal theses contaminants. Keeping in view of these impurities, it was decided to employ froth flotation technique to reduce these impurities and consequently increase its P_2O_5 content up to marketable grade.

Effect of grinding. The grind size range of ore under investigation was evaluated by grinding it in ball mill in different batches to obtain 60%, 70%, 80%, 90% and 100% minus 200 mesh size feed. Flotation of each batch was performed with arbitrarily selected values. Results obtained of each batch were analyzed separately for P_2O_5 content and reported in Fig. 1. It is obvious from this figure that both the grade and recovery of phosphate concentrate was improved gradually by decreasing the particle size from 60% to 90% passing 200 mesh size and further decrease in particle size affected the recovery adversely. The best results in term of grade and recovery were achieved when the rock was ground to 90% minus 200 mesh (75 µm) size. This feed size was considered as the optimum mesh of grind for the liberation of collophane and therefore selected for further investigation. In fact, most of mineral grains of cellophane are liberated from associated gangue minerals at this mesh size. It is notable that on further grinding of ore, although the grade of concentrate improved marginally from 27.02% to 27.50% P_2O_5 but recovery decreased significantly from 84.50% to 82.06%. It is probably due to the fact that excessive grinding generated slimes that affected the recovery (Santana, *et al*. 2011; Ahmed, 2007).

Effect of pulp density. Pulp density (percent solids of feed in the pulp) has a significant influence on the flotation results. The flotation efficiency of phosphate minerals is greatly enhanced during the collector conditioning stage at high solids. Without this high intensity conditioning step, better results are not achieved (Houot, 1982). The deslimed ore was initially conditioned at high solids (~ 70%) by the input of mechanical energy. The pulp density of conditioned ore was varied from 20% to 40% by diluting it with water in stainless steel cell. The results of pulp density variation versus grade and recovery of concentrate have been plotted in Fig. 2. It is clear from this Figure that the increase in pulp density from 20% to 40% decreases the grade slightly but increases the recovery initially and then decreases. Maximum recovery of 85.04% was achieved at a pulp density of 30% solids beyond which it began to decrease. It is assumed that at higher pulp density, coating by collector is not proper due to excess amount of solids. Moreover, the higher pulp density increases the pulp viscosity which inhibits proper air dispersion and bubble formation, thereby decreases the recovery. It was observed that lower pulp density (20%) at rougher stage although produced higher grade (28.07% P_2O_5) but the recovery was decreased (76.16%) which is not acceptable at rougher stage of flotation. However, in practice, the upper limit of pulp density which does not affect the recovery of rougher concentrate is selected (Gieseke, 1985). Therefore, this value was selected for the next study. It was found that at cleaning stage of flotation,

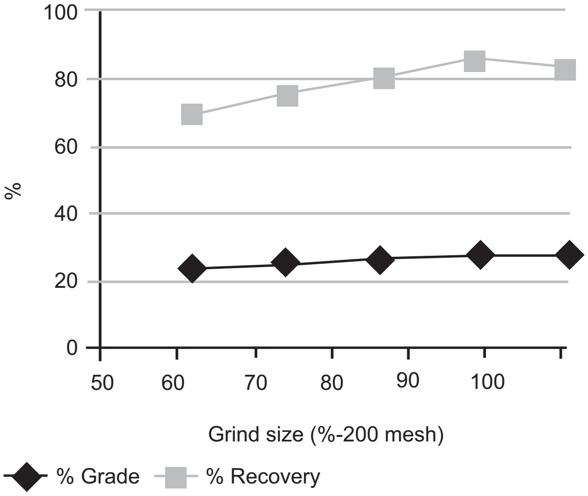

Fig. 1. Effect of grind size on the grade and recovery of rougher phosphate concentrate.

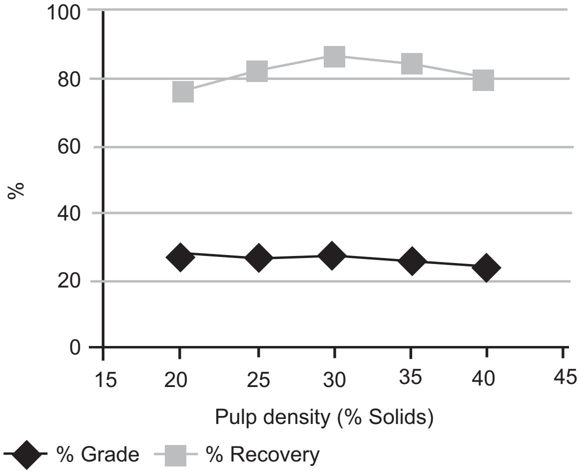

Fig. 2. Effect of pulp density on the grade and recovery of rougher phosphate concentrate.

the lower pulp density of 20% produced a higher grade concentrate with good recovery probably due to better drainage of froth.

Effect of pH. The pH of pulp is always a critical variable in flotation of phosphate minerals. Sulfuric acid, lime, sodium carbonate and sodium hydroxide are commonly applied as pH modifiers. The pH of pulp during anionic flotation of ore was varied from 8 to 10 in different batches. The pH was adjusted with sodium carbonate. The results obtained at various pH values have been presented in Fig. 3. It is clear from this Figure that the grade and recovery of rougher concentrate increased with increase in pH from 8 to 9 and then decreased. It was observed that the maximum grade (27.35% P_2O_5) and recovery (84.76%) is obtained at a pH of 9.0 at rougher stage and therefore selected. It was found that pH of the pulp plays a crucial role during the flotation of phosphate minerals and even a small variation from the optimum pH shows a significant change in grade and recovery. This effect may be attributed to the maximum stability of mineral-collector complex at specific pH. Same pH was maintained at cleaning and column flotation stage.

Effect of agitation. Flotation is a physico-chemical process that depends upon both physical and chemical factors. The flotation process uses chemical reagents to make certain minerals hydrophobic and other hydrophilic along with aeration to separate hydrophobic particles in suspension (Rao and Mohanty, 2002). The agitation speed of flotation cell was varied from 900-1200 rpm. Data collected regarding the effect of impeller speed has been shown in Fig. 4. It is obvious from this Figure that an agitation (aeration) speed of around 1100 rpm gave better metallurgical results at rougher stage. It is because the collector-coated mineral particles containing liberated as well as partially exposed (middling) particles floats at this rpm by attaching them on rising air bubbles and thus increases the recovery. While on the other hand, at cleaning stage, a lower agitation speed of 1000 rpm improved the grade because it allows the middling particles to remain in pulp thus produces a cleaner concentrate of greater purity.

Effect of collector. The most important aspect of a flotation investigation is to establish the best collector or a combination of collectors (Sis and Chander, 2003). The different types of collectors were tried for flotation of sedimentary phosphate minerals. Sodium oleate was found as best anionic collector in the flotation of sedimentary pebble phosphate minerals. Its effect on grade and recovery has been presented in Fig. 5. It is evident from this Figure that better results in term of grade (27.40% P_2O_5) and recovery (85.56%) were achieved at a dosage of 2.0 Kg/ton of oleic acid. It is notable on the other hand; excessive amount of collector adversely affects the grade and recovery of phosphate minerals due to the development of collector multi-layers on the mineral particles that reduce the selectivity. In cleaning stage, less amount of collector is required as compared to rougher stage because most of liberated particles were already coated. Likewise, fatty amine (cationic collector) showed good response during reverse

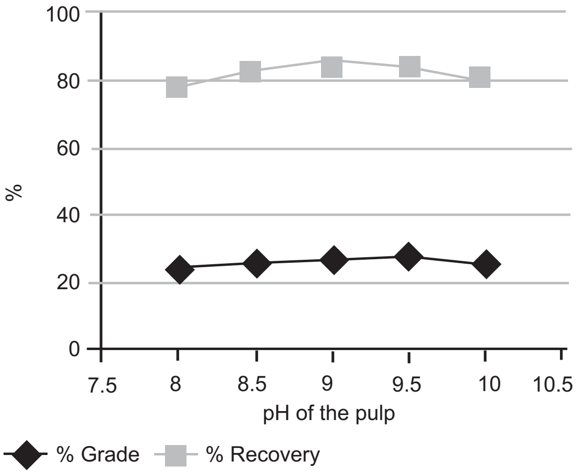

Fig. 3. Effect of pulp pH on the grade and recovery of rougher phosphate concentrate.

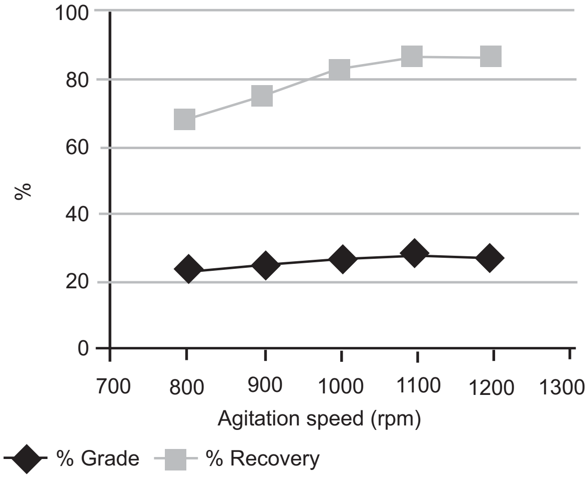

Fig. 4. Effect of agitation speed on the grade and recovery of rougher phosphate concentrate.

flotation of silicate minerals away from the sedimentary pebble phosphate minerals.

Effect of frother. Frother, being hetero polar, adsorbs on the air-water interface due its surface active action and reduces the surface tension of water and stabilizes the mineralized air-bubbles. Selection of a suitable frother for flotation of particular mineral is very important to improve the flotation efficiency in term of grade and recovery. The ideal frother or a combination of frothers selected should produce frothing conditions suitable for mineral transport to the froth phase and subsequent cell overflow, while also allowing drainage of entrained gangue particles. Polypropylene glycol was utilized as frother. Results obtained using different quantities of frother have been presented in Fig. 6. It is apparent from this figure that a dosage of 0.06 Kg/ton gives a reasonable grade (27.65% P_2O_5) and recovery (85.75%) and further increase shows no significant improvement in this regard. Similarly diesel oil responded well during reverse flotation.

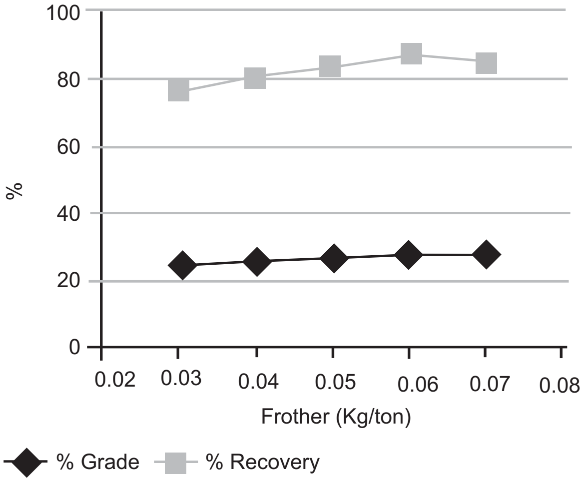

Fig. 6. Effect of frother on the grade and recovery of rougher phosphate concentrate.

Effect of depressant. Various inorganic and organic depressants were practiced to depress the associated gangue minerals. These include sodium silicate, sodium carbonate, sodium polyphosphate and polyacrylate. Sodium carbonate added to maintain the pH of pulp also helps to depress iron oxide and clay particles (Boulos, *et al*. 2014). Sodium silicate was added to depress the silicate gangue material. The use of sodium silicate as gangue depressant increases the selectivity of flotation by rendering the gangue minerals hydrophilic thus prevents their flotation. Results obtained using different quantities of sodium silicate depressant have been presented in Fig. 7. It was observed that quartz being the main gangue mineral present in this rock along with carbonates, clay minerals and slimes were depressed by it. It is obvious from this figure that with the increase in amount of depressant, the grade is improved (27.93 P_2O_5) first without disturbing the recovery (85.95%), but after 0.3 Kg/ton, the grade improved (28.03 P_2O_5) but recovery decreased (84.32%) probably due to the over coating of some middling particles. It was found that at cleaning stage, lesser amount of depressant (0.15 Kg/ton) was needed to get

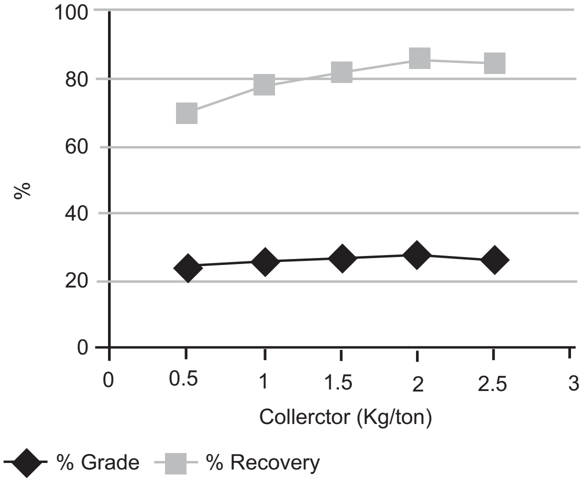

Fig. 5. Effect of collector on the grade and recovery of rougher phosphate concentrate.

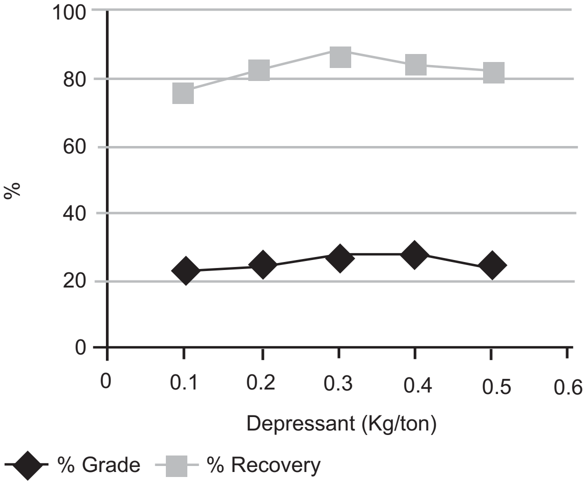

Fig. 7. Effect of depressant on the grade and recovery of rougher phosphate concentrate.

good results as compared to rougher stage. It is because the gangue minerals present in cleaning flotation slurry are in lower quantity than in rougher flotation feed.

Effect of conditioning time. The conditioning time and point of reagents addition usually have a large influence on process metallurgy. Proper conditioning time allows the surfaces of the mineral particles to react with the reagents. Addition points of collectors, frothers, activators and depressants can vary widely, depending upon their types, the mineral associations and water quality (Day, 2002). The optimum points of reagent addition were determined after conducting some preliminary tests. Results obtained for different conditioning time periods are shown in Fig. 8. It is apparent from this figure that conditioning time of 6 minutes is sufficient for the contact with the mineral particles for an optimum grade (28.20% P_2O_5) and recovery (85.98%) of the concentrate. It was found that a prolonged conditioning time has adverse effect on the performance. It peels off the coating of collector (calcium oleate layer) on the mineral particles resulting in lower flotation grade and recovery.

Effect of flotation time. The rate at which the mineralized froth is removed has a large influence on flotation kinetics (Wills, 1992). The time required for flotation of phosphate minerals was determined by collecting incremental concentrates separately after every two minutes time intervals until the froth was completely barren. The froth-scraping pattern was maintained consistent and separate concentrates were removed at equal time intervals. The metal distribution in each incremental concentrate was determined by using the weights and assays for that fraction. This data was graphically plotted as cumulative recovery versus cumulative flotation time in Fig. 9. The result indicated that flotation time of 12 minutes is sufficient to completely barren the froth. The highest grade (28.23% P_2O_5) and recovery (86.04%) was achieved after 12 minutes. This information was used for subsequent flotation tests. The walls of the cell and impeller shaft were periodically washed with water from a wash bottle to return adhering froth to the pulp and to maintain the pulp level.

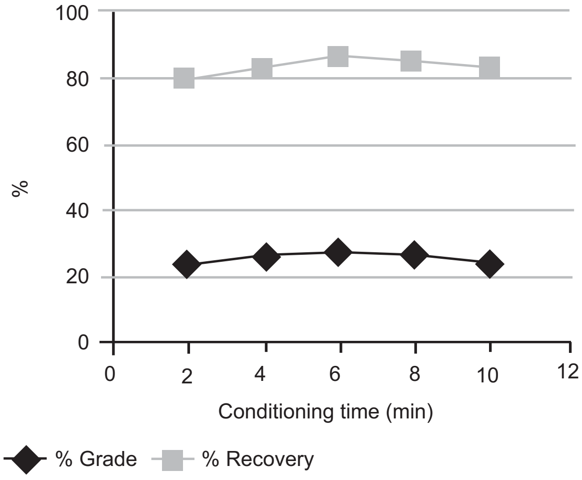

Fig. 8. Effect of conditioning time on the grade and recovery of rougher phosphate concentrate.

Effect of cleaning rougher concentrate. The rougher flotation tests were carried out to float phosphate minerals away from the gangue minerals comprising of silica and dolomite as the main impurities. Few flotation tests were carried out at optimum conditions to have enough concentrate to conduct cleaner flotation tests. It was found from chemical analysis that rougher concentrate contain an average 28% P_2O_5 content. It was again floated in cleaner flotation cells after regrinding to further improve the grade by removing the attached gangue particles. In cleaner flotation, additional amount of reagents was added for the newly liberated fine particles as given in Table 2. The pulp density was slightly lowered down to 20% solids and agitation speed to 1000 rpm to get higher grade without considering the recovery because cleaner tailings was circulated. The silica, carbonates, clays and other impurities were reduced. It can be seen from Table 2 that cleaning of rougher concentrate produces higher grade phosphate

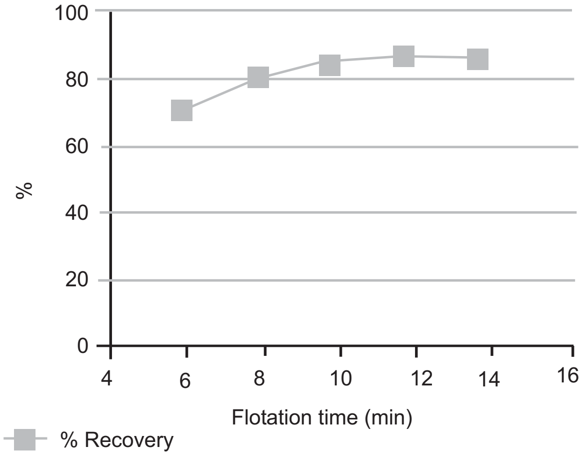

Fig. 9. Effect of flotation time on the grade and recovery of rougher phosphate concentrate.

Table 2. Flotation parameters and optimum conditions of different stages of flotation

Flotation parameters	Optimum conditions			
	Rougher flotation	Cleaner flotation	Column flotation	Reverse flotation
Particle size of flotation feed	90%-200#	100%-200#	100%-400#	95%-200#
Agitation speed (aeration)	1100 rpm	1000 rpm	-	1000 rpm
Pulp density (% solids)	30%	20%	15%	20%
pH of pulp (slurry)	~9.0	~9.0	~9.0	~7.0
Anionic collector-I (oleic acid)	2.0 Kg/ton	1.0 Kg/ton	2.0 Kg/ton	-
Frother-I (polypropylene glycol)	0.06 Kg/ton	0.03 Kg/ton	0.06 Kg/ton	-
Depressant-I (sodium silicate)	0.3 Kg/ton	0.15 Kg/ton	0.3 Kg/ton	-
Cationic collector -II (fatty amine)	-	-	-	0.1 Kg/ton
Frother-II (fuel oil)	-	-	-	0.05 Kg/ton
Depressant-II (acetic acid)	-	-	-	0.07 Kg/ton
Conditioning time	6 min	6 min	6 min	5 min
Flotation time	12 min	15 min	12 min	10 min

concentrate assaying 30.33% P_2O_5 with recovery of 82.03%.

Desliming and flotation of fines. Desliming is a necessary requirement for successful phosphate upgradation by flotation. Desliming removes fines (<38µm or 400 mesh) from flotation feed before flotation (Hussin and Ahmed, 2007; Abdel-Khalek *et al*., 2000). But during desliming step about 9-10% phosphates content are lost in these fines. The chemical analysis of these fines shows that these have up to 21.56% P_2O_5 content. These fines are wasted in conventional flotation process which decreases the total recovery of phosphate values. Column flotation was applied in order to recover them instead of their discard. The result shows that it is possible to recover them economically. Tests were performed using oleic acid as a collector for the phosphate minerals and sodium silicate as a depressant for their associated gangues. At these conditions a product assaying 29.87% P_2O_5 and with recovery of about 85% (7.83×100/9.21) was obtained from fines (slimes) feed containing 21.56% P_2O_5. Such a grade and recovery were not obtained by applying the conventional froth flotation technique, even after cleaning the rougher concentrate. Recovery of more than 85% of phosphate values in the column concentrate from the disposed slimes has improved the economic viability of the beneficiation process by increasing the recovery from 82.03% in cleaner concentrate to 89.86% in mixed concentrate (Table 2).

Cationic flotation of cleaner concentrate. The column flotation concentrate was mixed along with cleaner concentrate in order to maximize the recovery of phosphate content. The combined cleaner concentrate was sent to attrition cells and scrubbed with hot water at 50-70% solids, then rinsed with water to remove the attached reagents. This feed was sent to conditioning tank. The washed combined concentrate was subjected to cationic flotation (reverse flotation) using fatty amine collector and diesel oil as frother. Sodium acetate was used as a phosphate depressant. The results of cationic flotation tests are summarized in Table 2 which describes that mixed cleaner concentrate yielded a product assaying 32.85% P_2O_5 at about 88.14% recovery with reverse flotation. Higher-grade final phosphate concentrate was achieved by reverse flotation. The overall recovery has been improved by recycling of fines. The flow sheet developed for the process is shown in Fig. 10 which includes recycling of slimes (fines) and their addition into cleaner stages of flotation.

Lock cycle flotation test. At the last, locked cycle flotation tests were carried out for development of process flowsheet and to establish metallurgical balance sheet. In each complete cycle test, rougher concentrate was ground and middling in the form of cleaner tailings was circulated to previous step in the subsequent test cycle to reach equilibrium condition. Addition of a small amount of collector was required after regrinding of middling products. It is evident from the P_2O_5 distribution between the cleaner concentrate and the cleaner tailings (Table 3) that the better grade concentrate is produced after cleaning stage of flotation. Reverse flotation of cleaner concentrate further improved the grade of final concentrate. The locked cycle flotation tests have revealed that Tarnawai phosphate rock

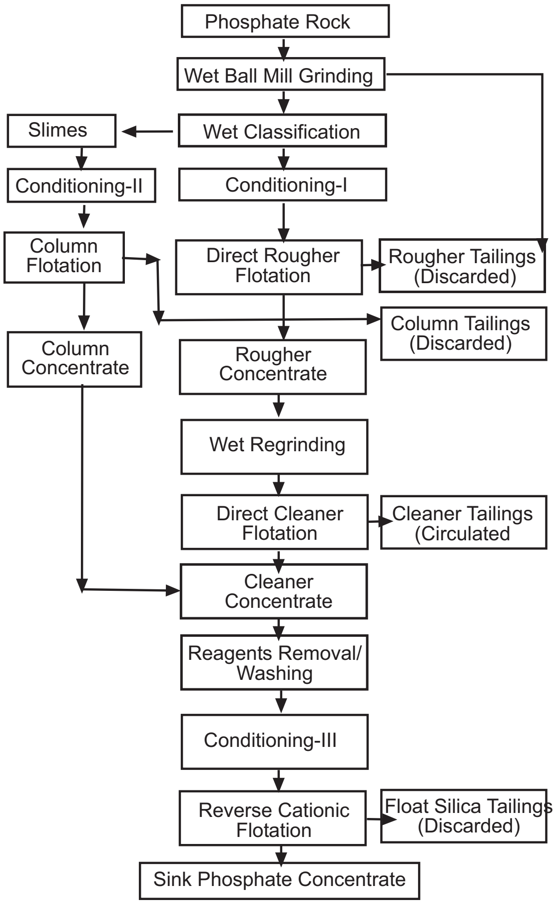

Fig. 10. Flowsheet developed for the beneficiation of phosphate rock of Tarnawai.

Table 3. Metallurgical balance sheet of a typical phosphate rock flotation test.

Flotation products	Weight (%)	Grade (%) P_2O_5	Recovery (%) P_2O_5
Sink phosphate concentrate	59.16	32.85	88.14
Float phosphate tailings	6.26	6.06	1.72
Mixed phosphate concentrate	(65.42)	(30.29)	(89.86)
Column phosphate concentrate	(5.78)	(29.87)	(7.83)
Column phosphate tailings	3.64	8.36	1.38
Cleaner phosphate concentrate	(59.64)	(30.33)	(82.03)
Cleaner phosphate tailings	7.57	11.68	4.01
Rougher phosphate concentrate	(67.21)	(28.23)	(86.04)
Rougher phosphate tailings	23.37	4.48	4.75
Phosphate fines (slimes)	9.42	21.56	9.21
Head sample of Phosphate rock	100.00	22.05	100.00

assaying 22.05% P_2O_5 can be upgraded to commercial grade phosphate concentrate i.e. more than 32% P_2O_5.

Chemical analysis of final concentrate. Table 1 presents the chemical composition of phosphate rock before and after beneficiation process. It indicates the impact of up-gradation as is clear from comparison of ore and concentrate composition. The silica content of the concentrate is 4.96% which has been reduced significantly from 20.86% present in investigated ore. Other undesirable impurities have also been minimized by flotation process. As a consequence of up-gradation, P_2O_5 content has been raised from 22.05% to 32.85% in the final phosphate product. The final dried phosphate concentrate produced was found to be free of flotation reagents. It meets the specification of chemical and fertilizer grade.

Conclusion

The P_2O_5 content of investigated phosphate rock of Tarnawai area, Hazara division is 22.05% which is quite enough to beneficiate it economically. The two stage direct and reverse flotation technology was found to be suitable and effective technique for the up-gradation this siliceous ore. However the performance and efficiency of phosphate rock up-gradation by flotation is sensitive to many parameters. Desliming of the fine particles prior to direct flotation has greatly improved the quality and recovery of concentrate. The use of column flotation enables to the achieve recovery of concentrate closer to perfect. The reverse flotation has improved the grade of final concentrate to a level that would not be possible using only direct flotation. The overall flotation process produced a commercial grade phosphate concentrate assaying 32.85% P_2O_5 with acceptable recovery of 88.14%. The obtained concentrate can be utilized for the production of chemicals and fertilizer.

Conflict of Interest. The authors declare no conflict of interest

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