# Removal of Impurities from Tailing (Quartz) Obtained from Bitlis Kyanite Ore by Flotation Method

**Fatma Deniz Ayhan***
**Halime Abakay Temel**
Department of Mining Engineering, Dicle University
21280 Diyarbakır, Turkey
E-mail: dayhan@dicle.edu.tr*

**Volkan Bozkurt**
Department of Mining Engineering, Eskişehir Osmangazi University
26480 Eskişehir, Turkey

**Abstract**

*Today, waste mountains are the most important result of the deformed industrialization. Our waste producing society approaches in two different ways to eliminate the problem. These approaches are as disposal and revaluation of the material and production of concentrates from the tailing. In this study, the removal of impurities from tailing (quartz) obtained from Bitlis kyanite ore by flotation method and the production of quartz products suitable specifiations, required in glass industry were investigated. For this purpose, the quartz was washed by using a 0.038 mm screen for removing of chemical materials from the quartz surface and three groups experiments were made. In the first group experiments, the flotation of impurities was performed in acidic medium. The effects of pH, depressant amount, collector type, collector amount and frother type on the flotation were investigated in the first group experiments. In the second group experiments, the magnetic separation and flotation (in acidic medium) of impurities were made. In the third group experiments, the magnetic separation and flotation (in neutral medium) of impurities were made. As a result, the quartz product obtained from the second group experiments was met the suitable properties, used in glass industry.*

**Keywords:** Quartz, tailing, Bitlis kyanite ore, flotation, magnetic separation

## *1. Introduction*

In today, industrialized and progressive countries are faced with environment pollution that is one of the most important problems. Industry continous must be produced new products to meet the necessities of life and the demands of development and social living. But, while the demands are met, selection of products and technology that produces little disposal and preference of raw materials that are not threatening both nature and human health, and utilization of nature source with high productivity and revaluation of disposals that are not as pollution source are an universal responsibility. Industrial minerals, such as silica sand, feldspars and kyanite, are often contaminated with impurities of unwanted components, particularly in form of iron, titanium and aluminium oxides. These impurities cause serious problems during their use in various applications, such as the production of colorless or optical glass, optical fibers and high purity ceramics. The usage and place of floated material for glass production are determined by the iron content ($Fe_2O_3$) of quartz sand (Farmer et al., 2000). Quartz used for glass production in plain glass quality and in glassware glass objects quality should be contained <0.05% $Fe_2O_3$ content and < 0.02 $Fe_2O_3$ content, respectively (Ay et al., 2000).

The stability of aqueous silica sols is of particular interest to colloid science (Kline ve ark. 1996, Rubio ve ark. 1976, Healy, 1994). The surface chemistry of silica has been the subject of a recent comprehensive review (Bergna, 1994). The iron content can be reduced by a number of physical, physicochemical or chemical methods, the most appropriate method depending on the mineralogical forms and distribution of iron in the ore (Taxiarchaou et al., 1997; Farmer et. al., 2000; Ay et al., 2000). Anionic collectors commonly are used in iron oxides and quartz flotation; carboxylate, sulphonate, alkyl sulphate, hydroxamate (Fuerstenau et al., 1976). In 1939, Keck et al., reported the collecting power of fatty acids on hematite to be in order of oleic>lauric>myristic>palmitic>carpylic. They also found that trithydroxyethylamine linoleate, diglycol stearate and sodium oleyl sulphate floated hematite although these are not as potent as the fatty acids. Potassium octyl hydroxamate is as superior to fatty acids, in selectivity, in iron ore flotation system (Fuerstenau et al., 1967; Fuerstenau et al., 1970). The potential determining ions may be regarded as prime reason for the existence of the electrical double layer, the counter being drawn by electrostatic forces to maintain electroneutrality at the interfacial layer.

Electrokinetic (and potentiometric) methods are established that hydrogen and hydroxyl ions, $H^+$ and $OH^-$, are potential determining ions for iron oxides and quartz (Raghavan et al., 1975; Somasundaran et al., 1975). The points of zero charge for some iron oxides are; hematite: pH 6.7, magnetite: pH 6.5, cumingtonite: pH 5.2, Synthetic hematite: pH 8.1, goethite: pH 6.7 (Aplan et al., 1962). Both cationic and anionic collectors are employed in iron ores and quartz flotation. In general, the collectors are long-chained, with a minimum of ten carbon atoms present in the hydrocarbon portion (Uwadiale, 1990). Iron oxides are floated effectively in the usage of petroleum sulfonate in acidic medium (pH 3-4) (Akar et al 1997; Ayhan et al., 2004; Ayhan et al., 2005). In the oleate/hematite flotation system, the highest concentrate recovery is obtained around pH 8.0 under optimum conditions (Kulkarni et al., 1975; Kulkarni et al., 1980), recovery is dropped progressively down to <10% and <40% at pH 4.0 and pH 9.0, respectively. The oleate acid soap ion, (RCOO) $H^+$, which holds the same pattern in the oleate species distribution diagram, come to mind in this regard. The acid soap complex is represented a larger surfactant molecule and is more surface active than other oleate species (Kulkarni et al., 1980; Somasundaran et al., 1979). In this study, the removal of impurities from tailing (quartz) obtained from Bitlis kyanite ore by flotation method was investigated. Experimental results are presented here.

## *2. Experimental*

The sample used in this study was a tailing obtained from Bitlis kyanite ore by flotation. The quartz was about 60 kilograms. Bitlis kyanite ore deposit was located at Bitlis Massif, 6 km south of Bitlis, Turkey.The kyanite ore was slightly weathered, and the kyanite content and mineralogical composition were considered to be representative of rock throughout the deposit. The rock was soft and friable and easy to crush and grind. The mineralogical and microstructural research was performed X-Ray diffraction, optical microscopy, and scanning electron microscopy. X-Ray diffraction, analyses were performed on a Philips powder diffractometer employing $CuK_\alpha$ radiation (40 kV, 30 mA) in the range 2θ) = 10-70$^o$ at a goniometer rate of 2θ = 2$^o$/min. The best flotation conditions of the Bitlis kyanite ore were determined and the beneficiation procedure for possible refractory uses of Bitlis Massif (Turkey) kyanite ore was developed by Ayhan et al. (2003). Laboratory batch flotation experiments were undertaken to determine the optimum conditions for separating impurities from the quartz. Experimental studies were made at three groups. In the first group experiments, the best flotation conditions of impurities in acidic medium were determined. In the second group experiments, firstly, the quartz was subjected to high intensity wet magnetic separation process.

Then, the flotation of obtained non-magnetic product was performed at the best conditions of first group experiments. In the third group experiments, the non-magnetic product obtained from high intensity wet magnetic separation process was subjected to flotation process in neutral medium. The flotation of impurities experiments were performed in a Denver laboratory flotation cell fitted with a 1 liter stainless steel tank at a solids concentration of 30%. The solid concentration was determined according to pre-experiments. Cytec International supplied the flotation reagents. At the beginning of experiments, the quartz was washed by using a 0.038 mm screen because of the sample used in this study was a tailing obtained from the kyanite flotation. In all flotation tests, pH was controlled continously and tap water was used throughout the experiments. The floated and tailings fractions were filtered and dried in an oven at 100 ± 5$^o$C to constant weight and analysed for $SiO_2$ and $Fe_2O_3$ content using XRF (X-ray Fluorescence) spectrometry at MTA (General Directorate of Mineral Research and Exploration, Ankara, Turkey). The high intensity wet magnetic separation experiments were performed with Master Magnets separator.

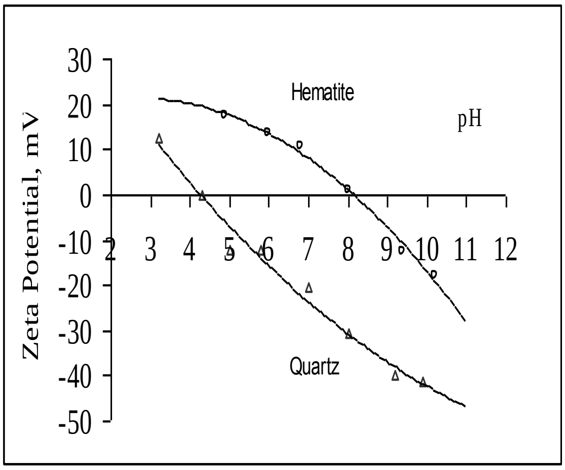
## *3. Result and Discussion*

At the end of petrographic examinations, it was observed that the kyanite, hematite and quartz in Bitlis kyanite ore were liberated in the particle size -0.1 mm. In addition, the relatively high amount of quartz detected by X-ray diffraction (Figure 1). Chemical analysis results of the quartz (Sample 1) used in the first group experiments and the quartz used in the magnetic separation process (Sample 2) are given in Table 1.

**Table 1.** Chemical analysis results of the Sample 1 and Sample 2.

| Content (%) | Sample 1 | Sample 2 |
|---|---|---|
| $SiO_2$ | 71.8 | 78.5 |
| $Fe_2O_3$ | 1.8 | 0.6 |
| $Na_2O$ | 0.1 | 0.1 |
| $K_2O$ | 0.08 | 0.08 |
| $Al_2O_3$ | 19.6 | 17.1 |
| $TiO_2$ | 0.01 | nil |
| CaO | nil | nil |
| MgO | nil | nil |

As it can be seen in Table 1, it was evident that the only significant impurity in the quartz was the iron content; the $Al_2O_3$ was acceptable, but could be reduced if necessary.




The adsorption of many collectors onto oxide and silicate surfaces was through electrostatic interaction and, therefore the isoelectric point of the mineral in question was generally critical (Smith at al., 1976; Fuerstenau at al., 1976; Fuerstenau at al., 1985). Collectors are prepared for attachment to air bubbles to mineral surfaces (Klassen, 1963). The surface charges of quartz and hematite were measured against pH to promote a theory for the flotation mechanism. Zeta potentials were determined using a Zeta-Meter (ZM-77 manufactured by Zeta-Meter, USA) employing a flat cell. About 1 g/l of mineral suspension was prepared in $10^{-3}$ M $KNO_3$ supporting electrolyte solutions and conditioned for 1 h at room temperature (22 ± 2°C). The zeta potentials of quartz and hematite as a function of pH are given in Figure 2. The results indicate that the isoelectric point of quartz is at pH 4.2 and that of hematite is at pH 8.

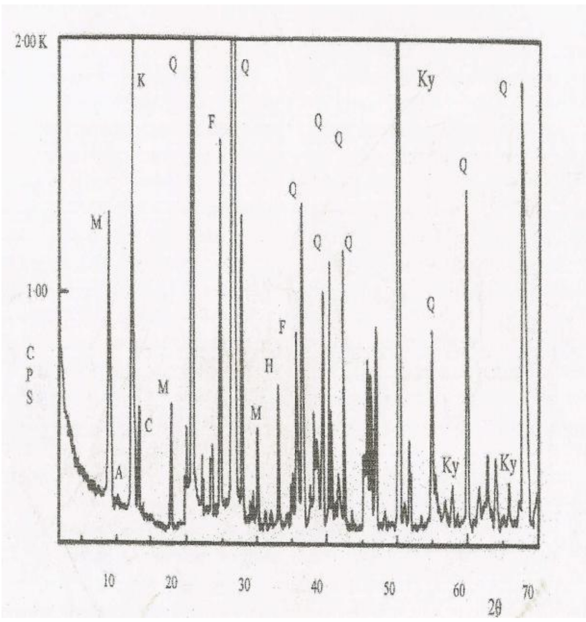



Figure 1. X-ray diffractograms of Bitlis Masif kyanite ore. Principal phases are: Ky, kyanite; Q, quartz; K, kaolin; H, hematite; F, feldspar; M, mica; C, calcite; and A, amphibole

**Figure 2**. The effect of pH on zeta potential of quartz and hematite

The removal of colourful and iron impurities from quartz sand could be made by using both magnetic separation and flotation method. The melting temperature was fallen by the impurities placed on quartz sand and the removal of impurities was performed with flotation method (Kurşun et al., 1995; Akar, 1994). In the first group experiments, the effects of pH, depressant amount, collector type, collector amount and frother type on the flotation of impurities were investigated. The removal of hematite from Bitlis-Hürmüz kyanite was made succesfully by Ayhan et al. (2005). The usage of HF as depressant reagent had negative effect on environment (Eddy et al., 1972; Immo, 1981). Therefore, the removal of impurities from quartz was investigated in both acidic medium and neutral medium.

According to various studies (Abdel-Khalek et al., 1994; Bayraktar et al., 1999; Bayraktar et al., 1997;Keck et al., 1939), petroleum sulphonate promoters were widely used in silica glass sand and feldspar flotation to remove iron oxides. Investigated flotation test parameters for the floating of impurities from quartz in the first group experiments are given in Table 2. Test results are given in Figure 3, 4, 5, 6 and 7. The evaluation of flotation results was made according to the $SiO_2$ content and recovery of the tailings.

**Table 2.** Investigated flotation test parameters of the first group experiments

| **Parameters** | |
|---|---|
| pH | 2, 2.5, 3, 3.5, 4 |
| Depressant amount | 100, 150,200,250,300, 350,400,450,500,550, 600, 650, 700,1000 g/t |
| Collector type | Armac C,T,TD, Aero 840,845,830,801,825 |
| Collector amount | 200,300,400,450,500, 550,600,700 g/t |
| Mixing speed | 1000 rpm |
| Frother type | MIBC, DF 250, Pine oil, AF 76 |
| Frother amount | 50 g/t |
| Conditioning time | 5+5+5 min |
| Flotation time | 2 min |
| Depressant type | HF |

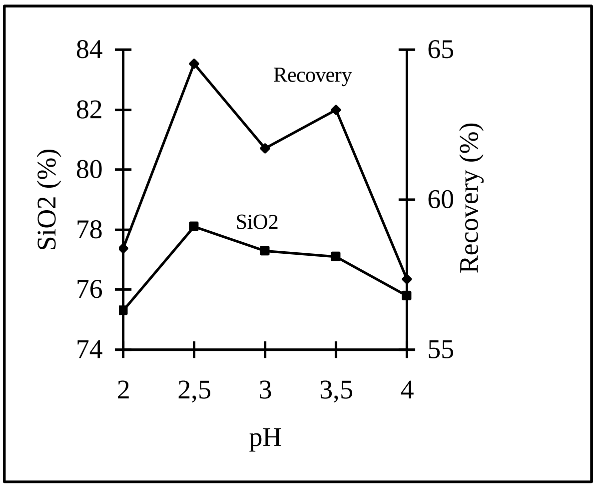



**Figure 3.** The effect of pH on the $SiO_2$ content and recovery of tailing

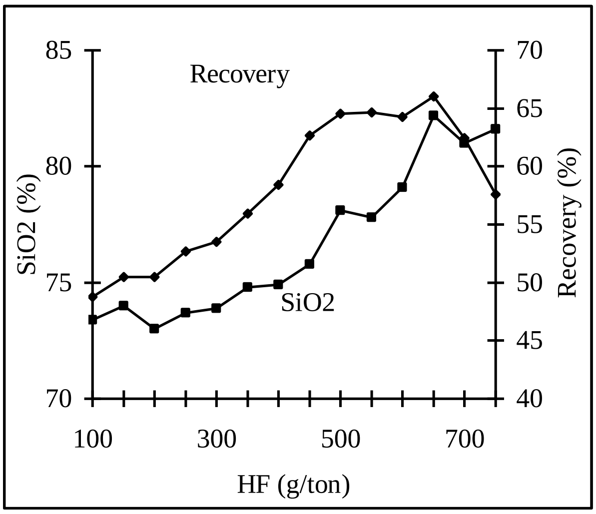



**Figure 4.** The effect of depressant amount on the $SiO_2$ content and recovery of tailing

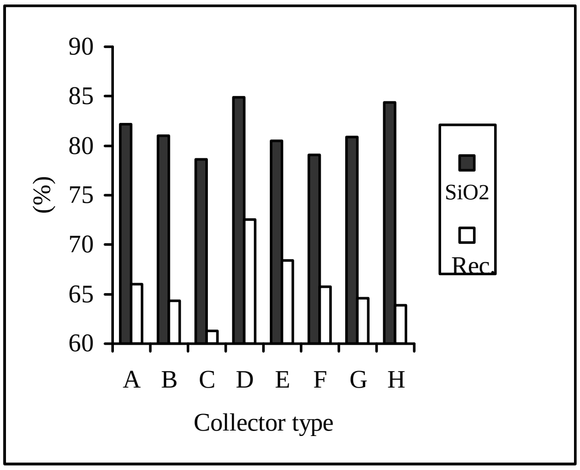



**Figure 5.** The effect of collector type on the $SiO_2$ content and recovery of tailing (A: Armac T, B: Armac TD, C: Armac C,D: Aero 840, E: Aero 845, F: Aero 830,G: Aero 801, H: Aero 825,)

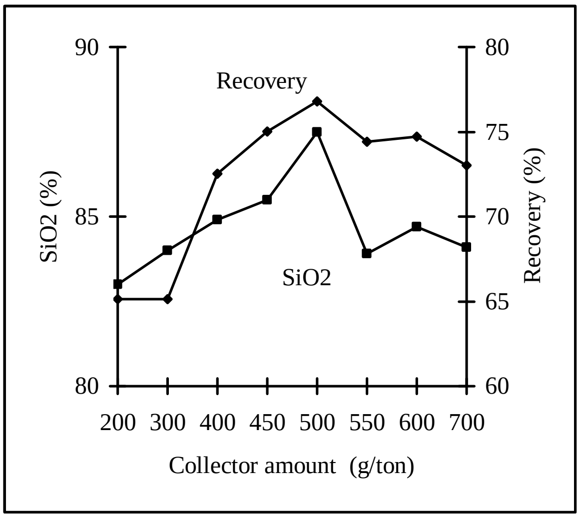



**Figure 6.** The effect of collector amount on the $SiO_2$ content and recovery of tailing

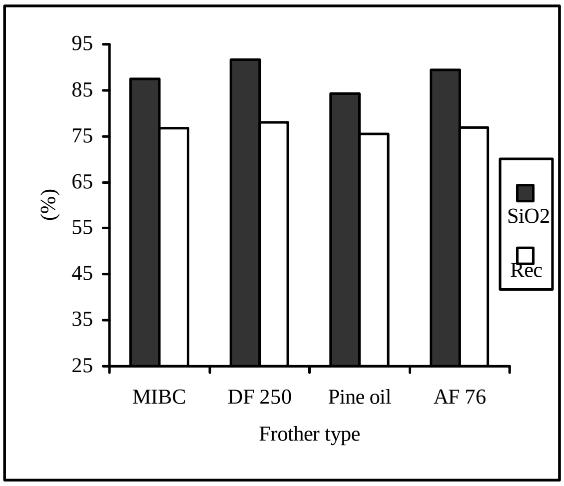



**Figure 7**. The effect of frother type on the $SiO_2$ content and recovery of tailing

It was found that the best flotation conditions of the first group experiments were as pH: 2,5 (with $H_2SO_4$; NaOH), HF: 650 g/t, Aero 840: 500 g/t, DF 250: 50 g/t. The best conditions also were used in the second group flotation experiments. The sink quartz products obtained from the first and second group experiments were subjected to 3 and 2 cleaning flotation processes at the same experimental conditions, respectively. Results of cleaning flotation of the first and second group experiments are given in Table 3 and Table 4.

**Table 3.** Results of cleaning flotation of the first group experiments

| Products | Wt.% | $SiO_2$ % | Recovery % |
|---|---|---|---|
| Concentrate | 49.67 | 98.8 | 68.35 |
| Middling 3 | 4.33 | 73.6 | 4.44 |
| Middling 2 | 4.0 | 61.5 | 3.43 |
| Middling 1 | 3.0 | 42.0 | 1.75 |
| Tailing | 39.0 | 40.56 | 22.03 |
| Total | 100.00 | 71.8 | 100.00 |

**Table 4.** Results of cleaning flotation of the second group experiments

| Products | Wt. %. | $SiO_2$ % | Recovery % |
|---|---|---|---|
| Concentrate | 67.00 | 99.3 | 84.75 |
| Middling 2 | 5.33 | 82.0 | 5.57 |
| Middling 1 | 4.67 | 55.5 | 3.3 |
| Tailing | 23.0 | 21.76 | 6.38 |
| Total | 100.00 | 78.5 | 100.00 |

Experimental conditions of the third group experiments determined according to pre-experiments were as pH: 7, Aero 840: 150 g/t, $Na_2SiO_3$: 450 g/t, DF 250: 50 g/t. The sink quartz product obtained from the third group experiments was subjected to 2 cleaning flotation processes. Results of cleaning flotation of the third group experiments are given in Table 5.

**Table 5.** Results of cleaning flotation of the third group experiments

| Products | Wt. % | $SiO_2$ % | Recovery % |
|---|---|---|---|
| Concentrate | 56.33 | 86.61 | 62.21 |
| Middling 2 | 11.34 | 78.30 | 11.31 |
| Middling 1 | 11.00 | 34.90 | 4.89 |
| Tailing | 21.33 | 79.67 | 21.59 |
| Total | 100.00 | 78.50 | 100.00 |

Chemical analysis results of the quartz products obtained from three group experiments are given in Table 6.

**Table 6.** Chemical analysis results of the quartz products obtained from three group experiments

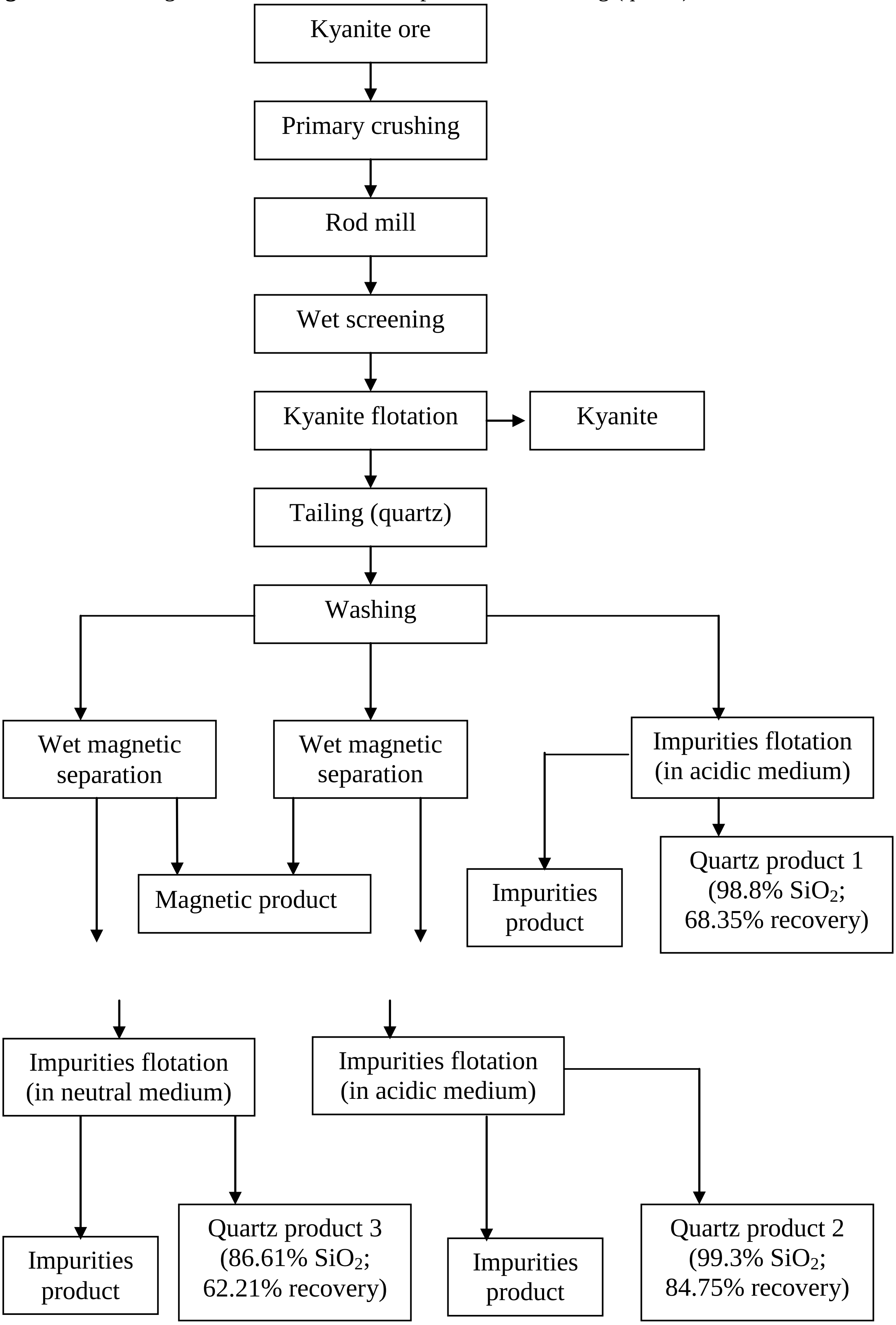
| Content (%) | Product 1 (First group) | Product 2 (Second group) | Product 3 (Third group) |
|---|---|---|---|
| $SiO_2$ | 98.8 | 99.3 | 86.61 |
| $Fe_2O_3$ | 0.05 | 0.01 | 0.01 |
| $Na_2O$ | 0.02 | 0.01 | 0.05 |
| $K_2O$ | 0.01 | nil | 0.01 |
| $Al_2O_3$ | 0.03 | 0.04 | 10.9 |
| $TiO_2$ | nil | nil | nil |
| CaO | nil | nil | nil |
| MgO | nil | nil | nil |

As it can be seen in Table 4, 5 and 6, the $Fe_2O_3$ contents of quartz products obtained from the second and third group experiments were found to be appropriate levels. The recovery of quartz product obtained from the second group experiments was higher than that obtained from the third group experiments because of the floating of kyanite was low at pH 7. Therefore, the $Al_2O_3$ content of quartz was not lowered sufficiently in the third group experiments. Flow diagram for the removal of impurities from tailing (quartz) obtained from Bitlis kyanite ore is given in Figure 7.

## *4. Conclusions*

The aim of this study was the removal of impurities from tailing (quartz) obtained from Bitlis kyanite ore by flotation. In this regard, three group experiments were made. The first group experiments results showed that a quartz product containing 98.8% $SiO_2$ and 0.05% $Fe_2O_3$ with 68.35% recovery was obtained by the flotation of impurities in acidic medium. The second group experiments results showed that a quartz product containing 99.3% $SiO_2$ and 0.01% $Fe_2O_3$ with 84.75% recovery was obtained by the magnetic separation and flotation of impurities in acidic medium. The third group experiments results showed that a quartz product containing 86.61% $SiO_2$ and 0.01% $Fe_2O_3$ with 62.21% recovery was obtained by the magnetic separation and flotation of impurities in neutral medium. The quartz product obtained from the second group experiments was met the suitable properties, used in glass industry.

**Figure 7.** Flow diagram for the removal of impurities from tailing (quartz) obtained from Bitlis kyanite ore



## *References*

Akar, A., 1994. Evaluation of Gördes Köprübaşı district feldspar deposit, Progress in Mineral Processing Tecnology, Editors Demirel, H., Ersayın, S., Proceedings of 5th Int. Mineral Process. Symp. Turkey, 243-249.

Ay, N., Arica, E., 2000. Refining Istanbul's Silica Sand, The American Ceramic Society Bulletin, August, 89-91.

Ayhan, F.D., 2003. Bitlis Masifi Disten Cevherinin Zenginleştirme Koşullarının Araştırılması, Ph.D Thesis, Mining Engineering, Çukurova University.

Ayhan, F.D., Abakay, H, 2005. Removal of hematite from Bitlis-Hürmüz kyanite for the achievement of concentrates suitable for refractory industry, Mineral Processing and Extractive Metallurgy (Trans.Inst.Min.Metall.C) (in press).

Ayhan, F.D., Bayat, O., Poole, C., 2004. The beneficiation of Bitlis Masif kyanite ore for use in ceramic materials, Mineral Processing and Extractive Metallurgy (Trans.Inst.Min.Metall.C), Vol. 113 CI.

Eddy, W.H., Collins, E.W., Browning, J.S., Sullivan, G.V., 1972. Recovery of feldspar and glass sand from South Carolina, waste granite fines, Brue of Mines, RI7651.

Farmer, A.D., Collings, A.F., Jameson, G.J., 2000. The Application of Power Ultrasound to The Surface Cleaning of Silica and Heavy Mineral Sands. Ultrasonics Sonochemistry 7, 243-247.

Fuerstenau M.C., Palmer B.R., 1976. Anionic flotation of oxides and silicates, in Flotation: A.M. Gaudin Memorial Volume, 1, Fuerstenau, M.C., Ed., 148-196.

Kline, S.R., Kaler, E.W., 1996. Aggregation of Colloidal Silica by n-Alkyl Sulfates. Langmuir, 12, 2402-2407.

Kursun, I., İpekoğlu, B., 1995. Türkiye kuvars kumu potansiyeline genel bir bakış, Endüstriyel Hammaddeler Sempozyumu, İzmir, Türkiye.

Immo, H., 1981. Flotation of feldspar, spodumene, quartz and mica from pegmatites in North Carolina, USA Proceedings of the 13 th Annual Meeting of the Canadian Min. Process. Ottowa.

Rubio, J., Kitchener, J.A., The Mechanism of Adsorption of Poly (ethylene oxide) Flocculant on Silica. J.Colloid Interface Sci., 57, n.1, 132-142, 1976.

Bergna, H.E., 1994. The Colloid Chemistry of Silica An Overview. In Advances in Chemistry Series, 234, American Chemical Society, Chapter 1, 1-47.

Healy, T.W., 1994. Stability of Aqueous Silica Sols. In Advances in Chemistry Series, 234, Chapter 7, 147-159.

Raghavan S., and Fuerstenau D.W. 1975. On the wettability and flotation concentration of submicron hematite particles with octylhydroxamate as collector, in Advances in Interfacial Phenomena of Particulate/Solution/Gas Systems: Application to Flotation Research, Somasundaran, P., and Grieves, R. B., Eds., AIChE Symposium Series, 150, 71, 59-67.

Somasundaran P. 1975. Interfacial chemistry of particulate flotation, in AIChE Symposium Series 150, 71, Somasundaran, P., and Grieves, R. B., Eds., 1-15.

Aplan F.F., Fuerstenau D.W., 1962. Principles of non-metallic mineral flotation, in Froth flotation 50th Anniversary Volume, Fuerstenau, D. W., Ed., AIME, 170-224.

Klassen V.I. Mokrousov V.A., 1963. An introduction to the theory of flotation, Butterworths, 492.

Kulkarni R.D., Somasundaran P., 1975. Kinetics of oleate adsorption at the liquid/air interface and its role in hematite flotation, in Advances in Interfacial Phenomena of Particulate/Solution/Gas Systems: Applications to Flotation Research, Somasundaran, P., and Grieves, R. B., Eds., AIChE Symposium Series 150, 71, 124-133.

Kulkarni R.D., Somasundaran P., 1980. Flotation chemistry of hematite/oleate system, Colloids and Surfaces, 1., pp. 387-405.

Taxiarchaou, M., Panias, D., Douni, I., Paspaliaris, I., Kontopoulos, A., 1997. Removal of Iron From Silica Sand by Leaching with Oxalic Acid. Hydrometallurgy 46, 215-227.

Uwadiale G.G.O.O., 1990. Selective oil agglomeration of Agbaja iron ore, Minerals&Metallurgical Processing, 7, No. 3, AIME, 132-135.

Somasundaran, P., Ananthapadmanabhan, K.P., 1979. Solution chemistry of surfactants and the role of it in adsorption and froth flotation in mineral-water systems, in Solution Chemistry of Surfactants, 2, Mittal, K. L., Ed., Plenum Pres, 777-800.