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Beneficiation of quartz waste by flotation and by ultrasonic treatment

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Abstract. Quartz is one of the most important industrial minerals used in glass and ceramic industries and high quality quartz are preferred. Impurities from quartz are usually removed by magnetic separation or flotation. This study is concerned to beneficiate the quartz from the quartz plant waste from Aydın-Çine region in Turkey by flotation and by ultrasonic bath treatment. The material with 94.8% Si is removed as a waste in the plant, after washing, grinding and classifying were applied. Flotation experiments showed that SiO₂ content increased to 98.3% with 80% recovery after removing the -53 micrometer size fraction by desliming. When ultrasonic bath treatment is applied the SiO₂ content increased from 94.8% to 99.2% after - 106 micrometer (30%) was removed as a slime. Results showed that better results were obtained with ultrasonic treatment. The aim of this study is to recover the quartz from waste and to add an economic value and therefore to reduce the waste stock for the environmental concern.

keywords: quartz, waste, beneficiation, ultrasonic bath treatment, flotation, impurities

1. Introduction

Industrial minerals, such as silica sand, kaolin and feldspars are often contaminated with deleterious impurities particularly in the form of iron, titanium and aluminium oxides. These industrial minerals are mainly used as raw materials for the production optical fibres, glass, ceramics and refractory materials. High quality quartz containing minor amount of iron and aluminium oxides are preferred. The specifications for different uses of silica sand are summarised in Table 1 (Chammas et al. 2001).

Iron minerals are generally separated from quartz by combination of magnetic separation and flotation process that feldspar is removed from quartz sands by a conventional froth flotation process by using amines as a collector and hydrofluoric acid as an activator which is harmful for the environment (Ozkan et al. 2001). In the literature, flotation of sand sample was subjected to sulfonate flotation to obtain sand concentrate having 0.11% Fe₂O₃ with 68% sand recovery (Önal et al. 2002). The other

study was also showed that using sulfonate type of reagents at pH 3 resulted a 99.5 % SiO_2 with 86% recovery and Fe_2O_3 content was decreased to 0.056 % by flotation after removing below 0.106 mm as a slime (Bayat et al. 2001). Vieira et al. (2007) has showed that particle size, even in a very narrow range, is very important for flotation results of quartz.

Table 1. Specifications of silica sand for different uses (Chammas et al. 2001)

Uses	Particle size (μm)	Chemical Composition
Glass-grade	100-600	SiO_2 98.5-99%, Al_2O_3 0.2-1.6%, Fe_2O_3 (<0.18% glass container) and Fe_2O_3 <0.04% (flat glass)
Fist Glass grade		SiO_2 99.8 %, < Al_2O_3 0.1 %, Fe_2O_3 <0.02 %
Optical Glass		Fe_2O_3 <10 ppm
Optical Fibers		Fe_2O_3 <1 ppm
Ceramic Grade	<75	SiO_2 97.5 %, Al_2O_3 < 0.55 %, Fe_2O_3 <0.2 %
Higher Premium ceramics	<45	Fe_2O_3 <100 ppm
Refractories		SiO_2 >97 %, Al_2O_3 < 0.1%, Fe_2O_3 <0.2 %, alkalis <0.3%
Foundry Grade		SiO_2 98.6.-99.6 %, Al_2O_3 0.08-0.5 %, Fe_2O_3 <0.0.3 %

On the other hand, the use of high power ultrasonic in industrial processes has increased last decade and the use of ultrasonic cleaning baths ranging from the small laboratory to large industrial cleaning tanks of several kilowatts is well known. Ultrasonic vibrations activate or accelerate many processes in liquid systems. The frequency range of 20-100 kHz is the most commonly used. Ultrasonic energy is form of mechanical vibration energy, propagates as waves through all material media including solids, liquids and gasses at characteristic velocity. Ultrasonic power can be applied externally as in a cleaning bath or by the insertation of an ultrasonic horn (solid probe) into slurry itself. Ultrasonic waves require only a liquid phase to transmit its energy to another system without the necessity of a special attribute that is the main benefit of ultrasound. Sound travels through a fluid as a three-dimensional pressure wave consisting of alternating cycles of compression and rarefaction. The rarefaction cycle is in the form of a negative pressure and the following compression cycle is a sudden burst of large and localised energy. The alternating behaviour of these cycles are known as the cavitation process, imposes significant effect on any solid phase within the liquid (Farmer et al. 2000; Önal et al. 2003; Altun et al. 2009). Numerous researchers evaluated ultrasound in chemical processes for cleaning of metal surfaces, acceleration of sedimentation and dewatering processes, waste water treatment and metal precipitation. One substantial benefit of ultrasonics in mineral processing can be the removal of surface coatings of clay and iron oxides from mineral surfaces. This is mainly achieved through the large, but very localised, forces produced by cavitation. Cavitation occurred by ultrasonic energy is a phenomenon of microbubble formation due to the degassing and change of phase to vapours. The

microbubbles are formed on the surface of the solids and assist in the separation of the solid and liquid due to the formation of gas liquid surfaces much lower surface energy compared to solid liquid surface (Farmer et al. 2000; Önal et al. 2003). The effect of energy released by cavitation is to help clean the surfaces of minerals suspended in slurry. Ultrasound can be used as a single process or as a pretreatment process or a combination with one of mineral processing techniques, such as to improve efficiency and/or selectivity of the flotation process (Cilek et al. 2009). Ozkan (2002) has found that applying ultrasound as a pre-treatment process or simultaneous treatment during magnesite flotation had positive effect. Also Ozkan and Kuyumcu (2006) and Altun et al. (2009) showed that ultrasonic coal and oil shales flotation yields more combustible recovery and lower ash values in concentrates than conventional flotation. Qi and Aldrich (2002) demonstrated that ultrasonic treatment is expediting precipitation, enhancing reagent adsorption and promoting the mechanical removal of the zinc hydroxide from the surface of the gypsum particles. The application of sonication to the reduction of iron oxide in a silica sand from 0.025% to less than 0.012% Fe₂O₃ is presented and the ultimate reduction in iron oxide contamination is reported to be dependent on the sonication power (Farmer et al. 2000). Önal et al. (2003) have showed that when ultrasonic treatment was applied during the sedimentation, it positively affected the sedimentation of clay and increased the settling rates by lowering the settling time. Ipek et al. (2001) and Sonmez et al. (2004) demonstrated that marketable product with high grade boron was obtained by ultrasonic sound waves as a process or as a pre-treatment before the magnetic separation. It has also been noted that ultrasonic treatment can be easily used at non-laboratory conditions with ultrasonic inducers and horns working at low power consumption. Although the investment expenses might be high for ultrasonic beneficiation but energy and operating costs are reported to be very low (Sonmez et al. 2004).

In this study, quartz tailing was experimented by using two different methods to compare them for removal of impurities. Reasonably high SiO₂ content was obtained from this sample with both methods and obtained material is suitable for a wide range of applications such as glass making.

2. Material and methods

2.1. Material

The material is a waste material of plant from Aydın-Çine region in Turkey. After crushing, grinding, classifying and washing were applied, about 1000 ton/year material is removed as a waste. The sample was analysed with X-ray Floresans Spectrometry by Cam-Ser A.Ş. The chemical analyses of material are shown in Table 2 that sample contains 94.8 % SiO₂ and main impurities are Al₂O₃ with about 3% and other alkalies. All impurities % increased as size fraction decreased. Table 2 also

shows the sieve analyses of the sample that 80% of the sample is below 425 micrometer and 8% is below 38 micrometer.

Table 2. Sieve and XRF analyses of sample

Size (micrometer)	Weight (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	TiO ₂ (%)	CaO (%)	MgO (%)	Na ₂ O (%)	K ₂ O (%)	LOI (%)
+850	6.00	98.81	0.55	0.045	0.008	0.05	0.04	0.07	0.12	0.31
-850+600	4.26	98.60	0.57	0.102	0.011	0.07	0.11	0.07	0.17	0.30
-600+425	7.68	98.66	0.60	0.078	0.011	0.06	0.17	0.07	0.17	0.18
-425+300	11.15	98.63	0.65	0.054	0.010	0.05	0.09	0.09	0.24	0.19
-300+212	14.47	98.16	0.93	0.054	0.016	0.05	0.04	0.15	0.36	0.24
-212+150	13.99	97.07	1.61	0.067	0.018	0.06	0.05	0.39	0.48	0.26
-150+106	13.47	94.79	2.97	0.381	0.025	0.08	0.06	0.97	0.62	0.10
-106+75	9.57	91.73	4.86	0.141	0.032	0.12	0.10	1.77	0.85	0.40
-75+53	6.52	88.75	6.45	0.226	0.047	0.21	0.23	2.18	1.12	0.77
-53+38	4.89	87.58	6.98	0.284	0.071	0.26	0.38	2.26	1.27	0.92
-38	8.00	83.83	9.72	0.091	0.162	0.12	0.42	1.45	1.81	2.04
Head	100.00	94.81	2.94	0.137	0.035	0.09	0.06	0.77	0.62	0.54

2.2. Flotation

Before the flotation tests, the sample was stirred, washed and then sieved on a 53 micrometer sieve and about 15% of slime was removed by this process. The analysis of the washed sample is shown in Table 3. This shows that SiO₂ content increased from 94.8% to the 97%, so that all impurities decreased. Therefore, flotation tests were applied to the washed sample. The particle size was below 425 micrometer in flotation experiments. Experiments were carried out in bench scale of 1 dm³ Denver type flotation cell. At the beginning of flotation, pulp is stirred at 900 rpm and 3 minutes of conditioning time was given for the reagents. Na Oleat was used as a collector for impurities and clay minerals at 2000 g/t dossages and 50 g/t pine oil was added as a frother. Flotation was performed for 10 minutes at 1200 rpm stirring rate. Flotation was performed at natural pH and no adjustment has been done.

Table 3. The analysis of washed sample

SiO ₂ , %	Al ₂ O ₃ , %	Fe ₂ O ₃ , %	TiO ₂ , %	CaO, %	MgO, %	Na ₂ O, %	K ₂ O, %	LOI, %
97.07	1.64	0.058	0.019	0.07	0.03	0.50	0.41	0.20

2.3. Ultrasound

Ultrasound was used as an alternative method to remove the surface coating of slime and clay type minerals from the quartz surface and beneficiate the quartz.

Ultrasonic bath experiments were performed in a stainless steel tank with 12 transducers in 400x300x450 mm dimensions and power supply with 500 W. Basically, in the ultrasonic bath an electrical current produced with high frequency a voltage by applying to crystals with piezoelectric properties that is obtained by high frequency vibrations. In the experiments, the tank was filled up to the determined level and it was run for 10 minutes to reduce the amount of solvent air and sample was added. Experiments were performed with 25% pulp density and 15, 30, 45, 60 minutes ultrasonic treatment time were chosen to see the effect of residence time to obtain optimum time. Then, pulp was screened through a series of sieves and sieve fractions dried, weighed and analysed.

Economical implication of the both processes in terms of practicality were not considered and the way of application of ultrasonic at industrial scale are not discussed in this paper, only laboratory test results are presented here.

3. Results and discussion

3.1. Flotation

Flotation tests were performed to float impurities from quartz. Flotation results are shown at Table 4 where first results are belong to 30% pulp density and second results are belong to 18% pulp density. Table 4 shows that 22% of sample was collected as a froth product with 4.84 % Al_2O_3 and 0.16 % Fe_2O_3 and 1.71 % Na_2O at 30 % pulp density. Therefore the non-float product was obtained with 80% recovery with 98.4% SiO_2 content and substantial amount of Al_2O_3 and Fe_2O_3 and Na_2O removal was achieved. But if 15% slime removal was also taken into account, overall recovery is 65%, which is low.

Özkan et al. (2001) obtained 99.4% SiO_2 with 83% recovery with NaOleat but it was a two stage flotation experiments where dodesil amin asetat (DAA) was also used as a second collector. When pulp density was reduced to 18%, lower grade and lower recovery was obtained and therefore this not improved the results.

Table 4. Flotation results

Products	Wt. %	Grade, %							SiO_2 Distribution, %
		SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	Na_2O	K_2O	
1.Float	21.8	92.34	4.84	0.162	0.048	0.14	1.71	0.76	20.77
1.Non-float	78.2	98.39	0.74	0.029	0.011	0.050	0.16	0.31	79.23
Feed	100	97.07	1.64	0.058	0.019	0.07	0.50	0.41	100.0
2.Float	25.4	95.47	2.63	0.126	0.026	0.09	0.77	0.53	24.95
2.Non-float	74.6	97.61	1.30	0.035	0.016	0.063	0.41	0.37	75.05
Feed	100	97.07	1.64	0.058	0.019	0.07	0.50	0.41	100.0

3.2. Ultrasound

Table 5 shows the SiO₂ grade of fractions and their distribution for the 0, 15, 30, 45 and 60 minutes of ultrasonic treatment. With or without any treatment SiO₂ content decreased as size fractions decreased. Without ultrasound application maximum SiO₂ content was 98.8% with maximum size fractions, +850 micrometer. When 15 minutes ultrasonic treatment was applied 99.4% SiO₂ was obtained with the fraction of -600+425 micrometer. When 60 minutes ultrasonic treatment was applied, 99.52% SiO₂ was obtained with the same fraction. When all treatment time was considered SiO₂ content decreased at -75 micrometer while +75 micrometer SiO₂ content increased. The mechanism of ultrasonic cavitation show that fine clay particles and iron oxides were removed from the quartz particle surface by the pull a part effect of ultrasonic sound waves.

Table 6 presents the Al₂O₃ content and distribution for all sieve fractions for 0, 15, 30, 45 and 60 minutes of ultrasound application. As size fraction decreased, Al₂O₃ content increased and, depending on the application time, about 35-45% of Al₂O₃ accumulated in below 38 micrometer size fraction. These results demonstrate that ultrasonic waves provide the dispersion in the pulp and contributed to removal of fines and clays out of quartz particles.

Table 7 demonstrates the Fe₂O₃ contents and distributions of all size fractions for 0, 15, 30, 45 and 60 minutes residence time. It shows that without ultrasound application Fe₂O₃ distribution was 38% in the -150+106 micrometer size fraction, but with application of ultrasound it decreased to 6%. It could be seen that 50-65% of Fe₂O₃ was concentrated on the -38 micrometer fraction which was 5% without ultrasound application. These results show that applying ultrasonic treatment enhanced the removal of iron oxides, from the quartz surface, especially from -150+106 micrometer fraction.

It can be seen from Tables 5, 6 and 7 that below 106 micrometer the Fe₂O₃ and Al₂O₃ contents are higher and as expected SiO₂ content is lower. Therefore if below 106 micrometer is separated, which is about 30% of the sample, than 70% of the sample with 99.2 % SiO₂ product could be obtained as a concentrate and below 106 micrometer fraction can be evaluated as tailings.

Figures 1, 2 and 3 are drawn to show the SiO₂, Al₂O₃, Fe₂O₃ content versus ultrasonic treatment time with +106 micrometer fraction which is evaluated as concentrate. Figure 1 shows the SiO₂ content for different ultrasonic treatment time. This shows that fines and clay particles were removed from quartz surfaces by using ultrasound waves and then by seiving efficient separation of quartz from the impurities was obtained.

It is also clear that as pulp's residence time in the ultrasound tank was increased SiO₂ content increased as well. But increasing the residence time to 60 minutes did not improve the results and similar results were obtained as 45 minutes. Fig. 2 demonstrates that Al₂O₃ content for different ultrasonic treatment time. Al₂O₃ content is reduced by application of ultrasound, but increasing the treatment time did not

improve the results and almost similar results were obtained for all residence time, only with 60 minutes residence time it increased slightly. Fig. 3 shows the Fe_2O_3 content of sample with treatment time. It could be seen that as treatment time increased a cleaner product have been obtained, but increasing it from 45 minutes to 60 minutes did not improve the results on the contrary Fe_2O_3 content is slightly increased.

These figures showed that ultrasound application time is certainly an important factor removal of impurities from the quartz particles and could be said that optimum time is 45 minutes for this particular waste material.

Table 5. SiO_2 content of sample with ultrasonic treatment

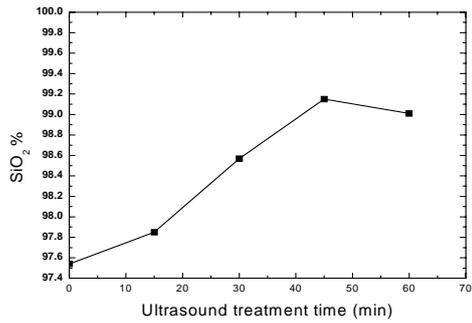
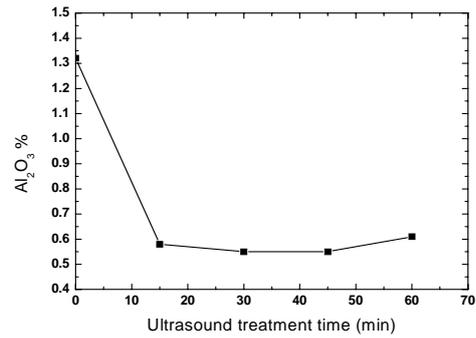
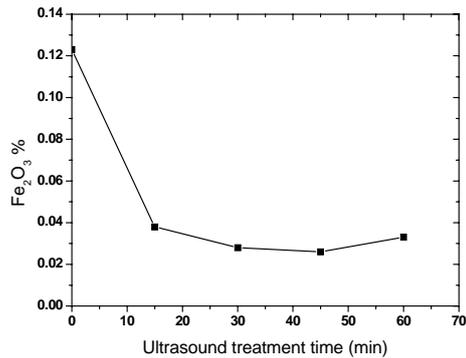
Size	SiO_2 Grade (%)					SiO_2 Distribution (%)				
Fraction	Ultrasound treatment time (minutes)					Ultrasound treatment time (minutes)				
(μm)	0	15	30	45	60	0	15	30	45	60
+850	98.81	99.24	99.33	99.34	99.33	6.25	6.55	6.84	6.05	6.21
-850+600	98.60	99.23	99.23	99.29	99.30	4.43	4.28	3.92	4.15	3.61
-600+425	98.66	99.39	99.44	99.48	99.52	7.99	8.27	7.81	7.96	7.18
-425+300	98.63	99.25	99.22	99.37	99.37	11.60	10.79	10.65	11.24	10.45
-300+212	98.16	98.95	99.09	99.06	99.03	14.98	13.74	13.60	14.22	15.26
-212+150	97.07	98.40	98.53	98.49	98.63	14.33	14.13	15.52	14.65	15.42
-150+106	94.79	97.54	97.57	97.34	97.36	13.46	13.44	13.39	13.88	17.05
-106+75	91.73	93.51	94.72	94.64	94.69	9.26	9.53	9.26	8.21	7.97
-75+53	88.75	86.61	87.32	87.00	87.33	6.11	4.86	5.41	5.79	4.77
-53+38	87.58	85.49	85.36	86.59	85.81	4.52	5.91	3.83	3.76	3.95
-38	83.83	81.35	80.70	80.85	80.44	7.07	8.63	9.99	10.27	8.69
Head	94.81	94.81	94.81	94.81	94.81	100.0	100.00	100.00	100.00	100.0

Table 6. Al_2O_3 content of sample with ultrasonic treatment

Size	Al_2O_3 Grade (%)					Al_2O_3 Distribution (%)				
Fraction	Ultrasound treatment time (minutes)					Ultrasound treatment time (minutes)				
(μm)	0	15	30	45	60	0	15	30	45	60
850	0.55	0.22	0.18	0.18	0.21	1.12	0.47	0.40	0.35	0.42
-850+600	0.57	0.22	0.18	0.21	0.17	0.83	0.31	0.23	0.28	0.20
-600+425	0.60	0.20	0.18	0.17	0.16	1.57	0.54	0.46	0.44	0.37
-425+300	0.65	0.29	0.24	0.22	0.23	2.46	1.02	0.83	0.80	0.78
-300+212	0.93	0.41	0.34	0.35	0.35	4.58	1.84	1.50	1.62	1.74
-212+150	1.61	0.71	0.66	0.67	0.61	7.66	3.29	3.35	3.21	3.08
-150+106	2.97	1.37	1.37	1.37	1.47	13.60	6.09	6.06	6.29	8.30
-106+75	4.86	2.33	2.32	2.36	2.33	15.83	7.66	7.31	6.60	6.32
-75+53	6.45	3.28	2.86	3.14	2.94	14.31	5.94	5.71	6.75	5.17
-53+38	6.98	3.36	3.42	3.25	3.96	11.61	7.49	4.95	4.55	5.88
-38	9.72	10.40	10.60	10.60	10.70	26.44	35.58	42.35	43.43	37.28
Total	2.94	2.94	2.94	2.94	2.94	100.00	100.00	100.00	100.00	100.00

Table 7. Fe₂O₃ content of sample with ultrasonic treatment

Size Fraction (μm)	Fe ₂ O ₃ Grade (%)					Fe ₂ O ₃ Distribution (%)				
	Ultrasound treatment time (minutes)					Ultrasound treatment time (minutes)				
	0	15	30	45	60	0	15	30	45	60
850	0.045	0.030	0.016	0.022	0.016	1.971	1.371	0.763	0.928	0.693
-850+600	0.102	0.039	0.028	0.032	0.079	3.172	1.164	0.766	0.927	1.989
-600+425	0.078	0.020	0.024	0.018	0.017	4.373	1.152	1.303	0.997	0.850
-425+300	0.054	0.036	0.023	0.018	0.019	4.395	2.709	1.709	1.408	1.383
-300+212	0.054	0.034	0.024	0.019	0.027	5.704	3.268	2.279	1.888	2.879
-212+150	0.067	0.044	0.032	0.025	0.025	6.842	4.371	3.490	2.573	2.704
-150+106	0.381	0.060	0.045	0.045	0.058	37.460	5.724	4.273	4.441	7.028
-106+75	0.141	0.091	0.076	0.072	0.073	9.849	6.416	5.142	4.325	4.252
-75+53	0.226	0.125	0.119	0.121	0.106	10.756	4.863	5.099	5.582	4.008
-53+38	0.284	0.161	0.146	0.135	0.132	10.137	7.709	4.529	4.050	4.211
-38	0.091	0.69	0.71	0.73	0.72	5.314	50.667	60.894	64.208	53.816
Total	0.137	0.137	0.137	0.137	0.137	100.00	100.00	100.00	100.00	100.00

Fig. 1. SiO₂ content versus ultrasonic treatment time with +106 micrometer size fractionFig. 2. Al₂O₃ content versus ultrasonic treatment time with +106 micrometer size fraction.Fig. 3. Fe₂O₃ content versus ultrasonic treatment time with +106 micrometer size fraction

As laboratory study showed that classifying very narrow size fractions did not change the results but below 106 micrometer SiO_2 content dropped dramatically. Therefore if below 106 micrometer is removed as tailings, totally +106 micrometer can be referred as concentrate with 99.2% SiO_2 . If industrial application is considered, -106 micrometer can be removed by hydrocyclones after 45 minutes ultrasound application. Although economical evaluation has not been done it can be easily said that it would be easier and more economical with ultrasound than flotation process.

4. Conclusion

In this study quartz plant waste was used as a sample and by stirring, washing and removing slime has already increased the SiO_2 grade from 95% to 97%. But it is still not suitable for specifications due to high Al_2O_3 , Fe_2O_3 , TiO_2 , Na_2O and K_2O and low SiO_2 content.

To increase the SiO_2 content and decrease the impurities, flotation was performed to the washed sample where 15% of the sample was removed as a slime. Na-Oleat was used as a collector in flotation at natural pH. 80% recovery with 98.3% SiO_2 content was obtained but when 15% slime was added, recovery can be calculated 65%. Although, recovery and grade is relatively low, obtained result is suitable for some certain specification.

When ultrasound was applied to remove the impurities from quartz; Fe_2O_3 content decreased from 0.123% to less than 0.026%, Al_2O_3 reduced from 1.32% to 0.546% and SiO_2 content was increased from 97.54% to 99.20%. If -106 micrometer is separated, 70% of sample with 99.2% SiO_2 can be obtained. The ultimate reduction of a contamination is dependent on ultrasound time and cleaner concentrates were obtained as the residence time was increased. Optimum results were obtained with 45 minutes ultrasonic treatment. The effect of ultrasonic waves on the dispersion of clay particles increased with increasing particle size fraction and ultrasonic waves penetrated better between coarser particles that leading to cleaner concentrates. Therefore, to use the process effectively and make the process beneficial, optimisation of ultrasonic treatment time and particle size are certainly critical factors.

When two methods were compared better grade and better recovery was obtained with ultrasonic treatment than flotation.

As a result, high grade quartz with reasonable recovery has been obtained and impurities-clay particles were removed from the surfaces of particles by using ultrasound waves. Obtained material is glass and foundry grade and suitable for manufacturing of these product.

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