

The effect of shape and roughness on flotation and aggregation of quartz particles

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Abstract: A combination of grinding and abrasion processes was applied to control the shape and roughness of quartz particles to investigate their roles in flotation recoveries and aggregation rates at different collector concentrations. The results showed that while the roundness values (R_o) of quartz particles varied in the range of 0.56-0.58 (R_o) at 480 and 1920 sec grinding, the roughness values of particles varied between 3.12-4.02 μm at 60 and 240 min abrasion. The flotation and aggregation tests showed while the flotation recovery increased from 31.3 % to 34.2 % in reverse proportion to their roundness values at 1×10^{-6} M DAH concentrations, a similar increasing trend from 34.1% to 38.1 % as a function of their roughness value from 3.12 μm to 4.02 μm . On the other hand, in the case of aggregation tests, while the turbidity values decreased from 40.6 NTU to 32.1 NTU at 1×10^{-6} M DAH concentrations for rounder particles, it was found as 36.2 NTU to 31.8 NTU for rougher ones. The overall results of this study indicated that tuning the morphology of quartz particles may be used to adjust both the flotation and aggregation rate of particles.

Keywords: quartz, grinding, abrasion, morphology, roundness

1. Introduction

In recent years, there is an upsurge of interest in understanding the contribution of the morphology of minerals in terms of particle-particle and bubble-particle interactions for improving the existing flotation and aggregation conditions (Guvan et al., 2015; Guvan et al., 2020; Uysal et al., 2021). In the literature, the contributions of many parameters have been studied to adjust the grinding and blasting conditions from the point of morphological characteristics of particles (Feng and Aldrich, 2000; Yekeler, Ulusoy, and Hicyilmaz, 2004; Koh et al., 2009; Guvan et al., 2015; Guvan et al., 2022). In most of these studies, the effect of grinding type has been extensively studied and their downstream effects on flotation characteristics of samples have also been presented.

For example, Feng and Aldrich (2000) showed that the selection of grinding type is of significant importance for the roughness of complex sulfide mineral particles. They showed that while rougher particles can be produced under dry grinding conditions, smoother particles could be obtained under wet grinding conditions. Accordingly, they showed that while the flotation kinetics increased as a function of roughness degree, the selectivity criteria decreased in the same trend. In another example of the effect of morphology on sulfide minerals, Vizcarra et al. (2011) found that while the angularity parameter of chalcopyrite particles was effective only on collector concentrations for low floatability rates, the collector concentration comes into the front at higher collector concentrations. In another example, Ulusoy et al. (2003) showed that while lower flotation recoveries were obtained for rounder particles, they gradually increased as a function of their angularity. More recently, in our previous reports on the effects of blasting on the morphology and floatability of quartz particles, it was found that while higher flotation recoveries were obtained for angular and rougher particles, they decreased in line with their morphological variations in terms of blasting pressure (Guvan et al., 2015). Wiese et al. (2015) investigated the effects of particle shape on entrainment. They found that spherical and elongated particles with a high aspect ratio resulted in less entrainment. Karakas and Hassas (2016)

investigated the effect of etching conditions on the roughness degree of glass beads and their effects on the interaction forces between particles under hexatrimethyl ammonium bromide (HTAB) medium. They found that roughness can be used to adjust the interaction forces between particles. In other words, while more attractive forces were measured for rough particles, they gradually decreased as the particles become smoother. More recently, Guven et al. (2016) investigated the effects of grinding and abrasion time on particle shape and roughness degree of alumina particles and their indirect effects on their flotation characteristics. As a result, it was found that while roughness and shape factors dominated the flotation characteristics of particles at low collector concentrations, shape factors became dominant at high concentrations. In another recent study, Turk et al. (2018) studied the effect of grinding time on the morphology of barite particles and their effects on their floatability. The results of their experimental studies showed that higher flotation recoveries could be obtained upon increasing the angularity of the particles. The role of particle roughness becomes less effective than the shape factor at high collector concentrations. In another recent study by Guven et al. (2020), the effect of grinding conditions on single and binary mixtures of chromite and serpentine was researched in terms of morphological variations and floatability of both single and their mixtures. The results of single mineral flotation and grinding tests showed that while the floatability of chromite increased as a function of its angularity degree, a reverse trend was obtained for serpentine minerals. On the other hand, the shape factor and floatability of the mixture were adjusted with the one with a higher density. In another study by Zhu et al. (2020) the effect of grinding conditions on the roughness degree of manganese minerals and their effects on floatability were investigated. They found that the particles with a high roughness degree showed higher flotation kinetics rate. Uysal et al. (2021) investigated the contribution of morphological indices on floatability and aggregation of sphalerite particles. They found that increasing the roundness value and roughness parameter resulted in higher flotation recoveries. The roughness of particles played a major role in their aggregation characteristics. Thus, the results reported in the literature showed that morphological parameters may vary based on the production method and structural properties of minerals in terms of their flotation and aggregation characteristics.

All in all, many studies are available in the literature on the contribution of both shape factor and roughness degree of particles on their floatability which were obtained with different grinding, blasting, or etching conditions. Although many studies have been related to the grinding conditions, to my knowledge, it's still unclear in the literature to systematically investigate the effect of morphology on both flotation and aggregation characteristics of samples. Therefore, this study aimed to determine the role of particle shape and surface roughness on the flotation and aggregation characteristics of quartz particles as a function of collector concentration in detail.

2. Materials and methods

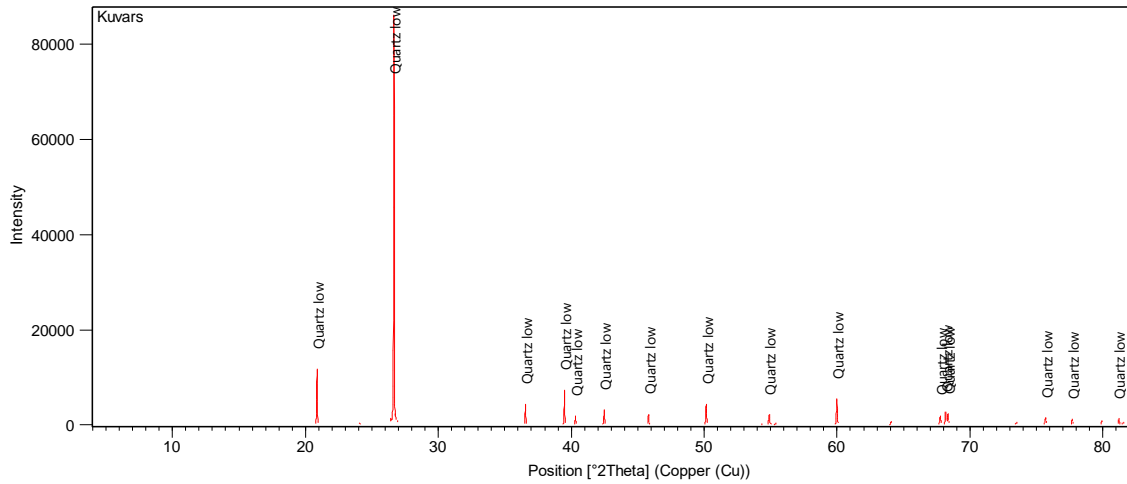
2.1. Materials

The quartz sample used in the experimental studies was provided by the ESAN mining company, Istanbul, Turkey. The purity of the sample was determined with X-ray Diffraction (XRD) method (Bruker D8 Advance Powder X-Ray Diffraction) and chemical analysis. The XRD patterns and chemical analysis of the sample shown in Fig. 1 and presented in Table 1, respectively, indicated that the sample was pure enough to carry out the experiments for investigating the contribution of morphology to flotation and aggregation characteristics of the sample.

2.2. Methods

2.2.1. Sample Preparation

The sample was first crushed by a series of crushers involving the jaw, cone, and roll crushers to obtain the particles of -2+1 mm in size for the grinding tests. Then, they were ground in a ceramic cylindrical ball mill with 5200 cm³ volume to prevent the possible contamination from decomposed grinding media. The properties of each grinding component are given in Table 2. In addition to this, to elucidate the contribution of the grinding process on particle size distribution, the powder/grinding media ratio (U) was kept constant as 0.5 throughout whole steps during different grinding times as 480, 960, and 1920 s. The value of the "U" parameter was calculated based on Equations 1-3 (Deniz, V., 2011).



In addition, as reported in previous studies for the effect of grinding conditions such as the values of void fraction (U) and ball filling (J) values, finer size ranges were obtained in accordance with the higher values of these parameters for different minerals (Deniz, V., 2012; Cayirli, S., 2014; Cayirli, S. 2018). Therefore, in this study, the value of the "J" parameter was selected as 0.2 in order to investigate the effect of grinding time on particle morphology while the values of the "U" parameter were chosen as 0.5 throughout grinding tests. After each grinding step, the sample was dry screened with laboratory-type screens with 74 and 38 μm apertures. Additionally, the samples within that size range were also wet screened for removing the possible very fine material on particles which would change the flotation and aggregation characteristics and morphological analysis (Güven et al., 2016; Güven and Celik, 2016; Uysal et al., 2021). The size distribution of samples was carried out with Malvern Particle Sizer 3000 and the d_{80} sizes of samples were 52.2 μm , 46.8 μm , and 42.2 μm respectively for increasing grinding time values.

Following the grinding step, the ground sample was divided into two parts with the quartering and coning method to investigate the effect of grinding time on shape factor and later abrasion processes for producing particles of different roughness values. As previously mentioned in the literature (Ulusoy, U., 2018, and Allen, T., 1997) although the reliability of this sampling method is a bit discussable compared to other methods such as "Scoop Sampling", "Table Sampling", "Chute Sampling", and "Spinning riffing", in order to increase the reliability of this sampling method, homogeneous sampling was made by taking samples from different sections of the sample in order to represent the sample. Thus, the shape factor as roundness values of ground particles was determined with ImageJ software. And, the selected particles with the lowest and the highest roundness parameter were selected and subjected to an abrasion process with silicon carbide (hereafter SiC) as a function of abrasion time. It is worth noting that during the abrasion process, only the abrasive material is mixed with the quartz samples for definite times resulting in very negligible changes in their particle size distribution. After these characterization steps, the samples were subjected to flotation and aggregation tests to determine the effects of morphology on the floatability and aggregation characteristics of particles.

2.2.2. Morphological Characteristics of Particles

Even though many papers reported the effects of different parameters such as grinding type and blasting (Ulusoy et al., 2003; Yekeler et al., 2004; Güven et al., 2015; Ulusoy, U., 2019; Ulusoy, U., 2018; Hassas et al., 2021), ball size (Kim et al., 2019) on morphological characteristics of minerals, to our knowledge, only a few studies reported the effect of grinding time (Ulusoy and Bayar, 2022; Güven et al., 2016; Güven et al., 2020; Uysal et al., 2021; Güven et al., 2021) on afore-mentioned indices.

In this study, a series of tests were conducted at different grinding times (4, 8, 16, and 32 min). The shape factors were determined on representative pictures of particles within -74+38 μm in size that was taken at the end of each grinding time. The morphological indices of samples were determined with the "Image Analysis" method by using a digital microscope with 50X magnification. In this context, although many parameters can be evaluated for morphological analysis of particles such as relative width, jaggedness, angularity, etc., regarding their contribution and role in both particle-particle and bubble-particle interactions, the value of the roundness parameter (Ro), in particular, was used for the evaluation of morphological characteristics for each particle (Mahmoud, A., 2010; Güven and Celik, 2016; Güven et al., 2020; Uysal et al., 2021). During these characterization tests, ImageJ software (Free of License) was used based on 2-D particle projections obtained from the micrographs of particles (Fig. 2). The roundness (Ro) values of about 120 particles were automatically calculated by the software and defined in Eq. 4 (Forssberg and Zhai, 1985):

$$\text{Roundness (Ro)} = \frac{4\pi A}{P^2} \quad (4)$$

where P is the perimeter and A is the area of particle evaluated by the software.

The scale of measurement was calibrated each time to obtain accurate and repeatable results for the roundness values calculated by the measured perimeter and the area of particles (Equation 1). In addition, the threshold of each picture was taken by automatic selection based on colors and adjusted settings like the limits of an area, circularity, etc. Thus, this feature provides an advantage for giving



Fig. 2. The image processing steps in ImageJ software (1-Original; 2-Threshold; 3-Analyzed). The pattern of the quartz sample

the ability to work with many particles, which are hard to be processed manually. Following their roundness characterization, the particles were subjected to the abrasion process by using SiC as a function of abrasion time following the same procedure reported in previous studies (Güven et al., 2016; Güven and Celik, 2016; Turk et al., 2018; Uysal et al., 2021). The surface roughness values of abraded particles at different abrasion times were determined with the contact profilometer device (Mitutoyo SV-2100 model, Japan). This instrument measures the average roughness (R_a) of surfaces by scanning across the surface of pellets made of $-74+38 \mu\text{m}$ -sized particles. The pellets were prepared under constant pelletizing pressure (10 bar) and time (5 min).

2.2.3. Characterization of particles in terms of shape and roughness

This study focused on the contribution of morphological parameters to flotation and aggregation characteristics of particles, roundness and roughness parameters of the particles were determined for different grinding times and abrasion times respectively. The results of morphological characterization and representative pictures of particles at different grinding times are shown in Fig. 3a in which the decrease in roundness values was initially abrupt and then became gradual as a function of grinding time, s (0.572-0.560).

Although the variation in roundness was not found considerable compared to the results of a previous study with blasted quartz particles reported in Güven et al. (2015), it was found in line with both the previously reported values for quartz particles as a function of grinding media (Ulusoy et al., 2003) and grinding time for different materials such as alumina and chromite which can be well attributed to the same breakage mechanism inside ball mill as a function of grinding time (Güven et al., 2016; Güven and Celik, 2016; Güven et al., 2020); From that point of view, it can be suggested that the grinding mechanism plays a significant role on particle shape besides grinding type and when it comes to abrasion or attrition like in ball mill grinding, the variation on roundness values became less significant compared to blasting (Güven et al., 2015).

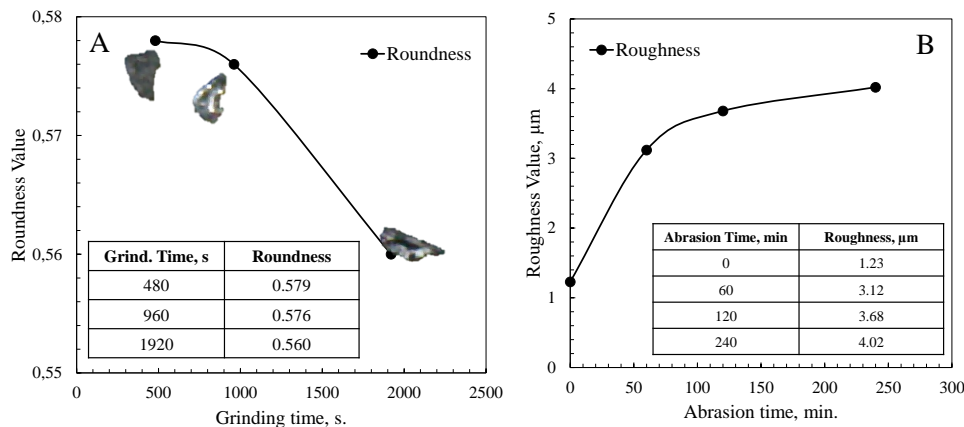


Fig. 3. (a) The effect of grinding time on roundness values of quartz particles (b) The effect of abrasion time on roughness values of quartz particles

Following the grinding tests, to evaluate the effect of roughness parameters on flotation and aggregation processes, the particles were abraded with SiC for different abrading times. The results are shown in Fig. 3b. As seen from the Fig., while a considerable increase was obtained at 60 sec of abrasion as 3.7 μm , it increased up to 4.0 μm at higher abrasion times. In the literature, while a significant increase was obtained for the roughness of the glass bead particles in 60 min of abrasion (from 6.7 μm to 9.1 μm), the value of roughness gradually decreased to 6.9 μm after 120 min of abrading time (Güven and Celik, 2016). However, in another example for sphalerite, while the roughness of sphalerite particles abraded for 15 min was found as 2.8 μm , it increased to 3.3 μm after 60 min of abrading and decreased to 3.0 μm after 75 min (Uysal et al., 2021). From this point of view, it can be seen that, although the same processes are performed and the same procedure was followed for producing rough surfaces, the effect of abrasive material on roughness will change based on the structural properties of the target material.

2.2.4. Zeta Potential Measurements

Zeta potential measurements were conducted with a microprocessor-equipped Zeta-Meter 3.0+ model instrument (Zeta-Meter Company, USA). The measurements were carried out under 75 V with a cell constant of 0.71 cm^{-1} . The procedure for these measurements was as follows: i) the addition of 0.5 g sample (under 38 μm where d_{50} and d_{80} sizes of the sample were 14.5 μm and 22.4 μm respectively) to 50 cm^3 of desired collector concentration (solids ratio 1% (w/v)) and mixing using a magnetic stirrer at 360 rpm for 5 min, ii) keeping the suspension for 5 min to allow the coarse particles settle down, iii) transfer of the suspension to the measurement cell. Ten measurements at each collector concentration were performed, and the average value of the measurements was obtained for both samples. An average error of measurements was ± 2 mV. All measurements were conducted using distilled water, the pH value of suspensions was measured before and after the measurements, and the pH of the suspension was adjusted to 9.5 ± 0.03 with 0.001 M NaOH.

2.2.5. Micro-Flotation Studies

Single mineral flotation experiments were carried out in a 155.5 cm^3 micro-flotation column cell (30x220 mm) with a ceramic frit (average pore size of 15 μm) which was mounted on a magnetic stirrer (the speed was fixed at 360 rpm.) and a magnetic bar ($r=1.5$ mm, $h=15$ mm) was used for the agitation. The experiments were performed with 1 wt. % solid rate, in the presence of a commercial collector "Dodecyl amine hydrochloride" ($\text{CH}_3(\text{CH}_2)_{11}\text{NH}_2\text{HCl}$ with 97% purity) produced by Alfa Aesar company. The samples were initially conditioned with the collector solution for 5 min and pH was adjusted to 9.5 ± 0.02 with 0.001 M NaOH (Sigma Aldrich, ≥ 97 % purity) to avoid precipitation of amine before the flotation experiments and maximize the efficiency of the collector. After conditioning, the suspension was transferred to the flotation cell and the samples were floated for 1 min by using N_2 gas at a flow rate of 50 cm^3/min . Following the flotation tests, both float and sink products were gravimetrically measured. The flotation recovery (ε) was calculated using Eq. 6:

$$\varepsilon = \frac{m_1}{m_1 + m_2} \times 100\% \quad (6)$$

where m_1 the mass of floated products is, and $m_1 + m_2$ is the mass of feed (including floated and non-floated products).

In addition to the tests as a function of amine concentration, another series of tests were carried out with particles of low, middle, and high roundness and roughness values respectively under low and high DAH concentrations for investigating the effect of particle morphology (roundness and roughness) on flotation recoveries of particles. All measurements were carried out three times, and the average values were used for evaluating the flotation characteristics of quartz samples with different morphology.

2.2.6. Aggregation Tests

As well-known, increasing the aggregation rate of particles will result in increased flotation recovery (Wan-Zhong et al., 2011; Güven et al., 2016; Uysal et al., 2021). Considering that, in these series of tests, the aggregation characteristics of quartz samples were determined by turbidity values under the same

conditions that were previously utilized in the micro-flotation experiments as collector concentration, pH, and conditioning time to mimic the micro-flotation conditions without bubble medium. To prove their repeatability, all measurements were performed at least three times and their standard deviation was ± 5 NTU. These measurements were made following the procedure explained in standard ASTM-D7315 with a 430IR model turbid meter (WTW, GmbH, Germany). The volume of the measuring unit was selected as 20 ml and due to the fast settling rate of particles, the ratio of the solid (w/v) was selected as 0.625 % to record the turbidity of solutions as a function of time.

3. Results and discussion

3.1. Effect of collector concentration on quartz flotation, aggregation, and zeta potential measurements

Before tests for determining the effect of particle morphology on flotation and aggregation characteristics of quartz, a series of flotation and aggregation tests were conducted as a function of amine concentration. In addition, the zeta potential measurements were conducted to provide the surface potential characteristics of samples in amine solutions. The combined presentation of the results of these tests is shown in Fig. 4.

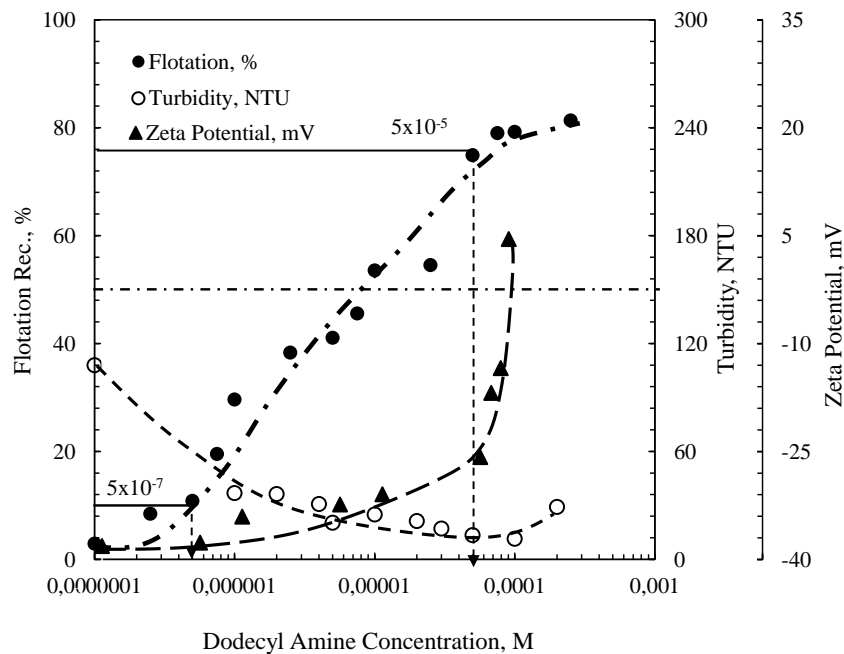


Fig. 4. Flotation and aggregation characteristics of quartz sample as a function of amine concentration along with zeta potential measurements

As shown in Fig. 4, regarding the results of flotation experiments, while negligible increases were obtained up to 5×10^{-7} M DAH concentration, a marked increase in the flotation recovery was obtained at higher concentrations over that concentration. This trend can also be ascribed to the formation of wetting films (that are generally referred to as flotation films) which can be characterized by zeta potential values in presence of the collector and the interaction between bubble and particle (Huang and Yoon, 2020). On the other hand, the turbidity results shown in Fig 4, indicated that while the particles were found in the dispersed form at lower concentrations such as 1×10^{-7} M, they started to decrease at 5×10^{-7} M concentration which also points out the same concentration. In the light of these findings, it was found that both flotation and aggregation characteristics of quartz minerals increased at an intersection concentration of amine as 5×10^{-7} M.

3.2. Effect of particle morphology on flotation and aggregation

As mentioned in the "Materials and Methods" section, besides the influence of other shape factors such as elongation rate, relative width, chunkiness, etc., in this study, the effect of the roundness parameter

is specially investigated due to its significant role on forming the dominant mechanism on bubble-particle and particle-particle interactions (Güven et al., 2016; Türk et al., 2018; Uysal et al., 2021; Güven et al., 2021).

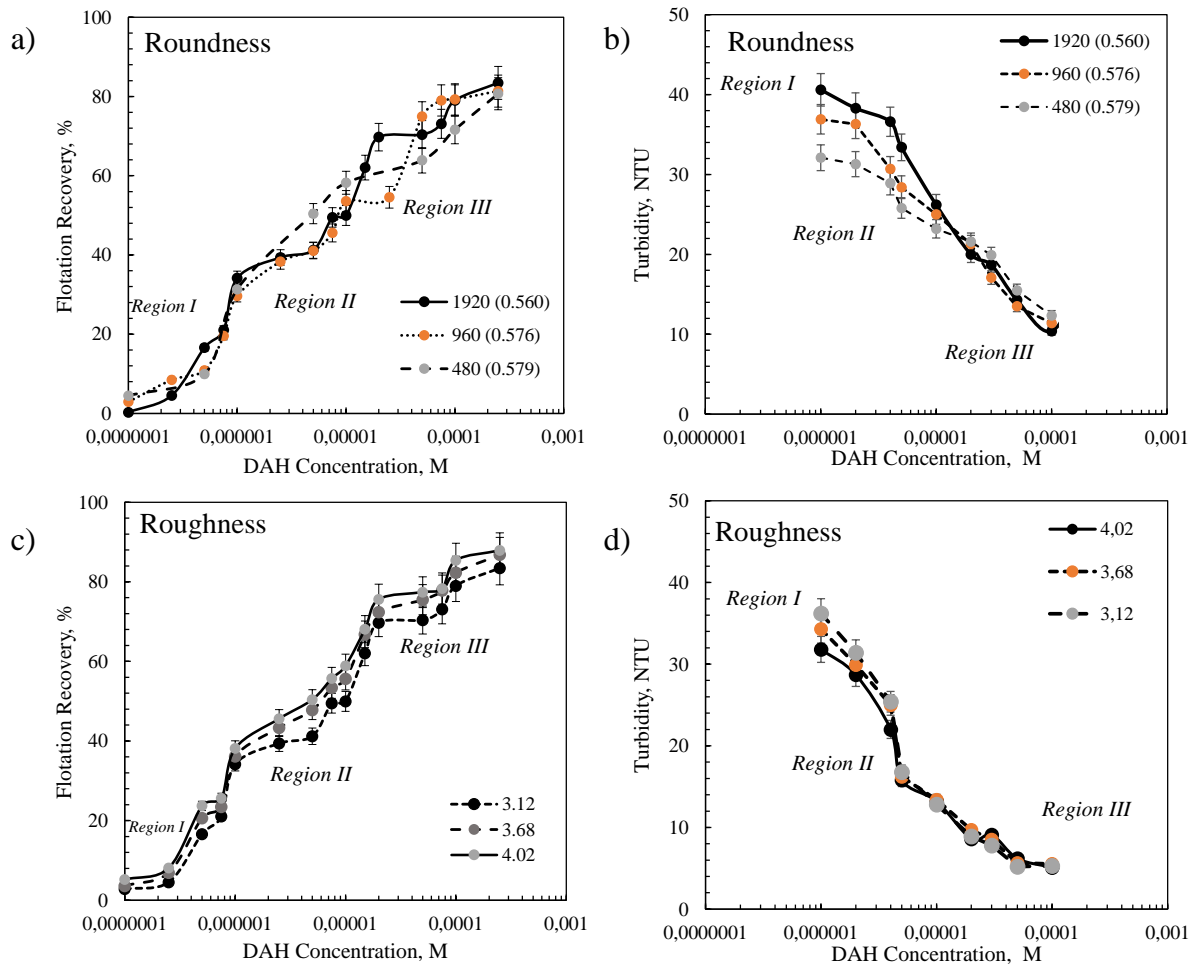


Fig. 5. (a) The effect of roundness on flotation recovery values (b) The effect of roundness on the aggregation rate of particles (c) The effect of roughness on flotation recovery of particles (d) The effect of roughness on aggregation characteristics of the quartz sample

Thus, in this study, morphologically characterized particles at three different grinding times were selected based on their roundness values (0.56, 0.57, and 0.58 after 480, 960, and 1920 sec grinding, respectively), and the micro-flotation experiments were conducted with these selected materials at different collector concentrations to investigate the effect of roundness on quartz flotation recoveries (Figs. 5a and 5c).

Following the effect of collector concentration on flotation and aggregation characteristics of quartz samples regardless of their morphological features (Fig. 4), the relation of morphological indices on the same processes was investigated by conducting experiments with particles of different roundness and roughness values. As mentioned in the "Materials and Methods" section, many studies were presented in the literature on these effects on the flotation of quartz and other minerals. For example, Ulusoy et al. (2003) investigated the effect of different grinding media on the morphology of quartz and accordingly their effects on the wettability degree of quartz. In discordance with this study, the wettability of particles was evaluated by the critical surface tension values. As a result, they found that the wettability degree varies by the roundness values of particles, it changed in reverse proportion with roughness values. In other words, the wettability degree became the lowest in terms of their roundness values for the products of ball milling (0.912), and it became the highest in terms of their roughness degree (4.49 μm). In this study, a similar trend was obtained for the contribution of roundness to the flotation

experiments, but a reverse trend was obtained for the effect of the roughness parameter on floatability which can be attributed to the range of particle size and the evaluation method studied in the previous report. Thus, the results of these series of experiments can be separated into three regions. In the first region (labeled "Region I"), while the roundness value varied between 0.56-0.58, the flotation efficiency varied between 31.3% and 34.2% at 10^{-6} M *DAH* concentration. Similarly, in the second and third regions (labeled by "Region II and Region III) in Fig. 5a, the flotation recovery varied between 58.0-69.0% and 80.1-83.4%, respectively. These results in turn showed that while the range of variation was higher at lower *DAH* concentrations, its role in these results decreased gradually at higher *DAH* concentrations.

In addition to the roundness value of particles, the evaluation of the roughness parameter indeed would provide a wide focus for commenting on the contribution of morphology to flotation characteristics of quartz minerals. Accordingly, the results of experimental studies carried out with abraded particles showed that while the roughness value varied between 3.12- 4.02 μm , in Region I, the flotation recovery varied between 34.1% and 38.1 at 10^{-6} M *DAH* concentration as shown in Fig. 5c. However, at higher *DAH* concentrations such as 1.5×10^{-5} M *DAH*, the flotation recovery values varied between 66.8-68.1 % which in turn showed that while the flotation characteristics of quartz sample can be adjusted by its roughness value at lower concentrations, it became almost independent from its morphological indices. Thus, the results of this study also demonstrated that while the morphological indices of particles can adjust the flotation conditions at lower collector concentrations, the collector itself would be dominant at higher concentrations. In sum, the contribution of morphological parameters on the flotation of quartz particles showed that while the flotation recovery increased up to 1.1 times at low *DAH* concentrations such as at $1 \cdot 10^{-6}$ M in the case of rounder particles, it was found as 1.2 for rougher ones. In conclusion, these results indicated that tuning the morphology in terms of roundness and roughness parameters would lead to improvement in the flotation recoveries.

On the other hand, the results of the aggregation characteristics of samples under the same conditions with flotation experiments (Figs. 5b and 5d) showed that at lower *DAH* concentrations such as 1×10^{-6} M labeled in Region I, while the turbidity value was measured as 40.6 NTU for the angular particles (0.56), it was found as 32.1 NTU for rounder particles (0.58). From that point of view, it can be clearly understood that while the angular particles were found a bit more dispersed in suspension, aggregates of particles were formed with rounder particles. This trend continued up to 1×10^{-5} M *DAH* concentration and while the turbidity value was measured as 26.2 NTU for angular particles, it was found as 23.2 NTU for rounder particles. However, above that concentration, a reverse trend was obtained. In other words, lower turbidity values were obtained for angular particles such as 11.4 NTU at higher *DAH* concentrations shown in Region III. It increased to 12.4 NTU for rounder particles. In the series of tests for investigating especially the roughness values of particles, it can be seen that while lower turbidity values were obtained for rougher particles regardless of their degree and collector concentration, higher turbidity values were measured for smoother ones. Therefore, it can be clearly understood that tuning the roughness would not only increase the flotation recoveries by bubble-particle interaction but also increase the aggregation rate by particle-particle interactions. In addition, if a correlation is made between the effects of amine concentration on flotation and aggregation of particles (see Fig. 5), it can be considered that while roughness plays a major role in both flotation and aggregation tests at lower *DAH* concentrations than 5×10^{-7} M, the roundness parameter could come into the front at higher concentrations.

4. Conclusions

A systematic grinding procedure was followed for the grinding of quartz particles for isolating the effects of grinding conditions in terms of morphological variations. Thus, this study provided detailed information about the flotation recovery, aggregation rate, and their relation with the particle morphology. In this manner, the quartz particles were ground in a ball mill as a function of grinding time and then abraded with SiC as a function of abrading time to produce particles with different shapes and roughness degrees. The results of these series of tests showed that while the roundness parameter varied in the range of 0.52-0.58 upon 1920 sec of grinding time, the flotation recovery increased from 31.3 to 34.2%. Likewise, when the roughness values of the abraded particles increased from 3.12 μm to 4.02 μm , the flotation recoveries increased from 34.1% to 38.1%. However, in terms of aggregation

characteristics, when the particles were more angular, the turbidity value was measured as 40.6 NTU, it decreased to 32.1 NTU upon rounder particles at the same *DAH* collector concentration. In addition, lower turbidity values were obtained for rougher particles regardless of their degree and collector concentration, and higher turbidity values were measured for smoother ones. Overall, the results of this study clearly showed that adjusting the morphology of particles will lead to stronger interactions between particles which then resulted in higher flotation recoveries and aggregation rates.

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