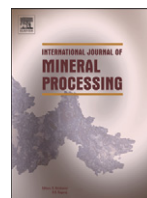




Contents lists available at ScienceDirect

## International Journal of Mineral Processing

journal homepage: [www.elsevier.com/locate/ijminpro](http://www.elsevier.com/locate/ijminpro)

## Dependence of morphology on anionic flotation of alumina

Onur Guven, Firat Karakas, Nurgül Kodrazi, Mehmet S. Çelik\*

Istanbul Technical University, Mining Faculty, Mineral Processing Engineering Department, Maslak, 34469, Istanbul, Turkey

## ARTICLE INFO

## Article history:

Received 31 December 2015

Received in revised form 17 June 2016

Accepted 20 June 2016

Available online xxxxx

## Keywords:

Flotation

Alumina particles

Surface morphology

Shape factor

Roughness

## ABSTRACT

Morphological features of particles upon size reduction affect flotation recoveries in terms of kinetics, bubble-particle interactions and even suspension properties. The significance of surface roughness and shape factor of particles on flotation recovery and their underlying mechanism has been shown to be of importance. Towards this aim, a series of micro-flotation tests have been performed with original, ground and ground-abraded alumina particles in the presence of sodium dodecyl sulfate collector in order to demonstrate the influence of morphological features on flotation recoveries. The flotation results are discussed with the help of literature data on shape and roughness of various types of particles.

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## 1. Introduction

In recent years, morphological properties come into prominence for explaining flotation recoveries of different particles like talc (Kursun and Ulusoy, 2006), quartz (Ulusoy et al., 2003; Rezai et al., 2010; Guven et al., 2015<sup>b</sup>), particles of different aspect ratios including wollastonite (Wiese et al., 2015), barite, calcite (Ulusoy et al., 2004), glass beads (Koh et al., 2009; Guven et al., 2015<sup>a</sup>; Karakas and Hassas, 2016; Hassas et al., 2016), complex sulphide ores (Feng and Aldrich, 2000) and anthracite (Xia and Li, 2015). As mentioned in a recent review, these variations can be addressed in two main classes of surface morphology such as “Shape factors” and “Roughness” (Mahmoud, 2010). In most of these publications, increase in flotation recoveries for many types of minerals was attributed to a decrease in Roundness parameter. This general finding was also proven by recent investigations (Verrelli et al., 2014; Hassas et al., 2016) in terms of bubble-particle attachment and induction times. Apart from the effect of roundness, some investigations also involved only surface roughness effect for the evaluation of overall flotation results (Ducker et al., 1989; Feng and Aldrich, 2000; Guven et al., 2015<sup>a</sup>; Hassas et al., 2016). In most of these studies it was found that the presence of roughness leads to some enhancement in flotation recoveries in terms of both experimental data and theoretical assumptions. However, apart from smooth spherical particles, one cannot only induce either shape or roughness on a particle through grinding process. Thus a careful procedure is required to isolate the effect of

shape from roughness in order to interpret hydrophobicity dependent flotation recoveries.

The studies on alumina flotation with SDS mostly focused on the effects of pH and adsorption of SDS onto alumina particles (Somasundaran and Fuerstenau, 1966; Fuerstenau and Pradip, 2005; Adak and Pal, 2005). The effect of surface morphology, to our knowledge, has never been considered in the flotation of alumina particles. Thus in this study, different morphology modification procedures as grinding and abrasion were applied to induce changes on shape and roughness of alumina particles followed by flotation in the presence of SDS.

## 2. Experimental studies

## 2.1. Materials

## 2.1.1. Alumina particles and their preparation

Aluminum oxide particles – 106 + 74 μm in size were supplied by ETI Aluminum Industries, Turkey. The pre-analysis of the sample performed by X-ray fluorescence (XRF) technique revealed that the sample was composed of 99.1% of Al<sub>2</sub>O<sub>3</sub>, 0.01% SiO<sub>2</sub>, 0.008% Fe<sub>2</sub>O<sub>3</sub>, 0.15% Na<sub>2</sub>O and 0.007% CaO per weight. The average B.E.T surface area of the delivered sample (with d<sub>50</sub> size of 75 μm) is 80 m<sup>2</sup>/g.

In order to obtain particles with – 74 + 53 μm in size for use in flotation studies, a mixture of ceramic balls of 30, 25, and 20 mm in diameters which weighed about 816 g was used in a cylindrical ceramic mill of 13,112 cm<sup>3</sup> under wet conditions. The reason for selecting a low ball charge in this mill design was to reduce overgrinding of relatively close feed size of – 106 + 74 μm. Different grinding times in the range of 30 s

\* Corresponding author.

E-mail address: [mcelik@itu.edu.tr](mailto:mcelik@itu.edu.tr) (M.S. Çelik).

to 10 min were tested to determine the effect of grinding time on particle morphology and in turn flotation recovery. Thus, in each grinding step, all the materials were wet screened through 74 and 53  $\mu\text{m}$  sieves to obtain samples in  $-74 + 53 \mu\text{m}$  size for both micro-flotation studies, shape factor and roughness analysis.

For roughening the alumina surfaces without altering the original size, about 5 g of alumina particles were dry abraded in 22  $\text{cm}^3$  test tubes and mixed on a mechanical wrist action shaker in the presence of 1 g fine silicon carbide ( $d_{50}$ : 15  $\mu\text{m}$  (Mohs Hardness Scale: 9.0–9.5) for 30, 60, 90, 120, 450, 600 and 1440 min in order to obtain particles with different roughness degrees. The reason for using such fine material was to avoid any size reduction during abrasion process. After each abrasion test, the abraded alumina particles were wet screened through a 53  $\mu\text{m}$  sieve for controlling the particle size fed to flotation process and also for removal of silicon carbide from alumina surfaces. All the materials were water washed several times, dried, and stored in nylon bags.

## 2.2. Morphological characterization of particles

### 2.2.1. Image analysis

Ground and abraded alumina particles were imaged with a binocular microscope of 500 $\times$  magnification, where Roundness of particles was determined by Image Analysis technique. For shape factor analysis, Image J software (Free of License) was used based on particle projections obtained from the micrographs of particles. The processing of the images simply based on taking the threshold of each picture as to automatically select the particles with the color difference. In our previous studies, a different software namely Leica QWin Image Analyzer was conducted for shape factor analysis (Güven et al., 2014, 2015b), however in order to decrease the tolerance of human based error and due to the finer size range of the particles, Image J software was selected for shape analysis. The advantage of using this software was the ability to evaluate more particles for shape factor analysis which produces more reliable results on shape factor. The Roundness values required to evaluate flotation recoveries were obtained as follows.

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

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

### 2.2.2. Roughness analysis

The roughness degree of both ground and abraded alumina particles at different times were determined by Zeiss Axio CSM700™ Optical Profilometer device. In this instrumental analysis, after imaging of particles by binocular microscope with 20 $\times$  magnification, the roughness evaluation was made on the threshold of these images. The analysis was made based on the differences between profile heights for each selected area on particles. Roughness characterization included average roughness (Ra) and other roughness parameters such as skewness (Rsk), kurtosis (Rku), however only average roughness value which presents the average of absolute values for surface heights deviations was considered in the evaluation of flotation recoveries as a function of roughness values. It is worth to note that at least 10 particles were selected and the average values of these measurements were used and presented. The procedure for roughness measurement is shown in Fig. 1.

## 2.3. Micro-flotation experiments

The micro-flotation tests were carried out using a 150  $\text{cm}^3$  micro-flotation column cell (25  $\times$  220 mm) with a ceramic frit (pore size of 15  $\mu\text{m}$ ) which was mounted on a magnetic stirrer as described elsewhere (Hancer and Celik, 1993). Moreover, an additional feed unit with 10  $\text{cm}^3$  volume was used in order to stabilize the washing water

for flotation. Sodium dodecyl sulfate (SDS) ( $\text{C}_{12}\text{H}_{25}\text{NaO}_4\text{S}$ , M, 288.38 g) with  $\geq 98.0\%$  GC was supplied from Fluka Company and used as collector. In addition, throughout all flotation tests, 40 ppm MIBC (Methyl isobutyl carbinol) was used in order to stabilize the froth. During flotation, 1 g of alumina particles in  $-74 + 53 \mu\text{m}$  size was conditioned in collector solutions of desired concentrations for 3 min. The pH value of the medium was measured as  $6.48 \pm 0.1$ . High purity nitrogen gas was used for aeration to maintain gas flow at a rate of 50  $\text{cm}^3/\text{min}$  throughout all the entire flotation experiments. The amount of both float and sink products was determined gravimetrically.

Besides micro-flotation experiments, the bubble particle interactions were also monitored by fast cam instrument where three types of particles, i.e. original (relatively spherical), ground (angular) and abraded (rougher) have been used. A similar procedure was followed throughout the monitoring experiments where the particles were introduced into a small beaker of water prior to the experiment (Verelli et al., 2011). Then a aliquot of particles was sucked up using a Pasteur pipette and transferred to a second pipette which was truncated with knife for use as orifice. The bubble was generated with atmospheric air which is blown from a 2 ml syringe with a needle bent in order to provide horizontal capillary rise. A schematic presentation of the system is provided in Fig. 2.

The liquid medium was  $9.76 \times 10^{-5}$  M SDS solution which was contained in a glass-walled tank with 26  $\times$  76 mm in size. The interactions were recorded on a Photron Ultima Fastcam high-speed video camera operating at 2000 frames/s). Video recordings were processed using the Photron Fast Cam Viewer software.

## 3. Results and discussion

### 3.1. Morphological characterization of ground and abraded particles

In Fig. 3, the roundness and roughness values of both ground and abraded particles are presented under representative pictures taken from micrographs. Compared to glass beads produced under similar conditions (Güven and Celik, 2015), the range of variation in shape factors is not wide for alumina particles according to their initial irregular structure. The roundness value was found to vary in the range of 0.876–0.811 for ground and abraded particles at different treatment times.

The evaluation of roughness can be divided into two regions as grinding and abrasion. In the first region, besides the variation of roundness values, the roughness of particles changes between 0.707  $\mu\text{m}$  to 0.524  $\mu\text{m}$  in the first 2 min and then progressively increased to 0.680  $\mu\text{m}$  after 5 min. These characteristics of the material can be explained with the increasing amount of the debris adhered to the particles that produces height variations on surfaces. In the second region, the roughness of particles increased from 0.690  $\mu\text{m}$  up to 1.074  $\mu\text{m}$  upon abrasion for 60 min (SiC60), however a critical decrease was obtained in the time span of SiC120–SiC450 to 0.720  $\mu\text{m}$ . In addition, at abrasion times higher than 450 min which is denoted as SiC1440, the roughness of particles increased to 0.906  $\mu\text{m}$  whereas the roundness value remained constant as 0.834.

It is worth to note that the use of silicon carbide for roughening the surfaces of alumina particles is not effective compared to that of glass beads (Güven and Celik, 2015). This can be related to their close hardness values on Mohs scale which are 9.0 and 9.5, respectively. Thus, the roughness of alumina particles varies from 0.520 to 1.074  $\mu\text{m}$  whereas this range was reported for glass beads in between 4.316 and 9.029  $\mu\text{m}$ .

In literature, plethora of research was conducted to illustrate the effects of different mechanisms like grinding (Ulusoy et al., 2004; Rezai et al., 2010; Feng and Aldrich, 2000; Wiese et al., 2015), etching (Dang-Vu et al., 2006), abrasion (Güven et al., 2015a), blasting (Güven et al., 2015b) on different shape factors and roughness. As mentioned in the “Introduction” section, most of these studies demonstrated that upon

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