

The surface features of lead activation in amyl xanthate flotation of quartz



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ABSTRACT

It is confirmed in this study that silica can be activated with lead (II) ion and floated with potassium amyl xanthate in the alkaline region as evidenced from micro-flotation experiments. This conclusion is complimented with the results from electrophoresis experiments and by solution chemistry analysis. The PbOH^+ and $\text{Pb}(\text{OH})_2$ (aq) species which are believed to be responsible for silica activation were found to precipitate at the silica surface. These island-like precipitate structures were clearly observed using AFM and SEM imaging. With amyl xanthate addition, lead amyl xanthate was formed as revealed by FTIR. Contact angle measurements further confirmed the nature of lead activation in silica flotation with amyl xanthate collector.

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1. Introduction

Due to the excellent characteristics quartz is widely used in the production of glass, ceramic, refractory material, and optical communication (Ding, 2010). As one of the most abundant minerals in the earth's crust, quartz frequently associates with other minerals, such as feldspar, talc, pyrite, hematite, smithsonite, and apatite (Zhou, 2013). The separation of quartz from other minerals has attracted increasing interest in recent years, especially for the separation of feldspar and quartz due to their similar tectosilicate structure (El-Salmawy et al., 1993; Vidyadhar and Hanumantha Rao, 2007). To obtain high purity quartz, froth flotation is generally used. Quartz can be satisfactorily floated by anionic collectors, such as sodium oleate (Sun et al., 1992), sodium alkylsulfonate (El-Salmawy et al., 1993; Fuerstenau et al., 1968), and sodium alkyl sulfate (Wang and Hu, 1988), with metal ions as activators. Metal ion activation has been a classic pretreatment method for quartz flotation (El-Salmawy et al., 1993).

Quartz is also a typical gangue mineral in many flotation systems such as beryl (Fuerstenau et al., 1965b), sphalerite (Duarte and Grano, 2007), and other sulfide minerals. Although quartz possesses hydrophilic surface properties in nature and cannot be floated by anionic surfactants alone at any pH, hydrophobicity can be induced by adding polyvalent cations to the system. For example, quartz can be completely floated from pH 5.8 to 8.5 with Pb^{2+} as activator and amyl xanthate as collector. In the case of Zn^{2+} , the activation pH range is from pH 7.5 to 8.1 (Fuerstenau et al., 1970; Fuerstenau et al., 1965a). Quartz can also be activated by Cu^{2+} or Ni^{2+} and floated with xanthate in the pH region

pH 7 to 10 (Fornasiero and Ralston, 2005). With oleate as collector, Ca^{2+} , Mg^{2+} , and Fe^{3+} are usually used for activation in quartz flotation. The results have shown that the effective flotation was attributed to the formation of a surface metal hydrous-oleate complex (Sun et al., 1992). Fe^{2+} , Al^{3+} , Pb^{2+} , Mg^{2+} , Mn^{2+} and Ca^{2+} are also shown to function as activators with long chain sulfonate as collector (Fuerstenau et al., 1963). Due to the slight dissolution of metal ions from metallic sulfides or oxides, the quartz can be activated inadvertently and floated by anionic collectors, which leads to unexpected presence of quartz in the concentrates (Fornasiero and Ralston, 2005).

To obtain better separation of quartz from other minerals in quartz production, or to prevent the inadvertent activation of quartz when present as gangue, extensive fundamental studies have been carried out in the last fifty years to study polyvalent cation activation effect in the flotation of quartz. M. C. Fuerstenau and his co-workers (Fuerstenau et al., 1970; Fuerstenau et al., 1963; Fuerstenau et al., 1965a; Fuerstenau et al., 1965b) have done much research on anionic quartz flotation with metal ion activation. Electrophoretic, metal ion adsorption experiments and flotation tests were performed. In the case of lead activation, it was found that due to the adsorption of the lead species, quartz was positively charged from pH 7 to pH 11 in the presence of 1×10^{-4} M lead ions (Fuerstenau et al., 1970). Complete amyl xanthate flotation was achieved in this pH range. Correlation of the lead species distribution with the flotation response shows that $\text{Pb}(\text{OH})^+$ is probably responsible for the activation. A basic lead xanthate complex may be functioning as the collector (Fuerstenau et al., 1965a), which has been suggested by other researchers (Fornasiero and Ralston, 2005; James and Healy, 1972a, 1972b; Laskowski and Castro, 2012). However, there is no direct evidence to verify the adsorption of active species and subsequent collector adsorption.

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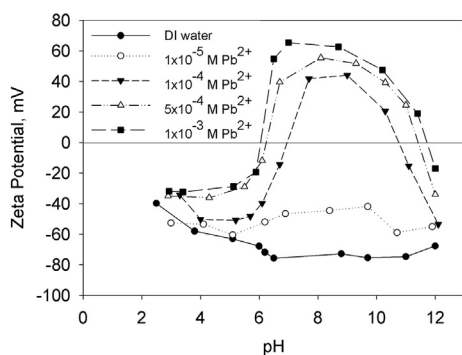


Fig. 1. Zeta potential of unactivated and activated silica as a function of pH.

In this study, AFM, together with SEM, has been utilized to detect the changes in the silica surface state when lead and amyl xanthate were used to achieve effective silica flotation. By imaging the silica surface, the precipitation of lead and/or the formation of a lead-xanthate compound might be detected. Diffuse reflectance infrared Fourier transform spectroscopy (DRIFT) was used to further identify the formation of the lead-xanthate compounds. In conjunction with the electrophoretic measurements, flotation tests and contact angle measurements, the mechanism of amyl xanthate flotation of quartz with lead as activator first studied by M. C. Fuerstenau and his co-workers will be better understood.

2. Experimental section

2.1. Chemicals and materials

All solutions in this study were prepared using Millipore Milli-Q water (18 M Ω ·cm). Potassium amyl xanthate (90%, TCI), lead nitrate (99%, Alfa Aesar) and 4-methyl-2-pentanol (MIBC, 98%, Sigma-Aldrich) were used as received, without further purification. Silica ($-10\ \mu\text{m}$, U.S. Silica) was used for the electrophoresis measurements. Coarse silica (40×100 mesh, Acros) was used for the micro-flotation. An IR/UV grade fused silica disk (20×2 mm, Gold Dragon Optics Co., Ltd., China) was used in AFM, SEM and contact angle measurements.

2.2. Electrophoresis measurements

Electrophoretic mobility of the silica was performed using a Zeta potential analyzer (Zeta PALS, Brookhaven Instruments Corporation, Holtsville, NY). Lead nitrate solutions of varying concentrations were prepared in a beaker. The pH was adjusted with sodium hydroxide and hydrochloric acid solutions, and then silica powders ($-10\ \mu\text{m}$) were dispersed in the lead nitrate solutions and a suspension with 0.025% silica was prepared. The suspensions were stirred for 20 min and then about 10 mL transferred to the Zeta PALS cell. The Zeta potentials of silica particles were measured. The instrument automatically calculated the mobilities of the particles and converted the results to zeta potential according to the Smoluchowski equation. To ensure precise zeta potential determination, five runs of 30 cycles per run were conducted for every sample and the average values were reported.

2.3. Micro-flotation tests

The flotation response of silica was determined using a 125 mL Hallimond tube (20×220 mm) with a fine frit ($10\ \mu\text{m}$) and a magnetic stirrer. Silica samples (40×100 mesh) of 2 g were used in each micro-flotation test. 150 mL lead nitrate solution of desired pH was first prepared and then the silica particles were added. The solution was conditioned for 20 min, and then additional 5 min was applied after amyl xanthate was added as collector. After collector adsorption, 0.02 mL of

MIBC was added as frother. The silica suspension was transferred to the Hallimond tube. The air was set to achieve the desired air flow rate of 40 mL/min. The hydrophobic silica particles attached to the bubbles and floated to the top of the tube. The flotation time was 5 min. The float and sink fractions were filtered, dried, and weighted.

2.4. Contact angle measurement

Similar to the silica treatment in micro-flotation, the fused silica disk was activated by lead and amyl xanthate was added for adsorption. The silica disk was taken out from the solutions and blown dry by high purity nitrogen. Then the contact angle of silica was measured by a contact angle goniometer (RAME-HART, Inc. NRL U.S.A.) using the sessile drop method. For each sample, at least five drops were measured and the average value from five measurements was calculated.

2.5. AFM measurement

A Nanoscope AFM with Nanoscope IV controller (Veeco Instruments Inc., Santa Barbara, CA) was used with an E-type scanner. Triangular beam silicon nitride (Si_3N_4) cantilevers (Veeco Instrument Inc., Santa Barbara, CA) with pyramid-shaped tips were used. The sample holder was cleaned using acetone, ethanol, and water in order, and gently dried with ultra high purity N_2 gas. The cantilevers were subsequently cleaned in a UV chamber for 30 min prior use. The silica disk treated with 150 mL lead and amyl xanthate solutions, was mounted on a piezo scanner. The contact mode was applied for imaging of the silica surface. The AFM instrument was kept in an acoustic and vibrational isolation chamber. The images of the silica surface were obtained at a scan rate of 1 Hz and a scan area of $5 \times 5\ \mu\text{m}$. The images were processed offline using Nanoscope vs. 5.31R1 software. Flattening and low pass filtering were applied to remove noise. Image processing software, Gwyddion 2.15, was used to calculate the area covered by lead-containing precipitates and lead-xanthate compounds at the silica surface.

2.6. SEM analysis

The silica disk was firstly treated with 150 mL lead and amyl xanthate solutions and then analyzed using SEM. The SEM image of treated silica was collected using FEI Quanta 250 SEM. The energy spectra were analyzed using EDAX GENESIS energy dispersive spectroscopy.

2.7. DRIFT measurement

Diffuse reflectance infrared Fourier transform spectroscopy (DRIFT) equipped with a diffuse reflectance unit (PIKE Technologies) was used to identify adsorbed collector at the silica surface. The infrared spectra were recorded for all samples on the air dried 40×100 mesh silica

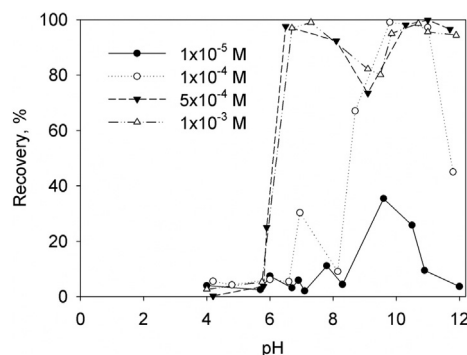


Fig. 2. Relationship between flotation recovery and pH with various lead and amyl xanthate additions (the lead concentration equals the amyl xanthate concentration).

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