



Flotation of quartz using ionic liquid collectors with different functional groups and varying chain lengths



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ARTICLE INFO

Article history:

Received 11 February 2016

Revised 20 June 2016

Accepted 21 June 2016

Available online 25 June 2016

Keywords:

Ionic liquids

Quartz

BHQ ore

Flotation

FTIR

XPS

ABSTRACT

The application of imidazolium, ammonium, and pyridinium based ionic liquids (ILs) as the flotation collectors of quartz has been investigated. The flotation studies have been carried out for pure quartz, quartz-hematite synthetic mixture and a low grade banded hematite quartzite (BHQ) ore while the trend of floatabilities has been explained in terms of the structural differences of the ILs. The flotation of pure quartz using the ammonium and pyridinium based IL collectors has exhibited an excellent performance; where about 100% quartz could be floated at a collector concentration of 5×10^{-5} M. The results are found better compared to the imidazolium ILs and conventional quartz collectors like dodecylamine (DDA) and cetyl trimethyl ammonium bromide (CTAB). The ammonium and pyridinium based collectors also show better iron grade and recovery compared to the imidazolium collectors during the reverse flotation of the synthetic hematite-quartz mixture and BHQ ore. Using these collectors, an iron ore concentrate of ~62% Fe at ~75% recovery could be obtained from the BHQ ore containing ~38% Fe. In the Fourier Transform Infra Red spectroscopy (FTIR), the peak intensity at 2900 cm^{-1} due to the presence of alkyl groups signifies the extent of adsorption of the collectors on quartz. The N/Si ratio and C—C peak area as determined by the X-ray photoelectron spectroscopy (XPS) results show the relative adsorption of the IL collectors. The pyridinium based IL collector show better adsorption strength compared to all others ILs under study. The better flotation results in the case of the ammonium and pyridinium collector are attributed to the presence of higher alkyl chain length inducing better hydrophobicity.

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1. Introduction

Quartz is one of the most common impurities associated in most of the natural occurring ores. It needs to be removed in order to achieve a suitable concentrate for the economic extraction of the metal values. It is often found that the lower grade ores have complex mineralogical textures and the mineral values are liberated at finer sizes. Froth flotation is one of the best methods for the selective separation of desired minerals that are liberated at the finer sizes. In this technique, the collector plays an important role in enriching the quality of the desired mineral species. In this context, new engineering innovations are introduced in the flotation methodologies while new collectors are being developed for obtaining improved outcomes.

In iron ore, where quartz is the major impurity, reverse flotation is preferred for the concentration of iron values. Several cationic

collectors such as dodecylamine chloride (DDA), cetyl trimethylammonium bromide (CTAB), ether or ester amines, and quaternary ammonium salts are used as the flotation collector of quartz. The interaction of amines with quartz has been established through several characterization techniques such as FTIR and XPS (Vidyadhar and Rao, 2007). The hydrogen bond geometries and the adsorption energies as calculated using the density functional calculations for different amines and their derivatives on the quartz surface reveal that ester amines are stronger surfactants compared to the ether amines and primary alkyl amines (Rath et al., 2014). Many researchers have investigated the use of alternate economically viable collectors for the flotation of quartz (Yuhua and Jianwei, 2005; Weng et al., 2013; Xia et al., 2009; Araujo et al., 2005). Different new collectors such as α -Bromodecanoic acid, N-dodecylethylenediamine, gemini and commercial collectors like AERO 3030C, Flotigam DAT, and Armoflot 64 have been synthesized and applied for better separation of quartz and concentration of mineral values (Barbaro et al., 1997; Pereira and Peres, 2005; Liu et al., 2009; Luo et al., 2015; Sekuli et al., 2004).

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Ionic liquids which possess low volatility and high ionization temperature, have several applications in varied fields. They are used as phase-transfer catalysts, solvents, lubricants, gas capturing agents, coating materials, or chemical sensors (Bradarić et al., 2003; Weng et al., 2006; Pernak et al., 2006; Garvey and Dietz, 2014; Ferreira et al., 2014; Gao et al., 2014). The functional heads such as ammonium, imidazolium, pyridinium, piperidinium, pyrrolidinium etc. which contain quaternary nitrogen atom are considered to be the most common cations of ionic liquids (Chen et al., 2013; Seddon, 1996; Lagrost et al., 2003; Blahušiak et al., 2013; Mancini et al., 2013; Tong et al., 2013). However, the literature related to the application of ionic liquids in mineral flotation is scanty. In this context, recently, Sahoo et al. (2014, 2015a, 2015b) have applied Aliquat-336, TOMAS (trioctylmethylammonium salicylate), and THEX (tetrahexylammonium chloride), in the flotation of quartz from low grade iron ores. The studies have indicated that, an iron grade containing 63–65% Fe with 65–68% Fe recovery could be achieved from a BHQ ore containing ~38% Fe. No extra frother was used in these studies. The adsorption of these reagents was confirmed by surface potential, FTIR, and XPS studies. Moreover, these IL collectors were found to perform better than the conventional collectors such as DDA and CTAB.

In continuation of the work related to the ILs as flotation collectors, the present paper explores the application of ionic liquids with functional groups such as imidazolium, pyridinium and organic group attached to the quaternary nitrogen atom in the flotation of quartz. The intention is to study the effect of functional groups other than the alkyl ammonium head group as covered earlier (Sahoo et al., 2014, 2015a, 2015b).

2. Materials and methods

2.1. Sample

The quartz samples assaying 98.3% SiO₂ used for the flotation studies were handpicked from Kalahandi, Odisha (India). The samples were crushed and ground to below 100 μm and further treated with dilute hydrochloric acid to remove any contaminated iron. The sample was washed several times with distilled water and dried prior to using it in the flotation studies. Selected hematite rich iron ore fines were obtained from one of the iron ore mines located in Goa, India. The sample was ground to below 100 μm using a laboratory ball mill. The small concentration of magnetite particles present in the ore was removed by low intensity magnetic separation. The nonmagnetic portion containing hematite as the major mineral was used in the flotation studies. Synthetic mixtures of hematite and quartz were prepared by vigorously mixing the samples in the required proportions. The low grade BHQ ore was collected from Hospet area, Karnataka. The purpose of selecting BHQ ore is that the sample contains mostly hematite and quartz as the dominating mineral phases. The sample was reduced to below 100 μm for the flotation studies. Table 1 shows the particle size distribution of the quartz, hematite and BHQ sample used for the flotation studies.

All the ionic liquids and starch were purchased from Sigma-Aldrich and used as such without any purification. The chemical

structures, molecular weights, and abbreviations as used in this manuscript are given in Table 2. Out of the five ILs used, cetyl pyridinium bromide (CPA) and benzyl dimethyl tetradecyl ammonium chloride (BDTA) have melting points of 67–71 °C and 56–62 °C respectively, and hence cannot be termed as Room Temperature Ionic Liquids (RTIL). The ILs were diluted with methanol (0.1% weight by volume) while being used as collectors in flotation.

2.2. Flotation

Quartz, synthetic mixture, and BHQ ore were subjected to flotation studies in a Denver D-12 sub-aeration flotation machine having a 1L capacity cell. Different ILs as mentioned in Table 2, DDA, and CTAB were used as the flotation collectors while soluble starch (1% weight by volume) was used as a depressant for the iron oxides. The solid concentration was maintained at 10% and the suspension was agitated for 3 min at 1500 rpm. Freshly prepared starch and collector were added into the cell orderly with 1 min interval of conditioning time. Further, a conditioning time of 5 min was maintained after all the reagents were added and the froth was collected for a time period of 2 min. The concentrate and tailings were dried, weighed, and analyzed for the iron content in order to determine the grade and recovery. The flotation studies of the pure quartz mineral system were carried out using distilled water while laboratory tap water was used for the BHQ ore. The compositions of the raw materials used are given in Table 3.

For FTIR and XPS studies, the sample of pure quartz was further ground to below 5 μm by a laboratory pulverizer. The finely ground sample was mixed with KBr and then pressed to prepare small pelletized discs for the FTIR analyses. FTIR studies in the range of 400–4000 cm⁻¹ were conducted in a Shimadzu FTIR instrument while the XPS studies were carried out using the equipment S/N: 10001, Prevac, Poland. All the IL-treated quartz samples were prepared by mixing quartz in an aqueous solution of the ionic liquid having a concentration of 2 × 10⁻⁵ M with the help of a laboratory stirrer. At the end, the particles were separated by filtration, thoroughly rinsed with distilled water and dried at room temperature. Poly vinyl alcohol (PVA) was used as a binder in making the pellets for XPS. The binding energies of the treated samples were determined from the spectra taken with Al Kα (hν = 1486.6 eV) radiation and a hemispherical energy analyzer. A pressure of 6 × 10⁻¹⁰ mbars was maintained during the experiments.

3. Results

3.1. FTIR and XPS study

The FTIR results of pure quartz and quartz treated with ionic liquids are shown in Fig. 1. In pure quartz spectra, the peaks are observed at 1160, 795, 650 and 520 cm⁻¹. The peak around 3400 cm⁻¹ marks the presence of hydroxyl group. The quartz treated with ionic liquids show two additional peaks around 2900 cm⁻¹ due to the presence of CH₂, CH₃ stretching bonds that confirm the adsorption of the ionic liquids onto the quartz surface. The spectra of quartz-CPA show the high intensity of alkyl peak indicating high adsorption, whereas OMI showing a less intense peak may be attributed to the less adsorption onto quartz surface. It is observed that the peak intensity of CH₂ group in DMI is more than HMI, which is clearly because of the presence of a longer alkyl chain in the former. The peaks of quartz remain unchanged in all the spectra confirming that only physisorption has taken place.

The XPS spectra peak positions of quartz and quartz treated with ionic liquids are shown in Table 4. The adsorption of the quaternary nitrogen is confirmed by the presence of N (1S) peak at

Table 1
Particle size distribution of quartz, hematite and BHQ used for flotation.

| Size, μm | Quartz wt% | BHQ wt% | Hematite wt% |
|-----------|------------|---------|--------------|
| –100 + 75 | 8.9 | 7.7 | 7.2 |
| –75 + 45 | 17.3 | 16.8 | 14.7 |
| –45 + 38 | 18.3 | 16.6 | 17.9 |
| –38 | 55.5 | 58.9 | 60.2 |

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