



Role of sodium carbonate in scheelite flotation – A multi-faceted reagent

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ABSTRACT

Even though sodium carbonate is a reagent frequently used in flotation, its role is mostly described as a buffering pH modifier and a pulp dispersant. In the case of scheelite flotation, literature has shown that sodium carbonate improves flotation performance but little is said as to selectivity against gangue minerals. There is a consensus that sodium carbonate neutralizes calcium-bearing minerals through surface carbonation but this does not explain why it should depress said minerals in a specific order. Furthermore, the addition of depressants such as sodium silicate or quebracho could be triggering new mechanisms. Through batch flotation testwork on a skarn scheelite ore, single mineral flotation and contact angle measurements, this article aims at demonstrating that sodium carbonate is a multi-faceted reagent, which serves as a buffering pH modifier, a pulp dispersant precipitating calcium ions in suspension and a depressant for calcite and calcium-bearing silicates. Based on the kinetics, calcium surface site density, calcium activity and surface reactions, fluorite would be the first mineral to be depressed, then calcite, then silicates and finally scheelite and possibly apatite if sodium carbonate is overdosed, likely independently from pH and definitely independently from the co-pH modifier. Sodium silicate acts mostly synergistically and partially antagonistically with other depressants, notably sodium silicate and quebracho.

1. Introduction

A typical reagent regime for scheelite flotation is called the “lime flotation process”, which involves the addition of lime and sodium carbonate as co-pH modifiers, sodium silicate as a depressant and oleic acid (or sodium oleate) as the main collector (Vazquez et al., 1976; Yongxin and Changgen, 1983; Martins and Amarante, 2012).

The presence of lime could be considered as ill-advised as it was commonly observed that calcium had deleterious effects in fatty acid flotation systems (Kupka and Rudolph, 2017). Indeed, Arnold et al. (1978) and Gao et al. (2015) showed that scheelite recovery could decrease drastically at very high concentrations of Ca^{2+} or Mg^{2+} when using sodium oleate as a collector, especially since it is sensitive to water hardness (Leja, 1982).

This is where sodium carbonate comes along: this reagent precipitates calcium and magnesium ions into CaCO_3 and MgCO_3 if the pH is high enough, as well as heavy metal ions (Zheng and Smith, 1997; Allan and Woodcock, 2001; Rashchi et al., 2004; Yuhua et al., 2011; Feng and Luo, 2013; Forssberg et al., 1993), limiting their presence in the pulp. Additionally, sodium carbonate, namely its carbonate ion, is sensitive to pH and can end up precipitating onto mineral surfaces (Zheng and Smith, 1997; Rashchi et al., 2004) such as fluorite (Miller

and Hiskey, 1972; Bahr et al., 1968; Hanna and Somasundaran, 1976) or calcite (Vazquez et al., 1976). This means it could end up depressing said minerals, which are known to contaminate scheelite concentrates due to their similar floating properties (Kupka and Rudolph, 2017).

Sodium carbonate (or soda ash) is therefore a buffering pH modifier (Allan and Woodcock, 2001; Yuhua et al., 2011; Bulatovic, 2015), a pulp dispersant (Zheng and Smith, 1997; Allan and Woodcock, 2001; Rashchi et al., 2004; Yuhua et al., 2011) and a depressant at the same time (Zheng and Smith, 1997). In the case of scheelite flotation, it has been shown that sodium carbonate improves both grade and/or recovery (Martins and Amarante, 2012; Kupka et al., 2017; Filippova et al., 2017; Liu et al., 2016; Gordon et al., 1984) but the mechanism has not been distinctly reported in the literature for scheelite. Indeed, there is a consensus that sodium carbonate neutralizes some minerals by precipitating onto their surfaces forming calcium carbonate, this has been shown through infrared for fluorite (Bahr et al., 1968; Hanna and Somasundaran, 1976) and demonstrated for quartz through the bubble pick up method (Martins and Amarante, 2012). As far as the authors are aware, in other flotations, López-Valdivieso et al. (2000) proved the conversion of the surface of celestite to strontium carbonate through FTIR and zeta-potential measurements while Rahimi et al. (2017) showed surface precipitation of sodium carbonate onto calcite through

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zeta potential, contact angle and FTIR measurements in their cationic flotation of pyrolusite. However, selectivity is hardly ever discussed and this does not explain why sodium carbonate should improve flotation performance. Finally, the addition of depressants such as sodium silicate or quebracho could be triggering new mechanisms (Kupka and Rudolph, 2017; Kupka et al., 2017; Filippova et al., 2017) but the interaction between these reagents is never mentioned.

As a consequence, this article aims at describing the effects of sodium carbonate in the froth flotation of scheelite in order to test the general hypothesis on its mechanism and to determine potential interactions with depressants.

2. Material and methods

2.1. Single minerals

Scheelite and calcite were the feed materials for single mineral flotation (microflotation). Scheelite came from the Mittersill mine, Austria. The target size fraction was 32–63 μm . X-ray diffraction determined that the sample was composed of 96 wt% scheelite with pyrite, molybdenite and other minerals as contaminants. Calcite came from Straßberg, Germany and the 63–100 μm fraction was selected. X-ray diffraction showed that it was composed of 97 wt% of calcite, 1 wt% of quartz and minor amounts of fluorite, ilmenite and others.

2.2. Microflotation

Microflotation was conducted in a Hallimond tube designed at the TU Bergakademie Freiberg. 1 g of the single minerals was added to an aqueous solution of 10^{-2} M KCl, stirring with a rotation speed of 400 rpm. 3–5 min were allocated to pH conditioning with NaOH. After achieving stable pH, the depressant was added before the collector with a conditioning time of 2 and 3 min respectively. The flotation suspension was transferred to the Hallimond tube and agitated for another 3 min with a rotational speed of 800 rpm using a magnetic stirrer. Airflow rate was set to 20 cm^3/min and microflotation lasted 2 min. If not otherwise stated, pH is set at 9 with NaOH and sodium oleate is used as a collector at a dosage of 1×10^{-4} mol/l. All experiments were conducted at least twice, up to five times, the number of repetitions being driven by the mass loss, which had to be strictly inferior to 10%.

2.3. Batch flotation

The ore used for batch flotation tests contains 0.51% scheelite, 1.70% calcite, 0.28% apatite and various silicates (quartz, micas, plagioclases and hornblende). The ore has a d_{80} of 63 μm after milling. Froth flotation tests were conducted with 500 g of ore at 33% pulp density in a Magotteaux® bottom-driven flotation cell and at constant pH of 9.5. Each had only a rougher stage, lasted seven minutes and involved three concentrates with a 5 s scraping rate, 450 rpm impeller speed and an air flowrate of 5 L/min. All samples were split and sent to ICP-MS and XRF for chemical assays (done by ALS Global®). It should be noted that all calcium grades given in this article have been recalculated by removing calcium contained in scheelite. The feed and tailings water samples were sent to ICP-OES for cation assays (done at the Technische Universität Bergakademie Freiberg, Germany).

The flotation testwork itself was set up using DesignExpert® software (Version 10.0.5.0) from Stat-Ease, Inc. The testwork included repetitions of the experiments. DesignExpert® was then used to analyze the data through ANOVAs and create response models with predictive properties.

2.4. Contact angles

Contact angle measurements were conducted on epoxy resin grain mounts of scheelite and calcite with a Dataphysics OCA 50 instrument

equipped with the SCA 20 software. The roughness of the mineral samples investigated is under 5 nm. The pure scheelite crystal came from Malakka, India whereas the pure calcite crystal came from Mittersill, Austria. The single crystal was conditioned in a background solution of 10^{-2} mol/l KCl and the reagents (also prepared with KCl) were added one after the other after setting the pH at 9 with NaOH. Sodium oleate is used as a collector at a single dosage of 1×10^{-4} mol/l. The grain mount was then dried with an air blower and placed on the instrument. Measurement was done with the sessile drop method with water droplets of a constant volume of 1 μL . Once the measurement was complete, the substrate was polished and cleaned in a plasma cleaner before being used again. Each point displayed on the diagrams corresponds to at least 12 to 20 measurements unless the angle is under 10° , then it corresponds to 5 measurements.

2.5. Reagents

Sodium oleate from Carl Roth® was used as a collector at a single dosage of 200 g/t in order to avoid multicomponent technical grade collectors with effects difficult to evaluate. Flotanol 7197® from Clariant Mining Solutions® was used as a frother at 20 g/t. Three pH modifiers were used: sodium hydroxide (NaOH), lime (CaO), and sodium carbonate (Na_2CO_3) from Alfa Aesar®. Sodium chloride (NaCl) from Carl Roth® was used for microflotation. Depressants included sodium metasilicate nonahydrate ($\text{Na}_2\text{O}_3\text{Si}\cdot 9\text{H}_2\text{O}$) from Aldrich Chemistry® and extract of quebracho from Baeck GmbH & Co. The dosages of water glass and quebracho were adapted based on previous flotation testwork conducted on the ore in use: dosages of sodium silicate were 500, 1000 and 2000 g/t and quebracho 15, 30 and 60 g/t, while sodium carbonate was added at a constant dosage of 100 g/t. All reagents are of analytical grade.

3. Impact as a co-pH modifier

Different pH modifiers can be used in scheelite flotation, such as sodium hydroxide, sodium carbonate or calcium oxide. As described before, sodium carbonate can be considered a buffering co-pH modifier, even though its addition fulfills several roles. In a short series of scheelite batch flotations, the authors compared the impact of the pH modifier type by using NaOH and CaO alone or in combination with sodium carbonate (abbreviated SC in the figures).

The authors chose not to use sodium carbonate alone because it would require much higher dosages in order to set the pH and would therefore invalidate any comparison with the other pH modifiers where “only” 100 g/t of sodium carbonate were used. In the literature, there are conflicting results on the impact of sodium carbonate on its own:

- In the flotation of a scheelite skarn ore, Filippova et al. (2017) stated that added alone, sodium carbonate had little to no impact on the flotation (it slightly reduced the tungsten recovery) and does not yield any improvement in selectivity;
- In the flotation of five different American scheelite ores, Dean and Schack (1964) stated that “sodium carbonate was more effective than sodium hydroxide as an overall pulp-regulating reagent” but do not give any details of why one was superior to the other;
- In the flotation of a Brazilian scheelite ore, Oliveira and Sampaio (1988) compared the tungsten grade and recovery under the influence of different pH modifiers, namely Na_2CO_3 , NaOH + Na_2CO_3 , NaOH and CaO. NaOH alone was the worst performing pH modifier while mixing it with sodium carbonate offered the best performance. There is no information on selectivity in the article;
- By analyzing De Castro et al. (1996)’s data, it appears that sodium carbonate compared to sodium hydroxide in the flotation of celestite (SrSO_4) against calcite showed no differences in grade or in selectivity but offers higher recoveries of both minerals (however only in the absence or at low dosages of depressants);

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