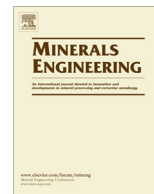




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A study of the reprocessing of fine and ultrafine cassiterite from gravity tailing residues by using various flotation techniques

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ABSTRACT

This study investigates the reprocessing of once disposed tin tailings from a historic German tin operation through different surface properties based processing techniques. Froth flotation and agglomeration flotation, by using commercially available cassiterite collectors from Clariant, were chosen as processing techniques. Isooctane as a nonpolar oil was used to promote the collection of ultrafine ($-10\ \mu\text{m}$) cassiterite by selective agglomeration, and thus, size enlargement. Results indicate that by using sulfosuccinates as a collector, around 80% of the fine ($-50\ \mu\text{m}$) to very fine ($-20\ \mu\text{m}$) cassiterite can be recovered (representing $\sim 50\%$ of the total cassiterite in the tailings sample). Agglomeration flotation experiments showed inferior results for recovering the ultrafine cassiterite (for feed fractions in the $-25\ \mu\text{m}$ size range). Oil/froth interaction and increased pulp ion content are considered as the main contributing causes.

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1. Introduction

Historically, the dominant beneficiation strategy to recover cassiterite (SnO_2) from tin ores has been gravity separation (including jigs, spirals and tables), which could be followed by sulfide flotation for further upgrading (Bulatovic, 2010). As gravity concentration techniques are very sensitive to particle size, the recovery of cassiterite by these methods becomes increasingly inefficient, if cassiterite particle sizes are below $40\ \mu\text{m}$ (Falcon, 1982). Taking the brittle nature of cassiterite into account, it is not surprising that in such gravity concentration circuits up to 30 to 40% of the tin could subject to tailings, due to the generation of such fine cassiterite during upstream comminution operations (Angadi et al., 2015).

Consequently, caused through decades of processing, the tailing dams of old tin operations could still contain substantial amounts of cassiterite, mostly as fine ($-50\ \mu\text{m}$) particles. Nowadays, due to improvements in processing technologies, including advanced gravity concentration and cassiterite froth flotation (Angadi et al., 2015), such deposits, can be considered as tin reserves. For example, the development of selective collectors for

cassiterite led froth flotation become a complementary processing technique for the treatment of products from gravity circuits in many tin beneficiation plants (Gruner and Bilsing, 1992; Bulatovic, 2010).

One of the main problems in froth flotation, which has to be taken into consideration, is that with increasing percentages of very fine ($-20\ \mu\text{m}$) or even ultrafine ($-10\ \mu\text{m}$) particles the process performance is rather inefficient. Potentially improving the performance for ultrafine particle systems attained a great deal of attention in research. Many investigations by means of developing a physicochemical separation technique suitable for processing of such particle systems have been described (Sivamohan, 1990; Laskowski, 1992). Some of those methods, which even have been performed for cassiterite particles, are: selective flocculation (Warren, 1982; Bilgen and Wills, 1994) and oil-assisted flotation techniques like agglomeration flotation (Schubert et al., 1966) and two-liquid flotation (Zambrana et al., 1974; Marinakis and Shergold, 1988). Besides the problems caused by high amounts of $-20\ \mu\text{m}$ particles, also surface alteration of the cassiterite particles, due to the long period of disposal, is uncertain, and thus, might affect the response to surface-based separation processes.

The main focus of this study is to interpret the process behavior of (fine to ultrafine) cassiterite from tin tailing residues through different flotation methods. Conventional froth flotation (CFF)

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and oil-assisted agglomeration flotation (OAF), using commercially available cassiterite collectors from Clariant, were chosen as processing techniques. Different particle size fractions as well as additional analyses were applied to show and better understand influencing parameters, which may affect the recovery of (ultra-fine) tailings cassiterite during either processing technique.

2. Experimental approach

2.1. Material characterization and preparation

Tailings for this study were chosen from the historic tin mining operation near Ehrenfriedersdorf in the German Erzgebirge (Ore Mountains). Reported cassiterite recovery by gravity concentration for the last 50 years of operation (till 1991) was 55 to 70% and nearly 3,000,000 t of (mainly table) tailings were piled up and disposed during that time frame (Hösel, 1994).

A representative tailings sample (drill core) was taken and characterized in terms of particle size distribution, measured by laser diffraction (Sympatec HELOS), and mineral locking properties, measured by Automated Mineralogy (Mineral Liberation Analyzer MLA). Measurements demonstrate that the material is relatively fine, with approximately 80% being smaller than 100 μm and more than 20% being in the ultrafine size range below 10 μm . The data indicates that the overall cassiterite content in the tailings body is around 0.2%, where more than 80% of the cassiterite is below 40 μm , and a substantial amount below 10 μm (Fig. 1). In terms of locking, most of the cassiterite particles analyzed are showing a high degree of liberation by free surface. Other minerals present in the drill core material included various silicates (~95%), oxides, sulfides, phosphates and carbonates.

In order to prepare different size fractions for the CFF and OAF experiments, classification steps were conducted on the tailings sample (Table 1). First, particles +200 μm were removed by wet sieving. Using a hydrocyclone, the material was further sized (cut size 25 μm) into an underflow (referred to as “H1_{underflow}”) and overflow (referred to as “H1_{overflow}”) product. A part of the “H1_{overflow}” fraction was again sized by a second hydrocyclone step (cut size 10 μm) providing a deslimed very fine fraction (referred to as “H2_{underflow}”). The different size fractions prepared were subsequently used as feeds for the CFF and OAF experiments. The SnO₂ grade of the fractions was determined by XRF on representative samples (Table 1).

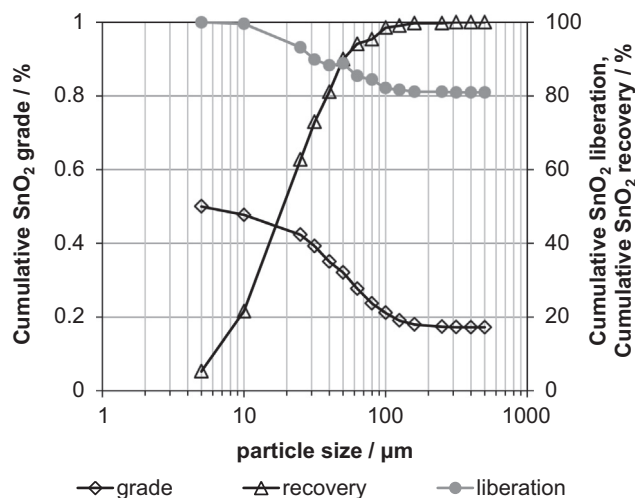


Fig. 1. Cumulated cassiterite grade, recovery and liberation within the tailings sample with respect to particle size (obtained by MLA, including virtual sieving).

Table 1

Properties of various size fractions created during material preparation and used for flotation testwork (particle size distribution parameter for all particles (D_{10} , D_{50} , D_{90} , Specific Surface) and for cassiterite particles only (D_{10,SnO_2} , D_{50,SnO_2} , D_{90,SnO_2}), cassiterite grade (c_{SnO_2}) as well as mass fraction (w) and mass fraction of cassiterite (w_{SnO_2}) related to original tailings sample).

| Fraction/properties | Underflow of 1st hydro-cyclone step | Overflow of 1st hydro-cyclone step | Underflow of 2nd hydro-cyclone step |
|--|-------------------------------------|------------------------------------|-------------------------------------|
| Designation | H1 _{underflow} | H1 _{overflow} | H2 _{underflow} |
| D_{10} (μm) | 15.3 | 2.0 | 4.2 |
| D_{50} (μm) | 60.1 | 10.5 | 17.5 |
| D_{90} (μm) | 153.3 | 35.2 | 44.4 |
| Spec. surface ($\text{m}^2 \text{g}^{-1}$) | 1.3 | 11.7 | 2.7 |
| D_{10,SnO_2} (μm) | 7.3 | NA | 2.8 |
| D_{50,SnO_2} (μm) | 22.1 | NA | 5.9 |
| D_{90,SnO_2} (μm) | 43.7 | NA | 14.3 |
| c_{SnO_2} (%) | 0.23 | 0.18 | 0.18 |
| w (%) | 51.6 | 62.9 | 19.4 |
| w_{SnO_2} (%) | 62.9 | 33.5 | 19.6 |

2.2. Flotation testwork

Single-stage, laboratory-scale CFF and OAF tests were performed using a GTK LabCell (Outotec). A two liter cell was used (~22% solid content in tap water) with rotor speed 1000 min^{-1} and air flow 4 l/min set constant. The pulp level height was adjusted shortly below the cell lid, to avoid pouring out of the pulp but emphasize pulp recovery instead of froth recovery effects. The pulp pH was adjusted using aqueous solutions of NaOH and H₂SO₄. Three different collectors were provided by Clariant (Table 2). For sulfosuccinamates (SC) as a collector pH 3 was used as well as lower frother dosage, due to the strong frothing properties of that collector (Angadi et al., 2015). For the other collectors, pH 5 was used (Bulatovic, 2010). Each parameter set was repeated once, and the average is considered. Sulfide flotation prior cassiterite flotation was not conducted. To measure concentrations of Fe and Ca ions prior and after each experiment (including conditioning and flotation), the supernatants of pulp samples were analyzed by ICP-OES (Thermo Scientific iCAPTM 6300).

The chosen OAF test parameters (frother, depressant, oil/pulp agitation) were based on literature, where intense, high density and lengthy pulp/oil agitation was reported as effective parameters for tin ore (deslimed at 20 μm) from the former Altenberg tin operation (Erzgebirge, Germany) (Schubert et al., 1966). Thus, pulp conditioning was performed in an extra agitation unit (anchor stirrer, 1700 min^{-1}). After reagent conditioning, emulsified isooctane as nonpolar oil was added to the pulp. Emulsification was performed

Table 2

Reagents used for testwork.

| Reagent type | Name | Description | Provider |
|--------------|---------------------------|---|---------------|
| Collector | Flotisor 7579 | Sulfosuccinamates (SC) | Clariant |
| | Flotisor 7186 | Phosphonic acids (PA) | |
| | Flotisor 7253 | Carboxylic acids (CA) | |
| Depressant | Sodium hexafluorosilicate | Na ₂ SiF ₆ | Sigma Aldrich |
| Frother | 1,2-Octanediol | C ₈ H ₁₈ O ₂ | Alfa Aesar |
| Oil | Isooctane | C ₈ H ₁₈ | Carl Roth |

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