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# Contribution of chloride ions to the sulfidization flotation of cerussite

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# ABSTRACT

The effect of the addition of chloride ions on the sulfidization flotation of cerussite was investigated by micro-flotation tests, surface adsorption experiments, zeta potential measurements, and X-ray photoelectron spectroscopy (XPS) analysis. The micro-flotation test results indicated that the addition of chloride ions prior to sulfidization significantly increased the flotation recovery of cerussite. The maximum and minimum percentages of increase in flotation recovery were 16.63% and 10.51%, respectively. Surface adsorption experiments of sulfide demonstrated that there was a higher adsorption concentration of *S* species on the mineral surface, and its adsorption efficiency was enhanced. The results of zeta potential measurements revealed that the addition of Na<sub>2</sub>S alone. The XPS analysis results indicated that more lead sulfide formed on the mineral surface after the addition of chloride ions into the pulp solution. These results consistently showed that the addition of chloride ions played a significant role in the sulfidization of cerussite, resulting in a better flotation performance.

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# 1. Introduction

Lead, an important base metal, is primarily extracted from natural sulfide ores (Nowak and Laajalehto, 2000; Santhiya et al., 2002; Feng et al., 2015). Thus, galena is undergoing steady depletion due to rapid economic and technological developments. Lead oxide has been developed as an alternative source of lead to satisfy its future market demands. Cerussite, a major mineral source of lead oxide, is a semi-soluble salt mineral with a standard solubility product constant of  $7.40 \times 10^{-14}$  (Powell et al., 2009; Shirota et al., 2011); its surface hydration is more extensive than the corresponding sulfide minerals, resulting in poor cerussite floatability (Fuerstenau et al., 1987; Herrera-Urbina et al., 1998, 1999). Therefore, lead oxide minerals are frequently treated with sulfidizing agents prior to flotation to improve their surface hydrophobicity by converting the lead oxide surface into lead sulfide compounds that will respond to flotation.

Similar to copper and zinc oxide minerals, sulfidizationflotation using sodium sulfide as a sulfidizing agent yields optimal performance for the commercial treatment of lead oxide minerals, during which sulfidization is the most critical stage (Kongolo et al., 2003; Gush, 2005; Mehdilo et al., 2014; Li et al., 2014). Malghan (1986) reviewed the role of sodium sulfide in the flotation of metal oxide minerals and discussed variables influencing the sulfidization process. Meanwhile, Malghan also proposed a method to measure sodium sulfide in pulp solutions. Marabini and Cozza (1988) demonstrated the activation role of sodium sulfide in the xanthate flotation of cerussite through transmission IR spectroscopy. Herrera-Urbina et al. (1998, 1999) also investigated the interaction of cerussite with aqueous hydrosulfide and amyl xanthate through zeta potential measurements and electrochemistry analysis. Although there are many studies on the sulfidization flotation of metal oxide minerals, there is a common problem in the sulfidization process because an insufficient or excess addition of sulfidizing agents has detrimental effects on flotation performance (Fa et al., 2005; Irannajad et al., 2009; Lee et al., 2009). It is known that the high solubility of metal oxide minerals causes dissolution of large amounts of metal ions from the mineral lattice; these ions can precipitate the added sodium sulfide as metal sulfide. Thus, sulfidization on the mineral surface only proceeds upon exhaustion of aqueous metal cations. With further addition of sodium sulfide, the surface transformation reaches equilibrium. Subsequently, some sulfides remained adsorbed on the sulfidized cerussite surface, resulting in oversulfidization of metal oxide minerals, which inhibits the subsequent xanthate flotation of metal oxide minerals due to the following reasons. First, as a strong reducing agent,





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excess sulfide ions will decrease the potential of the mineral/solution interface below the value required for the formation of metal xanthate. Second, metal sulfide is more easily formed than metal xanthate when sulfide ions and xanthate ions competitively adsorb onto the mineral surface. In addition, metal xanthate formed on the mineral surface may be replaced by excess sulfide ions in pulp solutions because metal sulfide is more insoluble than metal xanthate. Moreover, excess sulfide ions cause the mineral surface to be strongly negatively charged, inhibiting the adsorption of negatively charged xanthate onto the sulfidized mineral surface.

Given these limitations, researchers developed a control system for the sulfide concentration to optimize the addition of sulfidizing agents (Gush, 2005). The flotation of lead oxide minerals without preliminary sulfidization was also considered, i.e., the development and utilization of various selective chelating agents (Marabini et al., 1989; Cozza et al., 1992; Zhu et al., 2007). Meanwhile, Popoy and Vučinić (1992a,b, 1994) reported that cerussite flotation recovery could be improved through decantation of the pulp solution or prolonged agitation in the lead ion solution before xanthate addition. Furthermore, mechanochemical sulfidization (Yuan et al., 2012; Ke et al., 2013), hydrothermal sulfidization (Liang et al., 2012; Li et al., 2014), and sulfidization roasting (Li et al., 2010) were proposed for pretreatment of metal oxides before the collection stage because these methods would not encounter the difficulty caused by excess sulfide ions in pulp solutions. Although these methods provided new ideas for the treatment of metal oxide minerals, they are restricted to laboratory research and may be unsuitable for industrial applications.

Chloride ions are one of the most abundant inorganic anions in groundwater and seawater and can readily form complexes with lead ions in aqueous solutions. At low concentrations of chloride ions, the dominant species is PbCl<sup>+</sup> in addition to Pb<sup>2+</sup> in the aqueous phase, which attaches to the cerussite surface through ligands. Meanwhile, chloride ions have a small ionic radius, strong penetrability, and a strong adsorption capacity onto metals; thus, they can easily combine with lead on the cerussite surface to form lead chloride complexes and can pass through the mineral surface to exchange carbonate ions  $(CO_3^{2-})$  with the inner cerussite (Bargar et al., 1998; Guo et al., 2015; Wierzbicka-Miernik et al., 2015). This surface is beneficial in that it provides numerous active sites for sulfide ions in the subsequent sulfidization process. Sodium chloride (NaCl), an inexpensive and widely available compound, is an appropriate source of chloride ions due to economic and environmental factors (Rusen et al., 2008). Therefore, NaCl may be used to pre-treat the mineral surface to provide favorable conditions for the subsequent sulfidization. However, minimal information is available regarding the effect of the addition of chloride ions in pulp solutions on the sulfidization flotation of cerussite. In this study, micro-flotation tests, surface adsorption, zeta potential measurements and XPS studies were conducted to investigate the contribution of chloride ions to cerussite surface sulfidization.

# 2. Experimental

#### 2.1. Materials and reagents

High-purity cerussite was obtained from the Yunnan Province of China after manual removal of gangue minerals. The sample was manually ground in an agate mortar and then sieved to produce a size range from -74 to  $+45 \,\mu\text{m}$  cerussite for microflotation tests and adsorption studies of sulfide ions, as well as a fraction with particle sizes of less than  $5 \,\mu\text{m}$  for zeta potential measurements. Nitric acid (HNO<sub>3</sub>) and sodium hydroxide (NaOH) were used to adjust the pulp pH. NaCl was selected as the source of chloride ions. Na<sub>2</sub>S·9H<sub>2</sub>O was added as a sulfidizing agent, and commercial-grade sodium amyl xanthate (NaAX) was used as the collector. All of the reagents, except for NaAX, used in the tests are of analytical grade. Pure deionized water was used for all of the experiments.

#### 2.2. Micro-flotation experiments

Micro-flotation experiments were conducted in a mechanical agitation flotation machine. The mineral suspension was prepared by adding 2.0 g of pure cerussite to 40 mL solutions. The desired amounts of NaCl stock solution were first added into the pulp solution for 5 min if necessary. Freshly prepared Na<sub>2</sub>S·9H<sub>2</sub>O solutions of various concentrations were added to sulfidize the cerussite surface for 5 min; subsequently, the pulp pH was adjusted to 9.5. Then the pulp was conditioned with NaAX for 3 min and floated for 5 min. After the flotation tests, the concentrate and tailings were filtered and dried before weighing. Flotation recovery was calculated based on the solid weight distribution between the two products. Micro-flotation tests were conducted in duplicate. The maximum standard error in the flotation recovery results was  $\pm 2.5\%$ .

### 2.3. Adsorption of sulfide ions on the cerussite surface

The adsorption experiment of sulfide ions on the cerussite surface was performed in a three-necked flask batch reactor with 500 mL of solution in a thermostatically controlled water bath equipped with a digitally controlled mechanical stirrer, a thermometer for temperature control, and a condenser to prevent evaporation loss. Typically, 5 g of the cerussite samples was added before heating the aqueous solution to 298 K. The desired amounts of NaCl stock solution were first added into the pulp solution that was conditioned for 10 min if necessary. Freshly prepared Na<sub>2</sub>S·9H<sub>2</sub>O solution with the required concentration was added to sulfidize the cerussite surface at natural pH, and 5 mL of the resultant solution was accurately withdrawn at specific time intervals during this process. Subsequently, a centrifuge was used for solid-liquid separation. The liquid obtained from the separation was stored in closed vials and analyzed for the total S concentrations using inductively coupled plasma-atomic emission spectrometry (ICPS-1000II, Shimadzu, Japan).

#### 2.4. Zeta potential measurements

Zeta potentials of the samples were measured using a zeta potential analyzer (ZetaPlus, Brookhaven, USA). All of the measurements were conducted in a  $5 \times 10^{-3}$  mol/L KNO<sub>3</sub> background electrolyte solution to maintain the ionic strength of the solution constant. The cerussite samples were ground to a particle size of less than 5 µm. The mineral suspension was prepared by adding 0.10 g of cerussite samples to 100 mL of electrolyte solution at a desired pulp pH, which was adjusted using 0.10 mol/L HNO<sub>3</sub> or 0.10 mol/L NaOH solutions. The obtained suspension was conditioned for a determined time using a magnetic stirrer with the system open to air after each addition of the desired reagent concentrations. The resultant suspensions were allowed to sit for 10 min to settle the coarser particles. It is assumed that the zeta potential of the coarser particles is the same value as that of the finer particles because the chemical nature for various sizes is similar (Ejtemaei et al., 2012; Duzyol and Ozkan, 2014; Liu et al., 2015). Subsequently, a pipette was used to collect ultra-fine particles located at the top of the suspension, and then the collected sample was transferred to a square electrophoresis cell for zeta potential measurements. The average value of the zeta potential of three independent measurements was reported, and the maxiDownload English Version:

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