

## The adsorption of sulfur dispersing agents on sulfur and nickel sulfide concentrate surfaces

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### ABSTRACT

The adsorption behavior of sulfur dispersing agents, including lignosulfonate, Quebracho, o-phenylenediamine (OPD), and humic acid on elemental sulfur and nickel sulfide concentrate was investigated. The charge changes on elemental sulfur surface were characterized by the measurement of the electrokinetic sonic amplitude (ESA) signal. The adsorption behavior of OPD was investigated by measuring the interfacial tension between liquid sulfur and nickel sulfate solution at 140 °C. The adsorption of lignosulfonate on the molten sulfur surface was calculated by the Gibbs Equation. The adsorption of lignosulfonate, Quebracho, and humic acid on the nickel concentrate was studied at ambient temperature. The adsorption mechanism of sulfur dispersing agents on both elemental sulfur and nickel concentrate were discussed. The stability of OPD in the nickel concentrate slurry was discussed.

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### 1. Introduction

Oxygen pressure leaching of nickel concentrate at medium temperatures (140–150 °C) suffers from a sulfur wetting problem. Liquid elemental sulfur forms during the leaching process in the autoclave and will cover unreacted sulfides resulting in low nickel extraction due to surface blockage. The addition of sulfur dispersing agents into the leaching solution is a successful method to solve the sulfur wetting problem in the pressure leaching of zinc concentrate (Kawulka et al., 1975). Owusu (1993) studied the interfacial phenomena in the liquid sulfur–sphalerite–aqueous solution system in the absence or presence of sulfur dispersing agents, including lignosulfonate and OPD. The sulfur dispersing agents have the effect of decreasing the work of adhesion between liquid sulfur and sulfide minerals. The following researchers (Hackl et al., 1995; Dreisinger et al., 2003) investigated the effect of sulfur dispersing agents on the interfacial properties in the liquid sulfur–chalcopyrite–aqueous solution system and the liquid sulfur–pyrite–aqueous solution system. Brown and Papangelakis (2005) investigated the influence of lignosulfonate and chloride on the interfacial properties of the liquid sulfur–pentlandite–aqueous solution system.

Xia et al. (1987) tried to explore the relationship between leaching behavior and the adsorption of lignosulfonate on the mineral surface. The adsorption of lignosulfonate on the mineral surface increased with the dosage increasing, which resulted in a higher leaching rate until the adsorption reached saturation. Owusu and

Dreisinger (1996) estimated the adsorption of lignosulfonate at the liquid sulfur–aqueous solution interface by the Gibbs Equation. Lignosulfonate was adsorbed both physically and chemically by sphalerite. OPD was adsorbed chemically through the interaction of the C–N functional group with metal ions, forming a metal-amine surface complex (Owusu, 1993; Owusu et al., 1993).

The adsorption behavior of sulfur dispersing agents, including lignosulfonate, Quebracho, OPD, and humic acid have not been studied in the system containing nickel concentrate. The main objective of this investigation was to study the adsorption behavior of lignosulfonate, Quebracho, OPD, and humic acid on elemental sulfur and a nickel sulfide concentrate sample. This includes understanding of the surface charge changes on the elemental sulfur before and after sulfur dispersing agent adsorption; to understand the adsorption of sulfur dispersing agent on the nickel concentrate surface by the measurement of the sulfur dispersing agent concentration; to understand the adsorption of OPD and lignosulfonate on the molten sulfur surface (140 °C) by the application of the Gibbs Equation, and; to understand the stability of OPD in the nickel concentrate slurry by the UV/Vis spectra changes; to understand the adsorption mechanism of sulfur dispersing agents during leaching by analyzing the leaching conditions.

### 2. Experimental

#### 2.1. Materials

Elemental sulfur was obtained from Alfa Aesar, at a purity of 99.5%. The original particle size of sulfur was –100 mesh

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(150  $\mu\text{m}$ ). The sulfur sample was ground by a ring-and-puck pulverizer to  $P_{90}$  of 29  $\mu\text{m}$  and stored in double bags at 4 °C (or below).

The nickel concentrate slurry was obtained from XSTRATA Nickel (Strathcona Mill). The main sulfide minerals were pentlandite, pyrrhotite, pyrite, and chalcopyrite. A sub-sample was washed with deionized water and sieved to pass –325 mesh (44  $\mu\text{m}$ ). Chemical assay showed the content of metals: 11.6% Ni, 44.4% Fe, and 1.8% Cu. The sample was washed with pH 2.0 sulfuric acid solution for 5 min, then washed repeatedly with deionized water and dried under vacuum. The nickel concentrate sample was put into polyethylene bags and stored in a refrigerator to avoid oxidation.

The specific surface area of the nickel concentrate was analyzed by the Quantachrome Autosorb-1MP BET analyzer under vacuum after outgassing at 40 °C for 17 h. The surface area of the nickel concentrate sample is 1.62  $\text{m}^2/\text{g}$ .

Lignosulfonate is a complex anionic polyelectrolyte whose structure contains sulfonate, free phenolic, primary and secondary alcoholic, and carboxylate groupings (Rosen, 2004). Three lignosulfonate samples were obtained from Borregaard Lignotech USA. BorrePAL series additives were 100% water soluble. Quebracho is the traditionally important commercial source of condensed tannins. Orfom<sup>®</sup> grade 2 Tannin was obtained from Chevron Phillips Chemical Company LP which contained 85–95% sulfited Quebracho and 5–15% water. OPD has the chemical formula  $\text{C}_6\text{H}_4(\text{NH}_2)_2$  which belongs to the aromatic amine family. Humic acid–potassium salt (D-616), the modified lignite, was obtained from Borregaard Lignotech USA. The humic acid used in this study is a sulfited product. The humic acid molecular structure is variable and strongly depends on the source of the humic acid. Carboxyls, hydroxyls and carbonyls are the major oxygen-containing functional groups in humic acid (Berkowitz, 1994). All the sulfur dispersing agents were used without pretreatment.

## 2.2. Electroacoustics

A Zeta Probe (Colloidal Dynamics, Warwick, RI) was used to investigate the effect of sulfur dispersing agents adsorption on the magnitude of the surface charge of elemental sulfur particles. 10 g sulfur powder and 70 g 0.01 mol/L NaCl background solution were put in a 250 mL conical flask, and conditioned in a shaker for 24 h. This sulfur slurry was further treated in a laboratory blender for 5 min and then transferred into a measuring cup. All suspensions were de-aerated in a Baxter vacuum oven at room temperature by gradually lowering the pressure and quickly returning back to the atmospheric pressure (Pawlik, 2005). Additional background solution was added to make sure that the total solution addition was 250 g. The measuring cup was placed on the Zeta Probe mixer, and the suspension was mixed at 160 RPM for 50 min. The pH of suspension was adjusted by addition of 2.0 mol/L HCl. Once the measuring mode was selected the measurements were performed by the apparatus automatically.

## 2.3. Interfacial tension measurement

The pendant drop method is suitable for the liquid–liquid interfacial tension measurement at high pressure and high temperature (Cheng et al., 1990). It was first used by Owusu (1989) to measure the liquid sulfur–aqueous solution interfacial tension under leaching conditions which is also used in this study. The measurements were carried out at 140 °C, 690 kPa over pressure by nitrogen.

Interfacial excess of lignosulfonate at the liquid sulfur–nickel sulfate aqueous solution interface were calculated by the Gibbs Equation (Chattoraj and Birdi, 1984; Owusu and Dreisinger, 1996). The maximum adsorption is obtained from Eq. (1) when the  $d\gamma/d \ln C$  value is the largest.

$$\Gamma_{\max} = -\frac{1}{RT} \cdot \frac{\partial \gamma}{\partial \ln C} \quad (1)$$

where  $\Gamma_{\max}$  is the maximum adsorption of lignosulfonate at the liquid sulfur–aqueous solution interface,  $\text{mol}/\text{m}^2$ ;  $R$  is gas constant, 8.32 J/(mol K);  $T$  is temperature, K;  $\gamma$  is the liquid sulfur–aqueous solution interfacial tension,  $\text{mN}/\text{m}$ ;  $C$  is the concentration of lignosulfonate,  $\text{mol}/\text{L}$ . It was assumed that both lignosulfonate and nickel sulfate (0.1–1.0 mol/L) dissociate completely.

The molecular area occupied by one lignosulfonate molecule was calculated from Eq. (2) (monolayer adsorption assumed) (Zhang and Yin, 2005):

$$A = \frac{1}{N_A \Gamma_{\max}} \quad (2)$$

where  $A$  is the molecular area of lignosulfonate,  $\text{m}^2$ ;  $N_A$  is Avogadro constant,  $6.022 \times 10^{23} \text{ mol}^{-1}$ .

## 2.4. Adsorption measurements

A UV-2401PC UV-Vis recording spectrophotometer by Shimadzu was used in the study. For uniform conditioning in the adsorption experiments, a Lab-line Orbit Environ Shaker was used at a moderate setting for the indicated amount of time. A centrifuge was used to separate aqueous solution from nickel concentrate.

The UV spectra of lignosulfonate and humic acid were not influenced by the acidity of the aqueous solution under the conditions of this study. The concentration of BorrePAL U, BorrePAL N, BorrePAL S, and humic acid were determined at wavelength 281 nm, 280 nm, 278 nm, 254 nm, respectively. The Acid–Butanol assay was used to determine the concentration of Quebracho in aqueous solution and the measurement was obtained at wavelength 550 nm (Waterman and Mole, 1994; Porter et al., 1986). The concentration of OPD in aqueous solution was determined by spectrophotometric determination method (Isaev and Ishkov, 2002).

## 2.5. The adsorption isotherm

Langmuir adsorption isotherm was used to fit sulfur dispersing agent adsorption. Langmuir equation is shown as Eq. (3) (Langmuir, 1916):

$$Q = \frac{q_m K_x C}{1 + K_x C} \quad (3)$$

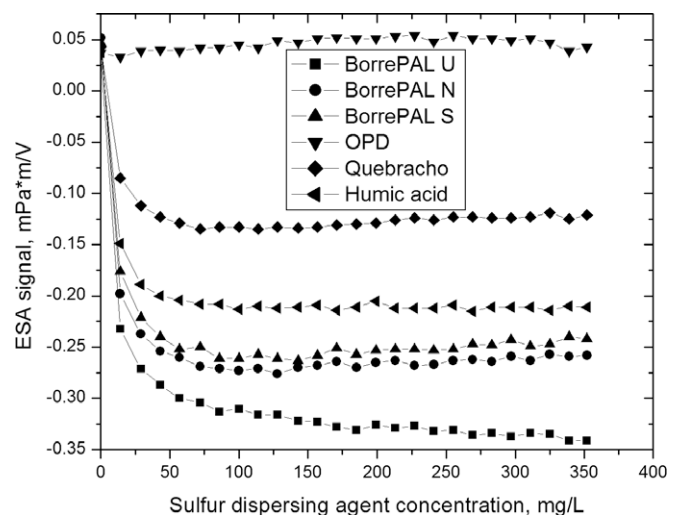


Fig. 1. Electroacoustic effect of sulfur in the presence of sulfur dispersing agent at pH 2.5.

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