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# Lignosulfonate, anionic surfactants and their mixtures influence on water solutions surface tension and zinc concentrate pressure leaching

**E B Kolmachikhina<sup>1</sup>, E A Ryzhkova<sup>1</sup>, D V Dmitrieva<sup>1</sup>, V V Sviridov<sup>2</sup> and S S Naboichenko<sup>1</sup>**

<sup>1</sup> Ural Federal University, 17, Mira St., Ekaterinburg, 620002, Russia

<sup>2</sup> Ural State Forest Engineering University, 37, Sibirskii trakt, Ekaterinburg, 620032, Russia

E-mail: e.b.khazieva@urfu.ru

**Abstract.** The formation of elemental sulfur during sulfide concentrate pressure leaching leads to the appearance of sulfur-sulfide granules, which significantly reduces zinc dissolution rate. The solution for this problem was the use of surfactants, which prevents the negative effect of molten sulfur, avoiding the granule formation during leaching. Lignosulfonates (waste from the woodworking industry) are most often used as reagents for sulfide concentrate pressure leaching. Influence of individual and mixtures of surfactants on surface tension of aqueous solutions, zinc extraction and size-grade distribution of cakes after sulfide zinc concentrate pressure leaching. Surface tension of aqueous solutions was analyzed by stalagmometric method. Leaching was performed in titanium autoclave in presence of lignosulfonate, sodium dodecylbenzenesulfonate and sodium dodecylsulfate. Solutions after leaching were analyzed on zinc by atomic absorption spectroscopy. Size-grade analysis of cakes after leaching was carried out by laser diffraction particle size analyzer. In this work, synergetic influence was observed of anionic surfactants and lignosulfonate on decreasing of aqueous solutions surface tension. The best results were obtained when LS-SDBS mixture was used, namely in the range of sodium dodecylbenzenesulfonate concentrations 400-600 mg / l. Usage of combined surfactants allowed to achieve high rates of zinc extraction and optimal particle size of cakes after leaching. At Ls and SDBS using zinc extraction was increased from 77,2 % up to 82,8 %, with the bulk of the cake (96,7%) having a particle size of -150  $\mu\text{m}$ . Combined surfactants usage (lignosulfonate and sodium dodecylbenzenesulfonate) can be recommended for applying at pressure leaching of sulfide zinc concentrates. The first section in your paper

## 1. Introduction

Autoclave technologies for production of non-ferrous metals from sulfide raw materials became commonly used due to their environmental friendliness, low grade raw material treatment opportunity and associated elements recovery [1-3]. Elemental sulfur formation during sulfide concentrates pressure leaching leads to appearing sulfur-sulfide pellets that results to emergency shutting-down of autoclave [4-5]. Lignosulfonates usage became solution for this problem.

However, lignosulfonates composition (molecular weight, content of hydroxyl, carboxyl, sulfonic groups) is inconstant and ranged depending on processed timber species, delignification conditions, metals ions addition [6]. In works of Lugovitskaya T.N. [7, 8] electrolytes composition influence on surface activity of lignosulfonates with variety of molecular weight were investigated in detail. It was



shown that high molecular weight fraction of lignosulfonates had a maximum effect on surface tension decreasing that can be connected with their less hydrolysis and dissociation in comparison with medium molecular and low molecular samples.

Besides that, surfactants mixture creation, which can enhance individual reagents impact is very important task for non-ferrous metals hydrometallurgy. It is known that combined addition of lignosulfonate and sodium dodecylsulfate leads to micelle formation critical concentration decreasing [9]. Interaction of dodecylbenzene sulfonate with polymethacrylic acid can be followed by formation of intermolecular aggregates [10] that leads to important changing of surface properties.

Combined surfactants (lignosulfonate and water soluble sulfonates) usage at pressure leaching of nickel pyrrhotite concentrates allowed to increase pyrrhotite decomposition degree, enhance of platinum group metal extraction from leaching cakes [4, 11]. Combined surfactants synergistic action allows to decrease solutions surface tension and this property is useful for ore and concentrates leaching intensification [12].

This work is devoted to investigation of simultaneous action of lignosulfonate and anionic surfactants (sodium dodecyl sulfate (SDS), sodium dodecylbenzene sulfonate (SDBS)) on solutions surface tension and zinc concentrates pressure leaching effectiveness.

## 2. Methods

Sodium lignosulfonate, sodium dodecylbenzene sulfonate and sodium dodecylsulfate were used for tests. Surface tension of individual and their mixtures solutions was determined by stalagmometric method at 298 K. Water solutions and sulfuric acid solutions ( $100 \text{ g}\cdot\text{l}^{-1}$ ) were used for tests; surfactants concentration in solutions was varied from 200 to  $800 \text{ mg}\cdot\text{l}^{-1}$ .

Pressure leaching was conducted in presence of individual surfactants and their mixtures. Zinc concentrates pressure oxidative leaching tests were carried out in 1l titanium autoclave at 418 K and oxygen pressure 5 MPa during 60 minutes. Zinc concentrate from Uchaly deposit had following composition, %: 44,2 Zn; 8,16 Fe, 1,22 Cu; 29,2 S.

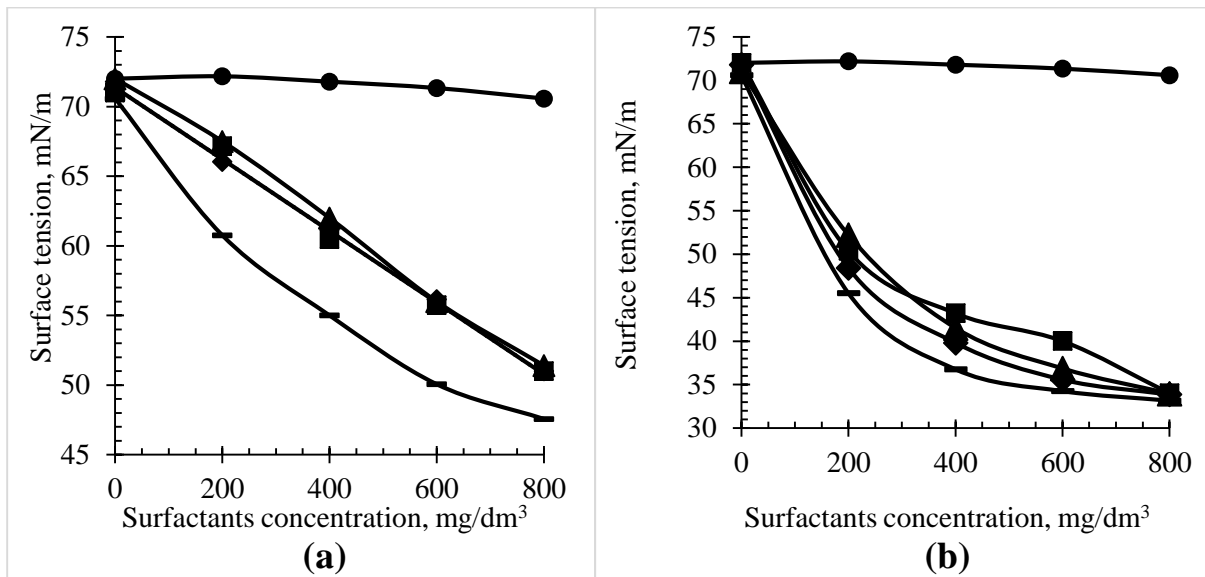
Filter effluent and washwater were analyzed to find out concentrations of Zn, Fe, Cu and  $\text{H}_2\text{SO}_4$  by atomic absorption spectrometer (novAA 300, Analytik Jena) and titration analysis. Mesh-size distribution of leaching residues was measured by laser light scattering particle size analyzer HELOS&RODOS (Sympatec GmbH).

## 3. Experiments results and discussions

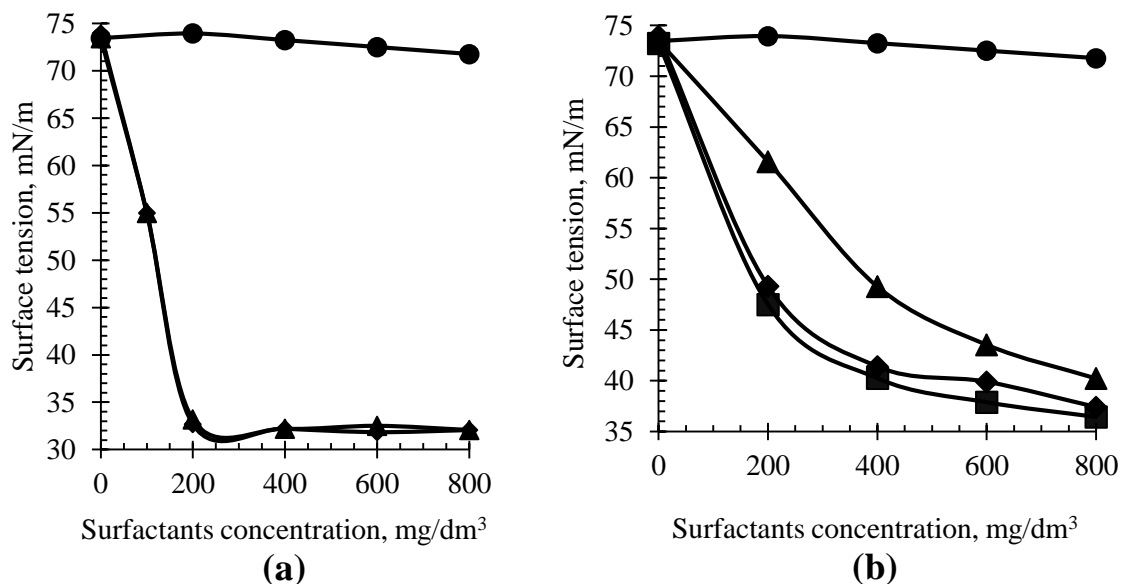
Decreasing of surface tension of water solutions in presence of individual and mixed surfactants is indirect measure of reagents effectiveness at pressure leaching. Tensiometric investigations (figures 1, 2) testify about low surface activity of lignosulfonates, surface tension of this solutions is within the range 70-72 mN/m. Anionic surfactants, SDS and SDBS, decreased water solutions surface tension intensively from 72 mN/m to 20.6 mN/m and 38 mN/m respectively at  $800 \text{ mg}\cdot\text{l}^{-1}$ .

Lignosulfonate action during pressure leaching appears to consist in stabilization of sulfur particles due to a structural mechanical barrier formation. Whereas high surface activity of anionic surfactants promote wettability increasing of sulfides by water solutions and by that wettability decreasing by molten sulfur.

According to fig. 1b in the range of SDBS concentration  $350\text{-}450 \text{ mg}\cdot\text{l}^{-1}$  a sharp change of slope corresponds critical concentration of micelle formation (CCM) adjusting ( $420 \text{ mg}\cdot\text{l}^{-1}$  [13]), similar curve shift on figure 1a was not observed because CCM for SDS equals to  $2.4 \text{ mg}\cdot\text{l}^{-1}$  [14]. At sulfuric acid addition to SDBS solution (figure 2b) surface tension remained as before, but sulfuric acid addition to SDS solution (figure 2a) led to significant decreasing of surface tension and CCM reaching as early as at  $\approx 200 \text{ mg}\cdot\text{l}^{-1}$ .



**Figure 1.** Surface tension changing of surfactants and their mixtures water solutions:  
 a) Concentration influence of Ls, SDS and their mixtures: ● – Ls; ▲ – SDS; ■ - 200 mg/dm<sup>3</sup> Ls+SDS; ◆ - 400 mg/dm<sup>3</sup> Ls+SDS; — - 800 mg/dm<sup>3</sup> Ls+SDS;  
 b) Concentration influence of Ls, SDBS and their mixtures:  
 ● – Ls; ■ – SDBS; ▲ - 200 mg/dm<sup>3</sup> Ls+SDBS; ◆ - 400 mg/dm<sup>3</sup> Ls+SDBS; — - 800 mg/dm<sup>3</sup> Ls+SDBS.



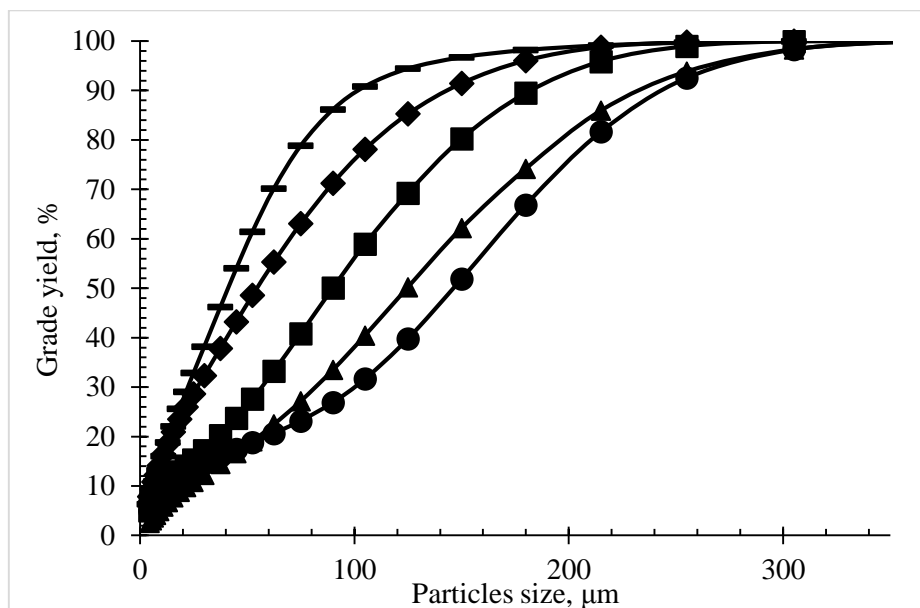
**Figure 2.** Surface tension changing of surfactants and their mixtures sulfuric acid solutions:  
 a) Concentration influence of Ls, SDS and their mixtures:  
 ● – Ls; ▲ – SDS; ◆ - 200 mg/dm<sup>3</sup> Ls+SDS;  
 b) Concentration influence of Ls, SDBS and their mixtures:  
 ● – Ls; ▲ – SDBS; ◆ - 200 mg/dm<sup>3</sup> Ls+SDBS; ■ - 400 mg/dm<sup>3</sup> Ls+SDBS.

Addition of Ls small quantity ( $200\text{--}400\text{ mg}\cdot\text{l}^{-1}$ ) to SDS solutions didn't lead to decreasing of surface tension (figure 1a), only at higher LS concentration ( $800\text{ mg}\cdot\text{l}^{-1}$ ) surface tension lowering on 10.0-11.3 % in comparison with individual SDS solutions was attained. In acidic media (figure 2a) synergetic effect of surface tension decreasing in presence mixture Ls and SDS was disappeared.

Significant synergetic effect was fixed in presence of mixture Ls and SDBS, where surface tension of water and sulfuric acid solutions was decreased at Ls concentration increasing in mixture. More noticeable effect appeared in the range of SDBS concentration  $400\text{--}600\text{ mg}\cdot\text{l}^{-1}$ . Moreover, CCM of mixture Ls-SDBS solutions was decreased at growing Ls concentration. For example, CCM of sulfuric acid solution of SDBS is equal to  $\approx 450\text{ mg}\cdot\text{l}^{-1}$ , and at the presence of  $200\text{ mg}\cdot\text{l}^{-1}$  of Ls in the mixture CCM was decreased up to  $250\text{--}300\text{ mg}\cdot\text{l}^{-1}$ . It is suggested [15] that reduction of surface tension of solutions in presence of surfactants can be related to hydrophobic interactions between surfactants hydrocarbon radicals and hydrophobic polymer chains, there is adsorption of low molecular surfactants on polymeric chain of Ls that causes surfactant CCM decreasing.

Results of leaching cake particle size analysis (figure 3) testify to significant stabilization action of Ls. At Ls presence content of fraction  $-150\text{ }\mu\text{m}$  increased from 80,2 to 91,5 % at the absence reagent and at presence of  $400\text{ mg}\cdot\text{l}^{-1}$  of Ls, respectively. Zinc extraction was enhanced from 44.9 % to 77.2 % (table 1). Optimal cake for flotation was received at addition  $800\text{ mg}\cdot\text{l}^{-1}$  of Ls, 97.5 % of cake particles had fineness  $-150\text{ }\mu\text{m}$ .

However, treatment of zinc cake with size less than  $10\text{ }\mu\text{m}$  can be complicated. It is known that Ls can lead to downsizing of sulfur particles during leaching. Obtained results confirm this strong dispersing action of Ls: content of fraction  $-10\text{ }\mu\text{m}$  was increased from 8.39 % in the absence of reagents to 14.11 % and 26.93 % at Ls addition  $400\text{ mg}\cdot\text{l}^{-1}$  and  $800\text{ mg}\cdot\text{l}^{-1}$ , respectively.



**Figure 3.** Size-grade distribution of cakes after leaching of zinc concentrate at presence of individual and composite surfactants: ■ – surfactants absence, ◆ – Ls  $400\text{ mg}/\text{dm}^3$ ; ● – SDS  $200\text{ mg}/\text{dm}^3$ , SDBS  $200\text{ mg}/\text{dm}^3$ ; ▲ – Ls  $400\text{ mg}/\text{dm}^3$ +SDS  $200\text{ mg}/\text{dm}^3$ ; — - Ls  $400\text{ mg}/\text{dm}^3$  + SDBS  $200\text{ mg}/\text{dm}^3$ .

**Table 1.** Surfactants and their mixtures influence on zinc extraction at sulfide zinc concentrate pressure leaching.

Reagents concentration, mg/dm <sup>3</sup>			Zinc extraction, %
Ls	SDS	SDBS	
-	-	-	49,2
400	-	-	77,2
-	200	-	52,4
-	-	200	58,8
400	200	-	76,6
400	-	200	82,8

In the context of autoclave technology of nickel pyrrhotite concentrates it is known [4, 11] that simultaneously addition of Ls with high molecular oil-products allows to receive cake with optimal fineness (10-150  $\mu\text{m}$ ) and increase velocity of pyrrhotite decomposition. Due to remarkable decrease of water solutions surface tension in presence of Ls and anionic surfactants mixtures, we have studied influence of these reagent of zinc extraction and fineness of leaching cake particles.

At addition of 200  $\text{mg}\cdot\text{l}^{-1}$  SDBS and SDS increasing of +150  $\mu\text{m}$  particles size up to 48.13 % was fixed, content of fraction of -10  $\mu\text{m}$  was equal to 9 %. Zinc extraction at addition of anionic surfactants was in the range 52-59 % (table 1). Simultaneous addition of SDBS and Ls (figure 3) allowed to receive disperse cake: 96.7 % and 13.2 % of -150  $\mu\text{m}$  and -10  $\mu\text{m}$  fractions, respectively; zinc extraction was increased by 5.6 %. Mixture Ls-SDS have not significant influence on cake particles fineness, -150  $\mu\text{m}$  fraction content was equal to 62.16 %, -10  $\mu\text{m}$  – 5 %. Zinc extraction was not changed in comparison with tests without Ls addition.

Consequently, surfactants mixture SDBS-Ls allows to enhance zinc extraction, receive optimal cake fineness due to decreasing of zinc sulfides wetting by molten sulfur (SDBS), stabilization of sulfur drops (Ls).

#### 4. Conclusion

The work is devoted to influence investigation of addition of anionic surfactants and Ls mixtures on pressure leaching zinc concentrates effectiveness. For the purpose of supplementary definition of mixtures effectiveness regularities of solutions surface tension changing was studied.

On the base of obtained results a following conclusions and recommendations was made:

- 1) simultaneous addition of Ls with SDS or SDBS leads to remarkable reducing of surface tension due to decreasing CCM;
- 2) employment of surfactants mixture allows to enhance zinc extraction from 77.2 % to 82.8 % and obtain cakes with optimal fineness (10-150  $\mu\text{m}$ ).

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