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Research article

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Oxidative degradation of lignosulfonates during pressure leaching of zinc concentrates

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Abstract: This study investigates the effect of preliminary autoclave oxidation with molecular oxygen ($T = 423$ K, $P_{O_2} = 0.6$ MPa, $\tau = 2$ h) on lignosulfonates differing in chemical composition and molecular weight distribution. Oxidation resulted in a reduction of hydroxyl groups and an increase in carbonyl groups, along with marked changes in solution properties such as redox potential, pH, specific conductivity, and surface tension at the liquid–gas interface. The functional activity of the initial and oxidized lignosulfonates was compared in terms of their ability to remove elemental sulfur films from the sphalerite surface under high-temperature oxidative pressure leaching conditions. The findings show that oxidative treatment decreases the effectiveness of lignosulfonates by diminishing their surface activity.

Keywords: high-temperature pressure leaching, sphalerite, surfactant, lignosulfonates, oxidative degradation, recovery, leaching.

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Деструктивные превращения лигносульфонатов при автоклавном выщелачивании цинковых концентратов

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Аннотация: Представлены результаты влияния предварительной окислительной обработки молекулярным кислородом в автоклавных условиях ($T = 423$ К, $P_{O_2} = 0,6$ МПа, $\tau = 2$ ч) образцов лигносульфонатов, отличающихся химическим составом и молекулярно-массовым распределением. Показано, что их окисление сопровождается уменьшением гидроксогрупп и увеличением в продуктах окисления карбонильных групп, а также изменением физико-химических свойств растворов — окислительно-восстановительного потенциала, pH, удельной электропроводности, поверхностного натяжения на границе жидкость/газ. Приведена сравнительная оценка функциональной активности исходных и окисленных образцов лигносульфонатов в части удаления

с поверхности сфалерита пленок элементарной серы непосредственно в условиях высокотемпературного окислительного выщелачивания. Установлено, что окислительная автоклавная обработка лигносульфонатов ухудшает их функциональный эффект (поверхностную активность).

Ключевые слова: высокотемпературное автоклавное выщелачивание, сфалерит, поверхностно-активные вещества, лигносульфонаты, деструкция, извлечение, выщелачивание.

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Introduction

High-temperature pressure leaching of sulfide concentrates containing zinc, lead, copper, and nickel is now widely practiced worldwide [1; 2]. Compared with the conventional roasting — leaching route, pressure leaching provides [3; 4]:

— clear environmental benefits, as it avoids SO_2 emissions by converting sulfur in the concentrate into elemental sulfur (S0), which can be safely stored and transported;

— greater processing flexibility, making it possible to treat low-grade sulfide ores and those with a high iron content;

— more efficient resource utilization, with the potential to recover not only the principal metals but also valuable by-products such as gallium, germanium, indium, silver, and cadmium.

Pressure leaching of sulfide concentrates is carried out at temperatures above 373 K and is accompanied by the formation of molten elemental sulfur films on the mineral surface, resulting in sulfur passivation and consequent inhibition or complete cessation of oxidative dissolution [5–7]. Certain surfactants can counteract

this shielding effect of sulfur, opening up the possibility of applying higher-temperature conditions and further intensifying autoclave processes [8; 9].

In hydrometallurgical practice, particularly in the processing of zinc, lead—zinc, and nickel—pyrrhotite concentrates, the most common surfactants are technical lignosulfonates (LS) — by-products of wood pulping [10–12]. Transitioning from low- to high-temperature leaching (410–425 K) boosts process throughput by a factor of at least 2.5. At the same time, more complete exposure of sphalerite inclusions in associated minerals such as pyrite and chalcopyrite provides an additional 2–5 % zinc recovery into solution.

Chemically, lignosulfonates are branched aromatic polymers with a wide molecular weight distribution (5000–80 000 Da), high functional diversity (hydroxyl, methoxy, carboxyl, carbonyl, and sulfonic groups), and pronounced polydispersity [13–15]. Fig. 1 shows a representative fragment of the lignosulfonate macromolecule.

The amphiphilic structure of lignosulfonates accounts for their surface activity: along with ionogenic

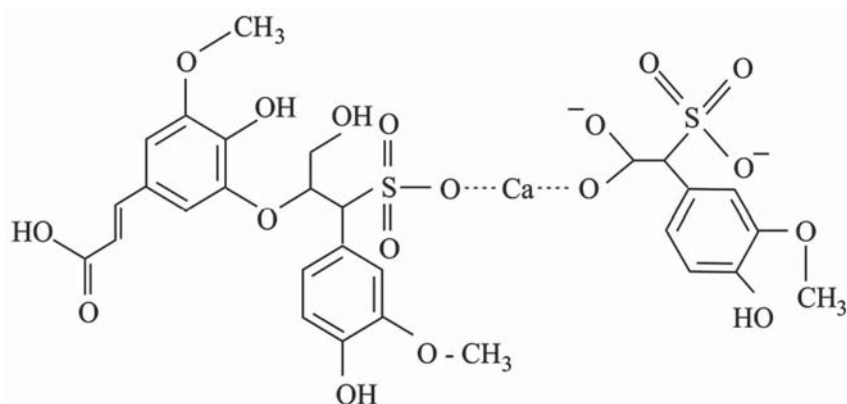


Fig. 1. Fragment of a lignosulfonate macromolecule [16]

Рис. 1. Фрагмент макромолекулы лигносульфонатов [16]

functional groups, they contain cross-linked aliphatic and aromatic carbon chains [17–19].

Surface activity and other physicochemical properties of liginosulfonates depend on both the type of wood used (softwood or hardwood) and the pulping conditions. Because liginosulfonates are by-products, their quality is not standardized.

Their use in hydrometallurgy is further complicated by the instability of their properties over time; efficiency also decreases as the duration of pressure leaching increases. This decline is likely due to chemical degradation of liginosulfonates under the harsh thermo-oxidative conditions applied to ores and concentrates [11].

To investigate this effect, in this work, liginosulfonate samples differing in chemical composition and molecular weight distribution were deliberately subjected to autoclave oxidation with molecular oxygen ($T = 423$ K, $P_{O_2} = 0.6$ MPa, $\tau = 2$ h). Their functional performance was then evaluated directly under high-temperature oxidative pressure leaching of a zinc concentrate. This allowed us to assess how oxidative treatment influences the structure and efficiency (i.e., surface properties) of liginosulfonates during pressure leaching.

Materials and methods

The study employed the following samples and reagents:

1. Liginosulfonates (LS). Samples were obtained from the Solikamsk Pulp and Paper Mill (JSC “Solikamsk-bumprom”, Russia, TU 2455-028-00279580-2014) (LSNo.1) and from LignoTech (Norway) (LSNo.2). Their elemental and functional compositions are given in Table 1. The samples were oxidized in an autoclave with molecular oxygen at $T = 423$ K and $P_{O_2} = 0.6$ MPa for 2 h. The oxidation products of LSNo.1 and LSNo.2 — hereafter LSONo.1 and LSONo.2 — were subsequently characterized by physicochemical methods.

2. Zinc concentrate. A standard zinc concentrate from the Belousovsky Mining and Processing Plant was used. At least 90 % of the material was finer than 74 μm , with the following composition (wt. %): Zn — 48.9, Cu — 0.9, Fe — 8.7, S — 32.5. The principal minerals were sphalerite, present as liberated grains 1–100 μm in size (≥ 70 %), and pyrite containing fine inclusions of sphalerite and chalcopyrite (1–25 μm) amounting to 20–25 %.

3. Gangue minerals. Represented mainly by oxides of Si (0.3 %), Al (0.2 %), and Ca (0.3 %).

Table 1. **Elemental and functional composition of liginosulfonates**

Таблица 1. Элементный и функциональный состав лигносульфонатов

Element/group	LS samples	
	No. 1	No. 2
C	29.0	41.7
O	54.5	38.2
S	5.5	5.4
Na	6.6	0.8
K	0.04	—
Ca	—	4.0
SO ₃ H	12.11	12.3
OCH ₃	9.6	9.2
OH _{phen}	2.1	2.0

4. Sulfuric acid. Solution with a concentration of 140 g/dm³.

5. Oxygen. Technical-grade oxygen supplied from cylinders.

Elemental composition of liginosulfonates was determined with an Elementar Vario MICRO cube analyzer (Germany) with an accuracy of ± 0.5 wt. %. IR spectra of the initial and oxidized LS samples were recorded on a Bio-Rad FTS-175 FT-IR spectrometer in the range 400–5000 cm^{−1}, with a resolution of 0.5 cm^{−1} and an absolute error of ± 0.1 cm^{−1}. Gravimetric measurements were performed on Ohaus Discovery analytical balances (USA) with an accuracy of ± 0.0001 g. pH values were measured on a Mettler Toledo FiveEasy FE20 pH meter (Singapore), calibrated with NIST-traceable buffers (pH 4.01, 7.00, and 10.01). Surface tension of LS solutions at the liquid–gas interface ($\sigma_{\text{liq-g}}$, J/m²) was measured with a Reh binder tensiometer. The surface morphology of the leach residue (particles, grains, and granules) was examined by scanning electron microscopy (SEM) using a dual-beam Carl Zeiss Auriga CrossBeam dual-beam FIB-SEM (Germany) equipped with an Oxford Instruments INCA 350 EDS system and an X-Max 80 detector (UK). Imaging was performed in backscattered electron mode with compositional contrast at an accelerating voltage of 20 kV and a probe current of 1.2 nA.

Pressure leaching experiments were performed in a Vishnevsky-type autoclave (titanium, 1 dm³ capacity) equipped with stirring, temperature control, sampling,

and instrumentation. The reactor filling ratio was maintained at 0.60.

Experiments were conducted at a fixed temperature of 415 K, oxygen partial pressure of 0.4–0.5 MPa, and stoichiometric consumption of sulfuric acid relative to sphalerite (molar ratio $\text{H}_2\text{SO}_4 : \text{ZnS} = 1.0$). The LS concentration in the slurry was varied from 0.05 to 1.50 g/dm³.

After loading the concentrate, initial or oxidized LS samples (sealed in glass ampoules), and sulfuric acid solution, the reactor was sealed and the slurry heated to the target temperature. Once the set temperature was reached, oxygen was introduced via a regulator to establish the required partial pressure, and stirring was initiated. The hydrodynamic conditions in the leaching slurry were kept constant at $\text{Re} = 10^4$ (Reynolds number). The start of agitation and the breaking of ampoules with LS were taken as time zero for the leaching process.

Samples were collected at regular intervals (5, 15, 30, 60, and 120 min) for chemical analysis. The solutions were analyzed for Zn, Fe(II), Fe(III), and H_2SO_4 concentrations. After each run, the reactor was cooled, the slurry filtered, the solid phase washed with distilled water, dried, and analyzed for Zn, Pb, total sulfur (S_{total}), and elemental sulfur (S^0). The progress of oxidation in the presence of LS was monitored by zinc recovery (ϵ_{Zn} , %), iron recovery (ϵ_{Fe} , %), degree of acid neutralization (ϵ_{acid} , %), and conversion of sulfide sulfur to elemental sulfur (ϵ_{S} , %). The leach residues were further characterized for particle size distribution and the fraction of sulfur–sulfide aggregates.

Results and discussion

Before comparing the functional activity of the initial and oxidized lignosulfonates (LS) under high-temperature oxidative pressure leaching, it is useful to examine their composition and physicochemical properties.

As reported earlier [20], the weight-average molecular masses of LSNo.1 and LSNo.2 were 9250 and 46300 Da, respectively. Elemental analysis (Table 1) showed that LSNo.2 contained 13 % more carbon and 16 % less oxygen compared with LSNo.1. The cationic composition of LS in sulfite liquors also differed: LSNo.1 contained sodium ions (6.6 %), whereas LSNo.2 contained calcium ions (4 %).

The IR spectra of the initial samples (Fig. 2) exhibited broad absorption bands at $\nu = 3420 \text{ cm}^{-1}$ and $1510\text{--}1610 \text{ cm}^{-1}$, attributable to hydroxyl groups and

aromatic ring vibrations. Methoxy groups ($-\text{OCH}_3$) were confirmed by absorptions in the range $\nu = 1039\text{--}1042 \text{ cm}^{-1}$ [21–23]. A broad band at $\nu = 1210\text{--}1190 \text{ cm}^{-1}$, along with medium and weak absorptions at 655 cm^{-1} and $540\text{--}520 \text{ cm}^{-1}$, indicated the presence of sulfonate groups. Peaks in the region $\nu = 1675\text{--}1640 \text{ cm}^{-1}$ corresponded to carboxyl groups conjugated with the benzene ring. In LSNo.1, a weak band at $\nu = 1720\text{--}1715 \text{ cm}^{-1}$ was assigned to non-conjugated carbonyl groups ($\text{C}=\text{O}$), which were absent in LSNo.2. These findings confirm that the two samples differ in chemical composition and molecular weight distribution.

The LS samples were then subjected to oxidative treatment ($T = 423 \text{ K}$, $P_{\text{O}_2} = 0.6 \text{ MPa}$, $\tau = 2 \text{ h}$), and the structural features of the oxidation products and the physicochemical properties of their aqueous LSO solutions were determined.

Comparative analysis of the IR spectra of the initial LS and their oxidation products (LSO) (Fig. 2) revealed new absorption bands at $\nu = 1720$ and 1640 cm^{-1} , corresponding to stretching vibrations of non-conjugated and conjugated carbonyl groups ($\text{C}=\text{O}$). At the same time, the intensity of the hydroxyl band at $\nu = 3420 \text{ cm}^{-1}$ decreased.

Under more severe oxidation conditions (higher temperature, longer duration, or elevated oxygen pressure), the band at $\nu = 1510 \text{ cm}^{-1}$, characteristic of skeletal vibrations of the aromatic ring, disappeared.

This indicates that oxidation of LS leads to the formation of carboxyl, carbonyl, and orthoquinone groups and, under harsher conditions, to chemical degradation of aromatic rings with the formation of lower carboxylic derivatives.

Oxidation also significantly altered the physicochemical properties of LS solutions. Measurements of specific conductivity (κ), redox potential (Eh), pH, and surface tension ($\sigma_{\text{liq-g}}$) as a function of LSO concentration (Table 2) showed the following trends:

— as LSO concentration increased from 0.01 to 0.64 g/dm³, pH decreased from 4.3–5.4 to 3.4–3.5, while Eh rose from 185 to 385 mV and conductivity increased from $(12\pm 20) \cdot 10^{-5}$ to $(370\pm 440) \cdot 10^{-5} \mu\text{S/m}$;

— compared with the initial LS, the oxidized samples exhibited greater surface activity: the minimum surface tension ($\sigma_{\text{liq-g}} \approx 0.068 \text{ J/m}^2$) for LSNo.1 was observed at 0.16–0.32 g/dm³, whereas for LSNo.2 it was reached only at higher concentrations ($>0.16 \text{ g/dm}^3$).

Thus, the observed changes in the structure and physicochemical properties of LS indicate that their

Table 2. Physicochemical characteristics of solutions of the initial and oxidized LS

Таблица 2. Физико-химические характеристики растворов исходных и окисленных ЛС

Sample	LS concentration, g/dm ³	$\kappa \cdot 10^5$, $\mu\text{S/m}$		pH		$\sigma_{\text{liq-g}} \cdot 10^3$, J/m ²		Eh, mV	
		LS	LSO	LS	LSO	LS	LSO	LS	LSO
LSNo.1	0.01	7	12	4.4	5.4	80.0	78.1	280	185
	0.02	10	19	5.5	5.2	76.7	79.6	275	195
	0.04	14	34	6.0	5.1	75.2	72.6	240	215
	0.08	22	55	5.7	4.6	75.9	70.6	240	235
	0.16	39	104	5.6	4.2	75.2	68.2	220	245
	0.32	82	200	5.7	4.0	78.1	68.6	200	325
	0.64	130	379	5.8	3.5	75.2	69.1	200	385
LSNo.2	0.01	28	20	5.8	4.3	80.0	79.6	349	310
	0.02	37	26	6.6	4.3	82.3	81.0	280	300
	0.04	40	36	6.7	4.1	86.8	79.5	284	315
	0.08	44	65	6.8	3.9	88.2	79.6	264	320
	0.16	55	116	7.0	3.8	79.6	79.6	280	345
	0.32	70	320	7.3	3.7	79.6	72.3	279	345
	0.64	85	400	7.3	3.5	78.2	75.2	285	340

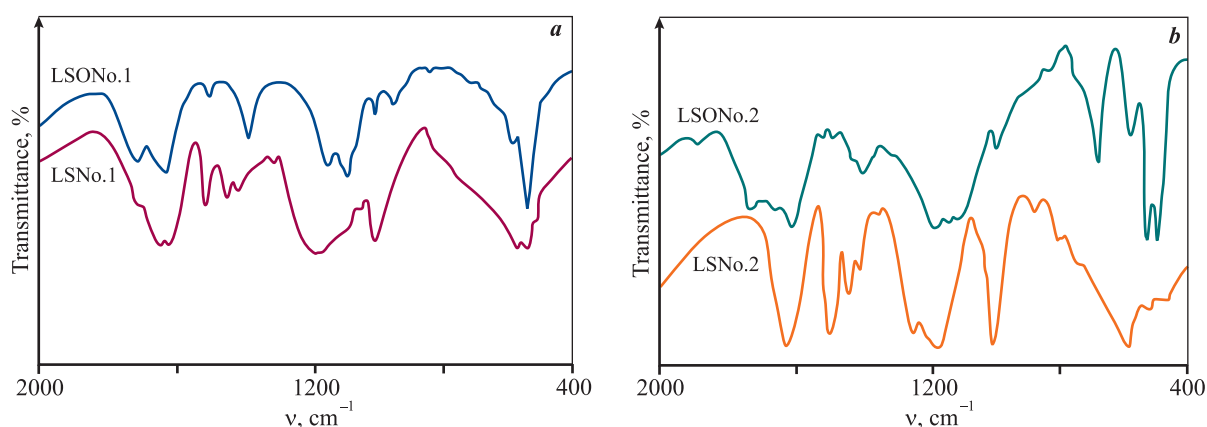


Fig. 2. IR spectra of the initial and oxidized samples of lignosulfonates: LSNo.1 (a) and LSNo.2 (b)

Рис. 2. ИК-спектры исходных и окисленных образцов лигносульфонатов ЛС№1 (a) и ЛС№2 (b)

efficiency may differ under high-temperature pressure leaching conditions.

Subsequently, the effect of the initial and oxidized LS samples on the performance of zinc concentrate pressure leaching was investigated.

Preliminary tests were carried out without lignosulfonates in the leaching slurry (baseline case). A sharp

decrease in the oxidation rate indicated onset of sulfur passivation by molten elemental sulfur films within the first 15–20 min of oxidative treatment, at a mineral conversion of 35–40 %. The insoluble residues were dominated by spheroidal aggregates larger than 0.25 mm, consisting of unreacted sphalerite cores encased in poorly crystallized polygonal elemental sulfur.

Overall, zinc recovery into solution did not exceed 50 % after 2.0–2.5 h of leaching without LS.

Similar results were obtained in the presence of LSNo.1. Within the studied concentration range, the kinetic curves leveled off at 55–65 % conversion, reflecting sulfur passivation of the sphalerite surface. As in the baseline case, the residues contained sulfur–sulfide granules larger than 0.25 mm. Elemental sulfur films on sphalerite were also observed at low LSNo.2 additions ($<0.15 \text{ g/dm}^3$). Oxidation of sphalerite accelerated after 30–40 min of leaching and was significantly more intense in the presence of LSNo.2.

By the time sphalerite had been leached for about 100–140 min, most of the added LS were expected to have undergone oxidation, so that oxidized forms would predominate in solution. This made it relevant to evaluate their functional activity directly under these conditions. After 3 h of leaching, zinc recovery with LSNo.1 was 9–10 percentage points lower than with unoxidized LS (at comparable concentrations of 1.0 g/dm^3), amounting to only 62–63 %.

The most favorable results were obtained with the high-molecular-weight LSNo.2. At concentrations of $0.25\text{--}0.50 \text{ g/dm}^3$, zinc recovery reached 90–91 % within 2 h. Beyond this point, the leaching rate slowed, reflecting zinc release from sphalerite inclusions enclosed in pyrite grains. At insufficient LSNo.2 concentrations ($<0.15 \text{ g/dm}^3$), sulfur passivation limited sphalerite oxidation to 72–75 %. This was confirmed by residue morphology: fine gangue particles, spherical particles 15–25 μm in size con-

sisting of unleached sulfides coated with elemental sulfur (Fig. 3, *a*), and sulfur–sulfide granules larger than 0.5 mm.

Concurrent oxidation of LS during leaching impaired their functional efficiency, as confirmed by experiments with pre-oxidized samples. After 2 h of leaching, zinc recovery in the presence of LSNo.2 was 18–19 percentage points lower than with unoxidized LSNo.2. The reduced interfacial activity of oxidized samples was accompanied by the formation of sulfur–sulfide aggregates 0.10–0.15 mm in size in the residues. Similar degradation of LS and lower metal recovery have also been reported in the literature [11; 12].

Balance tests were conducted under conditions optimized for the composition and concentration of LS (Table 3). As expected, LSNo.2 provided the most effective performance: after 2 h of leaching, 90.5 % Zn and 38.1 % Fe were extracted into solution, while acid neutralization reached 85.9 %. The residue yield was 39–41 % with an elemental sulfur content of 45–46 %, and no sulfur–sulfide granules were detected (Fig. 3, *b*).

Thus, testing of different LS samples under pressure leaching conditions was consistent with the predictive assessments obtained earlier from physicochemical studies of lignosulfonates and confirmed the high functional efficiency of the high-molecular-weight sample (LSNo.2). The unsatisfactory performance of low-molecular-weight LS, as well as their oxidation products, is attributed to an insufficient interfacial wedge effect at the sphalerite–sulfur boundary.

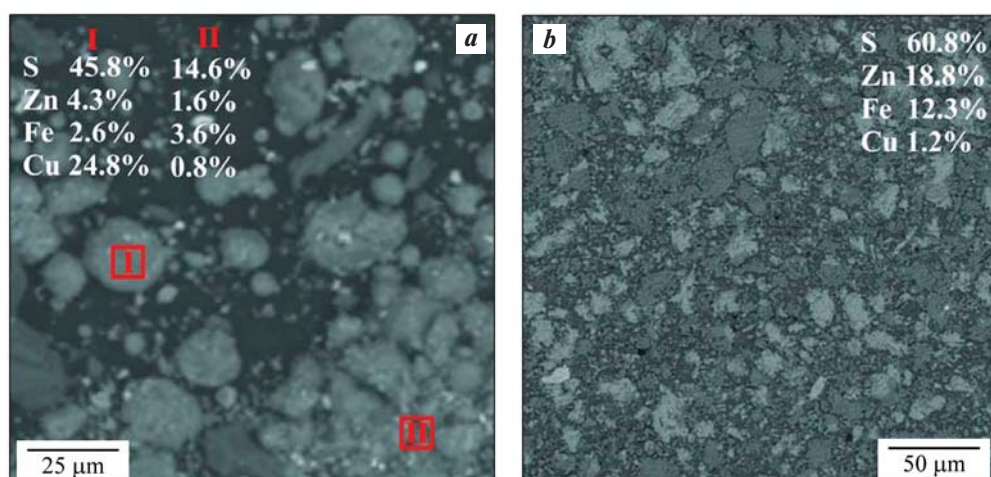


Fig. 3. SEM images and elemental analysis of leach residues obtained with LSNo.2 (*a*) and LSNo.2 (*b*)

Рис. 3. СЭМ-изображения и результаты элементного анализа кека, полученного при выщелачивании в присутствии образцов ЛС№2 (*a*) и ЛС№2 (*b*)

Table 3. Parameters of zinc concentrate pressure leaching in the presence of various LS samples

Таблица 3. Показатели автоклавного выщелачивания цинкового концентрата в присутствии различных образцов ЛС

LS samples	Test conditions*		Content in solution, g/dm ³				Recovery, %			Granule formation
	LS concentration, g/dm ³	τ, min	Zn	Fe(II)	Fe _{общ}	H ₂ SO ₄	Zn	Fe**	H ₂ SO ₄	
—	0.00	180	51.0	2.3	2.7	70.9	54.0	16.0	50.2	Yes
LSNo.1	0.25	180	61.5	2.9	3.2	58.5	65.1	18.9	58.9	Yes
LSNo.1	1.00	180	67.8	3.3	3.5	54.7	71.8	20.8	61.6	Yes
LSNo.1	1.00	180	58.6	3.1	3.5	62.9	62.1	20.8	55.8	Yes
LSNo.2	0.10	120	68.5	3.3	4.0	45.3	72.5	23.8	68.2	Yes
LSNo.1	0.25	120	85.5	5.4	6.4	20.1	90.5	38.1	85.9	No
LSNo.2	0.25	120	68.5	4.3	4.8	4.6	72.5	28.8	69.4	Yes

* Conditions: $T = 413\text{ K}$, $P_{O_2} = 0,5\text{ MPa}$, $[H_2SO_4] = 140\text{ g/dm}^3$.
** From pyrite (according to phase analysis of residues).

Conclusion

The investigation of the physicochemical properties of LS used to remove elemental sulfur films from the sphalerite surface during pressure leaching of zinc-bearing sulfide feed is not only of theoretical interest but also provides a foundation for optimizing oxidation processes and subsequent operations for the processing of solutions and leach residues.

The effect of LS of different composition, including samples pre-oxidized with molecular oxygen, was evaluated during sulfuric acid pressure leaching of zinc concentrate from the Belousovsky Mining and Processing Plant under the following conditions: $T = 415\text{ K}$, $P_{O_2} = 0.5\text{ MPa}$, $[H_2SO_4]_0 = 140\text{ g/dm}^3$, $Re = 10^4$, $H_2SO_4 : ZnS = 1.0$.

High-molecular-weight LS (46300 Da) proved to be the most effective both in removing elemental sulfur films from the sphalerite surface and in ensuring intensive and near-quantitative zinc dissolution. At LSNo.2 concentrations above 0.15 g/dm^3 , stable leaching proceeded without the formation of sulfur–sulfide aggregates. Within 120 min of leaching, 90.5 % Zn and up to 38 % Fe were recovered into solution. The leach residues, which accounted for 39–41 % of the mass, contained no less than 45 % elemental sulfur.

The results demonstrated that LS efficiency depends not only on their molecular-weight distribution but also on the chemical transformations they undergo under pressure leaching conditions. Compared with un-

oxidized LS, the oxidation products were less effective in removing elemental sulfur films from the sphalerite surface: zinc recovery decreased by 10–12 percentage points and did not exceed 72–75 %.

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