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# A COMPREHENSIVE STUDY OF THERMODYNAMICS AND ELECTROCHEMICAL PROCESSES INVOLVING MODIFIED IRON(II)-BEARING SPHALERITE IONS WITH THIOL COLLECTORS

## Introduction

Contemporary trends in the mining sector necessitate the adoption and introduction of technologies that promote sustainable regional development [1], while minimizing the ecological impact [2–4]. Prioritizing efficient energy utilization [5–7] and reducing waste generation in mining operations [8, 9] are imperative. Furthermore, the mining industry is in urgent need for digitalization of its processes [10, 11], including the implementation of intelligent transport management systems [12].

In mining production, all technological processes follow a common chain [13]. Product quality control is paramount at every stage, spanning from mining operations [14] to metallurgical processing [15–17]. The use of mining technologies with backfilling can lead to increased ore dilution [18], while dilution of minerals with backfill material can have negative effects on the enrichment process [19]. The study outlined in reference [20] is dedicated to the development of resilient backfill materials that resist degradation, ultimately reducing ore dilution.

The reagents used in the enrichment alter the chemical composition of the pulp resulting in complex compounds [21], characterized by increased mobility and a potential negative impact on the environment [22]. Modern approaches to mitigating the environmental effects of industrial waste are outlined in [23].

Drawing from industrial experiences in the extraction of sulfide ores, particularly lead–zinc and copper–zinc ores, which fall within the polymetallic ore group, it becomes evident that enhancing technologies at all stages of mining production is of great importance. This improvement is a key to enhancing enrichment quality during concentrate production and the subsequent extraction of metals [24–26].

Variations in the material and mineralogical composition of ores entering the processing plant, in conjunction with the existing reagent flotation regimes have a significant impact on both the quality and quantity of plant performance [27]. Recent research endeavors [28] have illuminated the profound influence of material composition, particularly in the case of polymetallic ores containing zinc such as sphalerite. Within this context, the presence or absence of specific components and their diversity play a significant role in shaping the flotation properties of sulfide ores. Moreover, some of these components may even serve as activators, further underscoring the intricate nature of the flotation process [29].

The beneficiation of copper–zinc sulfide ores and polymetallic ores typically employs collective–selective and selective beneficiation schemes utilizing froth flotation techniques. Primary collectors such as xanthates and alkali metals dithiophosphates, in combination with non-organic salts such as copper sulfate, zinc sulfate, iron sulfate, etc. serve as essential modifiers for flotation of non-ferrous ore processing [30]. The influence of thiol collectors on the flotation properties of sphalerite, both in the presence of non-organic modifiers and the combined effect of organic and non-organic reagent on

*This article presents a comprehensive study of interactions between sphalerite surfaces and various compounds, including iron sulfate, butyl xanthate and sodium dibutyl dithiophosphate, shedding light on their effects on the kinetics of sphalerite flotation, through a combination of thermodynamic and potentiometric analyses. It delves into impact of non-ferrous sulfates and thiol collectors on flotation of sphalerite, both in isolation and in the presence of iron-containing compounds. These investigations provide valuable insights into the complex dynamics of mineral flotation processes and offer crucial information for optimizing mineral processing in the mining industry.*

**Keywords:** flotation, sphalerite, potassium butyl xanthate, sodium butyl dithiophosphate, thermodynamics, electrochemistry, iron(II)-bearing sphalerite, modified sphalerite surface, isobaric–isothermal potential calculation, potentiometric studies of sphalerite in aqueous solutions

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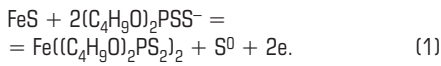
sphalerite, is a subject of widespread discussion in numerous articles, this underscores the continued relevance and significance of this research [31].

Natural activation processes often characterized by oxidation phenomena, occur within the ore deposit itself and are accentuated during extraction. This natural activation is further heightened as the extracted material comes into contact with oxygen during the separation process, and it continues during subsequent crushing stages. In the technological enrichment of sphalerite, these natural activation processes are considered, and mechanical activation methods, such as additional grinding are employed to enhance their effects [32]. Additionally, the presence of water-soluble copper minerals such as  $(\text{CuSO}_4 \cdot 5\text{H}_2\text{O})$  within the sphalerite composition itself contributes to its activation.

For another thing, the flotation activity can be significantly altered through the incorporation of various additives. One such activation additive that enhances the recovery of valuable components is copper sulfate [33, 34]. Preparatory activation treatment of sphalerite with copper ions has been shown to elevate collector sorption, thereby strengthening its affinity for the mineral surface [23]. Research efforts have also identified reagents that can either inhibit certain processes or reduce the extraction of specific minerals, known as depressors [35]. There is a substantial body of research dedicated to investigating the activation abilities of sulfhydryl collectors during the flotation-based enrichment of sulfide minerals, including innovative developments and enhancements to existing technologies for the flotation-based enrichment of polymetallic ores containing zinc (e.g. copper–zinc and lead–zinc). This underscores the ongoing relevance of the subject [36].

This article reports the outcomes of thermodynamic and potentiometric investigations into the interaction between sphalerite surfaces and iron sulfate, potassium butyl xanthate and sodium dibutyl thiophosphate. Additionally, it explores the impact of these reagents on the sphalerite flotation kinetics.

The thermodynamic system under investigation is represented as  $\text{ZnS} \mid \text{FeS} - \text{ROCSS}^- - (\text{RO})_2\text{PSS}^- - \text{H}_2\text{O}$ . The partial thermodynamic stability diagram for this system,  $\text{ZnS} \mid \text{FeS} - (\text{RO})_2\text{PSS}^- - \text{H}_2\text{O}$  was constructed based on the referenced methodology. This methodology enabled the calculation of the isobaric-isothermal potential for the primary reaction involving the interaction between iron(II) sulfate and dibutyl dithiophosphate ions:

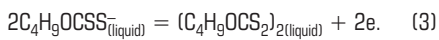
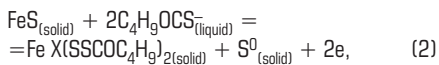


Figures 1 and 2 display the results of calculations for the combined thermodynamic stability diagram of the ZnS]FeS – ROCSS<sup>-</sup> – (RO)<sub>2</sub>PSS<sup>-</sup> – H<sub>2</sub>O system. These calculations were conducted during the transformation of the sulfate sulfur (FeS) into elemental sulfur at pH levels of 8 and 10. The choice of pH values is based on the technological parameters of sphalerite flotation in copper–zinc flotation processes for pyrite copper–zinc ores. Additionally, it takes into account the absence of zinc and iron(II) hydroxide precipitation at pH 10, as previously established [37, 38]. These factors determined the equilibrium concentration of Fe<sup>2+</sup> cations, used in the thermodynamic system calculation at pH 10.

Thermodynamic calculations indicate that at pH 8 (see Fig. 1) during the initial transformation of sulfate sulfur from iron(II) sulfate (pyrrhotite) into elemental sulfur, the primary oxidation product of pyrrhotite, found on the surface of sphalerite, is Fe<sup>2+</sup>. Under more oxidizing conditions, Fe<sup>2+</sup> undergoes further oxidation, forming Fe(OH)<sub>3</sub>.

The equilibrium concentration of Fe<sup>2+</sup> cations (10<sup>-6</sup> g-ion/l) was determined by the absence of iron(II) hydroxide precipitation when an alkali medium (pH 8) and iron(II) sulfate were mixed. This concentration value can be attributed as one of the possible reasons for the formation of these iron chemical compounds. Considering the solubility equilibrium constants  $K_{sp \text{ Fe(OH)}_2(\text{Fe}^{2+}, 2\text{OH}^-)} = 1 \cdot 10^{-15}$  and  $K_{sp \text{ Fe(OH)}_3(\text{Fe}^{2+}, 3\text{OH}^-)} = 5 \cdot 10^{-10}$  [39], the selected concentration of Fe<sup>2+</sup> cations (10<sup>-6</sup> g-ion/l) at pH 8 is lower than equilibrium concentration of the same ions, which would lead to Fe(OH)<sub>2</sub> precipitation with  $K_{sp} [\text{Fe}^{2+}] = 1 \cdot 10^{-3}$  g-ion/l.

The analysis of the diagram revealed that reactions (2) and (3) play a crucial role in forming the adsorption layer of xanthate on the pyrrhotite surface:



Adsorption (2) is chemical adsorption, leading to butyl xanthate iron(II) complex formation, while (3) is associated with physical adsorption, leading to dixanthogenide formation.

Elevated oxidation–reduction potential (ORP) values in the liquid phase lead to the oxidation of iron(II) xanthogenate, resulting in its transformation into iron(III) — (4), as indicated by the relevant region on the diagram.



Based on the diagram, the conversion of mineral sulfate sulfur to its elemental form at pH 8 indicates that the chemical sorption of the collector is only feasible at high collector concentrations (approximately 1 · 10<sup>-2</sup> g-ion/l). Such high concentrations are not typical in the reagent schemes for flotation and sphalerite depression in the processing of copper–zinc and polymetallic ores.

Dixanthate can be found on the surface of pyrrhotite over a wide range of xanthate concentrations in the liquid phase.

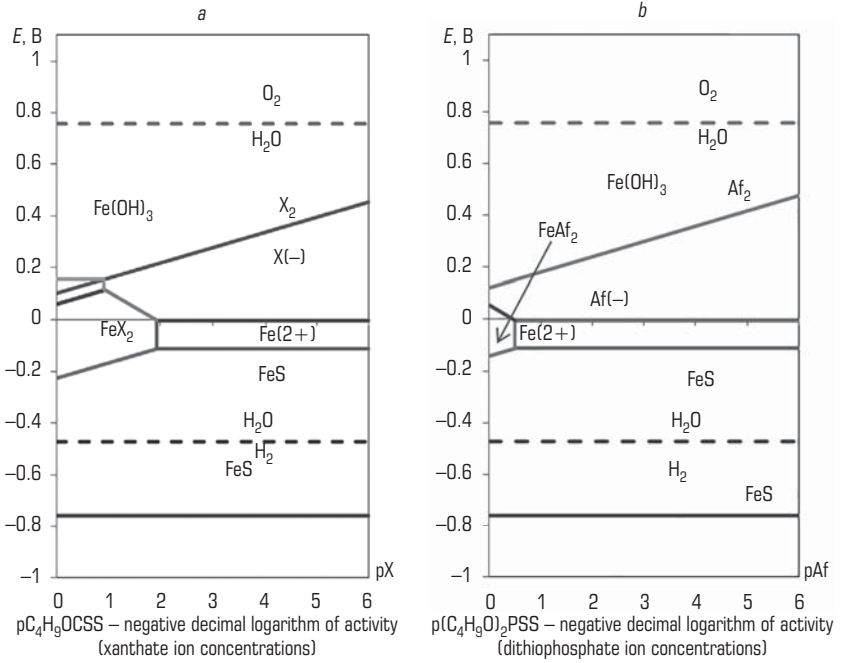


Fig. 1. Combined diagrams of thermodynamic stability for ZnS]FeS – CO<sub>2</sub> – C<sub>4</sub>H<sub>9</sub>OCS<sup>-</sup> – (C<sub>4</sub>H<sub>9</sub>O)<sub>2</sub>PSS<sup>-</sup> – H<sub>2</sub>O (pH 8) systems

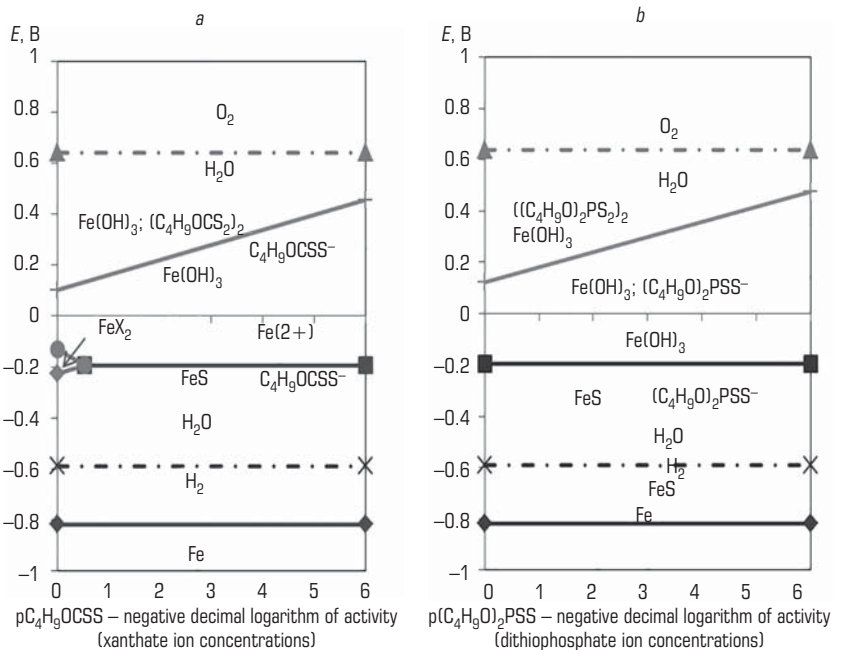
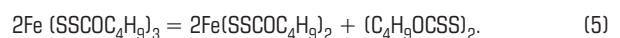


Fig. 2. Combined diagrams of thermodynamic stability for ZnS]FeS – CO<sub>2</sub> – C<sub>4</sub>H<sub>9</sub>OCS<sup>-</sup> – (C<sub>4</sub>H<sub>9</sub>O)<sub>2</sub>PSS<sup>-</sup> – H<sub>2</sub>O system at pH = 10

According to some studies, iron(III) butyl xanthate is an unstable solution that transforms into iron(II) xanthate and dixanthogenide (5):

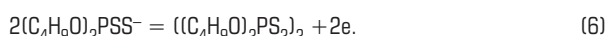


The switch to sodium dibutyl dithiophosphate bring no significant alterations in the composition of the adsorption layer of the collector on the surface of iron(II) modified by cations of sphalerite (see Fig. 1, b).

Due to differences found in solubility of butyl xanthate and iron(II) dibutyl dithiophosphate, the transition from xanthate to dithiophosphate ions results in a narrower zone for iron(II) dibutyl dithiophosphate compared to

iron(II) butyl. In both cases, the chemisorption of collectors ( $\text{Fe}(\text{SSCO}_4\text{H}_9)_2$  or  $\text{Fe}(\text{SSP}(\text{OC}_4\text{H}_9)_2)_2$ ) on pyrrhotite surface is possible when collector concentrations are high, which is atypical for sphalerite flotation. The subsequent analysis revealed that in both cases, in flotation concentration of collectors, only the physisorption of a collector was possible on pyrrhotite surface:  $(\text{C}_4\text{H}_9\text{OCS}_2)_2$  or  $(\text{C}_4\text{H}_9\text{O})_2\text{PS}_2$ .

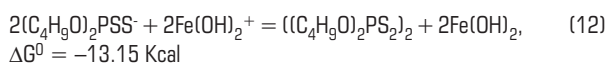
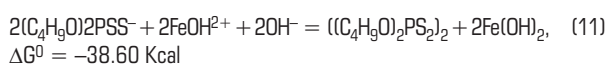
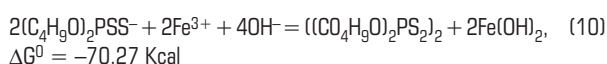
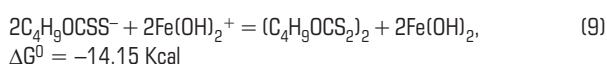
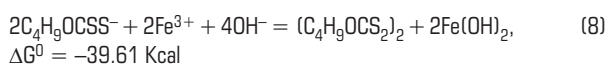
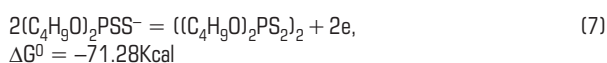
According to the diagrams in Fig. 2, in alkaline media ( $\text{pH} = 10$ ), in the case of sphalerite modified by iron(II) cations with xanthate, the formation of  $\text{Fe}(\text{SSCO}_4\text{H}_9)_2$  on sphalerite surface is only possible with collector concentrations much higher than typically used in flotation practice. However, with sodium dibutyl dithiophosphate, the thermodynamic conditions for the formation of  $\text{Fe}(\text{SSCO}_4\text{H}_9)_2$  on sphalerite surface are not met. This implies that at  $\text{pH} = 10$ , oxidation of xanthate ions and dithiophosphate ions into disulphides is thermodynamically possible by reactions (3) and (6) at  $\text{pH} 10$ :



At the same time, reaction (3) is more likely due to its lower standard electrochemical potential  $E^0 = 0.1 \text{ V}$  compared to (6) with  $E^0 = 0.122 \text{ V}$ .

By comparing the areas where  $\text{C}_4\text{H}_9\text{OCS}_2^-$  and  $(\text{C}_4\text{H}_9\text{O})_2\text{PSS}^-$  are present, along with the final product of pyrrhotite oxidation, which is iron hydroxide (III), at selected chosen pH value of the liquid phase, it is apparent that these ions can be oxidized either by oxygen from the liquid phase or by iron(III)-containing cations like  $\text{Fe}(\text{OH})_2^+$ ,  $\text{FeOH}^{2+}$ ,  $\text{Fe}^{3+}$  in equilibrium with  $\text{Fe}(\text{OH})_3$ .

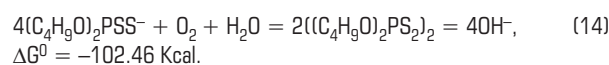
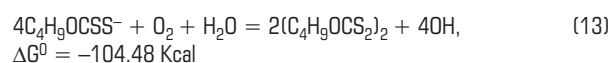
This is supported by the calculations of isobaric–isothermal potential for the reactions shown below:



The negative values of the isobaric–isothermal potential for these reactions indicate that under standard conditions, there is a propensity for the oxidation of butyl xanthate and dibutyl dithiophosphate ions by the iron-containing products resulting from the oxidation of iron(II) sulfate (pyrrhotite) that coats the surface of sphalerite.

By using  $\Delta G_0$  values for typical reactions, it was possible to calculate their equilibrium constants. These calculations helped determine the relationship between the equilibrium concentrations of xanthate and dithiophosphate ions, which, in turn, governed the possibility of simultaneous oxidation of both ions into their corresponding disulphides —  $(\text{C}_4\text{H}_9\text{OCS}_2)_2$  and  $((\text{C}_4\text{H}_9\text{O})_2\text{PSS})_2$ . The results of these calculations indicate that regardless of the composition of iron(III) containing cations involved in reactions (7)–(12), the concentration ratio  $[(\text{C}_4\text{H}_9\text{O})_2\text{PSS}^-]/[\text{C}_4\text{H}_9\text{OCS}_2^-]$  remains constant at 2.34. Consequently, in homogeneous reactions, such as (8) and (11), the oxidation process of dibutyl dithiophosphate ions into their disulphide  $(\text{C}_4\text{H}_9\text{O})_2\text{PSS}^-$ , requires 2.34 times the concentration of dibutyl dithiophosphate ions compared to the concentration of butyl xanthate ions needed for their oxidation into dibutyl ethanalamine.

Similar calculations were performed for the oxidation reactions of these ions with dissolved oxygen molecules in alkaline water (13) and (14). These calculations revealed that the concentration of dibutyl dithiophosphate ions is 5.5 times higher than the concentration of butyl xanthate ions:



The calculations demonstrate that when sodium dibutyl dithiophosphate is used, the presence of  $(\text{C}_4\text{H}_9\text{O})_2\text{PSS}^-$  in the liquid phase of sphalerite mineral suspensions, modified by iron(II) cations, is considerably larger than the presence of  $\text{C}_4\text{H}_9\text{OCS}_2^-$  when using potassium butyl xanthate in an alkaline environment. It is worth noting that the solubility of  $\text{Fe}(\text{SSCO}_4\text{H}_9)_2$  and  $\text{Fe}((\text{C}_4\text{H}_9\text{O})_2\text{PSS})_2$  is  $9 \cdot 10^{-9}$  and  $7 \cdot 10^{-7}$ , respectively. The precipitation of iron(II) butyl xanthate and iron(II) dibutyl dithiophosphate increases the concentration of dibutyl dithiophosphate by 8.8 times compared to butyl xanthate ions. The presence of a broad range of dibutyl dithiophosphate ions creates conditions for the development of  $\text{Fe}((\text{C}_4\text{H}_9\text{O})_2\text{PSS})_2$  layer on the pyrrhotite surface, which is generated on the surface of sphalerite after treatment with iron(II) ions.

In summary, the calculation results indicate that physisorption of a collector in the form of its disulphide is feasible in both low and alkali media.

Thermodynamic calculations alone cannot confirm the chemical interactions of collectors. A comprehensive study requires the application of experimental physicochemical methods.

#### Flotation kinetics of sphalerite modified by iron(II) ions and using thiol collectors

Kinetic studies are highly sensitive for revealing the mechanisms of action of flotation reagents [40]. The floatability of sphalerite with thiol collectors and modifiers such as ferric sulfate was comprehensively studied in [41].

The flotation experiments were conducted in the following manner. The sphalerite sample was mixed with pH-adjusted water (lime medium) in a flotation chamber for 5 minutes. Subsequently, iron(II) sulfate was introduced into the solution, and the mixture was stirred for additional 5 minutes. The amount of iron sulfate was reduced to 20 g/t of anhydrous iron(II) sulfate.

Next, 100 g/t of the collector compound (either potassium butyl xanthate or sodium dibutyl dithiophosphate) was added, and the solution was mixed for additional 3 minutes. The final component introduced was the frother which consisted of methyl isobutyl carbinol (MIBC) at a concentration of 40 g/t. The earlier studies [20] determined that the minimum amount of iron(II) sulfate required (20 g/t) would not result in the precipitation of iron(II) hydroxide.

**Figure 3** depicts the flotation kinetics of sphalerite with thiol collectors at different pH values of the mineral suspension, both with and without the presence of iron sulfate in the flotation cycle.

The presence of iron sulfate in sphalerite flotation with potassium xanthate had a significant impact on the flotation kinetics. In the absence of iron sulfate (see Fig. 3, a), the flotation kinetics was primarily influenced by the pH of the mineral suspension: the flotation speed essentially reduced with higher pH values, and the kinetics of flotation was far from conforming with Beloglazov's equation.

In the presence of iron sulfate (see Fig. 3, b), the flotation speed notably increased at all values of pH of the liquid phase, especially at pH 12. Iron sulfate appeared to activate sphalerite particles. A similar impact was observed at pH 10.

The use of sodium dibutyl dithiophosphate resulted in changes in sphalerite flotation, with and without iron sulfate. In the absence of iron sulfate (**Fig. 4, a**), the highest flotation rate was observed at pH 10, and the sphalerite recovery–time graph displayed an S-shaped curve at both pH 10 and pH 8. However, the presence of iron sulfate in sphalerite flotation with

sodium dibutyl dithiophosphate (Fig. 4, b) had a suppressive effect on sphalerite at pH 8 and 10. Nevertheless, a slight sphalerite activation was noted at pH 12.

The differences in the impact of iron sulfate on the flotation kinetics of potassium butyl xanthate and sodium dibutyl dithiophosphate at pH 8 and 10 can be attributed to variances in the collector adsorption layer on the mineral surface. Thermodynamic calculations indicate that the modified surface of sphalerite is more likely to form a chemical adsorption form of xanthate than dithiophosphate. Additionally, it is worth noting that only one physical form of adsorption for each collector can exist on the modified sphalerite surface at  $\text{pH} \geq 10$ . The enhanced activation of sphalerite by iron(II) ions during the flotation with xanthate may be linked to the oxidation of the ion form of the collector into xanthate. The appearance of disulfide necessitates higher redox potential values of the mineral suspension.

To summarize, the results of the kinetic studies using two thiol collectors align with the outcomes of thermodynamic calculations and electrochemical studies, providing further evidence of the potential influence of iron(II) ions on the composition and formation of the adsorption layer on the surface of activated sphalerite.

**Potentiometric studies of the sphalerite electrode in collector solutions, both with and without prior surface modification of the electrode using iron(II) ions**

The characteristics of sulfide mineral flotation are influenced by both thermodynamics and kinetics of electrochemical reactions, which result in the formation of surface compounds that dictate the wetting characteristics and flotation activity of minerals [40, 41].

The potential of the sphalerite electrode can change when the surface of sphalerite, whether treated or untreated with iron cations, comes into contact with the collector. This change indicates heterogenous reactions occurring on the surface. Thermodynamic calculations demonstrate that the surface modification of sphalerite grains with iron(II) ions is achievable in acidic, neutral and slightly alkaline environments. Consequently, potentiometric studies of the sphalerite electrode, both treated and untreated with iron(II) ions) in collector solutions at pH 8 and 10 were conducted. The experimental procedures for these studies were previously described in [37].

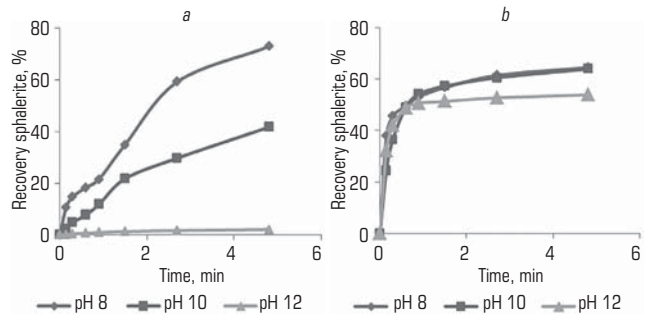
Figure 5 displays the potential of the sphalerite electrode over time when in contact with collectors at pH 8 and 10, with and without treatment with iron(II) cations. The presented results on the graphs correspond to the lowest concentrations of  $\text{p}4\text{C}_4\text{H}_9\text{OCSS} = 5.1193$  and  $\text{p}(\text{C}_4\text{H}_9\text{O})_2\text{PSS} = 5.2667$ .

The measurement series with an untreated electrode surface at pH 8 and 10 indicate a gradual increase in the electrode potential with time in potassium butyl xanthate and no change in the electrode potential over time in sodium dibutyl dithiophosphate solutions. This suggests that the sphalerite electrode interacts with xanthate-ions but does not interact with dithiophosphate ions.

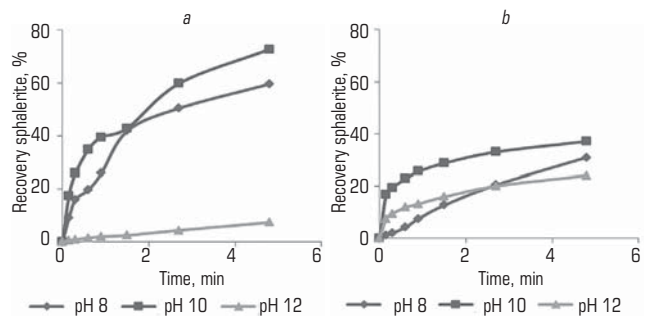
In the measurement series with the electrode treated by iron(II) cations at pH 8 and 10, a reduction in the negative potential values is observed. The most significant potential difference is observed when the sphalerite electrode is exposed to sodium dibutyl dithiophosphate. Interestingly, at pH 8, the electrode potential for potassium butyl xanthate is lower than that for sodium dibutyl dithiophosphate, even though their concentrations were the same. This behavior, as shown in the graphs, supports the existence of an interaction between the sphalerite electrode modified by iron(II) cations and both thiol collectors, and it aligns with the thermodynamic calculations.

**Conclusions**

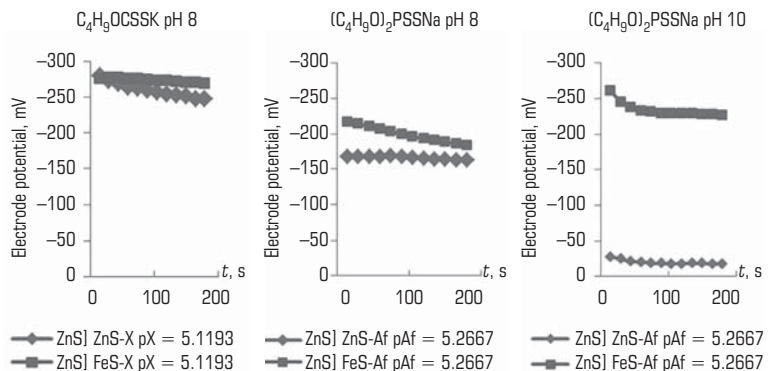
The thermodynamic calculations of surface interaction of sphalerite before and after treatment by iron(II) ions with butyl xanthate and dibutyl dithiophosphate of alkaline metals, as well as the potentiometric studies have shown that:



**Fig. 3. Kinetics of sphalerite flotation with potassium butyl xanthate without iron sulfate (a) and with the addition of iron sulfate (b)**



**Fig. 4. Flotation kinetics of sphalerite with sodium dibutyl dithiophosphate without iron sulfate (a) and with addition of iron sulfate (b)**



**Fig. 5. Sphalerite electrode potential versus time in solutions of potassium butyl xanthate and sodium dibutyl dithiophosphate at pH 8, 10**

- Treating the sphalerite surface with iron(II) cations and hydroxocomplexes at pH 8 leads to the formation of the collector adsorption layer. The specific composition of this layer depends on the collector used, resulting in the presence of either iron(II) butyl xanthate and xanthate  $(\text{ZnS}|\text{FeS}|\text{Fe}(\text{SSCOC}4\text{H}_9)_2|(\text{C}_4\text{H}_9\text{OCSS})_2)$  or iron(II) dibutyl dithiophosphate and sulfide  $(\text{ZnS}|\text{FeS}|\text{Fe}(\text{SSP}(\text{OC}_4\text{H}_9)_2)_2|((\text{C}_4\text{H}_9\text{O})_2\text{PSS})_2)$  on the sphalerite surface;

- According to the potentiometric studies of the sphalerite electrode in thiol collector solutions on weakly alkaline (pH 8), alkaline (pH 10) and strongly alkaline (pH 12) media, the electrode surface treated with iron sulfate exhibits more negative potential values than an untreated electrode in contact with the collector solutions only. This confirms the association between the presence of iron(II) sulfate on the sphalerite electrode surface and the interaction with xanthate or dithiophosphate ions.

- Iron(II)-containing compounds have a noticeable impact on the flotation kinetics of sphalerite with xanthate and dithiophosphate, particularly at pH 12.

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