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Analytical review of the theoretical prerequisites for developing flotation reagents based on sulfur-containing compounds and their compositions

Abstract: Innovative strategies for developing straightforward and efficient technologies are necessary due to the depletion of conditioned ore sources and the rising proportion of low-grade and technogenic raw materials utilized in production. Special emphasis is placed on flotation agents, as flotation is a primary method for processing low-grade, challenging thin scattered ores. An analytical evaluation of the evolutionary progression of theoretical foundations for formulating fundamental flotation agents and their combinations derived from sulfur-containing compounds utilized and suggested for application in the mining sector is offered. Collective flotation reagents, both traditional and contemporary, are crucial for effective processing, particularly in froth flotation and solid-liquid separation. The pursuit of novel and enhanced reagents is both technological and ecological, mirroring the dynamic difficulties faced by the business. This work aims to summarize and analyze the existing theory and practice of using collector mixes in sulfide flotation, encompassing both practical applications and research, to offer insights and recommendations for developing a predictive technique.

Keywords: sulfur-containing, flotation agent, composite collector, flotation.

Introduction

Reagents play a pivotal part in the flotation process. A defining aspect of the chemicals employed in the flotation process is their selective activity concerning various phase boundaries and, specifically, different mineral surfaces, contingent upon the conditions established in the aqueous medium of the pulp. This selectivity is invariably linked to a marked specificity of adsorption, adsorption-chemical, electrochemical action, or chemical reaction within the liquid phase of the pulp (Shubov, Ivannov & Shcheglova, 1990; Kenzhaliyev, 2019; Zhapbasbayev et al., 2021; Semushkina et al., 2023; Abikak et al., 2023; Argyn, Zoldasbay & Dosmukhamedov, 2024). These mechanisms alter the wetting conditions of individual mineral grains' surfaces, hence affecting their adherence to bubbles.

Flotation reagents can be classified based on their chemical composition into organic and inorganic groups, based on physical and chemical characteristics into soluble and insoluble in water, and based on their application across various industries such as chemical, petrochemical, oil refining, and metallurgy. Currently, the classification of reagents proposed by A.M. Gaudin (1957) is utilized and has been admitted. Gaudin classifies flotation reagents into the following categories:

- collectors - organic compounds that adhere to the surfaces of specific minerals, facilitating the formation of hydrophobic films;

- foaming agents - substances that concentrate at the water-air interface, aiding in the stabilization of air bubbles in a dispersed state;

- depressors - agents that diminish the flotability of minerals, inhibiting the attachment of collectors to mineral surfaces;

- activators - compounds that enhance the attachment of collectors to mineral surfaces, counteracting the effects of depressors;

- medium regulators - substances that influence the interaction of collectors by modulating the ionic composition of the pulp.

The primary agents for the flotation of non-ferrous minerals are collectors, which in the polar group possess a reduced sulfur atom (= S), in contrast to an oxidized sulfur atom, as seen in sulfuric acid derivatives.

Based on the atom to which the sulfur atom is attached, the entire category of collectors can be divided into two classifications: 1) xanthates, where the sulfur atom is bound to a carbon atom.

or 2) Aeroflot's, wherein the sulfur atom is connected to a phosphorus atom.

Xanthates were initially identified by Zeiss in 1822 and remained unutilized in beneficiation for nearly a century, serving solely in the production of fake liquor and the vulcanization of rubber. Keller introduced its application as a flotation reagent. The primary rationale for the collecting action of xanthates is the presence of xanthogenic acid ions, while alkali metal ions exert no influence on flotation. Butyl and ethyl xanthates are predominantly utilized in flotation practices.

Di-alkyl di-thiophosphates, or aeroflots, are thionic derivatives of di-alkyl phosphoric acid esters in which oxygen atoms are substituted with sulfur. Aeroflot is produced through the interaction of pentasulfuric phosphorus with alcohols at temperatures ranging from 50 to 80˚C. Potash, soda, or ammonium carbonate is included in the resultant di-thio-phosphoric acid for neutralization.

The polar group of di-thiophosphates resembles that of xanthates, differing only in the inclusion of phosphorus in place of carbon.

In flotation, many chemicals are typically employed concurrently, with their effects being interdependent and contingent upon their respective concentrations.

One principle in the formulation of selective reagent systems is the utilization of combinations of strong and weak collectors. Three approaches exist for the implementation of this principle concerning sulfide minerals: the utilization of ionogenic sulfhydryl collectors with varying hydrocarbon radical lengths and distinct solidophilic groups; the application of ionogenic sulfhydryl compounds (such as dithiophosphates and xanthates) as potent collectors, alongside non-ionogenic compounds (including lowpolarity thiono-carbamates, xanthogenic acid esters, and disulfides) and polar compounds as weaker collectors.

Research conducted by the Institute of Mining Engineering of the USSR Academy of Sciences since 1952 has empirically demonstrated the potential for enhancing the flotation process through the simultaneous application of xanthates with varying hydrocarbon radical lengths, alongside the amalgamation of long-chain xanthates with diethyl dithiophosphates. Experimental evidence demonstrates the establishment of a denser adsorption layer when utilizing combinations of collectors (ethyl xanthate, amyl xanthate, diethyl dithiophosphate) on arsenopyrite and galena. The optimal flotation outcomes are attained with the combination of amyl xanthate and di-ethyl di-thiophosphate at a mass ratio of 1:1, and with the compound of ethyl xanthate and amyl xanthate at a molecular ratio of 2:1. The concurrent use of multiple

collectors is proposed to enhance the formation rate of the adsorption layer due to the mineral surface's heterogeneity and the presence of sorption centers with varying affinities for different collectors, resulting in improved flotation kinetics (Ignatkina, 2009).

The findings of laboratory investigations on established sulfhydryl collectors and novel modified dithiophosphates from the Beraflot series for the flotation of monomineral fractions of pyrite, chalcopyrite, galena, and sphalerite are reported.

The enhancement of selectivity in flotation reagent modes for sulfide ores containing copper, zinc, lead, iron, and gold minerals relies on the utilization of a combination of weak and strong collectors from the same class, such as the simultaneous application of xanthates with varying hydrocarbon chain lengths and the integration of xanthates with di-thiophosphates. Combinations of weak and powerful collectors of various types—ionogenic and non-ionogenic—such as xanthates and thiono-carbamates; di-thiophosphates and thiono-carbamates; esters of xanthogenic acids; xanthates and apolar oils, among others.

The mechanics of thiol collector adsorption on sulfide minerals have been extensively studied over the years. Consequent to these investigations, several theories have emerged, specifically:

- the chemical theory proposed by (Taggart et al. 1934);
- the ion exchange theory articulated by Sutherland, Wark (1955), and Gaudin (1957);
- the neutral molecule theory posited by Cook and Nixon (1950);
- and an electrochemical model developed by several researchers (Рlaksin and Shafeev,1963; Tolun and Kitchener,1964; Majima and Takeda, 1968; Allison et al.,1972; Woods,1976; Richardson et al., 1984; and Chander, 1988; Fuerstenau, Natalie & Rowe, 1990).

The chemical theory posits that "all dissolved reagents operate through established chemical reactions between the reagent and the affected particle." This theory presupposes that the mineral or its oxidation product present at the surface is more soluble than the product resulting from the interaction between the mineral surface and the collector ions. Wark (1955) formulated the ion exchange theory based on the premise that sulfides are typically floated with coverage below a monolayer. Sutherland and Wark (1955) established that merely one per cent coverage is necessary for floating. The authors assert, "When the collector exists as a monolayer or less, no direct correlation with the solubility product is necessary." The ion exchange theory posits that xanthates adsorb onto sulfide surfaces through exchange with adsorbed ions, including carbonate, hydroxyl, and sulfur oxide.

The neutral molecule idea proposed by Cook and Nixon (1950) is founded on the hydrolysis of xanthate to xanthic acid in solution. This idea posits that xanthic acid is essential for the formation and release of water through the interaction of hydrogen ions from xanthic acid with adsorbed hydroxyl ions.

In the context of the electrochemical model, when a sulfide mineral is immersed in an aqueous solution, it generates a potential known as the resting potential. When the resting potential exceeds the reversible potential for xanthate oxidation, xanthate undergoes electrochemical oxidation to di-xanthogen on the mineral surface, while oxygen is released as hydroxyl. Pyrite, arsenopyrite, and pyrrhotite are classified within this category when ethyl xanthate is present (McFadzean, Castelyn & O'Connor, 2012).

A micro flotation investigation was conducted utilizing various ratios of ethyl and isobutyl chain lengths of xanthates, dithiocarbonates, and dithiophosphates as collectors, by these stipulations. Flotation recoveries and galena rate constants were analyzed about particle size to see whether the combinations of collectors yielded any advantageous effects relative to the employment of individual collectors. Combinations of ethyl xanthate and ethyl thiocarbamate showed a considerable enhancement in the rate and recovery of galena flotation relative to the use of individual collectors. The recovery rates diminish when employing a combination of ethyl xanthate and ethyl di thiophosphate. Nonetheless, isobutyl xanthate, when utilized as a sole collector, outperformed mixes in every instance.

Russian researchers examined the impact of collector mixes on the flotation process. Microradiography was employed to ascertain the galena surface coverage of xanthate mixes with varying alkyl chain lengths(Ignatkina, 2010; Bacharov et.al., 2005) . It was established that enhanced galena recovery resulted from a denser and more uniform surface coverage achieved through the application of a mixture of ethyl and butyl xanthate.

Adkins and Pearse (1992) saw enhancements in copper recovery rates from a mixed sulfide/oxide copper ore utilizing xanthate–di-thiophosphate combinations. Bradshaw and O'Connor (1994) discovered that combinations of cyclohexyl di-thiocarbamate with an additional di-thiocarbamate enhanced recovery,

grade, flotation rate, and coarse particle recovery, and decreased the necessary concentration for the batch flotation of pyrite ore.

Consequently, substantial experimental evidence indicates enhanced flotation performance when employing collector mixtures. Nevertheless, a comprehensive fundamental comprehension of the mechanics behind this phenomenon has not yet been attained.

Several explanations have been proposed to elucidate the improved flotation observed with mixes. Plaskin and Zaitseva (1960) ascribed improved performance to the concept of "collector-specific" locations on the mineral surface. Various sorption centers will exhibit distinct actions about the different collectors. They hypothesized that this led to increased adsorption kinetics, which consequently resulted in an enhanced flotation rate.

Hanson et al. (1985) posited that, in a binary xanthate-glycine system, glycine facilitated enhanced adsorption of xanthate by the creation of metal glycinate complexes, which subsequently react with the xanthate and ultimately adsorb onto the mineral surface. It is recognized that xanthate adsorbs in several layers on a mineral surface and requires minimal assistance from a co-collector. Bradshaw and co-authors have proposed a method that suggests either selective adsorption of collectors onto certain spots or the precise orientation of molecules, leading to enhanced surface coverage. Subsequently, the elevated reaction heat observed with mixes indicated that the presence of di-thiocarbamate enhanced the oxidation of xanthate to di-xanthate or that a more robust link was established when utilizing combinations of collectors. The di-thiocarbamate was also suggested to function as an anchor for the secondary adsorption of dixanthogen molecules.

The hypothesis of strong and weak sites, akin to that proposed by Plaskin and Zaitseva (1960), was introduced for a xanthate-di-thiophosphate mixture, wherein the stronger collector occupies the weaker, more oxidized sites, while the weaker collector occupies the stronger, less oxidized sites. This would yield a denser and more uniform surface coverage. Alternative recommendations have been suggested concerning the mechanisms involved in the use of collector mixes. This encompasses alterations in froth behavior, heightened collector adsorption, and enhanced particle hydrophobicity.

The advantageous effects of collector mixes diminish with increasing dosages and the predominance of a more potent collector.

Consequently, the evidence indicates that the theory about the interaction of flotation agents with minerals is continually evolving, thereby establishing the foundation for the formulation of novel, more effective flotation reagent compositions.

A series of studies examined the influence of the ratio of sulfhydryl ionogenic collectors, specifically butyl xanthate or isobutyl di-thiophosphate, in conjunction with thiocarbamates; the ratio of butyl xanthate combined with the sulfhydryl collector M-TP (a mixture of thionocarbamate and di-thiophosphate) on the flotation rate of non-ferrous minerals. It was determined that augmenting the amount of butyl xanthate in any mixture enhances the flotation rate of all monominerals (Ignatkina et al., 2015; Ignatkina, 2016). The application of reagent M-TF in conjunction with butyl xanthate on a copper-coal ore sample resulted in the selective separation of copper sulfides in the initial fractions, surpassing the efficacy of the usual reagent protocol utilizing butyl xanthate. The optimal flotation rate constant is achieved with a 4:1 and 3:1 ratio of M-TF to butyl xanthate, contingent upon the ore's material composition.

It is noteworthy that several enhanced modifications of Aeroflot, produced at the Institute "Mekhanobr" and CJSC "Mekhanobr-Orgsintez-Reagent," are currently being utilized effectively in industry.

The reagent IMA-1012A, an aqueous solution of sodium or ammonium salts of di-alkyl-di-thiophosphoric acids derived from fatty alcohols with a broad range of C9 - C12, is utilized at ALROSA's beneficiation facilities. This reagent enhances diamond recovery in the foam separation process, using 10 to 15 times less than butyl Aeroflot.

A variation of Aeroflot is endorsed and has been effectively utilized for several years at "Primorsky GOK." This reagent significantly enhances the selectivity of flotation for copper ores containing arsenic, yielding a copper concentrate with arsenic level below 1.0%. The introduction of Aeroflot decreased collector consumption by 4-5 times, with Aeroflot usage at 30-40 g/t compared to 150 g/t of butyl xanthate.

Currently, in light of the prevailing conditions in the non-ferrous metals market, research is being conducted on the broader application of reagents from the class of di-alkyl di-thiophosphates at Russian firms. A prototype batch of FRIM-9 reagent was manufactured and sent for industrial evaluation at the Erdenet facility.

Aeroflot, as an auxiliary collector to butyl xanthate, enhances the recovery of fine sulfide minerals and those containing precious metals (gold, silver), while also improving the selectivity in the separation of non-ferrous metal sulfides and pyrite.

Thus far, such tests have been conducted at "Urupsky GOK" CJSC, "Salairsky GOK" OJSC, and the facilities of "Kazakhmys Corporation." The utilization of a combination of xanthate with butyl Aeroflot (BTF) or xanthate with Aeroflot FRIM-9 enhances the efficiency of copper and zinc beneficiation by 3.0 - 4.0%, attributed to an increase in metal content within the concentrate (Ryaboy, 2011; Zhang, Nguyen & Zhou, 2018; Kenzhaliyev et al., 2021).

A substantial amount of laboratory research was conducted on ores processed at Kazakhmys Corporation facilities. Research indicates that copper recovery in rough concentrate can increase by up to 5.0% when employing a combination of xanthates and Aeroflot FRIM-9, and in certain instances, Aeroflot BTF.

Conclusions

Consequently, the aforementioned succinct review indicates that the theoretical foundations for the development of novel flotation reagents, particularly those comprising sulfur-containing compounds, are continually advancing. However, the challenge of a scientifically grounded selection of initial collectors and their formulations in establishing selective reagent protocols for the flotation of refractory ores persists, as their application is directly influenced by the qualitative and quantitative characteristics of the feedstock and its structure, necessitating a tailored approach in each instance.

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