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OPTIMIZED MULTICOMPONENT SURFACTANT COLLECTOR ENHANCES COPPER RECOVERY FROM COAL-PROCESSING WASTE

S.U. Xalilov[1], A.A. Kucharov[1,2,✉], F.M. Yusupov[1], N. Yodgorov[1], S.B. Sultonov[1], R.A. Toshboboyeva[1], A.R. Qurbonov[1] and S.K. Yusupov[1]

[1]The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan, Tashkent, 100071, Uzbekistan

[2]Pharmaceutical Education and Research Institute, Tashkent, 100114, Uzbekistan

✉Corresponding author: sciuzb@mail.ru

ABSTRACT

The research is centered on copper extraction from technical wastes formed during coal enrichment with the use of multicomponent surfactant flotation agents. This work deals with the colloid-chemical nature of the reagent-mineral-surface interaction and flotation. The multicomponent surfactant systems included sulfonate, carboxylate, amide, and phenol functional groups that could ensure selectivity of interaction with copper-bearing minerals. The presence of these active centers in the formulation was proved by spectroscopic studies. The results of flotation tests have shown a significant dependence of copper recovery on the concentration of the agent used. There was a certain optimum range where higher doses resulted in lower process efficiency. Among the studied multicomponent systems, one reagent showed excellent performance in flotation tests, confirming the necessity of proper functional group balance in the multicomponent system for selective extraction of copper-containing particles. In addition, the effect of pulp temperature and duration of flotation were found to have a strong effect on the process efficiency. Scientifically, these findings link the functional-group balance of multicomponent surfactant collectors with their flotation response, providing a colloid-chemical basis for designing more selective reagent systems. From a practical perspective, the optimized collector offers a promising approach for copper recovery from coal-processing waste while promoting waste valorization, secondary-resource utilization, and United Nations Sustainable Development Goal 12: Responsible Consumption and Production.

Keywords: Coal Processing Wastes, Copper Froth Flotation, Surface Active Agents, Composite Collectors, Colloidal Chemistry, Selective Adsorption, And Particle-Bubble Association

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INTRODUCTION

Coal processing and mineral beneficiation produce huge amounts of technically valuable wastes, which nowadays are regarded not only as an ecological load, but as a secondary source of valuable components as well.[1] As mentioned above, the generation of 1 ton of hard coal can result in the formation of around 0.4 tons of mining waste, and about 30% of mined materials are rejected as waste at coal preparation plants.[2-4] Annually, in the USA, only the production of coal impoundment waste equals 70-90 million tons, and in China 15% of total run-of-mine coal becomes waste during coal beneficiation. Such waste contains carbonaceous material, clay minerals, silicates, carbonates, pyritic materials and remnants of non-ferrous metals.[5-8] At the same time, there is a rise in the worldwide demand for copper caused by its usage for electricity grids, renewable energy systems, and power grid modernisation.[9,10] The forecast of the International Energy Agency claims that copper demand will grow up to 30% until 2040 in comparison with current values, so copper recovery from coal processing waste seems to be quite topical.[11-13] Nonetheless, small particle size, sliminess, heterogeneity of minerals, and the presence of only trace amounts of copper-containing minerals present difficulties when attempting to separate them via the traditional approach.[14-16] From the standpoint of colloid chemistry and physics of the surfaces, froth flotation is characterised by the presence of processes such as adsorption, changes in the nature of the electrical charge on the surface, regulation of hydrophobicity, dispersion of particles, collisions of bubbles with particles, and foam stability.[17,18] In particular, fine and ultrafine fractions smaller than 20

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microns create significant challenges for flotation due to the low weight and large specific surface energy of these particles and low efficiency of their collisions with bubbles.[19,20] In the current investigation, flotation reagents with LABSA, TEA alkyl sulfate, sodium oleate, GEPAN, and gossypol were explored for the extraction of copper from technical waste formed during coal enrichment processes.[17,21-24] In particular, the research is aimed at optimising the concentration of the collector, pulp temperature, and flotation time, along with providing an explanation of the separation process based on FTIR, AAS, and colloid chemistry.[25,26] Recent mineral-beneficiation studies have demonstrated that the chemical nature of the collector and the selected flotation scheme strongly influence mineral-separation selectivity and process efficiency.[27] The latest research shows that the selective extraction of valuable elements from coal processing waste utilising modern surfactant technology could enhance resource utilisation and help achieve United Nations Sustainable Development Goal-12: Responsible Consumption and Production. Hence, the current study attempts to resolve the lack of knowledge regarding the effect of functional group structure and operational parameters on multicomponent surfactant collectors' selective copper extraction from coal processing waste through the comparison of four surfactant collectors and optimisation of their concentration, pulp temperature, and flotation time.

EXPERIMENTAL

Materials

GMZ-2 coal-processing waste containing copper-bearing mineral phases was used as the feed material. Ore was crushed by wet ball milling to achieve a particle size of d80 ≈ 74 μm. Prior to flotation, desliming treatment was carried out to eliminate the ultrafine fraction < −20 μm, which made up around 5- 10% of the feed mass. Flotation agents were composed of LABSA, TEA alkyl sulfate, sodium oleate, GEPAN and gossypol as active ingredients. Four different compositions of flotation reagents, namely N-1, N-2, N-3 and N-4, were studied.

General Procedure

The experiments were conducted using a mechanical laboratory flotation cell without any pH-modifying agents at natural pulp pH. The GMZ-2 ore sample was crushed and mixed with water for the flotation pulp preparation, after which the pulp was conditioned with the chosen composite reagent. The amount of the collector was varied from 0 to 250 g/t to achieve the optimal reagent dosage. The effect of pulp temperature and flotation time on the flotation process was evaluated within the ranges of 10-40 °C and 0-16 minutes, respectively.

Detection Method

For the determination of functional groups within gossypol and for the composite reagents from N-1 to N-4, Fourier transform infrared spectroscopy (FTIR) was utilised. The range of FTIR spectra obtained was from 4000 to 500 cm^{-1}. Absorption ranges for –OH, COO^-, SO_3^-, C=O/C–N and aliphatic C–H functional groups were evaluated for reagents' chemical characteristics. The concentration of copper in aqueous solutions was measured using atomic absorption spectroscopy (AAS).

Analytical Discussion

Flotation recovery of copper was considered the key parameter when evaluating the flotation process performance. An investigation was made on the effect of collector dosage, pulp temperature, and flotation time to establish the optimal parameters for the flotation process. The highest performance in terms of flotation recovery was obtained at a collector dosage of 150–180 g/t, with the peak value being achieved at around 175 g/t. The optimal pulp temperature and flotation time were estimated to be 30 °C and 12–14 min.

RESULTS AND DISCUSSION

The floatability of multi-component surface-active agents was investigated for copper flotation from technical waste obtained from the beneficiation of coal. The findings are analysed from the point of view of colloid chemistry, taking into account functional groups determined by FTIR, recovery dependent on the concentration of the collector, and evidence provided by AAS analysis under optimized flotation conditions.

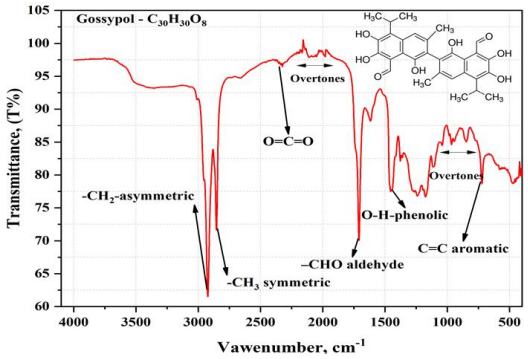

Fig.-1: FTIR Spectrum of Gossypol-Based Surfactant

Fourier transform infrared (FTIR) spectrum of gossypol ($C_{30}H_{30}O_8$) shows various types of functional groups involved in the mechanism of this compound as a composite flotation reagent. The wide peak ranging from 3400 to 3200 cm^{-1} represents phenolic O–H stretching vibrations, which show the presence of hydrogen bonding sites (Fig.-1). The presence of aliphatic groups is evident from the high absorption intensities of vibrations in the range of 2920 and 2850 cm^{-1}, whereas aldehyde and carbonyl vibrations have been assigned values between 1720 and 1650 cm^{-1}. Aromatic C=C skeleton modes fall within the region of 1600 to 1500 cm^{-1}, and vibrations of phenolic C–O stretching and O-H bending occur in the range of 1300 to 1000 cm^{-1}. From this data, it can be concluded that gossypol exhibits functional groups of polar coordinating nature along with non-polar aromatic/aliphatic groups.

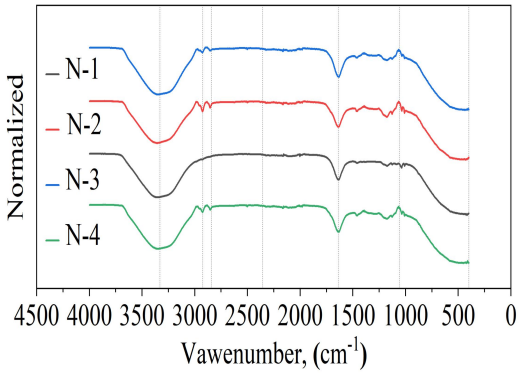

Fig.-2: Comparative FTIR Spectra of Composite Flotation Reagents N-1, N-2, N-3 and N-4

Comparative FTIR spectra of the flotation reagent complexes N-1 to N-4 demonstrate their simultaneous presence in the mixture (Fig.-2). The absorption maxima within the 3300-3400 cm^{-1} range represent phenolic hydroxyl groups –OH of gossypol origin. The peaks appearing within 1650-1680 cm^{-1} represent amide C=O/C-N vibrations corresponding to the GEPAN. The absorptions within 1550-1450 cm^{-1} characterise asymmetric vibrations of COO^- groups of oleate derivatives, while the maximum within 1180-1040 cm^{-1} is typical for sulfonate vibrations produced from LABSA. The preservation of the specific spectral features of each component proves their existence in the compound form. Moreover, the lack of new absorption bands and substantial changes in the absorption frequencies suggests that these reagents constitute a physically bound complex rather than the formation of a new substance. Such a structure might enhance the efficiency of the flotation process due to the simultaneous presence of sulfonate, carboxylate, amide and phenolic groups, providing good properties for reagent dispersion and modification of hydrophilic surfaces.

Copper recovery via flotation was carried out at collector concentrations varying between 0 and 250 g/t by means of N-1 to N-4 composite collectors. The study revealed that copper recovery increased gradually up to 150-175 g/t due to enhanced surface hydrophobicity of copper-bearing mineral particles as well as increased particle-bubble association efficiency. In comparison, the best performance was exhibited by N-3 collector, which attained a maximum recovery value of approximately 87-88% at 175 g/t, while N-1, N-4 and N-2 achieved approximately 83-85%, 78-80%, and 74-76%, respectively.

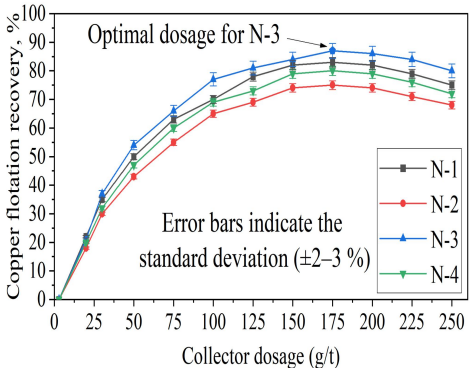

Fig.-3: Effect of Collector Dosage on Flotation Recovery of N-1, N-2, N-3 and N-4 Samples

However, past an optimal dosage of 175 g/t, especially in the range of 200 to 250 g/t, copper recovery was gradually reduced. Such trends could be due to excessive adsorption of the collectors, formation of multi-layers on the surface of mineral particles, non-selective activation of gangue mineral particles, and foam instability. Formation of multiple layers on the mineral particle surface might decrease the selective particle-bubble association of copper-bearing minerals and consequently result in entraining of gangue minerals into the froth product. Accordingly, Fig.-3 displays a unimodal dose–response profile with a distinct maximum at 175 g/t. The atomic absorption spectrometric results obtained for the control and optimized test systems are summarized in Table-1. The results extend previous investigations past investigations into surfactant-based coal washing processes, proving not only that the recovery of copper depends on the dose of the collector but also that it is influenced by the functional equilibrium of the multicomponent mixture used. Of all four mixtures investigated, N-3 proved to have the best response in the flotation process, which means that there is an optimal equilibrium between polar sulfonate, carboxylate, amide and phenolic functions and non-polar parts of the structure of this mixture. The decrease in recovery when the dose exceeds 175 g/t also proves that the more reagent is used, the worse the separation is achieved, possibly due to increased surface coverage and froth stability impairment.

Table-1: Atomic Absorption Spectrometric Determination of Copper in the Aqueous Phases of Control and Optimized Test Systems

Test system	Composition	Cu concentration (ppm)	RSD (%)
Blank	Deionized water	<LOD	–
Waste control	Water + GMZ-2 coal-processing waste	<LOD	–
Collector control	Water + N-3 collector at the test concentration	<LOD	–
Optimized system	Water + GMZ-2 waste + N-3 collector at 175 g/t	0.11–0.12	1–4

Based on FTIR, AAS, and flotation studies, it can be stated that the multicomponent surfactant enhances the copper recovery via selective adsorption, hydrophobic alteration of surfaces, and improved particle-to-bubble adhesion. However, this work only involves laboratory-scale studies and indirect verification of the reagent-mineral interactions; thus, further investigation is recommended to conduct XPS, zeta-potential, contact-angle, adsorption, flotation, and selectivity studies at pilot scale.

CONCLUSION

This study demonstrated the potential of multicomponent surfactant collectors for copper recovery from GMZ-2 coal-processing waste. The present study revealed that the multicomponent surfactants have significant potential to be utilised as copper collectors in GMZ-2 coal-processing waste. According to Fourier transform infrared spectroscopy results, sulfonate, carboxylate, amide, and phenolic groups are present in the tested collectors and can play the role of dispersing agents. From the total number of tested collectors, N-3 provided the highest copper recovery rate of 87–88%, while the optimal dosages of the collector, pulp temperature, and flotation time were 175 g/t, 30 °C, and 12–14 min, respectively. The observed decrease in the recovery rate at higher dosages (> 175 g/t) underlines the significance of dosage regulation. This might be related to the excess surface coverage and froth destabilisation. Atomic absorption spectroscopy revealed a measurable content of copper in a range of 0.11–0.12 ppm only in the

optimised system of waste and collector, while the rest of the control systems did not have sufficient concentrations to detect copper. Further research is needed to confirm the interaction mechanism by conducting zeta-potential, contact angle, adsorption, and X-ray photoelectron spectroscopy experiments. Complete copper mass balance and flotation tests on a pilot scale will also be required.

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CONFLICT OF INTERESTS

The authors declare that they have no known competing financial or personal interests that could have appeared to influence the work reported in this paper.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-12: Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

S.U. Xalilov https://orcid.org/0009-0007-9458-3268
A.A. Kucharov https://orcid.org/0009-0005-5619-8700
F.M. Yusupov https://orcid.org/0000-0002-6758-3445
N. Yodgorov https://orcid.org/0009-0000-9810-4067
S.B. Sultonov https://orcid.org/0000-0003-1996-1744
R.A. Toshboboyeva https://orcid.org/0009-0004-9042-3206
A.R. Qurbonov https://orcid.org/0009-0005-1896-8081
S.K. Yusupov https://orcid.org/0009-0005-3758-5799

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