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Organosolv lignin particles as an ecological reagent in the Kupfershiefer copper ore flotation

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Abstract: Mineral separation relies heavily on the process of flotation. This study explored the feasibility of using organosolv lignin nanoparticles and microparticles (OLP) as a greener alternative to xanthates in the flotation process for mineral separation. Xanthates are widely used but pose environmental and health risks. The efficiency of OLP as collectors was compared to collectorless flotation, resulting in approximately 50% copper recovery, indicating that OLP may not be a suitable replacement for xanthates. Further tests were conducted using a mixture of xanthates and OLP (birch nano and spruce micro) with varying substitution levels (20%, 30%, and 40%). The results demonstrated that increasing the dosage of OLP led to a decrease in flotation efficiency for copper. TOC analysis of the products revealed that high dosages (160 g/t) of birch nano and spruce micro as sole collectors showed beneficiation and selective recovery against copper. While OLPs did not prove effective as collectors, the study highlights their potential as substitutes for maltodextrin in selective flotation of the final concentrate. Two out of four tested OLPs were recommended for pilot scale testing.

Keywords: collector, particles, total organic carbon, organosolv, lignin

1. Introduction

The main process utilized in mineral beneficiation in the copper mining industry, is well known flotation. Advanced process exploits the differences in the electrochemical properties of mineral surfaces, between hydrophobic and hydrophilic surfaces (Ndoro and Witik, 2017). Particles of interest are separated from the pulp phase through the air bubbles, that selectively adheres to the surface of hydrophobic particles. Air bubbles attach and carry the hydrophobic particles, therefore forming a froth that is removed as a concentrate by skimming, while hydrophilic materials stay in the pulp phase, classified as tailings (Nguyen and Schulze, 2004).

Reagents play a crucial role in the flotation process, allowing to obtain products of the different desired compositions. One can distinguish reagents into collectors, frothers, depressants, activators, and regulators. Each type is used based on the process assumption and the processed ore characteristic. One of the frequently used collectors in the nonferrous minerals industry are xanthates, which are highly efficient (cost production, process efficiency) (Elizondo-Alvarez et al., 2021). Xanthates hydrolyze in aqueous solutions and flotation pulps to form toxic substances such as carbon disulfide (CS_2), which on account of its ability to accumulate in flotation plants, manage to pose a serious threat to worker safety and the environment (Shen et al., 2016). Reducing the use of xanthates in the process with suitable biodegradable and environmentally neutral reagents, could reduce the impact of the currently observed undesirable effects. A novel reagent was developed as a sustainable solution by using a biodegradable and environmentally friendly organosolv lignin. Lignin is one the most abundant biopolymer, thus renewable and sustainable resource that can present alternative to fossil resources. The global production of lignin is expected to reach over $913.1 million by the year 2025 (Bujwa et al., 2019).

The Concentrators exploited by KGHM Polska Miedź Inc. as the one of the major reagents' consumers. The deposit exploited by KGHM in the for-Sudetic monocline in the Legnicko-Głogowski

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Copper District (LGOM) is one of the largest copper deposits in the world, containing many accompanying elements in addition to copper and silver (Bartlett, et al., 2013). The deposit has a sedimentary genesis, which causes it complex lithological structure made of three main rock types: carbonates (dolomites, limestone), sandstone, and shale. Those types are divided into separate subtypes, such as dolomitic shale, and bituminous black shale. The polylithic character of the deposit and the lack of possibility of selective extraction of the consequent rock types cause problems in the enrichment process, due to the different chemical compositions of these lithological types. The concentrates produced in the process introduced in KGHM Polska Miedź S.A. are polymetallic containing mostly copper, and silver, but also gold, nickel, zinc, lead, and other elements which are recovered from the concentrate in the pyro-hydrometallurgical processes performed in the smelters. Those concentrates also contain impurities that are naturally occurring in the ore, of which the total organic carbon (TOC) is the most problematic for smelting, especially in the flash furnace process (Kijewski et al., 2010).

The shale is the most important source of valuable elements in the deposit. However, it also contains the highest amount of TOC where it varies between 0.2 and 8.0%, depending on the lithological series (Kijewski et al., 2010). The TOC content in the concentrates increases annually although ore copper grade decreases. This is caused by better floatability of the TOC in currently mined parts of the deposit (Kurzydło et al., 2021). Organic carbon is a naturally hydrophobic gangue that can unwantedly report to the copper concentrate, which is considered highly problematic and, its presence in the ore significantly deteriorates the efficiency of the flotation process (Skorupska et al., 2011). There were laboratories, pilot scale, and industrial scale trails utilizing the collectorless flotation process prior to the main flotation stages (pre-flotation), to remove the TOC before the next stages of beneficiation (Konieczny et al., 2015; Konieczny et al., 2013). When the laboratory tests have shown that there is a possibility to reduce the TOC in the semi-products and the ratio of TOC to copper was 2:1, the industrial trials, however, showed that utilizing the same process conditions, the ratio was reduced to 1:1 (Konieczny et al., 2015). For that reason, the pre-flotation process was not introduced in the industrial KGHM enrichment plants to date.

The low sugar maltodextrin is the reagent currently used in KGHM to produce selective final concentrate of differ TOC content (Kurzydło et al., 2021). The reduction of the TOC content in the process was tested and is currently performed at the final stage of beneficiation process in Rudna's concentrator for the concentrates with high and low TOC content (Skorupska et al., 2011). For that purpose, high amount of the maltrodextrin needs to be used at around 1-2 kg/Mg (Kijewski et al., 2010). The dextrins are known from their inhibitory properties of naturally hydrophobic materials (Foszcz and Drzymała, 2011; Miller et al., 1984; Nyamekye and Laskowski, 1991).

Lignin is a complex heterogenous polyphenolic compound that can be extracted from lignocellulosic biomass and has a great potential in numerous industrial applications. Organosolv pretreatment is one of the fractionation methods available for the lignocellulosic biomass and it yield high-quality lignin that is chemically unaltered and does not contain any sulphur (Soongprasit et al., 2020). In recent years the study of production and application of lignin nanoparticles increased significantly. The use of organosolv lignin micro- and nanoparticles as a flotation collector in mineral flotation was presented (Hruzova et al., 2010).

The aim of this study is to test the feasibility of using organosolv lignin nano- and microparticles as collector reagents in full relation to the fossil-based xanthates used continuously in the Polish copper mining industry. The products of the flotation with OLP as the collector were tested for TOC content to determine its potential substitution in the production of selective copper concentrates with low and high TOC content.

2. Materials and methods

2.1. Reagents currently utilized in KGHM Polska Miedź S.A.

KGHM Polska Miedź S.A. utilizes two types of xanthates: sodium ethyl xanthate (SEX) and sodium isobutyl xanthate (SIBX). Mixture of these two types of collectors were used in the laboratory tests. Xanthates are chemical compounds widely used in mineral processing as collectors. This type of collector is part of the ionizing – anionic – sulphydryl group of the reagents (Nguyen and Schulze, 2004).

In the industrial circuits of KGHM, different types of frothers are used depending on the concentrator. The industrial process which is implemented for the ore, which was obtained for the purpose of this work utilize the Nasfroth 245. The polyethylene glycol butyl ether is medium molecular weight frother, which distinguish itself with good selectivity, and is utilized when high recovery of the product is required [* Product Data Sheet. Nasfroth 245. Nadaco International Limited].

For the case of the work one of the reagents, which was not used during the test, but must be mentioned, is maltodextrin. The reagent is used in the industrial process for the regulation of the TOC particles (Skorupska et al., 2011; Miller et al., 1984).

2.2. Organosolv lignin particles (OLP)

OLP were provided by Luleå University of Technology, Sweden. Micro- and nanoparticles were extracted from lignin by organosolv pretreatment of birch and spruce wood chips. In particular, the organosolv pretreatment was performed at 183° C for 60 min in a 50 %, v/v (birch) or 60 %, v/v (spruce) ethanol-to-water solution. Farthest, with the use of vacuum filtration, solids were separated from liquor, which was collected for lignin isolation (Matsakas et al., 2020). To prepare lignin particles, a homogenization process was implemented, under previously described conditions (Kalogiannis et al., 2018). Precisely, the lignin was dissolved in a 75 % (v/v) ethanol-to-water solution. Then the lignin solution was homogenized with the use of APV-2000 (pressure homogenizer), at 750 bar. The particles were formed by solvent exchange method followed by freeze drying, yielding the lignin particles as a dry powder (Kalogiannis et al., 2018). This process resulted in 4 reagents: birch nanoparticles (BN), birch microparticles (BM), spruce nanoparticles (SN), and spruce microparticles (SM). The primary size of BN, BM, SN, and SM was determined the scanning electron microscopy and was 96.1±20.1 nm, 1.15±0.21 µm, 108.6±21.5 nm, and 1.00±0.21 µm, respectively. The hydrodynamic particles size determined by laser diffraction measurements showed significant aggregation in the SN and BN dispersion resulting in particles with D90 of 32.16 µm and 18.26 µm, respectively. The SM and BM showed D90 of 1.88 µm and 2.03 µm, which indicated that microparticles did not aggregate in the dispersion (Hruzowa et al, 2023).

2.3. Material – copper ore

Ore was provided by Legnicko-Głogowski Copper District of KGHM Polska Miedź Inc. The mined ore has a different lithological-mineralogical composition which is highly dependent on the mining operation region currently exploited. Selected type of ore has a variable lithological composition of 50-80% dolomite, 10-30% shale, and 6-30% sandstone. The copper content in the sample of approx. 1.7% was determined with iodometric titration method and TOC of approx. 0.7% was determined with thermal conductivity detector (TDC) in the external laboratory.

The main copper minerals in all types of ore are chalcocite and digenite, which generally occur together in concentrations up to 6% by weight (wt.%). Also abundant are bornite (up to 3.5 wt.%), chalcopyrite (up to 1 wt.%), covellite (also up to 1 wt.%), and locally, minerals from the tetrahedrite group. Accessory minerals include tenorite, azurite, cuprite, native copper, enargite, galena, sphalerite, smithsonite, loellingite, arsenopyrite, cobaltite, nickeline, native silver and stromeyerite (Nieć and Pietrzyński, 2007).

2.4. Methodology

Comparative flotation was carried out using standard flotation reagents such as sodium isobutyl xanthate and sodium ethyl xanthate, as a collector and Nasfroth 245, as a frother. The doses of the reagents corresponded to the standard doses being used in the mining industry in Poland.

Nasfroth 245 was used in each flotation trial. In each experiment, OLP was used as a complete replacement for the collector (xanthate) at different concentrations with a constant dose of frother. The OLP collectors such as birch lignin microparticles (BM), birch lignin nanoparticles (BN), spruce lignin microparticles (SM), and spruce lignin nanoparticles (SN) were used.

A 450 g representative sample of the ore was used as the feed for two separate laboratory flotation tests. A sample was ground in a 2 L ball mill with 375 ml of water for 32 minutes. After grinding, the slurry was mixed with 1600 ml of water. The diluted sample was divided into two filtrates with a density of about 1130g/dm3. The prepared filtrate was poured into a 1 L flotation cell.

The process protocol of flotation was summarized for comparative flotation, and OLP-based flotation (Table 1). Dosages of organosolv lignin particles used in collector tests were presented in Table 2. The dosage of 100% was equal to 160 g/Mg. The flotation process was carried out in a two-stage process. Starting with agitation, when first doses of froth and OLP collector were added, with no air outlet. During the flotation, five fractions were collected, and the elements were analyzed by a certified laboratory (CBJ Polkowice, Poland).

Table 1. Proces protocol for organosolv birch and spruce lignin micro- and nanoparticle flotation

	Comparative flotation (CF)		Lignin based flotation (OLP)	
Process step	Conditioning	Flotation	Conditioning	Flotation
Duration	0 min	6 min	0 min	6 min
Nasfroth (60g/Mg)	60%	40%	60%	40%
Xanthate (120 g/Mg)	60%	40%	-	-
Organosolv lignin (up to 160 g/Mg)	-	-	60%	40%

Table 2. Dosages of ONL particles in collector substitution tests

Dosage	
%	g/Mg
100	160
70	112
50	80

The ground and properly prepared feed was subjected to a flotation process in a laboratory mechanical flotation machine. The machine manufactured by Ganzhou Li Ang Machinery Co., Ltd, model 911MPE-D12-A flotation Cell consisted of a flotation chamber with a volume of 1dm^3 was used, operating at a rotor speed of 1150 rpm, a fixed parameter. The air flow rate was also a constant parameter, set to a maximum natural obtained flow of 1.0 dm^3/hour.

The flotation tests were conducted in such a way, in addition to the assumed differences, the process conditions and parameters were the same. The density of the flotation feed, which was 1130 g/dm^3, with an ore density of 2.7 g/cm^3, was controlled for each series of tests. Agitation and conditioning time without an air supply, was 5 minutes. After 1st minute, the collector was added, and after the 3rd minute, the frother was added, each with an appropriate amount. Scavenger began immediately after conditioning with the reagents, the air supply was set to maximum, while the constant level of suspension in the chamber was controlled by a peristaltic pump set to a flow rate of 15 ml/minute. The first concentrate (C1) was collected until the 2nd minute of flotation, and the second (C2) until the 6th minute. The next step was according to the scheme shown below (Fig. 1). The air and water supply were then stopped. During the one-minute break, successive doses of flotation reagents were added, and conditioning time were set. After conditioning, the collection of more concentrates continued. The total flotation time was 25 minutes, during which four concentrates and the final tailings were collected.

3. Results and discussion

For collectorless flotation, the total copper grade amounted to less than 7%, with a total recovery of 50%, whereas flotation with xanthate obtained almost 22% copper grade for 92% of total Cu recovery. The differences in the results are shown by the upgrading curve (Fig. 2), illustrating the enrichment process. A significant difference in the degrees of enrichment was shown. The curve depicting collectorless flotation presents low enrichment of the raw material.

In the first set of experiments BM and BN were compared. All experiments were carried out under similar conditions. The tested OLP were used as a collector in all cases. The approximate Cu recovery for used OLP remained below 51%. The second set of the experiment was carried out with the use of

SM and SN as a collector. The results presented the average selectivity towards Cu flotation, with total Cu recovery up to 53,59% for half (50%) dose of SM.

Figs. 3 and 4 show the copper enrichment curves for used OLP reagents. The most favourable results are presented for BN at the dosage of 50%. Total Cu recovery was obtained at a level of 47,19%, with a total Cu grade of 7,32%. BN obtained results were slightly worse, with total Cu recovery equal to 45,88%, with total Cu grade at 6,60%, were presented for 70% dose. Fig. 5 shows that result obtained with utilization of SN and SM are comparable to each other. For SN at a dosage equal to 70%, total copper recovery attained 48,31%, with total Cu grade at a level of 6,40%, while for SM at 100% dosage yielded a total grade of 6,10%, for total Cu recovery of 9,50%.

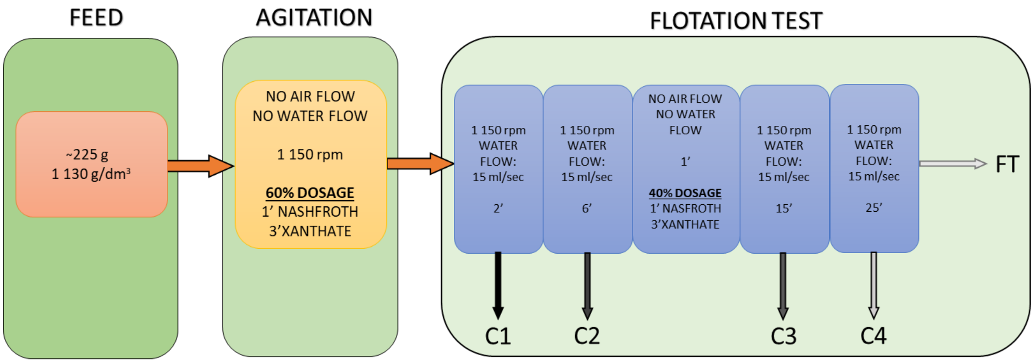


Fig. 1. Process diagram

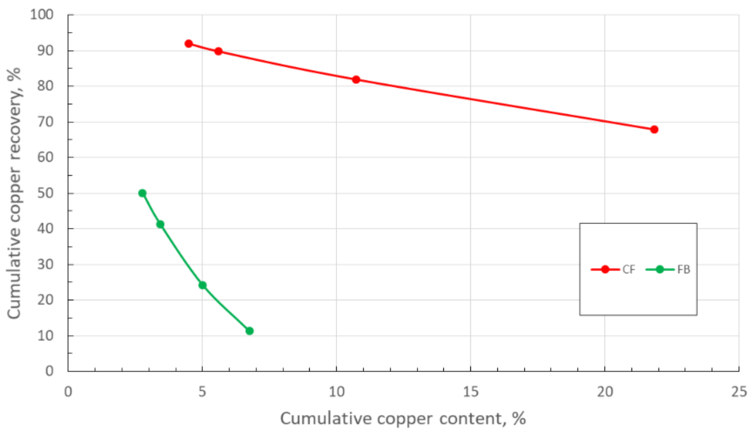


Fig. 2. Recovery-content curves for Cu: of comparative flotation (CF) and colllectorless flotation (FB)

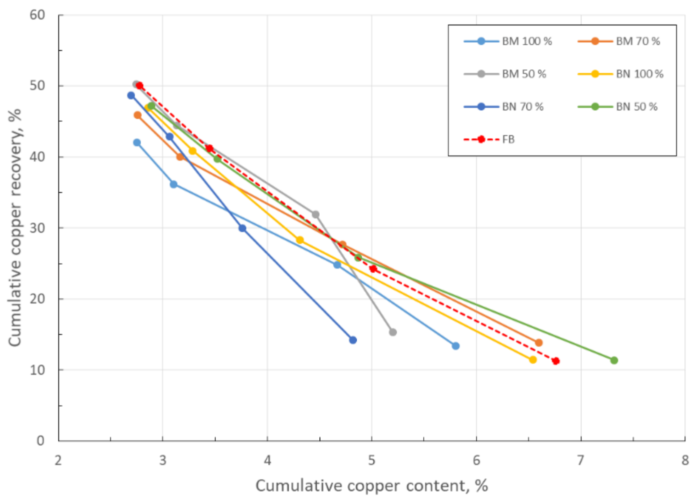


Fig. 3. Comparison of birch lignin microparticles (BM) and birch lignin nanoparticles (BN) curves

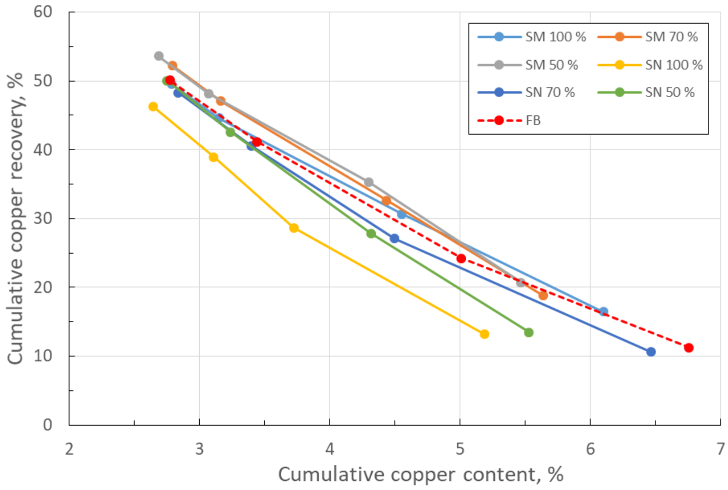


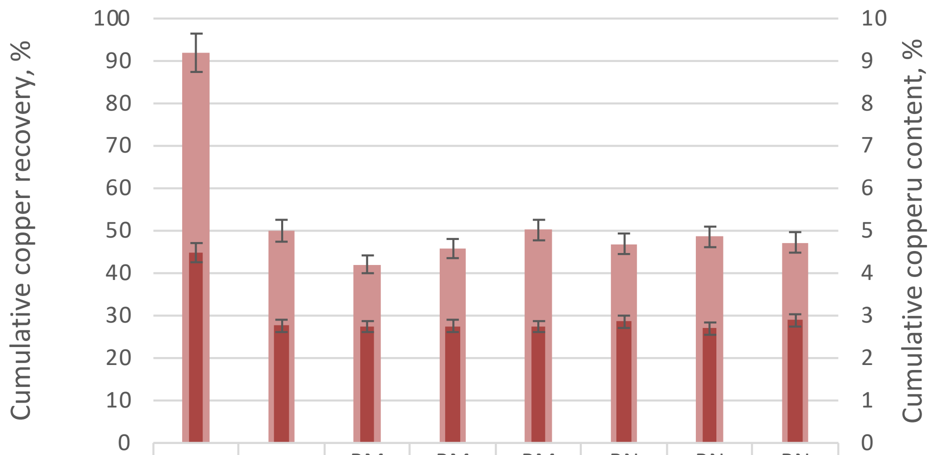
Fig. 4. Comparison of spruce lignin microparticles (SM) and spruce lignin nanoparticles (SN) curves

Comparing the results yielded in Figs. 5 and 6, undoubtedly significant deterioration in flotation performance is reported when OLP is used. Obtained results diverge, and the difference in presented results for comparative flotation, non-collector one, and with use of OLP reagents is notable. Few sets of experiments for BN and BM presented worse results than non-collector flotation. The same situation was presented for SN and SM. The degree of enrichment is unsatisfying and similar to collectorless flotation. The improvement of copper recovery is observed in the case of SM tests where the dosages of 70% and 50% increase recovery to 52.19% and 53.59% respectively (Fig. 6).

Analyzing the results (Fig. 6), with decreasing proportion of OLP reagents, increases the Cu content, approaching the value obtained in the comparison flotation. In addition, with the reduction of the proportion of BN, the Cu content increases, with SM continuing to deteriorate.

Based on the results obtained, the two reagents that show the best performance were selected. In the current series, BN and SM reagents were used to supplement the collector reagent dose. The OLP accounted for 40%-20% of the total collector dose. Xanthate was added as specified at 78g/Mg while frother was added at 60g/Mg.

Results presented in Fig. 7 shows that utilizing xanthate and OLP supplementation of the collector reduced copper recovery in the concentrate in comparison to the test when the mix of xanthates is used



	CF	FB	BM 100%	BM 70%	BM 50%	BN 100%	BN 70%	BN 50%
Copper recovery [%]	91.95	50.09	42.06	45.88	50.21	46.95	48.71	47.19
Copper content [%]	4.49	2.77	2.75	2.76	2.74	2.86	2.70	2.89

Fig. 5. Comparison of comparative flotation (CF), collectorless (FB), birch lignin microparticles (BM), birch lignin nanoparticles (BN)

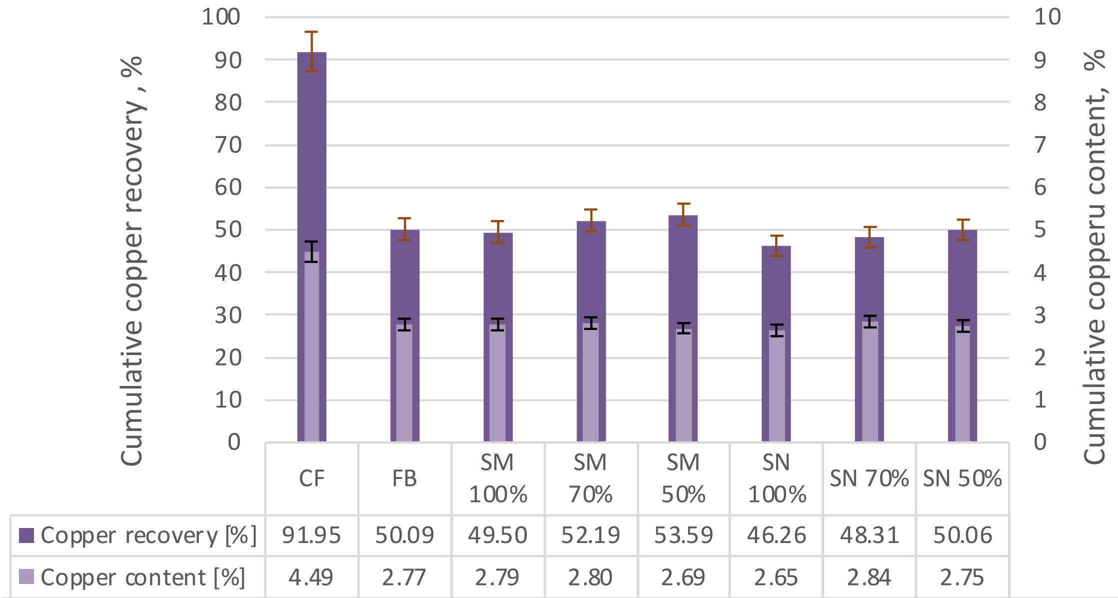


Fig. 6. Comparison of comparative flotation (CF), collectorless (FB), spruce lignin microparticles (SM), spruce lignin nanoparticles (SN)

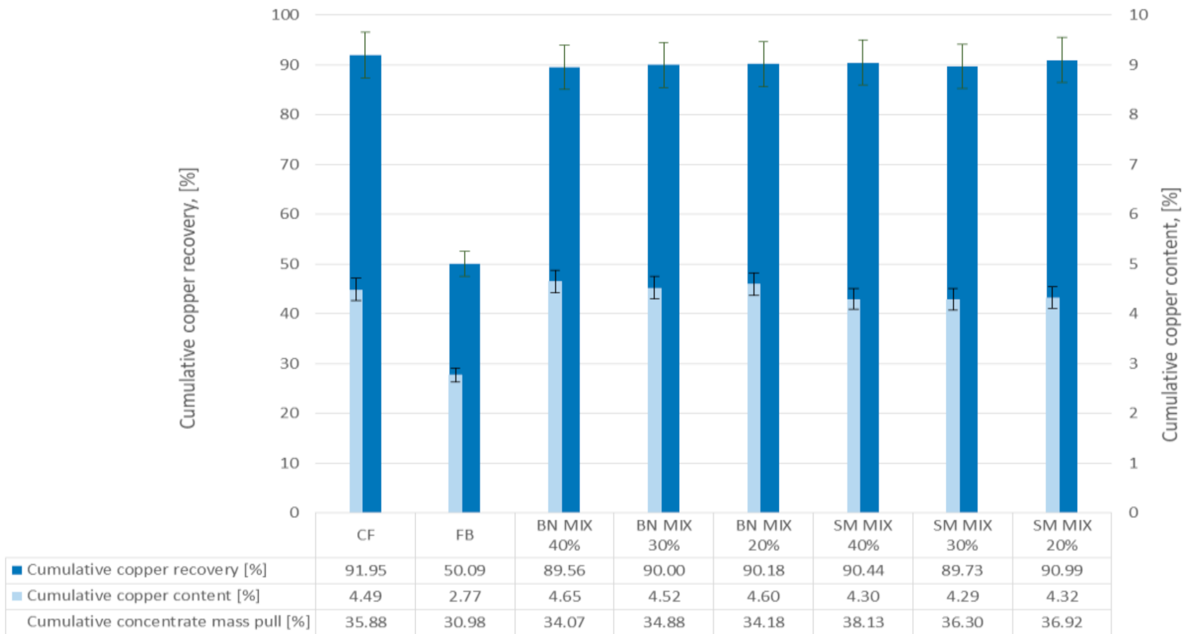


Fig. 7. Comparison of comparative flotation (CF), collectorless (FB), MIXTURES of Birch Nano (MIX BN), and MIXTURES of Spruce Micro (MIX SM)

solely (CF). In case of BM, mixtures with increased OLP concentration causes decrease in concentrate mass pull decreased with simultaneous increase in copper content (Fig. 7). In case of SM the contrary results are obtained.

3.1. Organic carbon activation

The experiment with dosages where the selectivity coefficient of copper content against gangue material content was the most promising was selected for TOC analyses. On grounds of a slightly similar result in the case of mixes with xanthates only test with SM were included in further TOC analyses.

The selectivity of organic carbon in comparison to copper was presented in Fig. 8, where content of those two components in the concentrates were demonstrated. One can see that flotation in which xanthates were utilized as main reagents, the recovery of TOC is lower than copper, nevertheless obtaining 90% of Cu recovery causes recovery of approx. 70% TOC. In comparison, the tests where no addition of collectors and addition of OLP as collector is performed, only approx. 50% of copper is recovered in 25 min test, with TOC recovery in range of 60-90%. The highest TOC recovery in the tests with SM and BN, where dosages were increased to 160 g/t. Fig. 8 shows that the utilization of those OLP reagents in high dosage allows to increase TOC recovery in the concentrate.

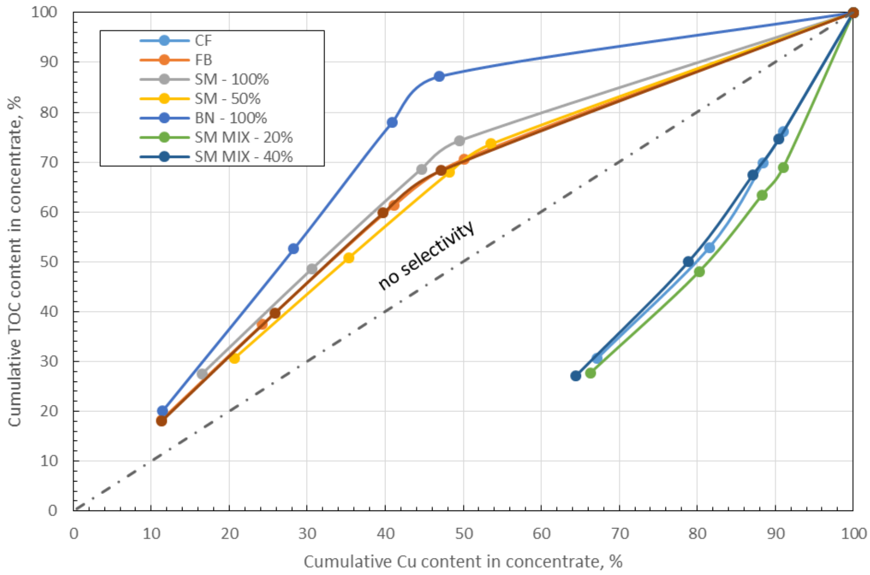


Fig. 8. Recovery of copper in concentrates vs. recovery of total organic carbon

4. Conclusions

This study evaluated the possibility of using OPL as a flotation reagent. The OLP did not show collecting properties toward copper minerals in the tested ore sample. When the OLP was used as a sole collector, the total Cu recovery was only 50%, which is not sufficient for the mining industry. Thus, OLP was studied as a flotation reagent that could partially replace xanthate during the flotation process. As a result, a mixture of xanthate and OLP was applied to the flotation process. The BN performed well in the case of process selectivity, where copper content in the concentrate increased but the recovery is lower due to lower mass pull. Because the utilization of the OLP caused the decrement of process efficiency in case of copper, the selectivity toward TOC was also examined by analyses for TOC in the flotation products. The tests with high dosage of BN and SM (approx. 160 g/t) showed that high dosage of this types of OLP reagents caused promotion of the TOC into concentrate and increased selectivity against copper in this product. Therefore, the results showed high potential in utilization of the OLP reagents as the substitute of currently used maltodextrin in the final concentrate selectivity flotation, where it would provide the selective separation of copper and TOC. In addition, the dosage of OLP was lower than the required dosage of maltodextrin (1-2 kg/Mg). Finally, BN and SM was recommended for further tests as a TOC collector. The trials will be performed in pilot scale installation.

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