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Satbayev University

Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ
НАУК РЕСПУБЛИКИ
КАЗАХСТАН
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NAS RK is pleased to announce that News of NAS RK. Series of geology and technical sciences scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of geology and technical sciences in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of geology and engineering sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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STUDY OF PURIFICATION OF COPPER ELECTROREFINING SOLUTION BY FLOW CENTRIFUGATION

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Abstract. Studies of purification of a copper electrodeposition solution by flow and decanter centrifugation with separation of a concentrated solution are carried out. The principle possibility purification of the solution is shown. The aim of research is to improve electrolyte purification technology and selective separation of non-ferrous metals - copper, nickel and zinc at regeneration and full utilization of copper electrolyte, that will increase productivity and profitability of operating enterprise producing cathode copper and get new, demanded and highly liquid goods of high value. The dependence of difference, density, viscosity, content and volume ratio of upper and lower drains on frequency of rotation of flow centrifuge separator is determined. The denser and more concentrated solution in content of sulfuric acid and non-ferrous metals is emitted into upper plum of centrifuge. Its quantity and composition depend on frequency of rotation of the separator of the centrifuge. At a separator speed of 12,000 rpm and 15,000 rpm, the difference in the individual element content of the solution was up to 10 %. The use of flow centrifugation to separate the concentrated part will make it possible to reduce the volume of solution sent to evaporation for recycling in production when obtaining cathode copper. Evaporation of the sulphuric acid solution is an energy consuming and time consuming operation, on which the profitability of the entire production depends.

Keywords: waste copper electrolyte, zinc sublimates, flow centrifugation, viscosity, density, neutralization, precipitation, decanter centrifuge

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Аннотация. Концентрацияланған ерітіндіні бөлумен ағынды және декантерлік центрифугалау әдісімен мыс электро тұндыру ерітіндісін тазарту бойынша зерттеулер жүргізілді. Ерітіндіні тазартудың негізгі мүмкіндігі көрсетілген. Зерттеу үшін Қазақстандағы түсті металдардың сынықтарын өңдейтін "Кастинг" ЖШС мыс балқыту зауытының пайдаланылған электролиті және құрамында мырыш бар өнеркәсіптік өнімі пайдаланылды. Тақырыптың өзектілігі айналымдағы электролитті тазарту технологиясын жетілдірумен байланысты түсті металл сынықтарын қайта өңдеу өндірісінің тиімділігін арттыру қажеттілігімен анықталады. Зерттеудің мақсаты-электролитті тазарту технологиясын жетілдіру және мыс электролитін регенерациялау және толық кәдеге жарату кезінде түсті металдарды — мыс, никель және мырышты селективті бөлу, бұл катодты мыс шығару бойынша жұмыс істеп тұрған кәсіпорынның өнімділігі мен рентабельділігін арттыруға және жоғары құны бар жаңа, сұранысқа ие және жоғары өтімді тауарларды алуға мүмкіндік береді. Жұмыста алғаш рет электролиттің концентрацияланған сульфат бөлігін бөлу үшін ағынды центрифугалау әдісін қолдану мүмкіндігі зерттелетін болады. Жоғарғы және төменгі ағындардың тығыздығының, тұтқырлығының, мазмұны мен көлемдерінің ағындық центрифуга бөлгіштің айналу жиілігіне қатынасы анықталды. Центрифуганың жоғарғы ағызында күкірт қышқылы мен түсті металдардың құрамы бойынша неғұрлым тығыз, концентрацияланған ерітінді бөлінеді, оның мөлшері мен құрамы центрифуга бөлгіштің айналу жиілігіне байланысты. Бөлгіштің айналу жиілігі 12000 айн/мин және 15000 айн / мин болған кезде ерітіндінің жеке элементтерінің құрамындағы айырмашылық 10 % дейін болды. Концентрацияланған бөлікті бөлу үшін ағынды центрифугалауды қолдану өндірісте кәдеге жарату үшін буландыруға катодты мыс алу кезінде жіберілетін ерітіндінің көлемін азайтуға мүмкіндік береді. Күкірт қышқылының ерітіндісін буландыру энергияны қажет ететін және ұзақ жұмыс болып табылады, оған бүкіл өндірістің рентабельділігі байланысты.

Түйін сөздер: қалдықтағы мыс электролиті, ағынды центрифугалау, тұтқырлық, тығыздық, бейтараптандыру, тұндыру, декантерлік центрифуга

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ИССЛЕДОВАНИЕ ОЧИСТКИ РАСТВОРА ЭЛЕКТРОРАФИНИРОВАНИЯ МЕДИ МЕТОДОМ ПРОТОЧНОГО ЦЕНТРИФУГИРОВАНИЯ

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Аннотация. Проведены исследования очистки раствора электроосаждения меди методом проточного и декантерного центрифугирования с отделением концентрированного раствора. Показана принципиальная возможность очистки раствора. Для исследований использован отработанный электролит и цинксодержащий промпродукт медеплавильного завода ТОО «Кастинг» в Казахстане, перерабатывающего лом цветных металлов. *Актуальность темы* определяется необходимостью повышения эффективности производства переработки лома цветных металлов, связанной с совершенствованием технологии очистки оборотного электролита. *Цель исследований* — совершенствование технологии очистки электролита и селективное выделение цветных металлов — меди, никеля и цинка при регенерации и полной утилизации электролита меди, что позволит повысить производительность и рентабельность действующего предприятия по выпуску катодной меди и получить новые, востребованные и высоколиквидные товары повышенной стоимости. В работе впервые будет исследована возможность применения для отделения концентрированной сульфатной части электролита способа проточного центрифугирования. Определена зависимость разницы, плотности, вязкости, содержания и отношения объемов верхнего и нижнего сливов от частоты вращения разделителя проточной центрифуги. В верхний слив центрифуги выделяется более плотный, концентрированный раствор по содержанию серной кислоты и цветных металлов, количество и состав которого зависит от частоты вращения разделителя центрифуги. При частоте вращения разделителя 12 000 об/мин и 15 000 об/мин разница в содержании отдельных элементов раствора составила до 10 %. Использование проточного центрифугирования для отделения концентрированной части позволит сократить в производстве объем раствора отправляемого при получении катодной меди на выпарку для утилизации. Выпарка сернокислого раствора является энергозатратной и продолжительной операцией, от которой зависит рентабельность всего производства.

Ключевые слова: отработанный электролит меди, проточное центрифугирование, вязкость, плотность, нейтрализация, осаждение, декантерная центрифуга

Introduction

Problems in the technology of non-ferrous scrap processing at the stage of electrorefined copper production are the accumulation of undesirable impurities of nickel, zinc and others in the recycled electrolyte, which deteriorate the grade of the copper cathode produced. Part of the electrolyte from the saleable baths is periodically removed from the electrolysis cycle and recycled. The productivity of the plant and its profitability depend on the efficiency of the electrolyte cleaning operation, therefore it is important to carry out research to improve the existing technology.

Different approaches have been proposed for the extraction of heavy metal cations from recycled electrolyte: extraction, sorption, membrane and combined approaches based on sorption and electrochemical methods (Dyussenova et al., 2018; Dyussenova et al., 2019; Kenzhaliev et al., 2017).

In the production of refined copper from minerals, most plants use a two-stage solution processing scheme – evaporation and crystallization of the electrolyte to produce copper sulfate and extraction of residual copper from the evaporated solution by electroextraction. After extraction of copper from solution, nickel is extracted as a sulphuric salt by evaporation, crystallisation and subsequent refining (Hoffmann et al., 2004; Junior et al., 2019; Krishnan et al., 2021; Kasikov et al., 2010). The method does not allow for sufficiently selective isolation of non-ferrous metals present in the solution.

There is a known method for processing copper electrolyte selected for regeneration to produce copper and nickel vitriol. In which, by triple neutralization of excess acidity with copper powder in the presence of oxygen, evaporation in a vacuum – evaporation unit, cooling with crystallization, separation of crystals from the mother liquor by centrifugation, copper sulfate is obtained. Cathode copper contaminated with arsenic and antimony and nickel solution are obtained from the mother liquor by electrolysis with insoluble lead — silver electrodes, which is sent for evaporation, cooling with crystallization and separation of nickel vitriol crystals from the filtrate (González de las Torres et al., 2021).

The reasons hindering the achievement of the mentioned technical result when using the known method include the fact that in the known method the electrolyte cleaning scheme is rather cumbersome, the

consumption of copper powder is high, undesirable micro impurities are distributed between copper and nickel vitriol, worsening the grade, and the electricity consumption is also high.

The method of copper electrolyte purification in which by neutralization of residual acid to 60 kg/m³, precipitation with barite concentrate, separation of precipitate from electrolyte by filtration, solution returned to copper electrolysis cycle and arsenic precipitate subjected to further treatment with sodium sulfate and sodium sulfide mixture is known (Patent SU 1643632. Golikov et al., 1991; Patent 4,157,946. Hyvärinen et al., 1979). In the method, only arsenic is purified, while other impurities are not removed from the copper electrolysis cycle.

In the conducted studies for the purification of spent copper electrolyte, the possibility of using flow and decanter centrifugation methods has been investigated.

The relevance of the topic is determined by the need to increase the efficiency of the production of non-ferrous scrap processing associated with the improvement of the technology of purification of recycled electrolyte.

The purpose of the research is to improve the technology of electrolyte purification and the selective isolation of non-ferrous metals — copper, nickel and zinc during the regeneration and complete utilization of the copper electrolyte, which will increase the productivity and profitability of the existing enterprise for the production of cathode copper and obtain new, in-demand and highly liquid goods of increased value.

Materials and methods of research

X-ray fluorescence analysis of the chemical composition was performed on a Venus 200 wave dispersion spectrometer (Panalytical B.V., Holland). Chemical analysis was performed using an Optima 2000 DV inductively coupled plasma optical emission spectrometer Optima (Perkin Elmer, USA). Semi-quantitative X-ray phase analysis was performed on a D8 Advance diffractometer Advance (BRUKER) using copper Cu-K α radiation at an accelerating voltage of 36 kV and a current of 25 mA. The speed of rotation of the flow centrifuge separator was determined using an IO-30 tachometer, with an accuracy of $\pm 2\%$. The density of the solutions was determined using a standard set of densimeters. The viscosity of the solutions was measured using a glass capillary viscometer VPZh-2 with a capillary diameter of $d=0.56$ mm.

The kinematic viscosity of solutions was calculated by the formula:

$$V = g/9.807 \times \tau \times K$$

where, K is the viscometer constant equal to $0.03186 \text{ mm}^2/\text{s}^2$;

V – kinematic viscosity, mm^2/s .

g is the acceleration of gravity at the measurement point, m/sec^2

τ is the liquid expiration time in seconds, s.

When calculating the kinematic viscosity of solutions, the average value of the liquid outflow time obtained as a result of 5 measurements was used.

Results and their discussion.

For the research, the spent electrolyte of the copper smelter of Casting LLP in Kazakhstan, which processes non-ferrous metal scrap, was used.

Chemical composition of spent electrolyte, wt. g /dm³: Cu 67.14; Ni 36.41; Fe 11.43; Zn 10.96; SO₄ 125.9; N 4.1; As; 0.03; Bi 0.002; Na 2.6; Pb 0.014; Sb 0.05; Si 0.047; Sn 0.0.

The method of flow-through centrifugation of spent electrolyte was tested using standardized equipment "Motor Sich-500" with fixed adjustment of centrifugation modes.

Flow-through centrifuge are also used for fast processing of large volumes of liquid. Centrifugation occurs in continuous or semi-continuous operation modes (Izatt et al., 2015).

Flow centrifuges can be used for both sedimentation (precipitation) of the target product and separation of two liquid fractions of different densities (Jamaal et al., 2015; Plath 1994; Schafflinger 1990).

According to the passport data, the maximum speed of rotation of the separator of the flow centrifuge "Motor Sich-500" is 8000.0 revolutions per minute.

To increase the speed of rotation of the separator, the centrifuge was upgraded at mi. For this purpose, the DSK-250 engine was replaced with a more high-speed and powerful DC-066 engine with a power of 660 W, which allows maximum rotation speed up to 25 000.0 rpm.

The speed of rotation of the separator rotor depends on the voltage. To establish the dependence of the speed of rotation of the separator of the flow centrifuge on the applied voltage, the speed of rotation was calibrated (Table 1).

Table 1-Dependence of the speed of rotation of the separator of the flow centrifuge on the voltage

Voltage, V	Revolutions, rpm
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100.0	8 000.0
125.0	10 000.0
150.0	12 000.0
175.0	15 000.0
200.0	17 000.0
220.0	17 000.0

Purification of the circulating copper electrolyte by flow centrifugation was performed at separator rotation frequencies from 8000.0 rpm to 15000.0 rpm. The extreme magnification caused the centrifuge to vibrate violently.

During the tests, the output volumes of the upper and lower drains, the metal content, the density and viscosity of the obtained solutions were determined.

The results of flow centrifugation of waste copper electrolyte are shown in Table 2.

Table 2-Flow centrifugation of waste copper electrolyte during flow centrifugation

Separator rotation, rpm	Name	Volume			Solution composition, g /dm ³								ρ*, g/sm ³	Δ ρ*, %
		dm ³	%	L:S	S		Cu		Ni		Zn			
					g/dm ³	Δ*, %	g/dm ³	Δ*, %	g/dm ³	Δ*, %	g/dm ³	Δ*, %		
8 000.0	Upper drain	1.0	25.0	1:3	9.05	2.84	18.45	7.89	16.25	4.83	4.52	5.11	1.33	0.1
	Lower drain	3.0	75.0		8.8		17.1		15.5		4.3		1.332	
10 000.0	Upper drain	0.62	14.0	1:6	9.38	5.03	18.7	6.85	16.22	3.31	4.42	5.23	1.335	0.3
	Lower drain	3.78	86.0		8.93		17.5		15.7		4.2		1.331	
12 000.0	Upper drain	0.44	11.0	1:8	8.92	8.4	17.85	10.1	16.0	8.01	4.44	5.71	1.335	0.3
	Lower drain	3.48	89.0		8.23		16.2		14.8		4.2		1.331	
15 000.0	Upper drain	0.3	8.0	1:11	8.84	10.6	18.04	10.2	16.0	7.38	4.45	5.95	1.335	0.08
	Lower drain	3.45	92.0		7.99		16.37		14.9		4.2		1.33	

Δ - content difference, %.

ρ* - density of the solution, kg /m³;

Δ ρ* - density difference, %.

The variability of the difference in the solution content, density and ratio of the volumes of the upper and lower drains of the flow centrifuge separator from the rotation speed is shown in Figures 1–3.

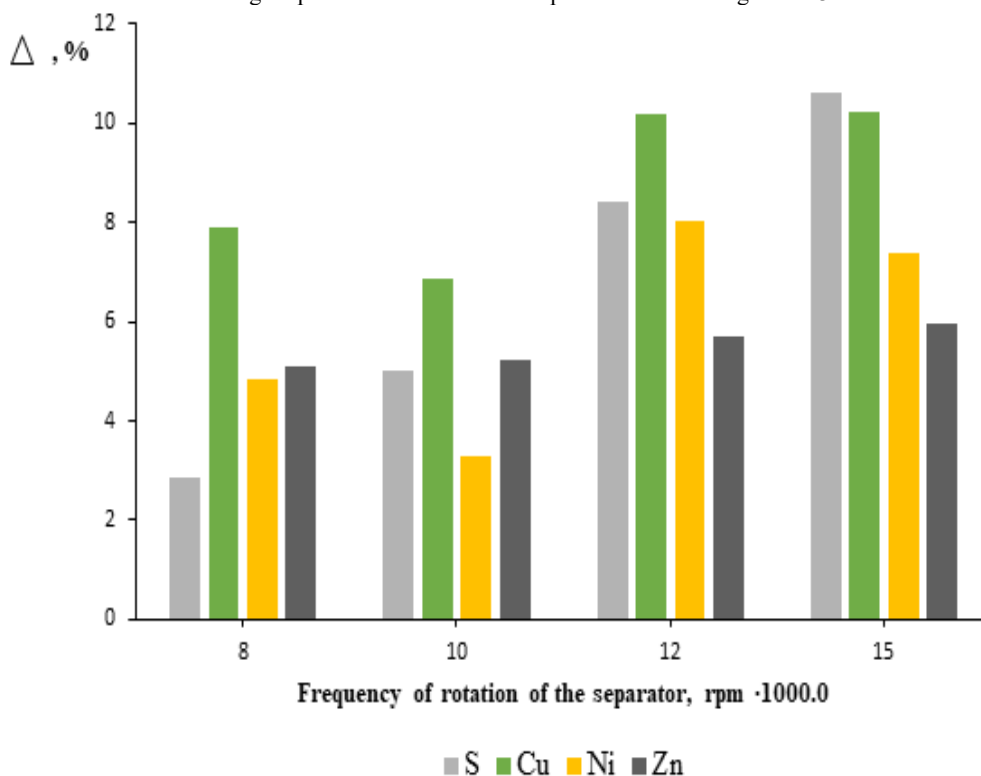


Figure 1–Dependence of the difference in the content of solutions in the upper and lower drains on the speed of rotation of the separator of the flow centrifuge

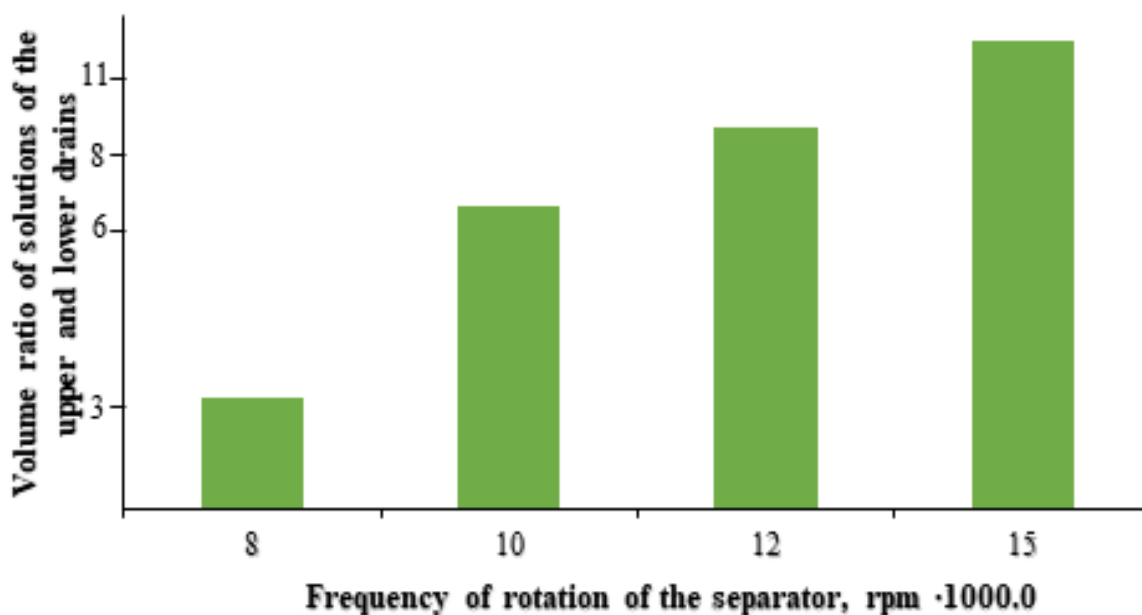


Figure 2–Dependence of the ratio of the volumes of the upper and lower drains on the rotation speed of the separator of the flow centrifuge

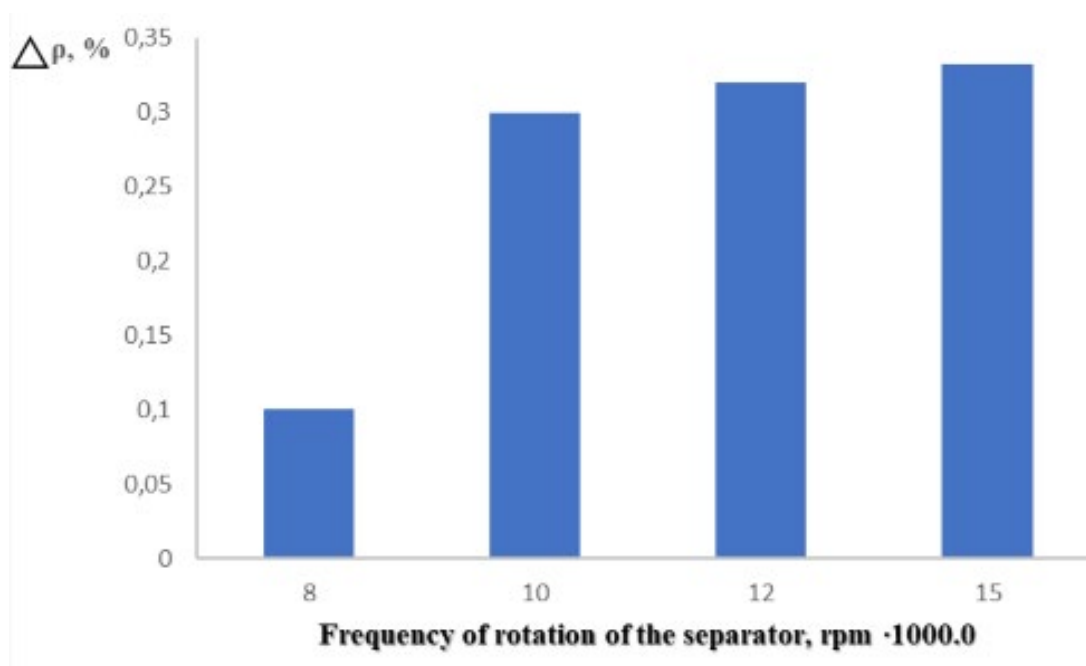


Figure 3–Dependence of the difference in the density of the upper and lower drains on the speed of rotation of the separator of the flow centrifuge

The dependence of the difference in viscosities and solutions (ΔV) of the upper and lower drains on the speed of rotation of the separator of the flow centrifuge is determined (Table 3 and Figure 4).

Table 3-Viscosity of solutions depending on the speed of rotation of the separator of the flow centrifuge

Rotation speed, rpm	Name	τ , average, s	V , mm ² /s
8 000.0	Upper drain	119.62	3.816
	Lower drain	117.1	3.737

10 000.0	Upper drain	119.62	3.816
	Lower drain	116.64	3.721
12 000.0	Upper drain	123.63	3.943
	Lower drain	121.12	3.864
15 000.0	Upper drain	119.62	3.816
	Lower drain	116.61	3.72

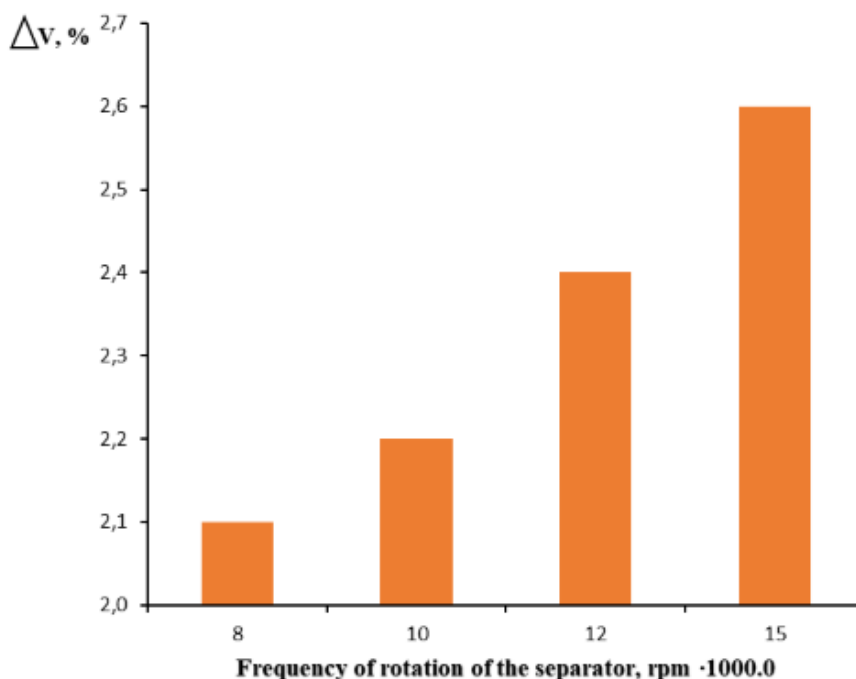


Figure 4–Dependence of the difference in the viscosity of the upper and lower drains on the speed of rotation of the separator of the flow centrifuge

Studies have shown that:

- a solution with a higher density is released into the upper drain. The difference in the density of solutions of the upper and lower drains is from 0.1 % to 0.333 %;

- a solution with a higher viscosity is released into the upper drain. The difference in the viscosity values of the solutions of the upper and lower drains is from 2.1 % to 2.6 %.

As the speed of rotation of the flow centrifuge separator increases:

- the difference in the content of solutions of the upper and lower drains increases. When the speed of rotation of the separator of the flow centrifuge was 12 000.0 and 15 000.0 rpm, for sulfur it was 8.4 % and 10.6 %; for copper it was 10.19 % and 10.2 %; for nickel it was 8.01 % and 7.38 %; and for zinc it was 5.71 % and 5.95 %, respectively;

- the difference in the volume of the upper and lower drains increases from 1: 3 at 8 000.0 rpm to 1: 11 at 15 000.0 rpm.

Selective isolation of nickel and copper-containing products from the sediments of neutralization of waste copper electrolyte by decanter centrifugation has been studied.

Precipitation and neutralization from spent copper electrolyte were obtained by step neutralization using zinc sublimates at the first stage.

Zinc sublimates are formed at a non-ferrous metal scrap processing plant in the process of fire refining of copper as a result of oxidation of excess zinc in anodic copper to the required standards, by purging the melts with compressed air and capturing them from the exhaust gases on bag filters.

Chemical analysis of zinc sublimates, wt. %: F 0.97; Al₂O₃ 0.15; SiO₂ 0.82; P₂O₅ 0.15; SO₃ 4.0; Cl 11.64; K₂O 0.93; CaO 0.36; Fe₂O₃ 0.29; NiO 0.05; CuO 7.86; ZnO 39.46; Br 0.19; MoO₃ 0.1; CdO 0.23; SnO₂ 7.16; WO₃ 0.36; PbO 19.4; Bi₂O₃ 0.04; other products (o.p.) 5.84.

X-ray phase analysis of zinc sublimates is shown in Figure 5.

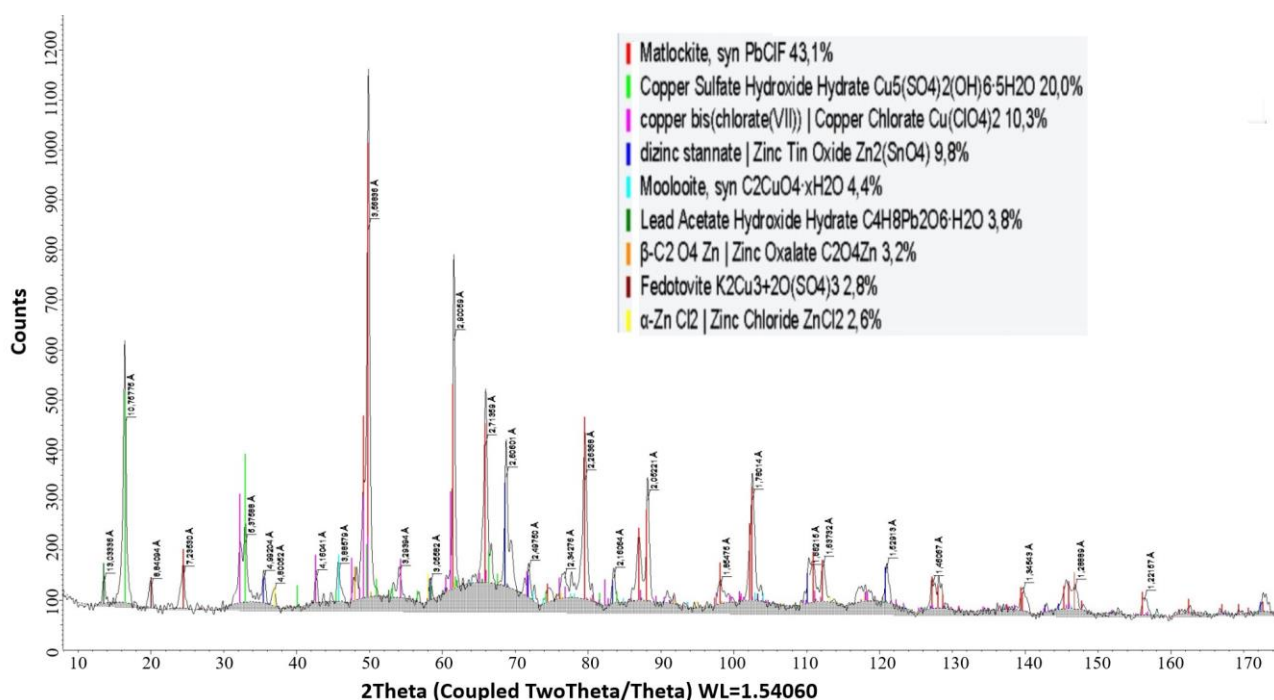


Figure 5–X-ray phase analysis of zinc sublimates

The first stage of neutralization of the spent copper electrolyte was carried out to a pH of 4.7 at L:S=5:1, a temperature of 20°C and a stirring time of 3 hours.

As a result of neutralization, a black precipitate of the composition, wt. %: F 0.44; Na₂O 0.71; Al₂O₃ 1.56; SiO₂ 4.08; P₂O₅ 0.41; SO₃ 11.12; Cl 0.72; K₂O 0.19; CaO 0.85; Fe₂O₃ 5.48; NiO 0.15; CuO 2.97; ZnO 4.8; Br 0.09; MoO₃ 0.23; CdO 0.08; SnO₂ 6.19; WO₃ 0.29; PbO 16.97; Bi₂O₃ 0.09; o.p. 4.7.

Sediment separation was carried out on OS-6MC decanter centrifuge at a rotor rotation speed of 2 500.0 rpm. The centrifuge provides separation into fractions of inhomogeneous liquid systems with a density of 2 g/sm³ under the influence of centrifugal forces.

X-ray phase analysis of the first stage of neutralization sediment is shown in Figure 6.

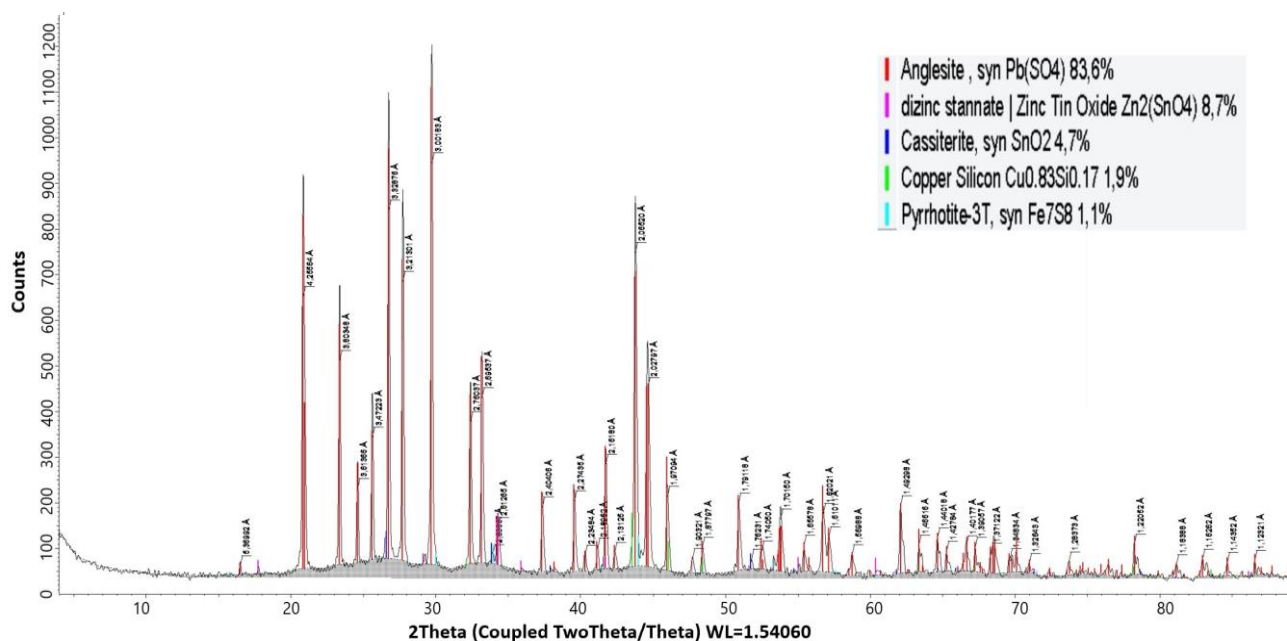


Figure 6–X-ray phase analysis of the sediment of the first stage of neutralization of spent copper electrolyte

The second stage was neutralized and potash was applied to pH 9.92. As a result, a sediment of the composition, wt. %: NiO 8.89; CuO 23.91; ZnO 13.4; o.p. 53.80.

During centrifugation, the sediment of the second stage of neutralization was divided into two parts - the lower part is blue and the upper part is light green.

Chemical composition of the lower part of the sediment, wt. %: CuO 51.88; ZnO 3.4; NiO 0.89; o.p. 43.83.

Chemical composition of the upper part of the sediment, wt. %: NiO 29.26; ZnO 44.09; CuO 0.34; o.p. 26.31.

X-ray phase analysis of the lower part of the neutralization sludge is shown in Figure 7.

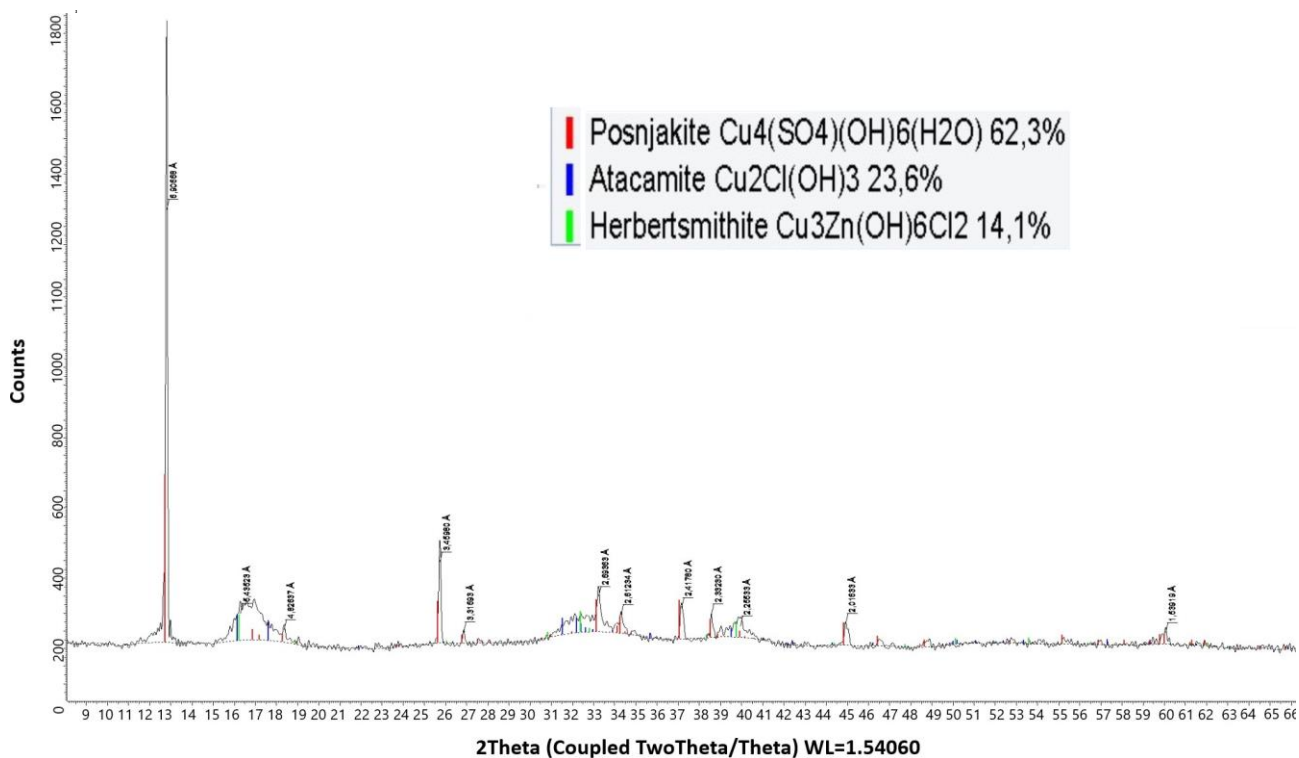
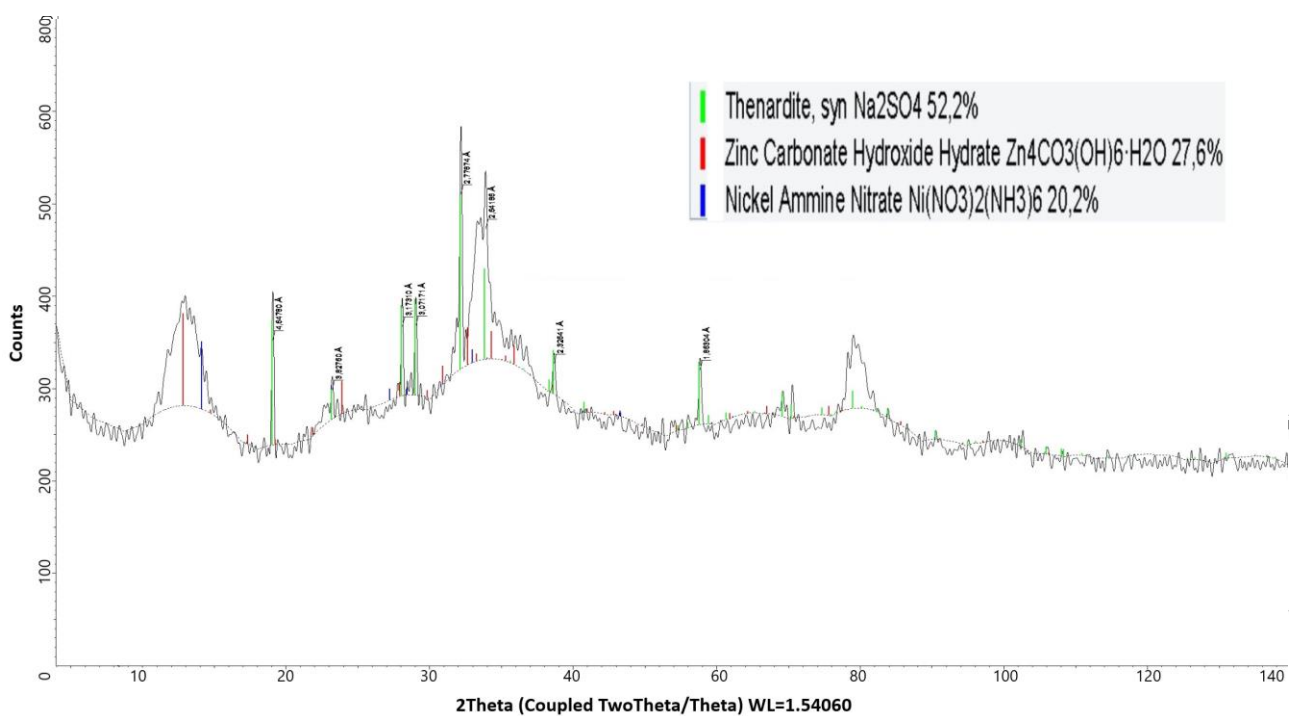


Figure 7–X-ray phase analysis of the lower part of the neutralization sludge

The lower part of the precipitate can be considered as a copper-containing concentrate and used in the preparation of a copper electrofining solution.

X-ray phase analysis of the upper part of the neutralization sludge is shown in Figure 8.



The upper part of the sediment can be considered as a nickel-zinc-containing product and used to product selective nickel and zinc concentrates after alkaline treatment.

To determine the regularity of neutralization sludge separation during decanter centrifugation, physical and chemical characteristics (viscosity and density) of copper, nickel, and zinc sulfuric acid solutions were determined (Table 4).

Synthetic solutions containing 20 g/dm³ of non-ferrous metals (CuO, NiO, and ZnO) and 50 g/dm³ H₂SO₄ were used for the studies H₂SO₄.

Table 4–Viscosity of non-ferrous metal solutions

Name of solutions	τ , medium, s	V, mm ² /s
Copper	42.63	1.357
Nickel	42.51	1.353.353
Zinc	42.345	1.348

The density of the solutions was determined using a set of densimeters.

Physical and chemical characteristics of sulfuric acid solutions of non-ferrous metals are given in Table 5.

Table 5–Physical and chemical characteristics of sulfuric acid solutions of non-ferrous metals

Name of solution	Content g /dm ³			p. g/sm ³	V. mm ² / s
	CuO	NiO	ZnO		
Copper	20			1.050	1.357
Nickel		20		1.051	1.353
Zinc			20	1.050	1.348

From the obtained results of physical and chemical characteristics of sulfuric acid solutions of non-ferrous metals, it follows that solutions with the same density have different viscosities.

The separation of neutralization sediments during decanter centrifugation can be explained by the difference in the speed of movement of hydrated non-ferrous metal molecules, which is related to the viscosity of solutions.

Conclusions

Purification of waste copper electrolyte is possible by flow centrifugation. In the upper drain of the centrifuge separator, a denser, more concentrated solution is released in terms of the content of sulfur and non-ferrous metals, the quantity and quality of which depends on the speed of rotation. At a rotation speed of 12 000.0 rpm and 15 000.0 rpm, the difference in the content of individual elements of the solution was up to 10 %.

As a result of decanter centrifugation and stepwise neutralization of spent copper electrolyte, selective separation of nickel and copper-containing products was obtained. It is determined that the selective separation of non-ferrous metal neutralization sediments during decanter centrifugation is related to the viscosity of solutions.

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