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<https://doi.org/10.31643/2019/6445.11>**LINNIK X. A., SHARIPOVA A. S., ZAGORODNYAYA A. N.***Satbayev University, Institute of Metallurgy and Ore Beneficiation, Almaty, Republic of Kazakhstan**e-mail: [xenija_linnik@mail.ru](mailto: xenija_linnik@mail.ru)***A SLURRY EMISSION OFF A PULP APPEARED AFTER METALLURGIC GASES WASH OF BALKHASH COPPER SMELTING PLANT BY SPINNING***Received: 21 January 2019 / Peer reviewed: 10 March 2019 / Accepted: 10 April 2019*

Abstract. A sulphuric acid is obtained of the metallurgic gases after smelting of feed materials and matte converting preliminary cleaned dry or wet method. A pulp appears after wet gases cleaning containing sulphuric acid solution, slurry and amorphous selenium. The sulphuric acid production at the Balkhash copper smelting plant differs from the identic one at the Zheskazgan copper smelting plant by cleansing solutions spinning off and extracting slurry out of them by sedimentation. By this reason Balkhash copper smelting plant discharge pulp 7-14 times as much as Zheskazgan copper smelting plant of clarified liquor. Pulp is a valued man-made raw material to extract rhenium off the solution, selenium iodine off the slurry. At present, a sorption technology of rhenium extraction off the solution is developed and tested in the pilot scale. Selenium is a second component worth mentioning considering its production at the Balkhash copper smelting plant. This article analyzes prospect researches of various methods testing of clear solutions obtaining, faults and benefits of straining by spinning. The results are provided of the slurry amount effect and amorphous selenium in the pulp, the speed and time of rotation of the centrifuge rotor. A rotor spinning velocity is established to be the main factor has impact on pulp separation into the clear solution and residue of every slurry compound, including amorphous selenium by spinning. This method according to calculations allows to a substantial energy costs saving comparing the pulp heating-straining.

Keywords: metallurgic gases, flushing, pulp, cleansing sulphuric acid, slurry, straining, spinning.**ЛИННИК К. А., ШАРИПОВА А. С., ЗАГОРОДНЯЯ А. Н.***Satbayev University, Металлургия және кен байыту институты, Алматы, Қазақстан, e-mail: [xenija_linnik@mail.ru](mailto: xenija_linnik@mail.ru)***БАЛҚАШ МЫС БАЛҚЫТУ ЗАУЫТЫНДАҒЫ МЕТАЛЛУРГИЯЛЫҚ ГАЗДАРДЫ ШАЙҒАН КЕЗДЕ ПУЛЬПАДАН ЦЕНТРУГАЛАУ АРҚЫЛЫ ПАЙДА БОЛАТЫҢ ШЛАМНЫҢ ШЫҒУЫ**

Түйіндеме. Балқаш және Жезқазған мысбалқыту зауыттарындағы мыс шихтасын балқытқанда және штейнді конвертлегендегі металлургиялық газдардан күкірт қышқылы алынады. Жезқазған мысбалқыту зауыттардан дан Балқаш мысбалқыту зауыттарындағы күкіртқышқыл өндірісінің ерекше айырмашылығы, айналмалы шайынды ерітінділер мен тұндырған кезде, онда шламның болмауымен айқындалынады. Соңдықанда, Жезқазған мысбалқыту зауытқа қарағанда, Балқаш мысбалқыту зауыты тазаланған пульпаны 7-14 еседей артық шығарады да басқа кәсіпорындарға тұндырылған ерітіндіні жібереді. Пульпа (қойыртпақ) – шламнан йод пен селенді, ерітіндіден ренийді бөліп алу үшін бағалы техногенді шикізат болып табылады. Ерітіндіден ренийді бөліп алудың сорбциондық технологиясы жартылай өндірістік ауқымда сынақталды және өндірілді. Басқа да көңіл бөлінген элементтің бірі, селен. Пульпадан тұнық ерітіндіні алу барысында, аморфты селеннен қиыншылықтар туындады. Мақалада, таза ерітінділерді алудың әртүрлі әдістерімен ізденіс үстіндегі зерттеу жұмыстарлы талданды әрі олардың кемшіліктері келтірілді. Демек, центрифугирлау әдісі, оның ішінде, осыған сай көрсеткіштер мен пульпаның құрамдық қасиеттері анағұрлым анық зерттелінді. Центрифугадағы ротордың айналу уақыты мен жылдамдығы, пульпадағы аморфты күйдегі селен және шламның үлесті әсерінің нәтижелері келтірілді. Яғни,пульпаны тұнық ерітіндіге бөлудің әсері және аморфты селенді қосқандағы шламдағы барлық шөгінділерді центрифугирлегендегі негізгі фактор, ротордың айналуы болып табылады. Есеп көрсеткітері бойынша бұл әдіс, пульпаны қыздыру-сүзу тәсіліне қарағанда, электрлі энергияның шығынын айтарлықтай қысартады.

Түйінді сөздер: металлургиялық газдар, шаю, пульпа (қойыртпақ), шайынды күкірт қышқылы, шлам, сүзу, центрифугалау.**ЛИННИК К. А., ШАРИПОВА А. С., ЗАГОРОДНЯЯ А. Н.***Satbayev University, Институт металлургии и обогащения, Алматы, Казахстан, e-mail: [xenija_linnik@mail.ru](mailto: xenija_linnik@mail.ru)***ВЫДЕЛЕНИЕ ШЛАМА ЦЕНТРИФУГИРОВАНИЕМ ИЗ ПУЛЬПЫ, ОБРАЗУЮЩЕЙСЯ ПРИ ПРОМЫВКЕ МЕТАЛЛУРГИЧЕСКИХ ГАЗОВ БАЛХАШСКОГО МЕДЕНЛАВИЛЬНОГО ЗАВОДА**

Резюме. На Балхашском медеплавильном заводе из металлургических газов от плавки медной шихты и конвертирования штейна, предварительно очищенных сухим и мокрым способами, получают серную кислоту. При мокрой очистке газов образуется пульпа, содержащая раствор серной кислоты, шлам, в том числе и аморфный селен. Сернокислотное производство Балхашского медеплавильного завода отличается от аналогичного Жезказганского медеплавильного завода отсутствием циркуляции промывных растворов и выделением из них шлама отстаиванием. Поэтому Балхашский медеплавильный завод сбрасывает на очистные сооружения пульпы в 7-14 раз больше, чем Жезказганский медеплавильный завод осветленного раствора. Пульпа является ценным техногенным сырьем для извлечения рения из раствора, селена и йода из шлама. В настоящее время разработана и апробирована в полупромышленном масштабе сорбционная технология извлечения рения из раствора. Селен – второй элемент, заслуживающий внимания, учитывая, что на Балхашском медеплавильном заводе налажено его производство. В статье дан анализ поисковых исследований по апробации различных методов получения чистых растворов, указаны их недостатки и преимущество центробежного фильтрования. Приведены результаты по влиянию количества шлама и аморфного селена в пульпе, скорости и времени вращения ротора центрифуги. Установлено, что основным фактором, влияющим на разделение пульпы на прозрачный раствор и осадок всех составляющих шлама, в частности аморфного селена, центрифугированием является скорость вращения ротора. Этот метод, как показали расчеты, позволит на порядок сократить расходы на электроэнергию по сравнению со способом нагревания-фильтрация пульпы.

Ключевые слова: металлургические газы, промывка, пульпа, промывная серная кислота, шлам, фильтрация, центрифугирование.

Introduction. A bulk copper from a feed material in Kazakhstan is produced at the Balkhash and Zheskazgan copper smelting plants (BCSP, ZCSP) from feed materials one of the main component of is local copper concentrates and man-made formations of non-ferrous industry. Apart copper the other non-ferrous and rare and precious metals as well can be found within processed feed material. Companion-elements are collected in various solid and liquid man-made formations in the process of obtaining of commercial copper. For example, sulfur, rhenium and osmium-187 basically are in the mixtures after cleansing of gases of metallurgy (industrial terms: cleansing sulfuric acid-CSA), lead in the powder and slurry of sulfuric production; gold, silver and selenium are in the copper anode slime. Today, copper, gold, silver, selenium, sulfuric acid (BCSP); copper, ammonium perrhenate, osmium isotope-187 and sulfuric acid (ZCSP) are extracted and obtained as commercial products from companion- elements contained in the raw materials at copper plants.

Sulfuric acid is obtained from the gases of metallurgy after feed material smelting and matte converting. The gases are preliminary cleared from removed feed materials, solid insoluble sublimations within the dry electrostatic precipitators; soluble sublimations and fine fraction of solid sublimations cleansed by sulfuric acid solutions in different kind of units.

In 2008 a sulfuric production unit (SPU) has started operating again in Balkhash, projected by Canadian experts of "Komitex". The equipment and cleansing of gases of metallurgy of this production unit is gradually differs of those SPU at ZCSP [1]. A wet gases cleansing is provided using fresh

solution of sulfuric acid at SPU of ZCSP, after cleansing of which is vanished to the sewage treatment plant like pulp. As a result, SPU of ZCSP vanishes a large bulk of pulp a day. The plant keeps under control the rhenium content and sulfuric acid in the CSA, a slurry in the vanished pulp, later selenium as well in the CSA initiated by The Institute of Metallurgy and Ore Benefication Co., Ltd. A chemical compound of CSA and the slurry are within the pulp (Table 1) was defined by the experts of The Institute of Metallurgy and Ore Benefication.

The elements were established to be found in both man-made formations that are of interest, firstly, for the needs of various industries. In particular is rhenium, selenium, iodine. Secondly, with regard to ecology is lead, mercury. Preliminary calculations testified that rhenium, selenium, iodine, and mercury are emitted into the sewage treatment plant along with the pulp in industrially significant quantities.

Since rhenium is one of the most important elements and most of it falls on the CSA, the first studies were aimed at developing the technology for its extraction [2-7]. As a result, a sorption technology was developed to produce ammonium perrhenate and rhenium acid. The technology has been tested on a semi-industrial scale, according to the test results, technological procedures have been developed, the production cost of ammonium perrhenate has been calculated [8].

Another element worth mentioning with regard to industrial production is selenium. This element is in the pulp, obviously, mainly in the component amorphous state of the red modification (hereinafter: amorphous selenium). This is testified

by the abundant suspension of red color in the CSA solution once the pulp is settled, but high selenium contents in the CSA solutions and BCSP slurries (Table 1) are not typical for sulfuric acid plants of other Non-Ferrous Industry Plants in Kazakhstan.

Table 1 – A pulp average chemical compound

Elements	BCSP Pulp	
	Cleansing sulfuric acid*	Slurry
	content,	
	g/dm ³	mass %
Re	0,008	0,14
Zn	0,216	0,35
Cu	0,0015	0,28
Cd	0,0024	0,15
Fe	0,0042	0,08
As	1,47	0,15
Se	0,031	4,60
Pb	0,0064	56,4
Mn	0,001	0,003
Sb	0,0009	0,05
K	0,013	Not determined
Na	0,202	Not determined
Mo	0,0003	0,001
SiO ₂	0,55	0,23
Cl	0,0210	Not determined
F	0,653	Not determined
Bi	0,004	0,32
Te	0,00065	Not found
Os	0,00012	0,001
Cr		0,01
Al		0,03
Ni		0,02
Ca		0,34
Hg		0,57
S		7,78
J		0,33
Br		0,01

Note: * sulfuric acid concentration – 57,05 g/dm³

A necessary requirement to extract any element from solutions by sorption or extraction is the purity of the solutions (transparency). Therefore, the pulp is subjected to repeat cleansing of slurry. For example, the main amount of it and amorphous selenium is separated in the septic tanks of the SPU at the BCSP. Then, once the CSA is taken to extract rhenium, the remaining amount of slurry is also separated by settling, fine after treatment is carried out by filtration. No filtration of CSA solutions after 2 stages of settling (even the selenium content in the CSA is not more than 2 mg / dm³) did not allow extracting rhenium from it in the previously existing production of ammonium perrhenate at the Balkhash Mining and Metallurgical Combine (BMMC, now

BCSP) by sorption anion exchangers. (The first industrial production of ammonium perrhenate from CSA in the former USSR, which was terminated in the 90s of the last century, first due to the absence of rhenium-containing raw materials, and later sulfuric acid due to equipment wear). For this reason, it was first sorbed using a KAD coal rank, and was removed from the eluates by sorption [9]. Experience of BMMC was taken into account in the development of extraction technology for the extraction of rhenium from the CSA for Zhezkazgan.

The slurry content in the pulp of the CSA of BCSP, as Table 2 shows, ranges from 0.034 to 14.35 g/dm³, the average - 2.62 - 3.51 g/dm³. The main element of the slurry is lead, the main phase is lead sulfate, as established by the IMOB, which is well within the literature data [10, 11].

Table 2 - The average content of slurry in the pulp and selenium in solutions of the filtered pulp

A slurry content in the pulp g/dm ³		
<i>top</i>	<i>bottom</i>	<i>average</i>
<i>2014 z</i>		
14,35	0,034	2,72
<i>2017 z</i>		
8,35	0,78	3,51
<i>2018 z</i>		
3,44	1,17	2,62
Se concentration in the solutions, mg/dm ³		
<i>top</i>	<i>bottom</i>	<i>average</i>
<i>2014 z</i>		
88,0	15,0	56,5
<i>2017 z</i>		
59,0	0,5	25,4
<i>2018 z</i>		
24	1,2	12,46

(The table shows the plant data of average monthly samples taken from the Venturi scrubber for 6 months. Average daily samples were arranged from medium-shift ones)

To separate the solid and liquid phases is known to be carried out by exposure methods (from the bulk of the solid phase), filtration (from both the bulk and fine fraction of the solid phase) under vacuum through various types of filter cloth and by gravity flow through the sand filters, sedimentation by coagulants and spinning [12, 13]. All these methods were tested in the laboratory in the BCSP production pulp in the development of technology for the extraction of rhenium from CSA [4].

The exposure at room temperature is established ineffective: the amorphous selenium deposition is very slow. An increase in temperature to 80 ° C dramatically reduces the phase separation time due to

the transfer of selenium from the red amorphous modification to the black crystalline [14].

The method of filtration of the pulp under vacuum using different filtering is not suitable for phase separation: amorphous selenium was distributed between the filter cake and the filtrate.

The filtration method through the quartz sand is of no use as well: a decrease in the pulp transmission rate due to the filling of the pores between the sand particles with lead sulfate and amorphous selenium; due to the loss of slurry, which is a valuable industrial raw material for the extraction of rhenium, selenium, and iodine from it.

The method of deposition using coagulants and flocculants to form amorphous selenium in the sediment also proved to be ineffective due to poor filtration.

By spinning, according to exploratory studies, a clear solution and residue was obtained and the precipitate containing slurry and amorphous selenium.

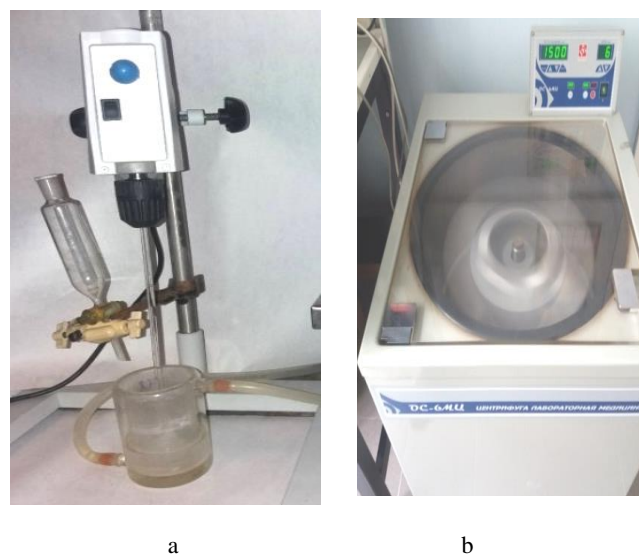
A semi-industrial tests for the extraction of rhenium from the CSA of BCSP, which was obtained according to the scheme: heating the pulp at high temperature - filtering the precipitate under vacuum, approved the cost of obtaining a pure solution is 60% of the production cost of 1 kg of ammonium perrhenate.

In order to reduce the cost of a pure solution production from the pulp and making the sediment of all components of the slurry, in particular, amorphous selenium, they chose a spinning method. Previously, information was elaborated to use this method in various industries [15-24], the availability of centrifuges of appropriate capacity, capable of processing the volume of pulp produced at the BCSP and the possibility of their acquisition for industrial operation [25]. The spinning method is revealed to be used more often due to ease of operation and process control, efficiency and obtaining precipitation with low moisture content. Especially is for sewage treatment of industrial enterprises. However, it is not applied at the non-ferrous industry plants of Kazakhstan. The enterprises of far and near abroad produce for about 3,000 types of centrifuges of different productivity.

An objective is to find the optimal conditions for pulp spinning of CSA of BCSP. Technological studies were carried out taking into account the properties of the pulp and process parameters. In particular, there are the amount of slurry and amorphous selenium in the pulp, the speed and time of rotation of the centrifuge rotor.

Experiments. Materials. Slurry obtained from BCSP pulp according to the scheme: heating - filtration - washing with water from sulfuric acid - drying at 105 °C. There is metallic selenium of the CT-0 grade, sulfuric acid of the HG qualification, nitric acid of the HG qualification, tin (II) chloride of the analytical grade qualification. Amorphous selenium obtained from a metal according to the method described in the book [26]. Model sulphate solutions of pulp with different content of slurry (0.1-14 g/dm³) and component selenium of red modification (0.03; 0.1; 0.2 g/dm³) at a constant concentration of sulfuric acid (50 g/dm³).

Amorphous selenium was obtained by dissolving the metal in concentrated nitric acid to form selenous acid, followed by its recovery with a solution of tin chloride. The dissolution and recovery operations were carried out in a temperature-controlled cell with stirring using a mechanical mixer with an adjustable speed, selenium was released in a Dastan RS-6MC centrifuge of Kyrgyz production (Figure 1b).



a - a cell for obtaining selenous acid and obtaining amorphous selenium from it, b - a centrifuge for extracting amorphous selenium from pulp

Figure 1 - Laboratory setup to obtain amorphous selenium from metal

Amorphous Selenium Production Method. Five grams of pre-grinded metallic selenium (Figure 2a) was dissolved in 0.23 dm³ of concentrated nitric acid with stirring. A solution of emerald color was received (Figure 2b). Then, from a separating funnel, the calculated amount of 10 wt. % of SnCl₂

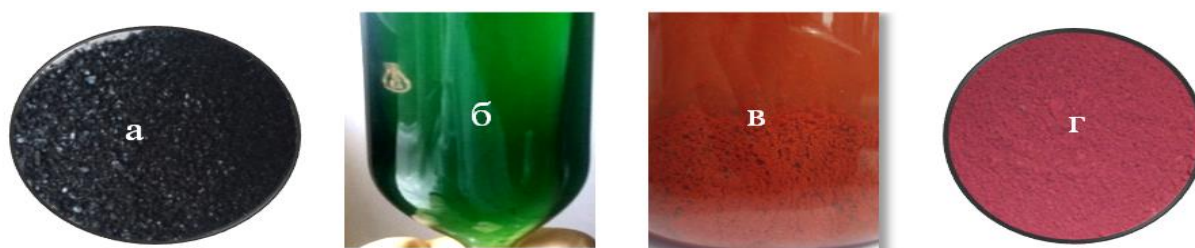


Figure 2 - Metallic technical selenium look (a), selenous acid solution (b), selenium precipitated from the selenous acid solution (c) and dry sediment of amorphous selenium (g)



Figure 3 - The original model pulp look with a slurry content of 0.1 - 14 g/dm³

solution was added. After it was added, a light suspension of red color appeared. As the amount of Sn Cl₂ solution increased, the amount of suspension increased, and the color intensity of the solution decreased. Tin chloride was applied until completely discolored solution and the red suspension fall has ceased. A bulky residue of amorphous selenium (Figure 2c) was isolated from the pulp by spinning. The mother liquor was elutriated from the cell, distilled water was poured in to wash the residue from the solution, and all was thoroughly mixed. The phases were separated by spinning. The residue was dried at atmospheric temperature. The dry sediment was grinded in a porcelain mortar (Figure 2d).

Results and discussion. The slurry content. Figure 3 shows the model pulp solutions look with a slurry content of 0.1; 0.5; 1; 2; 4; 6; 8; 10; 12; 14 g/dm³. Slurry weights are due to its actual content in the production pulp of CSA of BCSP.

Conditions for the experiments: selenium content - 70 mg/dm³, sulfuric acid - 50 g/dm³, solution volume - 0.2 dm³, centrifuge rotor speed - 2000 rpm, spinning time - 10 min.

Figure 3 shows that with an increase in the slurry content, the color of the pulp changes from light pink to dark grey. Under the conditions of the experiments, all types of pulp were divided into solution and residue. Solutions are colorless transparent, which indicates a complete transition of slurry and amorphous selenium in the sediment. Unfortunately, it was not possible to provide photographs of the cell after spinning the pulp because of the cell construction material.

They are made of white thick polymer material. Therefore, the sediments were photographed from the top of the cell. The amount of sediment at the bottom of the cell increases with increasing slurry in the pulp. The sediments are represented by the two layers: the bottom is dark, the top changes from pink to dark grey with black blotches. Figure 4 shows photographs of sediments with minimum and maximum slurry contents in the pulp (top view). In the first case, the top layer of sediment is red, in the second - dark, since the amount of selenium is very small, therefore its particles are entangled in the slurry.

Selenium content. The selenium content in the pulp was 0.03; 0.1; 0.2 g/dm³.

Conditions of the experiments are the slurry content - 1 g/dm³, sulfuric acid - 50 g/dm³, solution amount - 0.2 dm³, rotor speed - 2000 rpm, spinning time - 10 minutes. And in this case, the pulp was divided into two phases, similar to the previous series of experiments. However, the top layer of sediments in all experiments is red, the thickness of which is visually different. The photographs of residues are similar to the photographs of Figure 4a, so they are not provided.

Rotor speed and spinning time. The effect of these parameters was studied when the slurry content in the pulp was 1 g / dm³ and selenium was 0.03; 0.1; 0.2 g/dm³. The emphasis on the selenium content is made because of the lower rate of its release from the pulp, due to its amorphous state and lower specific weight compared with lead sulfate - the main component of the slurry.

Table 3 - Effect of rotor speed and spinning time on pulp separation

Spinning parameters		Selenium content, g / dm ³		
Rotation r/min	Time, min	0,03	0,1	0,2
		Solution specification		
500	2	Pale-pink	Intense pale-pink	dull, pink
	4	dull, colourless	Pale-pink	dull, Pale-pink tint
	6	dull	Pale-pink	dull, Pale-pink tint
	8	dull	Pale-pink	faint pink tint
	10	dull	dull, pink tint	faint pink tint
1000	2	dull, pink tint	dull, pink tint	dull, white-pink tint
	4	dull	dull	dull
	6	dull	dull	dull
	8	dull	dull	dull
	10	dull	dull	dull
1500	2	dull	dull	dull
	4	dull	dull	dull
	6	transparent, colourless	transparent, colourless	transparent, colourless

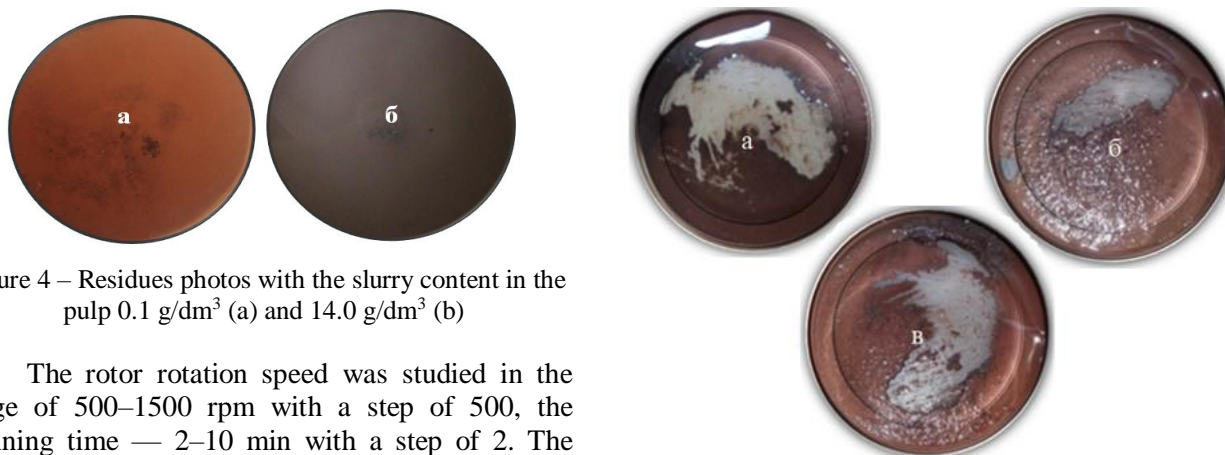


Figure 4 – Residues photos with the slurry content in the pulp 0.1 g/dm³ (a) and 14.0 g/dm³ (b)

The rotor rotation speed was studied in the range of 500–1500 rpm with a step of 500, the spinning time — 2–10 min with a step of 2. The process was controlled by turbidity and solution color. The results are provided in Table 3

As can be seen from Table 3, the main factor affecting the separation of the pulp into phases is the speed of rotation of the rotor. An increase in the rotor speed above 1000 rpm has a positive effect on both the spinning time and the separation of the pulp into phases, regardless of the selenium content in it. So when the number of rotations equals to 500 and 1000 rpm./min, complete separation of the pulp into phases is not achieved even after 10 min for any content of selenium in the pulp. As evidenced by the state of the solutions: dull, painted in pink colors of various shades. A complete separation of the pulp is achieved at a rotor speed of 1500 rpm. The separation into phases occurred within 6 minutes at any content of selenium with obtaining residues of various colors (Figure 5). The color of residues depends on the content of selenium in the pulp. So, when its content in the pulp is 0.03 g/dm³, the sediment is dark brown; at 0.1 and 0.2 g/dm³ -

Figure 5 - Residues obtained at a rotation speed of 1500 rpm and the content of amorphous selenium in the pulp, g/dm³: 0.03 (a), 0.1 (b) 0.2 (c)

the red color prevails. (White spots on the photos - the bottom of the ditch). (White spots on the photos - the bottom of the ditch).

Considering the volume of pulp discharged to the BCSP of 30–60 m³/h and the selected spinning conditions, a centrifuge with a capacity of 35 m³/h was chosen, the drive power - 45 kW/h, 45 kW. According to the calculations for the separation of 1 m³ of pulp with obtaining a clear solution and slurry sediment, the energy consumption was 1.3 kW, in monetary terms (when the cost of electricity is 10 tenge per 1 kW) is 13 tenge. In the case of pulp heating and filtration (at the same cost) is 143 tenge. Thus, at the same cost of 1 kW of electricity, the method of pulp spinning is more than 10 times more efficient than the method of heating - filtering.

Conclusions. The main factor affecting the separation of the pulp into phases with obtaining a clear solution and sediment of all components of the slurry, in particular amorphous selenium, by spinning is the speed of rotation of the rotor. Its increase leads to a separation period reduction of the pulp into phases. Preliminary calculations have shown that the implementation of this method will substantially reduce costs compared to the previously recommended method of heating-filtering of pulp.

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