

# KIMYO VA KIMYOVIY TEHNOLOGIYA

UDC 620.197.3

## STUDYING THE INFLUENCE OF CERTAIN FACTORS ON THE EFFECTIVENESS OF INHIBITING THE SCALE OF INHIBITORS IN THE WATERS OF THE KARAKALPAK REPUBLIC

G.R. Kaipbergenova<sup>1</sup>, H.H. Saitmuratova<sup>1</sup>, Kh.I. Kadirov<sup>1</sup>

<sup>1</sup>Tashkent Institute of Chemical Technology

**Annotatsiya.** Hozirgi vaqtda korroziyadan himoya qilish va mineral tuzlarning choʻkishini oldini olish uchun oltmish mingdan ortiq turdagi reagentlar taklif qilingan, ulardan faqatgina 7 foizi sanoat miqyosida ishlab chiqarilmoqda. Mahalliy xomashyo va sanoat ingibitorlari asosida arzon va sinergetik jihatdan samarali ingibitorlarni tayyorlash dolzarb vazifadir.

Mazkur ishda IOMS-1, Option, Zn-OEDP, "Nalko-2000" kabi sanoat ingibitorlari va oksietilidendifosfon kislotasi rux kompleksoni, karbamid-formaldegid qatroni va monoetanning vakuumda haydash kub qoldigʻidan iborat tarkib taqqoslanadi. 4-14 mg/l konsentrasiyalar chegarasida tavsiya etilgan kompozitsion tarkibning, Option va Nalco-2000 sanoat ingibitorlaridan samaradorligi kam emasligini va mos ravishda 95%, 94% va 95% ni tashkil etishi aniqlandi. Bunda, taklif etilayotgan preparat tarkibidagi import qilinayotgan reagentning miqdori uchdan bir qismni tashkil etish va tanlangan kompozitsion tarkib sinergik samarali taʼsir koʻrsatishi taʼkidlangan.

**Abstract.** Nowadays, to protect against corrosion and prevent the deposition of mineral salts, more than sixty thousand types of reagents have been proposed, which only 7% are produced on an industrial scale. The preparation of cheap and synergetically effective scale inhibitors based on mixed raw materials and industrial inhibitors is an actual task.

Comparative study of industrial inhibitors IOMS-1, Option, Zn-HEDP, "Nalco-2000" with inhibitory compounds consisting of the zinc complex of hydroxyethylidene diphosphonic acid (Zn-HEDP), carbide-formaldehyde resin, and vacuum distillation residue of monoethanolamine, have been proposed. The study of the concentration of inhibitors to prevent scaling in the range of 4-14 mg/l found that the proposed composition is not inferior in efficiency to the industrial inhibitors. The effectiveness of these drugs are 95%, 94% and 95%, respectively. At the same time, it is noted that the content of the imported reagent in the composition of the proposed drug is one third and the selected composition has a positive synergetic inhibitory effect.

**Key words:** scale inhibitor, hydroxyethylidene diphosphonic acid, zincate, hardness of water, inhibition efficiency, inhibition mechanism, residue of monoethanolamine, synergism

**Аннотация.** В настоящее время для защиты от коррозии и предотвращения отложения минеральных солей было предложено более шестидесяти тысяч типов реагентов, из которых только 7 % производятся в промышленных масштабах. Приготовление дешевых и синергетически эффективных ингибиторов солеотложения на основе местного сырья и промышленных ингибиторов является актуальной задачей.

В статье сравниваются эффективность промышленных ингибиторов ИОМС-1, Оптион, Zn-ОЭДФ, "Nalco-2000" с ингибирующим составом, состоящим из цинкового комплекса оксиэтилидендифосфоновой кислоты (Zn-ОЭДФ), карбамидно-формальдегидной смолы и кубового остатка вакуумной перегонки моноэтаноламина. Исследованием концентрации ингибиторов для предотвращения солеотложений в интервале 4 - 14 мг/л

установлено, что предлагаемая композиция по эффективности не уступает промышленным ингибиторам Оптион и “Nalco-2000”, эффективность данных препаратов составляет 95,94 и 95% соответственно. При этом отмечается, что содержание импортируемого реагента в составе предлагаемого препарата составляет одну треть и выбранная композиция оказывает положительный синергетический ингибирующий эффект.

Currently, the global production of polidentate compounds has reached the maximum number and amounts to 2.5-3.5 million tons per year. On average, 40% of these reagents produced, about 1.2 million tons, are used to obtain inhibitors of the deposition of mineral salts and corrosion. Widely used corrosion inhibitors Dodicor-4543; Dodicor-4712, Danox C1-252, Sepacorr ts 3201; KI 75w, Danox-CS 102 B and inhibitors of the deposition of mineral salts, such as IOMS-1, HEDP, NTP-3, HELAMIN, etc. The problem of protecting technological equipment from scaling and internal corrosion in water treatment remains important.

The construction of various enterprises in remote areas creates problems in water treatment. A similar problem exists in the Ustyurt Gas Chemical Complex in the Republic of Karakalpakstan.

The composition of the water in the Karakalpak Republic is highly generalized, the hardness of which varies from 7 to 18 mg equivalent / liter (Tables 1 and 2).

To protect against corrosion and prevent the deposition of mineral salts, sixty thousand types of reagents have been proposed, of which only 7% are produced on an industrial scale.

The main manufacturers are US factories: Monsanto Co (Dekvest preparations), Nalco (Visco preparations), Petrolite Corp. (SP preparations) and other German companies Joh Joh Benekiser, Swiss company Esso ( Korexit preparations), the English branch of Ciba - Geigy Ltd, and others.

**Table 1. Chemical composition of water in some regions of the Republic of Karakalpakstan**

№	Object name	The content of components, mg.eq/l							Total salinity mg/l
		Calcium	Magnesium	Total Hardness	Sodium and potassium	Total alkalinity	Sulphates	Chlorides	
1	Nukus	10,2	3,5	17,0	12,6	3,5	11,1	15,0	2298
2	Schukmana i	9,4	3,4	12,8	0,2	3,0	6,0	4,0	800
3	Turtkul	6,1	1,9	8,0	6,6	3,7	6,0	4,2	898
4	Biruni	4,9	3,0	7,9	6,9	3,9	5,0	6,0	720
5	Chimbay	5,6	4,0	9,6	6,5	2,6	8,2	5,3	780

**Table 2. Chemical classification of water in some regions of the Republic of arakalpakstan**

№	Name of the object	Classification by chemical composition				
		Rigidity	General mineralization	According to the predominant anion	According to the predominant cation	According to acc. conc. ions
1	Nukus	Very tough.	Solonov.	Chloride	sodium	III a type
2	Schukmanai	Very tough.	Promoted. mineral.	Sulfate	calcium	-“-
3	Turtkul	Avg tough.	-“-	-“-	sodium	III a type
4	Biruni	-“-	-“-	Chloride	sodium	III a type
5	Chimbay	Tough.	-“-	Sulfate	-“-	III a type

In particular, the product manufactured under the name ISB-1 is widely used, the basis of which is nitrile trimethylphosphonic acid [1]. NTF forms strong complexes with a number of cations and has all the properties of strong acids. The greatest effect is achieved at dosages of 4-5 g/m<sup>3</sup> of treated water.

Based on nitrilotrimethylphosphonic acid (NTP), multicomponent inhibitors “Difalon”, “Inkredol”, “IOMS-1”, and “Sinol IS-001” have also been created [2]. Promising among these inhibitors is "Inkredol." It is a green-yellow liquid, has complex-forming and crystal-destructive properties. In addition to NTP, Inkredol-1 contains additives that increase compatibility with mineralized waters, lower the pour point and reduce corrosion activity. Maximum efficiency is achieved at a dosage of 10-20 g/m<sup>3</sup>, freezing temperature minus 50°C, and corrosion rate 0.3 mm/year.

As a phosphorus-containing reagent, NTP or HEDP or Incredol-1 or SNPCH-5312 or SNPCH-5313 or SNPCH-5314 or SNPCH-5301 and others are used, and sodium nitrate (NaNO<sub>3</sub>) or potassium nitrate is used as an aqueous solution of alkali metal nitrate (KNO<sub>3</sub>) or ammonium nitrate (NH<sub>4</sub>NO<sub>3</sub>) [3].

The ratio of components can be regulated depending on the content of dissolved iron in produced water (Table 1).

**Table 3. The ratio of components depending on the content of iron ions**

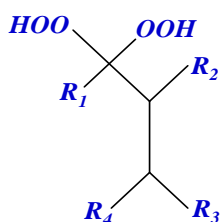
Iron ion content, mg/dm <sup>3</sup>	Ratio of components					
	NTP : KNO <sub>3</sub>	Incredol : NH <sub>4</sub> NO <sub>3</sub>	HEDP : KNO <sub>3</sub>	SNPCH-5321 : NH <sub>4</sub> NO <sub>3</sub>	SNPCH-5313 : NH <sub>4</sub> NO <sub>3</sub>	Incredol: NH <sub>4</sub> NO <sub>3</sub>
5	1 : 1	1 : 1	1 : 1	1 : 1	1 : 1	1 : 1
10	1 : 2	1 : 2	1 : 2	1 : 2	1 : 2	1 : 1
15	1 : 3	1 : 2	1 : 3	1 : 3	1 : 3	1 : 2
20	1 : 4	1 : 3	1 : 4	1 : 4	1 : 4	1 : 2

Oxyethylidene diphosphonic acid (HEDP) is an important **preservative** of organophosphonic acids. The component forms stable complexonates of practice with all cations, including alkaline and alkaline-earth metals. Along with the structure, type and number of functional groups, the inhibitory properties of the components also affect the molecular weight, optimal 1000, over 10.

Polyacrylamide (PAA) is an anionic type inhibitor. Its use is based on the ability to form a monomolecular film on the surface to be inhibited, which is a protective barrier for salt deposition. It is recommended to use hydrolyzed PAA with a molecular weight of 10000-20000, the optimal dosage is 10 - 20 g/m<sup>3</sup> of injected water [5].

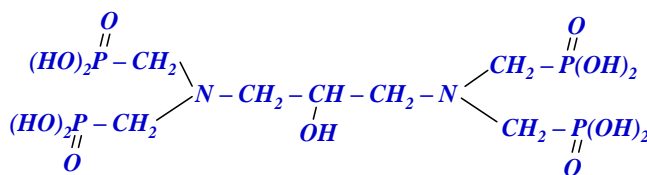
Sodium hexametaphosphate (GMPH) and sodium tripolyphosphate (TPPH) are inhibitors from the class of inorganic polyphosphates [6, 7]. To combat salt deposits, a 1% solution of sodium tripolyphosphate in fresh water in an amount of 20 mg / l was used. It has been established that with the supply of TPPF, the process of scaling is significantly slowed down, but does not completely stop.

It is proposed to use hydrolyzed polyacrylonitrile as inhibitors of inorganic salt deposits obtained by polymerization of acrylonitrile (HIPAN) in the presence of a peroxide initiator at elevated temperature [8], of the general formula:



where R<sup>1</sup> = lower alkyl, R<sup>2</sup> = H or methyl, R<sup>3</sup> = H or lower alkyl, R<sup>4</sup> = H, C<sub>1</sub>-C<sub>4</sub> or -CH<sub>2</sub>C(R<sub>5</sub>R<sub>6</sub>)CH<sub>2</sub>-, in an amount of 0.05-0.5% by weight of acrylonitrile and the process lead at a temperature of 50-60°C in the mass of monomer. It is recommended to use HIPAN with a molecular weight of from 20,000 to 100,000. The optimal dosage is 5-10 g/m<sup>3</sup> of treated water.

2-hydroxy-1,3-diaminopropane-N,N,N',N'-tetramethylene phosphonic acid (DTP-1) is an effective inhibitor, it is unlimitedly soluble in water. Low toxicity, freezing temperature up to minus 100 °C. Designed to prevent deposits of calcium sulfates, barium and calcium carbonates in wells and the productive part of the bottomhole formation zone. Dosages are 5-70 g/m<sup>3</sup> depending on the composition of the inhibited salts [9].



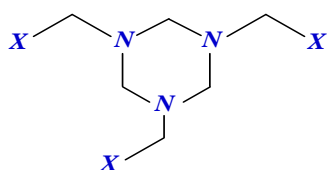
Polyalkylene polyaminopolyoxymethylene phosphonic acid - is the basis for the scale inhibitor PAF-13. R<sub>2</sub>N[(CH<sub>2</sub>)<sub>2</sub>NR]<sub>n</sub>R, R = CH<sub>2</sub>P(O)(OH)(ONa), R<sub>4</sub>=C<sub>4</sub>-C<sub>8</sub>, n=1-3. The preferred dosage of inhibitor to prevent deposition of calcium sulphate is 10 g/m<sup>3</sup> and calcium carbonate is 15 g/m<sup>3</sup> of treated water [10,11].

SP-181 is an imported scale inhibitor, which is a multicomponent composition. The basis of the inhibitor are organic phosphates. The recommended dosage of the reagent is 15-20 g/m<sup>3</sup> [12].

Scale Inhibitor "SP-203 W-P" - is a multicomponent composition, the active principle of which is organic phosphates. The highest dosages are necessary to prevent barium sulfate from 30-50 g/m<sup>3</sup> [13].

Corexide-7647 (K-7647) is a scale inhibitor, which is a multicomponent composition, the active principle of which are water-soluble polymers. Dosages to prevent deposition of calcium sulfate and calcium carbonate are 15 g/m<sup>3</sup>, and barium sulfate is 50 g/m<sup>3</sup> of treated water [14].

Water-soluble polymers based on maleic anhydride, for example, its copolymer with N-alkyl-N-vinylamides or N-vinyl lactams, were used as an inhibitor of calcium phosphate deposits [15].



N-phosphonomethylglycine or its salts N-phosphonomethylglycine in a mixture with chloride salts were used as an inhibitor of the deposition of mineral salts, obtained by hydrolysis of an intermediate product of the interaction of hexahydrotriazine derivatives with triacyl phosphite of the

formula  $P(OCOR^3)_3$ , where  $R^3$  is phenyl.

As an inhibitor of mineral salt deposits, it was proposed to use an inhibitor composition comprising phosphonocarboxylic acid  $((HO)_2P(O)C(R)CH_2COOH)COOH$ , for example, 2-phosphonbutene-1,2,4-tricarboxylic acid [16].

To prevent precipitation, including calcium phosphate, it was proposed to introduce into the water 0.1 - 200 g of an inhibitor consisting of a copolymer of 50 - 90% acrylic, methacrylic acids or their salts, 50 - 60% dialkyl ester of itaconic acid [17].

Uzbek scientists have also synthesized and prepared a number of compositions to prevent scaling in waters of different hardness [18 - 23].

In this work, we compare the efficiency of industrial inhibitors IOMS-1, Option, Zn-OEDP, "Nalco-2000" with our proposed composition consisting of a zinc complex of hydroxyethylidene diphosphonic acid (Zn-HEDP), a carbide-formaldehyde resin and a vacuum distillation residue of monoethanolamine in a ratio of 1 : 1 : 1.

Inhibitor concentrations were studied to prevent scaling in the range of 4-14 mg/l. As shown in table 3, the most effective inhibitors to prevent the formation of deposits of inorganic salts are inhibitors Option, "Nalco-2000" and the proposed composition. The highest effectiveness of these inhibitors in preventing scaling is 95%, 94% and 95%, respectively. With the water of the maximum hardness (Nukus), their effectiveness reaches 90% in concentrations of 12 mg/l, it should be noted that, the content of imported - expensive reagent in the composition of the proposed inhibitor is one third.

Since the increase in the efficiency of inhibition of scaling is insignificant at a concentration above 14 mg/l, further studies were carried out with a concentration of scaling inhibitors of 12 mg/l.

**Table 3. Comparative effectiveness of scaling inhibitor versus its concentration at 80 ° C in five regions of the Karakalpak Republic**

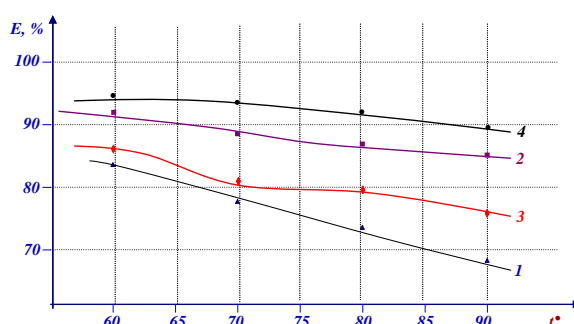
№	Inhibitor	Inhibitor dose, mg/l	Efficiency %				
			Hardness study of water, mg.eq/l				
			Nukus	Schukmanai	Turtkul	Biruni	Chimbay
			<b>17,0</b>	<b>12,8</b>	<b>8,0</b>	<b>7,9</b>	<b>9,6</b>
1.	IOMS-1	4,0	79	82	87	87	84
		6,0	80	83	88	89	86
		8,0	82	85	89	89	88
		10,0	83	88	90	90	89
		12,0	85	90	91	91	90
		14,0	85	90	91	91	90
2.	Option	4,0	83	84	89	90	88
		6,0	84	86	91	92	89
		8,0	86	87	93	93	90
		10,0	88	89	94	94	91
		12,0	89	90	95	95	92
		14,0	89	90	95	95	93
3.	Zn-OEDP	4,0	81	84	86	87	87
		6,0	82	85	88	88	88
		8,0	84	86	89	89	89
		10,0	86	88	90	91	90
		12,0	87	89	91	92	91
		14,0	87	90	92	93	91
4.	"Nalco-	4,0	87	89	91	92	89

	2000''	6,0	88	90	92	93	90
		8,0	89	91	94	94	90
		10,0	90	92	94	95	91
		12,0	91	93	95	95	92
		14,0	91	93	95	95	93
5.	The proposed composition	4,0	85	86	90	90	87
		6,0	86	88	92	91	89
		8,0	87	89	93	93	89
		10,0	89	90	94	94	91
		12,0	90	92	95	95	92
		14,0	91	92	95	95	93

From table 3 it is seen that among industrial scale inhibitors in concentrations up to 14 mg / l the most effective are: IOMS-1 and OEDP to prevent in the waters of Chimbay, Turtkul and Biruni, the formation of deposits of chloride salts of calcium and magnesium, Zn-OEDP and Option in water Schukmanai and Nukus to prevent the formation of calcium sulfate, Option and "Nalco-2000" for deposits of sulfate and chloride of calcium and magnesium. Inhibitors IOMS-1 and Zn-OEDP have low efficiency in the waters of the predominant chloride anions.

Temperature is one of the most important parameters affecting scaling during oil production. It is well known that temperature changes have a significant effect on the solubility of inorganic salts, which in turn affects the rate of formation of salt deposits and their inhibition efficiency.

The studies were carried out in the temperature range from 60 to 90 °C in dynamic conditions. The research results are presented in Fig. 1.



**Fig. 1. The dependence of the effectiveness of the scale inhibitor on temperature at a concentration of 12 mg/l:**

1-IOMS-1; 2- Option; 3-Zn-OEDP; 4 proposed composition

As shown in the figure, an increase in temperature negatively affects the process of inhibition of sedimentation. With increasing temperature, the effectiveness of inhibitors in preventing the formation of calcium carbonate and magnesium sulfate decreases, since the solubility of these salts decreases with increasing temperature. However, the decrease in the effectiveness of the attached drug with an increase in temperature is insignificant, while the effectiveness remains more than 90%. While preventing the deposition of chloride salts, an increase in temperature increases the effectiveness of inhibition, since the solubility of calcium and magnesium chlorides increases with increasing temperature.

In addition to changes in the solubility of salts with increasing temperature, there are other factors that may explain the change in the effectiveness of the inhibition of salts depending on temperature. With increasing water temperature, microcrystalline particles of salts move faster, and the speed of colliding particles increases, as a result, precipitation is accelerated.

The adsorption of scale inhibitors on the surface of salt microparticles is exothermic. Therefore, with increasing system temperature, the degree of adsorption of inhibitors decreases. The kinetics of the deposition of salt crystals is one of the factors affecting the effectiveness of scale inhibitors. In the study, it was assumed that the degree of supersaturation of solutions with ions is constant at all temperatures. Also, temperature affects the rate of precipitation of salt crystals and the degree of supersaturation, since the supersaturation of solutions with ions is considered to be the driving force of salt deposition.

Therefore, the influence of changes in these parameters is mutually compensated. Therefore, with increasing temperature, there is a decrease in the efficiency of inhibition of calcium carbonate and calcium sulfate deposits.

### Conclusion

From the literature review, it is found that polyphosphates (sodium hexametaphosphate, zinc, calcium and nickel heptamethaphosphate, polyphosphate compounds, etc.), nitrilomethyl phosphon or hydroxyethylidene phosphonic acid and their zinc and zinc are mainly produced as inhibitors of the deposition of mineral salts. aluminum salts, thiouryl derivatives (allylthiouryl, N,N'-diethylthiouryl, N,N'-diisopropylthiouryl, phenylthiouryl, diphenylthiourile, ditolultiourel, etc.) 1-octosethyl-1-xanthogenyl in a solvent cyclohexane, preparation you derived ketosulfides.

It is shown that the main factors affecting the process of inorganic salt deposition are: oversaturation with water ions, temperature, pressure, ionic strength, water incompatibility, evaporation, flow condition and hydrogen index.

The efficacy of industrial inhibitors IOMS-1, Option, Zn-HEDP, "Nalco-2000" is compared with inhibitory formulations consisting of the zinc complex of hydroxyethylidene diphosphonic acid (Zn-HEDP), carbide-formaldehyde resin and distillation residue of vacuum distillation of monoethanolamine: 1 in the ratio of 1 in the ratio of 1: 1 1.

A study of the concentration of inhibitors to prevent scaling in the range of 4-14 mg / l found that the proposed composition is not inferior in efficiency to the industrial inhibitors Option and Nalco-2000, the effectiveness of these drugs is 95%, 94% and 95%, respectively. If used in water, the maximum hardness of Nukus, the effectiveness of the proposed reagent reaches 91%. Moreover, it is noted that the content of the imported reagent in the composition of the proposed drug is one third and the selected composition exhibits a positive synergistic inhibitory effect.

### References

1. [https://ru.wikipedia.org/wiki/Nitrilotrimethylphosphonic\\_acid](https://ru.wikipedia.org/wiki/Nitrilotrimethylphosphonic_acid)
2. [yugtehtkom.pulscen.ru/.../31341635-ingibitor\\_oleotlozheni\\_difalon\\_inkredol\\_ioms](http://yugtehtkom.pulscen.ru/.../31341635-ingibitor_oleotlozheni_difalon_inkredol_ioms)
3. Pat. RF 2233376. Pavlychev V.N., Prokshina N.V., Dorofeev S.V., Gabdullin R.F., Safonova E.A., Khasanov F.F., Kalimullin A.A., Faskhutdinov R.A., Safonov E.N., Belyaev V.A. A method for preventing the deposition of mineral salts and iron sulfide. Claim 10/07/2002. publ. 07/27/2004.
4. [www.himprom.com/products/prod/detail.php?ELEMENT\\_ID=2979](http://www.himprom.com/products/prod/detail.php?ELEMENT_ID=2979).
5. Ragulin V. V., Latypov O. A., Mikhailov A. G. Modeling of salt formation during oil production and technologies for its prevention // Scientific and Technical Bulletin of Yukos. 2003, No. 6. / P. 42-47.
6. A.N. Fedenko. The use of sodium polyphosphate in chemical water treatment: features, practice, and economic efficiency. Journal "Heat Supply News", No. 11, (27), November, 2002. P.29-30.
7. Saushkin A.Z., Tokunov V.I. Process fluids and formulations to increase the productivity of oil and gas wells. Edition: Nedra-Business Center LLC.- M ./ 2004. -711 p.



8. Pat. 2393173 of the Russian Federation. The method of producing polyacrylonitrile. Terentyev A.O., Sakharova A.A., Vygodsky Y.S., Platonov M.M., Sapozhnikov D.A., Volkova T.V., Nikishin G.I. Claim 04/04/2008. Publ. 06/27/2010.
9. Ibragimov G.Z., Sorokin V.A., Khisamutdinov N.I. Chemical reagents for oil production. Reference worker. - M.: Nedra, 1986.- 240 p.
10. A.G. Re-purchase, Yu.S. Elizarova. Efficiency and prospects of using multicomponent scaling inhibitor mixtures. // Oil industry. - M.: /, 2003. / S. 82-84.
11. Guidelines for the use of antiscaling agents and corrosion inhibitors OEDF, AFON 200-60A, AFON 230-23A, PAF-13A, IOMS-1 and their analogues, tested and certified by RAO UES of Russia, at power plants. CO 34.37.536-2004. - 67 p.
12. Persiyantsev M.N. Oil production in difficult conditions. "Nedra business center." 2000.- 655 s. ISBN 5-8365-0052-5.
13. Pat. 4869845 (USA). Composition for water treatment. Declared 12.10.88; Publ. 09/26/89. RZhKhim 1989.16 I380.
14. A method of inhibiting salt deposition processes on the surfaces of water treatment equipment. Scale inhibition adentand method for usind some: U.S. Patent 6162391, IPC7 CO2F5 / 10, 09K 3/00. Kuritf Water Ind, Kowata Ken Ji, Limura Akira, Sato Shideru, Usui Urara, Sanaduchi Aya. No. 09/083007; Claim 05/21/1998; publ. 12/19/2000; Prior. 05/23/1997, No. 9 - 150367 (Japan); NPK 422/16 Eng.
15. V.N. Kizhnyayev, M.B. Astakhov, E.I. Biryukova. The effect of intermolecular interaction of monomers on the copolymerization of 1-vinylbenzimidazole with maleic acid // High molecular weight. connections. Ser. B. - 1992. -/ T.; 34, No. 10. S. 4-18.
16. Tsukanova T.V. The use of complex compounds in the preparation of additional water to optimize the water-chemical regime of boilers and heat supply systems. Abstract of dissertation for the degree of candidate of technical sciences.-M.:, 2007. - 27 p.
17. Zhon Zuo-mind, Jind Guan-hua, Tong Shou-yin, Dai Uon-Zhi. Jindx huadond zhondjianti. The manufacture of scale inhibitors adapted to high rigidity and high alkalinity. // Fine chem. Intermediates 2001. No. 4. P.21 - 24.
18. Kim F.O., Kadirov Kh.I., Mukhitdinov Kh.D. Condensation of urea and thiourea with formaldehyde. Reports of the Academy of Sciences of the Republic of Uzbekistan, 2003. No. 3; -/P. 56-59.
19. Kadirov H., Azamatov U., Turabjanov S.M. The initiator ishtirokida complexonlate synthesis. Uzbek chemical journal. 2014, No.6. From 7-11.
20. Kadirov H., Azamatov U., Turabjanov S., Ikramov A. Hydroxyethylidene diphosphon acid zincate asoside composite inhibitor olish. Chemistry and chemical technology. 2015. No. 3. S. 15-19.
21. Kadirov H., Azamatov U., Turabjanov S.M. New compositions of corrosion and scaling inhibitors. Composite materials, 2015; No.2. S. 53-57.
22. H.I. Kadirov, Z.S. Vohidov, T. Rizaev, U.R. Azamatov, A. Ikramov. Comparative tests of organophosphonate scale inhibitors. DAN RUZ.; 2015, No. 3. S.48-51.
23. Kadirov Khasan Irgashevich, Turabjanov Sadritdin Mahamatdinovich. Synthesis of zincate-oxyethenediphosphonic acid and the comparative results applying as scale inhibitors. Europäische Fachhochschule. European Applied Sciences. Stuttgart, Germany. ISSN 2195-2183. # 6. - / 2015.S. 66-69.



# PROBLEMS OF COEXISTENCE OF SUPERCONDUCTIVITY AND MAGNETIC ORDERING OF COPPER SUBLATTICES IN $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7-x}$ CERAMICS

Q.Kh. Bobomurodov<sup>1</sup>, O.Kh. Babakhanov<sup>1</sup>, Sh.S. Zamonova<sup>1</sup>, M.R. Sattorov<sup>1</sup>,  
S.Q. Bobomurodov<sup>2</sup>, R.A. Shokirov<sup>2</sup>

<sup>1</sup>Termez State University, <sup>2</sup>Tashkent State Technical University

**Annotatsiya.** Maqolada  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7-x}$  keramikasining mislik panjaralarida o'ta o'tkazuvchanlik va magnit tartiblanishlarining birgalikda hosil bo'lish muammolari ko'rib chiqilgan.

Ma'lumki, o'ta o'tkazuvchanlik ortorombik panjarali  $\text{YBa}_2\text{Cu}_3\text{O}_{7-z}$  keramikada, kislorod miqdori pasaygan sari, o'ta o'tkazuvchanlik holatiga o'tish harorati pasayadi va  $z > 0,6$  da o'ta o'tkazuvchanlik y o'qoladi, panjara esa tetragonal panjara bo'lib qoladi va shu bilan birga bir vaqtning o'zida,  $\text{Cu}(2)$  panjarasida antiferromagnetik tizim hosil bo'ladi.  $\text{YBa}_2\text{Cu}_3\text{O}_7$  keramikasida temir atomlari bilan mis atomlari bilan almashtirish (ya'ni  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7+y}$  ning qattiq eritmasining hosil bo'lishi) shunga o'xshash jarayonlar kuzatiladi:  $x$  ortib borganda,  $T_c$  pasayganda,  $x > 0,05$  ortorombik panjara tetragonal panjaraga aylanadi,  $x > 0,45$  bo'lganda o'ta o'tkazuvchanlik yo'qoladi. Eng diqqatga sazovar tomoni shundaki, mis maydonlaridagi temir atomlarining o'ta o'tkazuvchanligi va magnit tartiblanishi  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7+y}$  keramika tarkibida  $x$  ning shu sohalarida  $0,03 < x < 0,45$  qiymatlarida (ikkinchisi  $^{57}\text{Fe}$  izotopida Messbauer spektroskopiyasi usuli bilan aniqlangan).  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7+y}$  panjarasidagi temir atomlarining magnitlanganligi mis atomlarining magnit tartibiga bog'liqligi aniq emas.

**Abstract.** The article studies the problems of coexistence of superconductivity and magnetic ordering of copper sublattices in  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7-x}$  ceramics.

It is known that in superconducting ceramics  $\text{YBa}_2\text{Cu}_3\text{O}_{7-z}$  with orthorhombic lattice as the oxygen content decreases, the transition temperature in superconducting state  $T_c$  decreases, and at  $z > 0.6$  superconductivity disappears, the lattice becomes tetragonal and at the same time, antiferromagnetic ordering of sublattices  $\text{Cu}(2)$  appears. The substitution in ceramics of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  part of copper atoms by iron atoms (i.e., the formation of a solid solution of  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7+y}$ ) is accompanied by similar effects: as  $x$  increases,  $T_c$  decreases, at  $x > 0.05$  the orthorhombic lattice becomes tetragonal, at  $x > 0.45$  the superconductivity disappears. The most significant moment is the fact of coexistence in ceramics  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7+y}$  in the region of compositions  $0.03 < x < 0.45$  of superconductivity and magnetic ordering of iron atoms in copper nodes (the latter is established by Mossbauer spectroscopy on isotope  $^{57}\text{Fe}$  in a large number of works. However, it remains unclear whether the magnetic ordering of iron atoms in the  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7+y}$  lattice is related to the magnetic ordering of copper atoms.

**Key words:** Mossbauer spectroscopy, electric field gradient, semiconductor ceramics, orthorhombic, copper node.

**Аннотация.** В статье предложено решение проблемы сосуществования сверхпроводимости и магнитного упорядочения подрешеток меди в керамиках  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7-x}$ . Известно, что в сверхпроводящей керамике  $\text{YBa}_2\text{Cu}_3\text{O}_{7-z}$  с орторомбической

решеткой по мере уменьшения содержания кислорода происходит уменьшение температуры перехода в сверхпроводящее состояние  $T_c$ , и при  $z > 0,6$  сверхпроводимость исчезает, решетка становится тетрагональной и одновременно появляется антиферромагнитное упорядочение подрешетки Cu (2). Замещение в керамике  $YBa_2Cu_3O_{7-y}$  части атомов меди атомами железа (т. е. образование твердого раствора  $YBa_2Cu_{3-x}Fe_xO_{7+y}$ ) сопровождается аналогичными эффектами: по мере возрастания  $x$  уменьшается  $T_c$ , при  $x > 0,05$ , происходит перестройка орторомбической решетки в тетрагональную, при  $x > 0,45$  сверхпроводимость исчезает. Наиболее существенным является факт сосуществования в керамике  $YBa_2Cu_{3-x}Fe_xO_{7+y}$  в области составов  $0,03 < x < 0,45$  сверхпроводимости и магнитного упорядочения атомов железа в узлах меди (последнее установлено методом мессбауэровской спектроскопии на изотопе  $^{57}Fe$  в большом числе работ). Однако остается неясным, связано ли магнитное упорядочение атомов железа в решетке  $YBa_2Cu_{3-x}Fe_xO_{7+y}$  с магнитным упорядочением атомов меди.

It is known that in superconducting ceramics  $YBa_2Cu_3O_{7-z}$  with orthorhombic lattice as oxygen content decreases, the temperature of transition in superconducting state  $T_c$  decreases, and at  $z > 0.6$  superconductivity disappears, the lattice becomes tetragonal and at the same time, antiferromagnetic ordering of the Cu (2) sublattices appears [1,2]. The substitution in ceramics of  $YBa_2Cu_3O_7$  part of copper atoms by iron atoms (i.e., the formation of a solid solution of  $YBa_2Cu_{3-x}Fe_xO_{7+y}$ ) is accompanied by similar effects: as  $x$  increases,  $T_c$  decreases, at  $x > 0.05$  the orthorhombic lattice becomes tetragonal, at  $x > 0.45$  the superconductivity disappears [3]. The most significant moment is the fact of coexistence in the ceramic  $YBa_2Cu_{3-x}Fe_xO_{7+y}$  in the region of compositions  $0.03 < x < 0.45$  of superconductivity and magnetic ordering of iron atoms in copper nodes (the latter was established by Mossbauer spectroscopy on isotope  $^{57}Fe$  in a large number of papers [4-7]. However, it remains unclear whether the magnetic ordering of iron atoms in the  $YBa_2Cu_{3-x}Fe_xO_{7+y}$  lattice is related to the magnetic ordering of copper atoms (see, for example, [8]).

To solve this problem, it seems promising to use emission Mossbauer spectroscopy on isotope  $^{61}Cu$  ( $^{61}Ni$ ): after the decay of the mother nucleus  $^{61}Cu$ , the Mossbauer probe  $^{61}Ni$  is formed in the copper node, whose nuclear parameters allow reliable recording of magnetic ordering in copper nodes [9]. Two pairs of samples were used for investigations:  $YBa_2Cu_3O_{6.96}$  (orthorhombic modification,  $T_c = 92$  K),  $YBa_2Cu_3O_{6.1}$  (tetragonal modification,  $T_c < 4.2$  K) and  $YBa_2Cu_{2.8}Fe_{0.2}O_{7.03}$  (tetragonal modification,  $T_c = 50$  K),  $YBa_2Cu_{2.5}Fe_{0.5}O_{7.18}$  (tetragonal modification,  $T_c < 4.2$  K)..

Samples of  $YBa_2Cu_3O_{6.96}$  and  $YBa_2Cu_{3-x}Fe_xO_{7+y}$  were prepared by high-temperature solid phase synthesis.  $Y_2O_3$ ,  $CuO$ ,  $Fe_2O_3$  (enrichment by isotope  $^{57}Fe$  was 92 %) and  $BaCO_3$  were used as components. After sintering at  $900^\circ C$  for 20h in the air, the samples were annealed

in an oxygen current at 920°C for 70 "s with subsequent cooling at a rate of 5 K/min. The annealing of the sample  $\text{YBa}_2\text{Cu}_3\text{O}_{6.96}$  at 800°C for 2 h with constant pumping resulted in  $\text{YBa}_2\text{Cu}_3\text{O}_{6.1}$ .

Samples were doped with  $^{61}\text{Cu}$  by diffusion annealing at 450°C for 30 min in oxygen current (except for sample  $\text{YBa}_2\text{Cu}_3\text{O}_{6.1}$ , which was doped by diffusion annealing at 650°C for 30 min during pumping). No changes in structure,  $T_c$  value or oxygen content were observed for control samples. According to [10], the described procedure guarantees that the  $^{61}\text{Cu}$  isotope enters the copper nodes of the lattice.

The  $^{61}\text{Cu}$  ( $^{61}\text{Ni}$ ) Mossbauer emission spectra were shot at 80 and 4.2 K on an industrial spectrometer, the standard absorber was  $\text{Ni}_{0.86}\text{V}_{0.14}$  with a surface density of 1500 mg/cm<sup>2</sup>. Typical spectra are shown in Fig. 1.

In the Y system, copper atoms occupy two crystallography non-equivalent positions Cu (1) and Cu (2), populated as 1:2. In accordance with this, we represented the experimental Mossbauer spectra of  $^{61}\text{Cu}$  ( $^{61}\text{Ni}$ ) of the above ceramics as an overlay of two multiplets corresponding to the  $^{61}\text{Ni}^{2+}$  centers in Cu (1) and Cu (2) nodes. Each multiplet was described by a superposition of either five lines with relative intensities 10 : 4 : 1 : 6 : 9 (in the case of a pure quadrupole interaction), or twelve lines with relative intensities 10 : 4 : 1 : 6 : 6 : 3 : 3 : 6 : 6 : 1 : 4 : 10 (in the case of the combined quadrupole and Zeeman interactions), and the position of the multiplet lines was determined as the difference in the eigenvalues  $E_m^I$  of the Hamiltonian of the combined superfine interaction of the excited and the main states  $^{61}\text{Ni}$

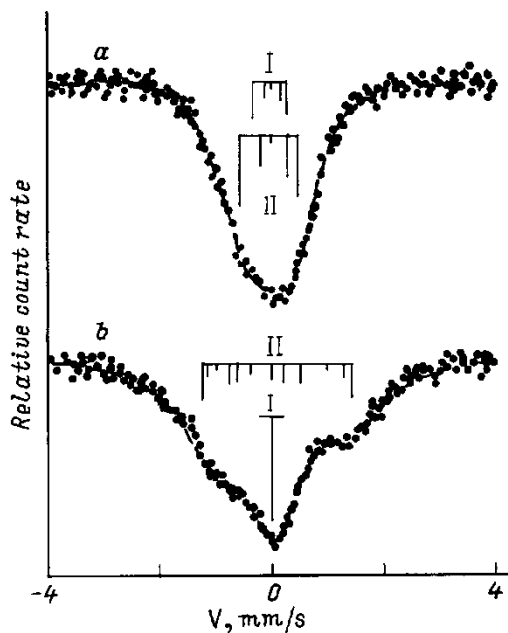
$$E_m^I = mg\beta_N H + \{eQU_{zz}/4I(2I - 1)\} \times \{3m^2 - I(I + 1)\} \{(3\cos^2\theta - 1)/2\},$$

where  $I$  is the spin of the nucleus,  $H$  is the magnetic field on the nucleus,  $U_{zz}$  is the main component of the electric field gradient (EFG) tensor on the nucleus,  $\theta$ — is the angle between the main axis of the EFG tensor and the direction of the magnetic field,  $m$  is the magnetic quantum number,  $Q$  is the quadrupole moment of the nucleus,  $g$  is the nuclear  $g$  is factor,  $\beta_N$  is the nuclear magneton. The above formula is valid for the axially symmetric EFG tensor both for  $gH \gg eQU_{zz}$ , and for  $H = 0$  (but in the latter case we should take ( $\theta = 0^\circ$ )).

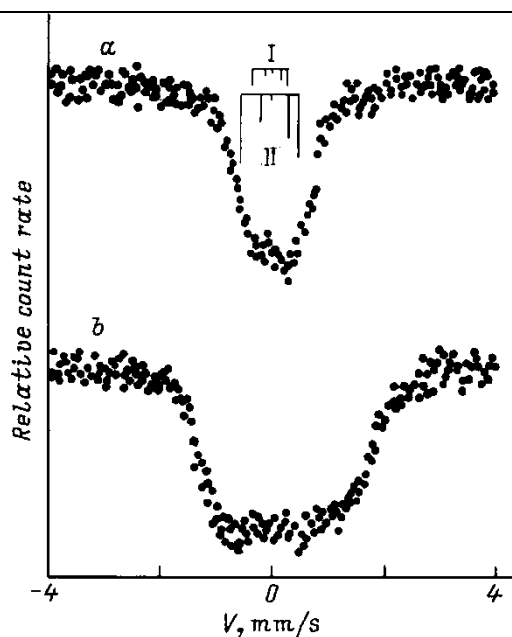
The computational spectrum was adjusted to the experimental method of least squares, and the fitting parameters were not the parameters of individual lines, but the Hamiltonian parameters  $H$  and  $U_{zz}\{(3\cos^2\theta - 1)/2\}$ , as well as the positions of the centers of gravity of the multiplets. Since no isomer shift is observed in the Mossbauer  $^{61}\text{Ni}$  spectra [11], we made sure that the center of gravity of the calculated multiplet does not deviate from zero speed by more than  $\pm 0.05$  mm/s.

Mossbauer spectrum  $^{61}\text{Cu}$  ( $^{61}\text{Ni}$ ) of superconducting ceramic  $\text{YBa}_2\text{Cu}_3\text{O}_{6.96}$  is a superposition of two quadrupole multiplets corresponding to the centers of gravity of  $^{61}\text{Ni}$  (1) and  $^{61}\text{Ni}$  (2). Fig. 1 indicates the positions of the components of the corresponding multiplets and their relative intensities. The ratio of areas under the spectra  $^{61}\text{Ni}$  (2) and  $^{61}\text{Ni}$  (1)  $P=1.95$  (5), which is close to the population ratio of nodes Cu (2) and Cu (1). The obtained parameters of the spectra are as follows:  $eQU_{zz} = 32$  (2) MHz for centers  $^{61}\text{Ni}$  (1) and  $eQU_{zz} = -54$  (2) MHz for spectra  $^{61}\text{Ni}$  (2) (here  $Q$  is the quadrupole moment of the  $^{61}\text{Ni}$  core in the main state).

Mossbauer spectrum of  $^{61}\text{Cu}$  ( $^{61}\text{Ni}$ ) semiconductor ceramics  $\text{YBa}_2\text{Cu}_3\text{O}_{6.1}$  (Fig. 1, b) is a superposition of a quadrupole multiplets corresponding to the  $^{61}\text{Ni}^{2+}$  nodes Cu (1) ( $|eQU_{zz}| < 30\text{MHz}$ ), and a multiplet corresponding to the  $^{61}\text{Ni}^{2+}$  nodes Cu (2). And the fine structure of the last spectrum is obliged by the origin to the combined superfine (Zeeman and electric quadrupole) interaction ( $eQU_{zz} = -48$  (3) MHz,  $H = 85$  (5) kOe,  $\theta = 90$  (10) $^\circ$ ). The ratio of areas under the spectra  $^{61}\text{Ni}$  (2) and  $^{61}\text{Ni}$  (1) remains equal to the ratio of the population of nodes Cu (2) and Cu (1) in the lattice  $\text{YBa}_2\text{Cu}_3\text{O}_7$  ( $P=1.97$  (5)). The spectra in Fig. 1 illustrate the possibilities of Mossbauer emission spectroscopy at the isotope  $^{61}\text{Cu}$  ( $^{61}\text{Ni}$ ) to observe the magnetic ordering of the copper sublattice of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-z}$  ceramics with a decrease in oxygen content.



**Fig.1. Mossbauer emission spectra of  $^{61}\text{Cu}$  ( $^{61}\text{Ni}$ ) at 80 K for  $\text{YBa}_2\text{Cu}_3\text{O}_{6.96}$  (a) and  $\text{YBa}_2\text{Cu}_3\text{O}_{6.1}$  (b) ceramics. The position of multiplets components corresponding to  $^{61}\text{Ni}^{2+}$  centers in Cu (1) (I) and Cu (2) (II) nodes is indicated.**



**Fig.2. Mossbauer emission spectra of  $^{61}\text{Cu}$  ( $^{61}\text{Ni}$ ) at 4.2K for  $\text{YBa}_2\text{Cu}_{2.8}\text{Fe}_{0.2}\text{O}_{7.03}$  (a) and  $\text{YBa}_2\text{Cu}_{2.5}\text{Fe}_{0.5}\text{O}_{7.18}$  (b) ceramics. For spectrum a, the position of the multiplets components corresponding to centers  $^{61}\text{Ni}^{2+}$  in nodes Cu (1) (I) and Cu (2) (II) is indicated.**

The Mossbauer spectrum of  $^{61}\text{Cu}$  ( $^{61}\text{Ni}$ ) superconducting ceramics  $\text{YBa}_2\text{Cu}_{2.8}\text{Fe}_{0.2}\text{O}_{7.03}$  is a superposition of two quadrupole multiplets (Fig. 2, a), whose parameters are close to the parameters of the corresponding spectra of the ceramics  $\text{YBa}_2\text{Cu}_3\text{O}_{6.96}$ , although the ratio of areas under the  $^{61}\text{Ni}$  (2) and  $^{61}\text{Ni}$  (1) spectra differs significantly from the expected value ( $P=4.0$  (4)). This is obviously due both to a decrease in the proportion of Cu (1) centers (due to partial substitution of a part of Cu (1) nodes by iron impurity atoms) and to the influence of iron impurity atoms on the parameters of the Mossbauer  $^{61}\text{Ni}$  spectra (which decreases the proportion of the undisturbed spectrum from  $^{61}\text{Ni}$  (1) atoms).

For ceramics  $\text{YBa}_2\text{Cu}_{2.5}\text{Fe}_{0.5}\text{O}_{7.18}$ , in which superconductivity is suppressed, a Zeeman splitting is observed in the  $^{61}\text{Cu}$  ( $^{61}\text{Ni}$ ) Mossbauer spectra (Fig. 2, b). Unfortunately, the resolution of the last ceramics spectra was insufficient for the extraction of components corresponding to  $^{61}\text{Ni}^{2+}$  centers in Cu (1) and Cu (2) from experimental spectra. So, as in the case of ceramics  $\text{YBa}_2\text{Cu}_3\text{O}_{7-z}$ , for ceramics  $\text{YBa}_2\text{Cu}_{3-x}\text{Fe}_x\text{O}_{7+y}$  there is an obvious correlation between the appearance of magnetic ordering of one of the copper sublattice and disappearance of the superconductivity phenomenon.

For ceramics  $\text{YBa}_2\text{Cu}_{2.8}\text{Fe}_{0.2}\text{O}_{7.03}$  the Mossbauer spectra  $^{57}\text{Fe}$  were also measured ( $^{57}\text{Co}$  in palladium was used as a standard source). In agreement with the literature data at  $T < 50$  K, the spectra are poorly resolved Zeeman multiplets corresponding to impurity iron atoms in nodes Cu (1) in the "spin glass" state. Thus, we must state that there is no correlation between the magnetic

ordering of impurity iron atoms in sublattice Cu (1) and the magnetic ordering of copper sublattice ceramics  $\text{YBa}_2\text{Cu}_{2.8}\text{Fe}_{0.2}\text{O}_{7.03}$ . However, the increase in iron concentration (transition to  $\text{YBa}_2\text{Cu}_{2.5}\text{Fe}_{0.5}\text{O}_{7.18}$ ) is accompanied by both the complete suppression of superconductivity and the appearance of magnetic ordering of copper sublattice. Since in the latter ceramics a part of iron atoms is stabilized in the Cu (2) sublattice, it is obvious that the appearance of the magnetic ordering of the copper sublattice should be associated with these iron atoms.

### Reference

1. Jorgensen J.D., Veal B.W., Paulikas A.P., Nowicki L.J., Grabtree G.W., Clans H., Kwok. phys W.K. Rev. B41, 1863 (1990). 43-46 P.
2. Yasuoka H., Shimizu T., Imai T., Sasaki S., Ueda Y., Kosuge K.. Hyperfine Interact 49,167(1989).12-14 P.
3. Xu Y., Suenaga M., Tafto J., Sabatini R.L., Moodenbaugh A.R., Zoliker P. phys. Rev. B39, 6667(1989). 89-93 P.
4. Qiu Z.Q., Du Y.W., Tang H., Walker J.C. Magn J. Mater. 78, 359(1989). 173-174 P.
5. Tamaki T., Komai T., Ito A., Maeno Y., Fujita T..Solid State Commun. 65, 43(1988). 25-27 P.
6. Suharan S., Chadwick J., Hannon D.B., Janes D.H., Thomas M.F. Solid State Commun. 70, 817 (1989). 62-64 P.
7. Takano M., Hiroi Z., Mazaki H., Bando Y., Takedo Y., Kanno R.. physicaC153/155, 860(1988). 40-43 P.
8. Hechel D., Nowik I., Bauminger E.R., Felner I. phys.Rev. B42, 2166 (1990). 52-58 P.
9. Masterov V.F., Nasredinov F.S., Saidov Ch.S., Seregin P.P., Bondarevskiy S.I., Sherbatyuk O.K. SFXT 5, 1339 (1992). 22-25 P.
10. Masterov V.F., Nasredinov F.S., Saidov Ch.S., Seregin P.P., Sherbatyuk O.K. FTT 34, 7, 2294 (1992). 81-83 P.
11. Love J.C., Obenshain F.E., Czjzek G. phys. Rev. B3, 2837 (1971). 99-101 P.
12. Bobomurodov Q.H, Razakov J.Kh, Bobomurodov S.Q, and Shokirov R.A. METHODS FOR RESEARCHING THE LOCALIZATION AND DELOCALIZATION OF CARRIERS IN  $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$  FILMS, Technical science and innovation: Vol. 2019 : Iss. 4 , Article 7. Available at: <https://uzjournals.edu.uz/btstu/vol2019/iss4/7>
13. Misrikhanov, M. Sh. and Khamidov, Sh.V. MATHEMATICAL POWER FLOW MODEL IN AN ELECTRICAL SYSTEM CONTAINING A SERIAL COMPENSATOR THYRISTOR CONTROLLED REACTIVE COMPONENT, Technical science and innovation: Vol. 2019 : Iss. 3 , Article 5. Available at: <https://uzjournals.edu.uz/btstu/vol2019/iss3/5>
14. Fugol I.Ya., Samovarov V.N.. FNT 22, 11, 1241 (1996). 35-37 P.
15. Salamon D., Abbamante P., Liu R., Klein M., Lee W., Gingsberg D. phys. Rev. B53, 2, 886 (1996). Fizika tverdogo tela, 1997, tom 39, № 10, 118-120 P.
16. Dmitriev V.M., Yeremenko V.V., Kachur I.S., Piryatinskaya V.G., Prihod'ko O.R., Ratner A.M., Kristenko Ye.V., Shapiro V.V. FNT. 21, 2, 219 (1995). 56-59 P.
17. Moeckly B., Lathrop D., Buhrman R. phys. Rev. B47, 1, 400 (1993)Jorgensen J.D., Veal B.W., Paulikas A.P., Nowicki L.J., Grabtree G.W., Clans H., Kwok. phys W.K. Rev. B41, 1863 (1990). 43-46 P.

RESEARCH OF INCREASING THE REACTIVITY COTTON  
CELLULOSE INTENDED FOR CHEMICALR.S. Sayfutdinov<sup>1</sup>, U.D. Mukhitdinov<sup>1</sup>, N.M. Eshpulatov<sup>1</sup><sup>1</sup>Tashkent Institute of Chemical Technology

**Annotatsiya.** Muammoning kelib chiqishi. Kimyoviy ishlov berish jarayonida paxta sellyulozasining reaksiya faolligi boshqa tsellyuloza o'z ichiga olgan o'simliklardan olingan tsellyulozalarga nisbatan ancha past. Paxta sellyulozasining reaksiya faolligini oshirish unga asoslangan mahsulotlar sifatini yaxshilaydi.

Ushbu ishning maqsadi kimyoviy qayta ishlangan paxta sellyulozasining reaksiya faoligini oshirish, olingan mahsulot sifatini yaxshilash va reaksiya tezligini oshirish orqali xorijiy davlatlardan olib kelingan yog'och sellyulozasini mahalliy paxta sellyulozasi bilan almashtirish imkoniyatini o'rganishdir.

Ushbu ishda paxta tsellyulozasi namunalarining tarkibi va tuzilishini aniqlash usullari qo'llanilgan. Elektr zaryadlari bilan ishlov berilgandan so'ng olingan namunalarning atsetilatsiyaga reaksiya faolligi o'rganildi. Reaksiya faollikning ortishi kompyuter tomonidan boshqariladigan XRD-6100 asbobida (Shimadzu, Yaponiya) qayd etilgan diffraksiya naqshlar asosida kristalli hududlarning pasayishi bilan hisoblab chiqilgan. Paxta sellyulozasining yuqori kuchlanishli elektr zaryadlari bilan ishlov berish orqali uning reaksiya faoligini oshirish uchun usul taklif etiladi. Paxta tsellyulozasining kristal maydonini kamaytirish uchun u elektr zaryad bilan faollashtirildi, bu esa paxta tsellyulozasining reaksiya faoligini oshirishga olib keldi. Optimal kuchlanish chegaralari, impulslar soni va kondansatori kerakli sig'imi topiladi, bunda paxta sellyulozasining maksimal reaksiya faolligiga erishiladi.

**Abstract.** Background of the problem. The reactivity of cotton pulp during chemical processing is significantly lower compared to celluloses obtained from other cellulose-containing plants. Increasing the reactivity of cotton cellulose improves the quality of the products based on it, and also increases labor productivity.

The aim of this work is to study the possibility of replacing wood pulp imported from foreign countries with domestic cotton cellulose by increasing the reactivity of cotton cellulose for chemical processing, improving the quality of the resulting product and increasing the reaction rate.

In this work, we used methods for determining the composition and structure of cotton cellulose samples. The reactivity of the obtained samples to acetylation after treatment with electric charges was studied. An increase in reactivity was evaluated by a decrease in crystalline areas based on diffractograms that were recorded on a computer-controlled XRD-6100 apparatus (Shimadzu, Japan). A method is proposed for increasing the reactivity of cotton cellulose by treating it with high voltage electric charges. To reduce the crystalline areas of cotton cellulose, it was activated by an electric charge, which led to an increase in the reactivity of cotton cellulose. The optimal voltage limits are found, the number of pulses and the required capacitance of the capacitor, at which the maximum values of the reactive activity of cotton cellulose are achieved.

**Key words:** Oxygen-alkaline cooking, cotton lint cellulose, electric charge treatment, X-ray diffraction analysis, reactivity, control.

**Аннотация.** В статье рассматривается реакционная способность хлопковой пульпы во время химической обработки значительно ниже по сравнению с целлюлозами, полученными от других целлюлозосодержащих растений. Повышение реакционной



способности хлопковой целлюлозы улучшает качество продуктов на ее основе, а также повышает производительность труда.

Целью данной работы является изучение возможности замены древесной массы, импортируемой из зарубежных стран, на отечественную хлопковую целлюлозу путем повышения её реакционной способности для химической обработки, улучшения качества получаемого продукта и повышения скорости реакции.

В работе использовались методы определения состава и структуры образцов хлопковой целлюлозы. Изучена реакционная способность полученных образцов к ацетилированию после обработки электрическими зарядами. Повышение реактивности оценивали по уменьшению кристаллических областей на основе дифрактограмм, которые были записаны на управляемом компьютером приборе XRD-6100 (Shimadzu, Япония). Предложен способ повышения реакционной способности хлопковой целлюлозы путем обработки ее электрическими зарядами высокого напряжения. Чтобы уменьшить кристаллические площади хлопковой целлюлозы, она активировалась электрическими зарядами, что приводило к увеличению реакционной способности хлопковой целлюлозы. Найдены оптимальные пределы напряжения, количество импульсов и требуемая емкость конденсатора, при которых достигаются максимальные значения реактивности хлопковой целлюлозы.

**Introduction.** Cotton fiber by its nature is crimped, therefore, in dry and wet conditions, they quickly assemble into lumps and nodules, forming flagella and ropes, as well as enveloped with weed impurities and become difficult to clean.

Due to the above specifics, cotton fiber requires additional mechanical processing - chopping or chopping. For grinding fibers, rolls, conical and disk mills are mainly used. [1-3].

A number of works [4-8] provide descriptions of various methods of cleaning lint, cooking and acidification in order to obtain uniformly pure cotton cellulose intended for chemical processing, in particular cellulose ethers.

The reactivity of cotton cellulose during chemical processing is significantly lower than that of other types of cellulose, since its structure consists of crystalline and amorphous sites. Chemical reagents easily react with functional groups in the amorphous region, however, these reagents are difficult to penetrate into crystalline regions. As a result, part of the cellulose enters into chemical reactions, while the other does not. This leads to the shutdown of the production line due to difficulties in passing through the filter (cellulose ethers).

A study of the scientific and technical literature in the field of increasing the reactivity of cotton pulp revealed a number of works aimed at solving this problem. For example, a method has been proposed, the essence of which is as follows: cellulose swollen in water is frozen at a temperature of -15-20 °C, followed by thawing, which ultimately leads to a decrease in crystalline regions in the structure [9]. By treating cotton cellulose with nitrogen containing substances [8, 10–14], as well as by partially esterifying cotton cellulose, a slight increase in the distance between cellulose macromolecules was achieved [15].

In the process of oxygen-alkaline cooking, as well as with other cooking methods, along with the refinement of natural cellulose, its structural changes occur. Research on the changes in the macro- and microstructure of cellulose fiber depending on the cooking conditions is necessary when choosing the optimal mode.

**Methods and materials.** In the work, physicochemical methods were used to determine the quality indicators of cotton pulp obtained under various conditions.

The characteristics of acetylation of cotton pulp were determined by the method proposed by the French company Speyshen, which is determined by multiplying the viscosity by filterability and divided by 1000.

The obtained cellulose samples were processed into triacetates, where the kinetics of acetylation was studied.

The study of changes in the crystalline and amorphous sections of cotton cellulose after treatment with electric charges was carried out by identifying samples based on diffractograms that were recorded on a computer-controlled XRD-6100 apparatus (Shimadzu, Japan).

**Results and discussion.** We have conducted studies on the activation of cotton cellulose by electric charge in order to reduce the crystalline areas that contributed to the decrease in the reactivity of cotton cellulose.

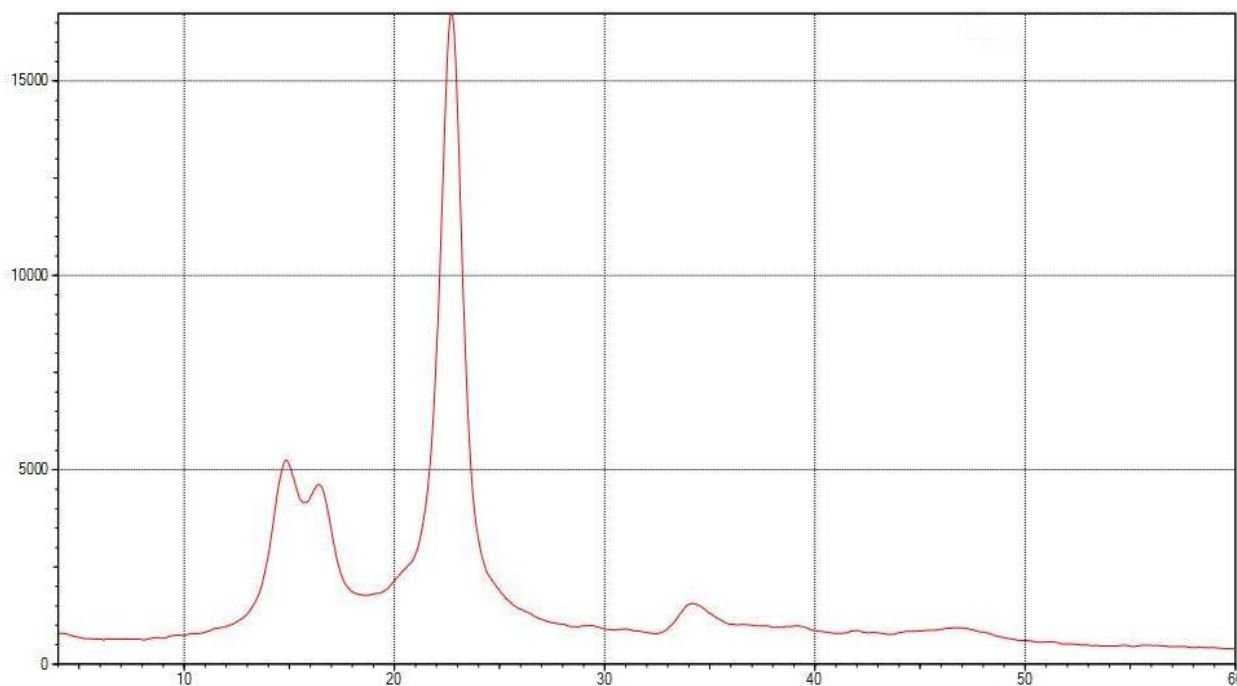
For the study, samples of cotton pulp were prepared without activation control (1), with water treatment (2) and electrolyte treatment (3). As the electrolyte, a solution of ammonium carbonate salt was chosen.

Visual information about the structural changes in cotton cellulose after treatment with an electric charge in the samples under study is provided by X-ray diffraction analysis.

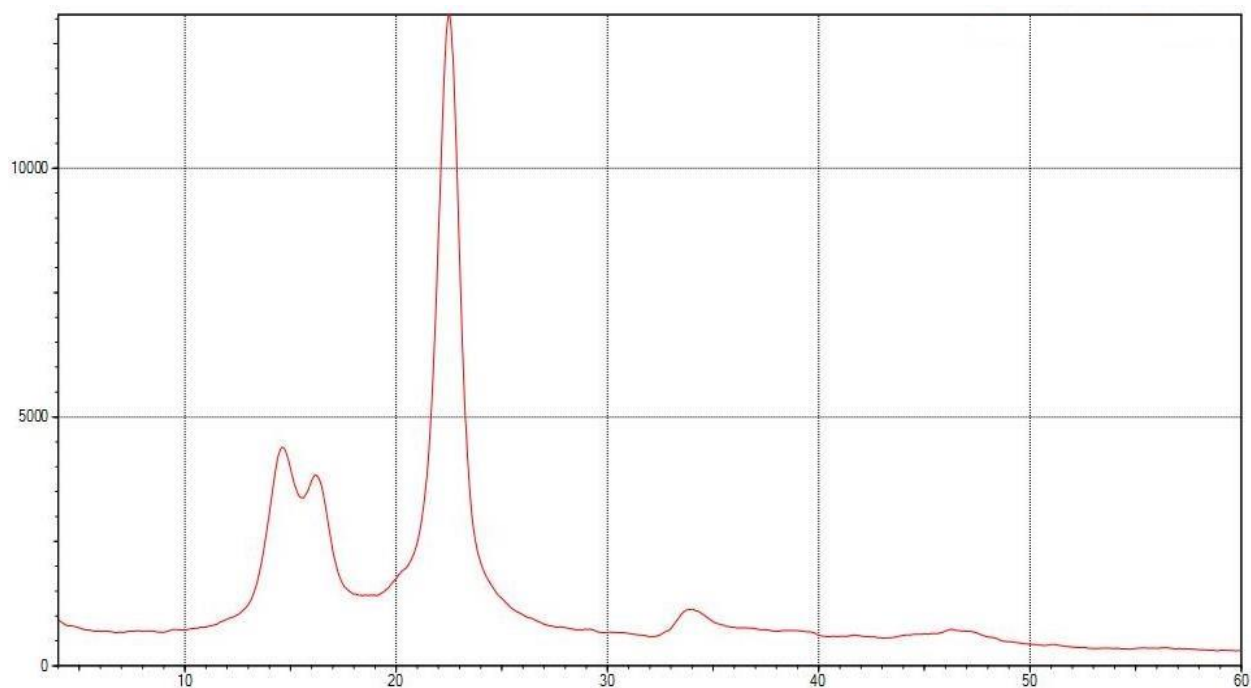
Structural changes in cellulose samples, as well as determining the degree of crystallinity / SC / cellulose, were studied by the most common x-ray method, which is based on a comparison of the intensity of X-ray scattering in crystalline and amorphous regions.

According to the results of studies, it was found that the maximum SC of cotton cellulose is observed in the control sample. At the same time, when processing by an electric charge without an electrolyte and with an electrolyte, partial destruction of intermolecular hydrogen bonds is observed.

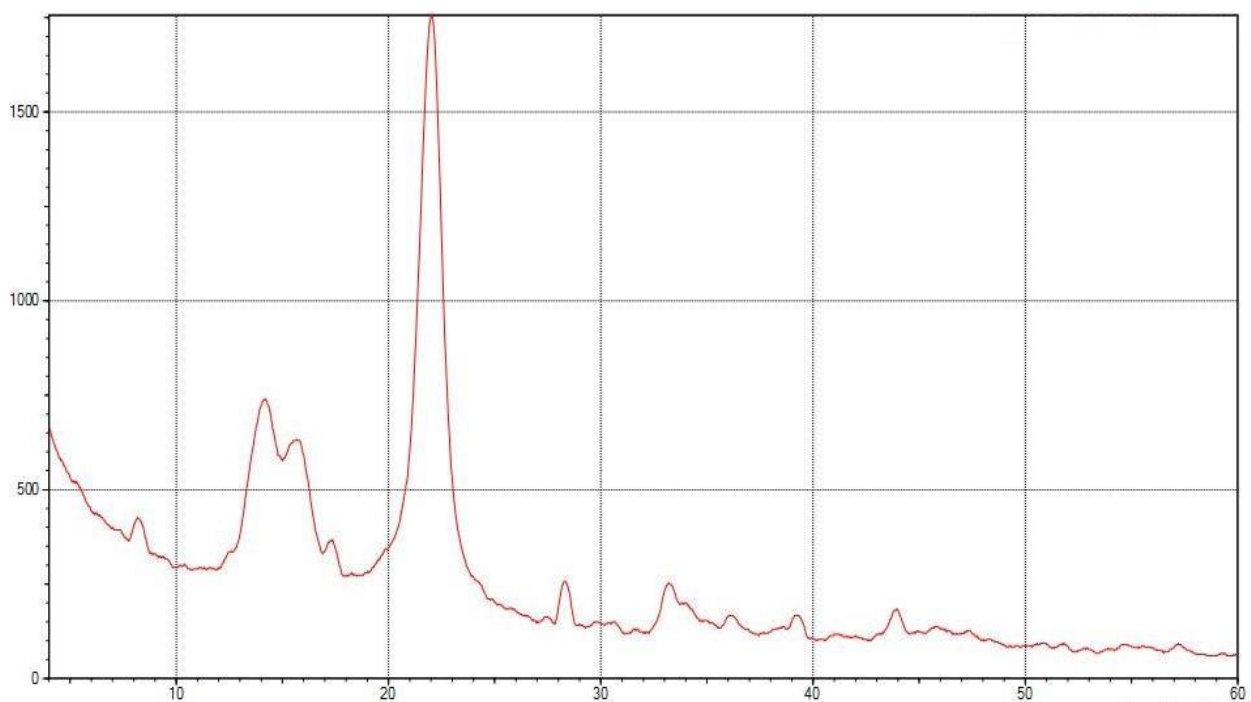
According to x-ray phase analysis (Fig. 1-3), it is possible to assess the degree of crystallinity of the obtained samples in comparison with the reference and initial microcrystalline cellulose (samples 1-3).



**Fig. 1.** X-ray diffraction pattern of sample 1



**Fig. 2.X-ray diffraction pattern of sample 2**



**Fig. 3.X-ray diffraction pattern of sample 3**

Since amorphization or a decrease in crystallite size leads to expansion of the diffraction pattern peaks, integration of the most intense peaks of crystalline cellulose and summation of the peak integrals, taking into account the background and amorphous peaks, makes it possible to

calculate the cellulose crystallinity index based on the data of X-ray diffraction patterns (Table 1).

**Table 1**

**Calculation data of crystallinity of cellulose samples based on x-ray phase analysis**

<b>Samples</b>	<b>Integrals of 4 crystalline cellulose peaks</b>	<b>The sum of the integrals of crystalline peaks of cellulose</b>	<b>X-ray diffractogram integral (crystalline, amorphous peaks and background)</b>	<b>Cellulose crystallinity index (sum (integrals of crystalline peaks of cellulose / integral of x-ray diffraction patterns) * 100)</b>
Sample-1	56.78942	282.47305	423.34631	66.72
Sample-2	67.56028	303.29433	668.62411	45.36
Sample-3	54.52621	268.3713	410.37879	65.40

In addition, the appearance in the X-ray diffraction pattern of the sample of 3 peaks of ammonium bicarbonate \* 2H<sub>2</sub>O in the form of separate crystalline peaks with sufficient intensity for calculation allows us to conclude that a certain amount of an admixture of ammonium bicarbonate is present in the sample. Given the sensitivity of the X-ray phase analysis method and quantitatively analyzing the diffraction pattern of sample 3, we found in it the presence of 4.79% bicarbonate. In this case, an abnormal decrease in the crystallinity index is observed for sample 3, despite the fact that the presence of a 5% impurity leads to the appearance of additional crystalline peaks.

The use of the Rietveld method for analyzing the diffractogram of sample 3, using the least squares method to refine and approximate the theoretical line of the entire diffractogram profile to its experimental profile, allows us to analyze the crystal structure and obtain reliable results when the reflections from the crystalline phases of microcrystalline cellulose and bicarbonate overlap.

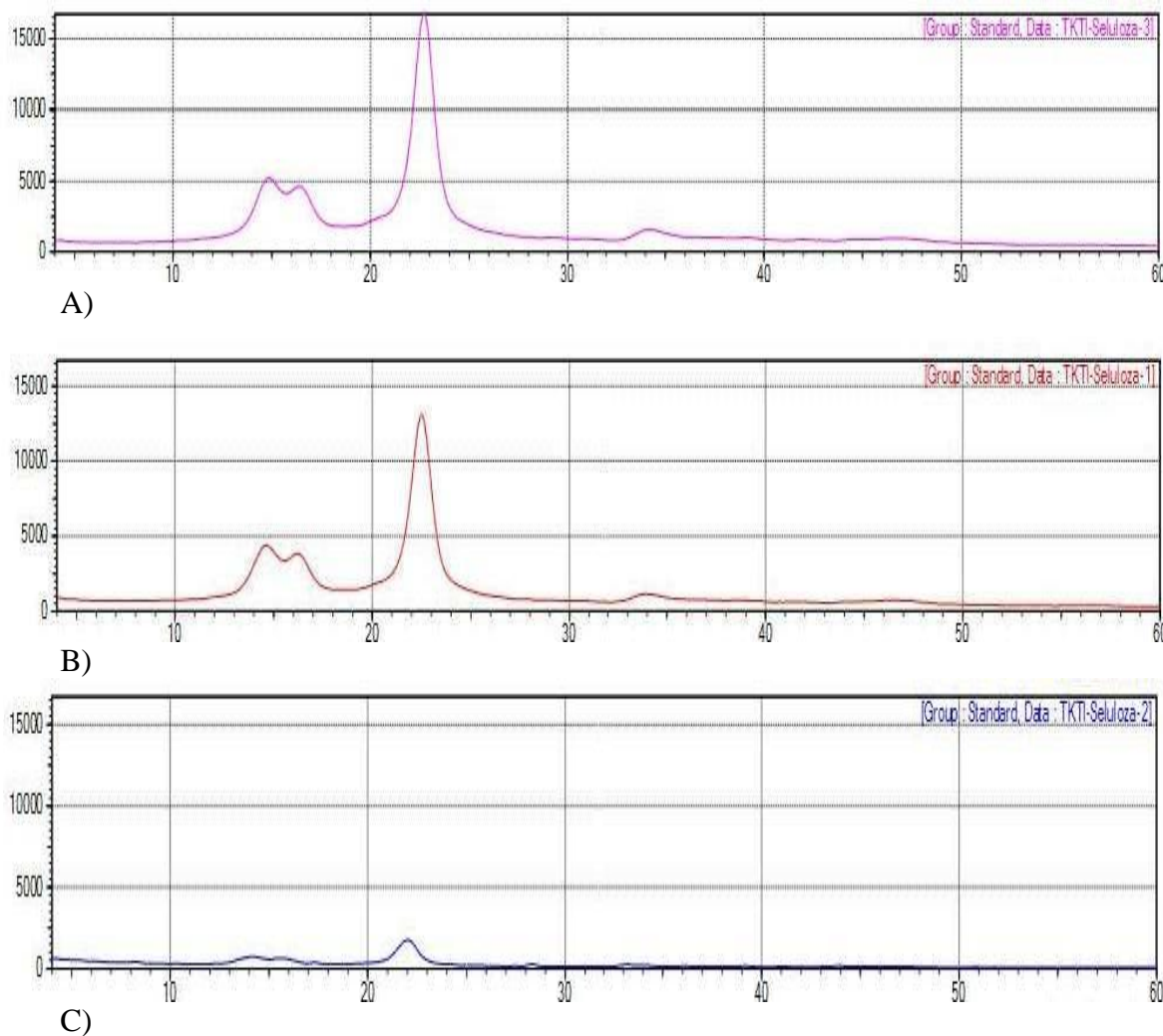
In the table. 2 shows the percentage of MK cellulose and amorphous cellulose based on the Rietveld analysis. Based on the data on the relative standard deviation of RNO (%), which does not exceed 5-9%, it can be concluded with a high degree of certainty that sample 3 has a more amorphous structure and a smaller crystallite size compared to the initial and reference MK cellulose, t. e. there is a decrease in crystallinity (MK cellulose content) from 62-67% to 49% (about 20%).

**Table 2**

**Ritveld analysis data for cellulose samples**

<b>Sample Components</b>	<b>%</b>	<b>RNO (%)</b>
<b>Sample 1</b>		
MK-cellulose	67.1	9.1
Amorphous cellulose	32.9	9.1
<b>Sample 2</b>		
MK-cellulose	62.2	5.4
Amorphous cellulose	37.8	5.4
<b>Sample 3</b>		

MK-cellulose	48.5	2.4
Amorphous cellulose	46.7	2.4
Ammonium carbonate (bicarbonate)*2H <sub>2</sub> O	4.79	0.26



**Fig. 4. Structures of cotton cellulose x-ray analysis.**

A) Control cellulose, B) Wet cotton cellulose,  
C) Cotton cellulose moistened in an electrolyte.

Identification of the samples was carried out on the basis of diffraction patterns, which were recorded on an XRD-6100 apparatus (Shimadzu, Japan), controlled by a computer. CuK $\alpha$  radiation ( $\beta$  filter, Ni, 1.54178 current and tube voltage regimes of 30 mA, 30 kV) and a constant detector rotation speed of 4 deg / min in increments of 0.02 deg were used. ( $\omega$  /  $2\theta$  coupling), and the scanning angle varied from 4 to 80 ° (Fig. 4).

Thus, we can conclude that during the treatment of cellulose with an electric pulse (sample 2), the structure of the cellulose practically does not change, i.e., the cellulose is not amorphized and is similar to the control sample (sample 1).

After chemical treatment with a bicarbonate salt followed by an electrical impulse, the cellulose is amorphized and the peak in region 220 disappears, which indicates the complete disappearance of the crystalline sections of cellulose (sample 3).

**Conclusion.** A method is proposed to increase the reactivity of cotton cellulose, the quality indicators of the resulting cellulose ethers are improved by treating cotton cellulose with an electric charge.

The dependence of the reactivity of cellulose on the voltage, number of pulses and capacitance of the capacitor is determined. Optimum parameters were determined experimentally: Discharge voltage 11-13 kV, number of pulses 22-24 and capacitor capacitance 0.6  $\mu\text{F}$ , respectively. According to the results of studies, the highest quality index of acetate films and fibers was observed for cellulose triacetate, obtained on the basis of cotton cellulose moistened with an electrolyte and treated with an electric charge in the optimal mode.

### References

1. Boldina V.A., Zaripova A.M. Vliyanie izmelcheniya linta na kachestvo xlopkovoy sellyulozi i ee reakcionnuyu sposobnost. - V kn.: Ximiya i texnologiya sellyulozi i volokno, vip. N 6, 1973; Tashkent, izd. "Fan", S.145-148.
2. Mirkamilov T.M. Issledovanie vliyaniya usloviy ximicheskoy ochistki siklonnogo puxa i korotkoshtapelnogo linta na ximicheskiy sostav poluchennoy sellyulozi i ee prigodnost dlya pererabotki na atsetilsellyulozu i visokokachestvennuyu bumagu. Avtoref.dis.kand.tex.nauk.-Leningrad, 1966; S.26.
3. Zaripova A.M., Golovnikova M.V., Tishaboev U., Igamberdiev I.I., Usmanov X.U. Vozmojnost ispolzovaniya zasorennix sortov linta dlya polucheniya visokokachestvennoy xlopkovoy sellyulozi. V kn.: Struktura i modifikatsiya xlopkovoy sellyulozi. Vip. 3, izd."Fan", UzSSR, - Tashkent, 1966,; S.11-15.
4. Akim L.E., Mirkamilov T.M., Usmanov X.U. Poluchenie visokokachestvennoy xlopkovoy sellyulozi iz siklonnogo puxa dlya pererabotki eeyego, atsetilsellyulozu i bumagu. - V kn.: Struktura i modifikatsiya xlopkovoy sellyulozi. Tashkent, 1966,; izd. "Fan", S.32-37.
5. Tishaboev U. Vliyanie texnologicheskogo rejima polucheniya sellyulozi iz xlopkovogo linta na ee ximicheskiy sostav i reakcionnuyu sposobnost k viskozoobrazovaniyu. Avtoref.diss.kand.tex. nauk. Tashkent, 1970,; S.22.
6. Eroxin N.G. Razrabotka texnologii polucheniya visokooblagorajennoy xlopkovoy sellyulozi dlya proizvodstva atsetatov sellyulozi. V kn.: Ximiya i texnologiya proizvodnix sellyulozi. VNIIS, g. Vladimir, 1964,; S.141.
7. Zaripova A.M., Plovnikova M.V., Tishabaev U., Igamberdiev I.I., Usmanov X.U. Vozmojnost ispolzovaniya zasorennix sortov linta dlya polucheniya visokokachestvennoy xlopkovoy sellyulozi. V kn. Struktura i modifikatsiya xlopkovoy sellyulozi. Vip. 3, izd."Fan",- Tashkent, 1966,; S.11-15.
8. Sayfutdinov R.S. Razrabotka ximicheskoy texnologii ispolzovaniya otxodov xlopkovodstva dlya proizvodstva drevesno-strujechnix plit i sellyulozi. Avtoref.dis.dokt.tex.nauk. Tashkent, 1998, S.49.
9. Primkulov M.T., Ismoilov S.N., Umarova V.K. // Bug'doy somoni, sholi va g'o'za poyalaridan sellyuloza olish va ularning strukturasini suvda bo'kishi usuli orqali o'rganish. // Kompozitsion materiallar, 2015g. №1. Toshkent. B 62-64.
10. Nikitin N.I., Klenkova N.I. Vliyanie slabogo alkilirovaniya na svoystva sellyuloznogo volokna. // Jurnal prikladnoy ximii, 1951.24. 296.

11. Klenkova N.I., Kulakova N.A. Deystvie pervichnix aminov jirnogo ryada na strukturu i reaksionnuyu sposobnost xlopkovix selluloznix volokon. // Jurnal prikladnoy ximii. 1965;38, 4, S.919.
12. A.S.573524 Sposob polucheniya xlopkovoy sellulozi. Mirkamilov T.M., Turaev E., Djalilov A.T., Askarov M.A./- Opubl.v BI N 35, 1977.
13. A.S.604888 Sposob polucheniya xlopkovoy sellulozi. Mirkamilov T.M., Turaev E., Djalilov A.T., Askarov M.A./-Opubl.v BI N 16, 1978.
14. Bozorov O.N. Texnologiya pererabotki nizkosortnogo linta dlya polucheniya efirov sellulozi. Avtoref.diss. kandidata texnicheskix nauk. – Tashkent, 2005, S.22.
15. Akim E.L., Mirkomilov T.M., Akim L.E., Usmanov X.U. Vliyanie chastichnogo oksietilirovaniya na skorost atsetilirovaniya xlopkovoy sellulozi, poluchennoy iz siklonnogo puxa i korotkoshtapelnogo linta. V kn. Trudi Leningradskogo instituta SBP. vip. 23,- M., 1970. izd. Lesnaya promishlennost; S.59-62.

UDC 621.643-777.6

## MULTI-STAGE ANALYSIS AND MODELING OF A TWO-STAGE QUASI-DEVICE PNEUMATICSEPARATION OBJECT

**A. Artikov<sup>1</sup>, D.T.Karabaev<sup>1</sup>, L.Akobirova<sup>1</sup>, Z.Akbarkhodjaev<sup>1</sup>, S.Oymirov<sup>1</sup>,  
M.T. Abdirova<sup>2</sup>**

<sup>1</sup>Tashkent Chemical Engineering Institute,

<sup>2</sup>Almaty State College of Energy and Electronic Technologies

**Anotatsiya.** Ushbu ishda ko'p bosqichli, tizimli tahlil yordamida yirik, mayda-butun mahsulotlar pnevmo separatsiya qonuniyati ko'rib chiqildi hamda karbonat kalcii ya'ni bor mahsulotining pnevmo separatsiyasi yordamida hosil bo'ladigan fizik-kimyoviy effekti va hodisalarning bog'liqligi aniqlangan. Ushbu jarayonlarning asosini chala kuydirilgan hajmli mahsulotning aralashmasidir. Modellash tirishda chala kuydirilgan zona 1 ya'ni, gazli va qattiq fazalardan qismlardan ekanligi aniqlangan. Modellash tirish jarayonida raqamli ekranda hisobotlar, vaqtinchalik grafiklar orqali namayon bo'lishi kuzatildi. Grafiklar shuni ko'rsatadiki umumiy massa ichidagi maydalangan mahsulotlar konsratsiyasi ma'lum bir intervalda to'g'ri chiziqli ya'ni to'yingan holatda bo'lar ekan. Tajriba shuni ko'rsatadiki, yuqori sifatli maydalangan mahsulotlarni olish nazarda tutilgan.

**Abstract.** Based on the multi-phase system analysis, we determined general pneumatic separation regularities of the dried crushed target product and detected the main interrelationships between physical and chemical effects and phenomena that takes an important place in the process of pneumatic separation with simultaneous crushing of the target product (calcium carbonate) of chalk in bulk materials and on this basis identifies the possibilities of the pneumatic separation process in the flowing fluidized volume, and proposes rational methods for intensifying the process. The principal device here is the fluidized volume moving unit. The fluidized length, the fluidized layering zone and the inlet area. The volume zone that is fluidized is divided into two components: the gas phase and the solid phase. The solid phase, in turn, consists of the first, second, third crushed target product particles, etc. components.

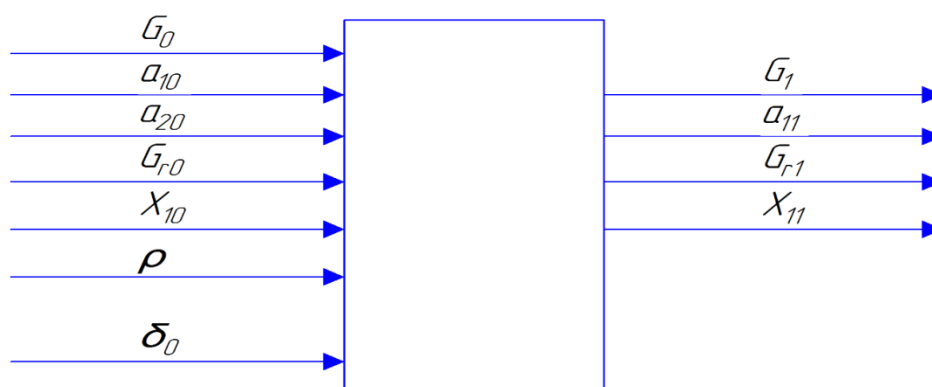


**Key words:** system, analysis, physicochemical, effects, and phenomena, model, product, zone, interval, graph.

**Анотация.** В статье на основе многоступенчатого системного анализа отражены общие закономерности пневмосепарации сушеного измельченного целевого продукта и выявлены основные взаимосвязи физико-химических эффектов и явлений, имеющих важное место при протекании процесса пневмосепарации с одновременным измельчением целевого продукта – мела (карбоната кальция) в сыпучих материалах и на этой основе определены возможности процесса пневмосепарации в фонтанирующем псевдооживленном объеме, а также предложены рациональные пути интенсификации режимов функционирования процесса пневмосепарации с одновременным измельчением целевого продукта в фонтанирующем псевдооживленном объеме. Основным объектом исследования здесь является аппарат с фонтанирующим псевдооживленным объемом, имеющим зону псевдооживления и зону уноса. Зона псевдооживления разделяется на две составляющие: газовая и твердая фазы. В свою очередь, твердая фаза состоит из частиц первого, второго, третьего измельченного целевого продукта и др. компонентов

Advancement of the conceptual bases of the pneumoseparation process with simultaneous grinding of the target product in the gushing fluidized volume, by means of mathematical modeling of the class of processes considered, predetermines further development of functional methods and methods of mathematical modeling of interconnected effects and phenomena on separate hierarchical measures.

The principal device here is a fluidized volume flowing device. If the system-installation is divided into the elements considered above at the first stage of the multi-stage system analysis [1, 2], then the fluidized volume apparatus is also divided into a number of elements: the fluidized volume body, the gas inlet, the grid, the fluidized zone and the entry zone. The volume region that is fluidized is divided into two components: the gas phase and the solid phase. The solid phase, in turn, consists of the first, second, third crushed target product particles, etc. components (Figure 1).



**Fig.1. Pneumatic separation unit of dried crushed target product in flowing fluidized layers as modeling object.**

Input parameters of the pneumatic separation object of the dried crushed target product are: flow rate of solid material  $G_0$ , size of solid particles  $\delta_1$ , gas flow rate  $G_f$ , pressure in the apparatus  $P$ , concentration of the crushed target product at the inlet  $a_{10}$ . Output parameters: consumption of dried material (calcium carbonate) of chalk  $G_{ch}$ , concentration of component -

dried material (calcium carbonate) of chalk  $a_{11}$ , gas consumption  $G_g$ , concentration of studied components in gas  $a_{11}$ .

For the advancement of calculation methodology and the design of drying process plants in flowing fluidized layers of bulk materials, particularly chalk (calcium carbonate), let's consider the object as a cybernetic system[3,4]. The pneumatic separation apparatus in gushing fluidized layer volume of dried crushed target product is shown in Fig. 1.

The device (e.g.) is fed with a material (calcium carbonate) of chalk with a certain concentration of moisture. The wet paste-like material (calcium carbonate) of the chalk covers the surfaces of glass beads that are in a fluidized state at the bottom of the drying process in a gushing fluidized volume. With the supply of hot gas, fluidized layering takes place and part of the material remains in the unit, while part is carried away with air. The material balance equation for the selected component (e.g. for  $a_1$  chalk to be dried (calcium carbonate)) is compiled:

$$\frac{dm_1}{d\tau} = g_{10} - g_{11} + \Delta g_1 \quad (2.1.)$$

Where,  $m_1$  is the mass of material (calcium carbonate) of chalk in a fluidized volume determined by the equation:

$$m_1 = m_0 * a_1 \quad (2.2.)$$

Here  $m_0$  is the total mass (calcium carbonate) of chalk in a fluidized layer;  $a_1$  is the concentration of dried material (calcium carbonate) of chalk in a fluidized layer;  $g_{10}$  and  $g_{11}$  - the initial and final costs of converting the material (calcium carbonate) of chalk into dried material (calcium carbonate) of chalk, respectively the transition of its gas phase separating from the glass balls and the consumption of material in the machine with fluidized layer volume;  $\Delta g_1$  the amount of material that goes into the gas phase and is defined as:

$$\Delta g_1 = k_1 * (1 - a_1).$$

In the process of pneumatic separation of the dried crushed target product in the flowing fluidized layer machine, the hydrodynamic structure of flows can be attributed to the model of ideal multi quasi-device mixing, in each quasi-device, in each fluidized quasi-device layer there is as if the same concentration of materials exists.

To find  $m_1$ , we must calculate the mass of fluidized layer  $m_0$  by equation:

$$m_0 = V_{fl} * \rho_{fl} \quad (2.3.)$$

Where  $V_{fl}$  - is the fluidized layer volume equal to  $V_{fl} = S_c * h_{fl}$ ; ( $S_c$  cross section of the layer;  $h_{fl}$  -height of fluidized layer in a fountain fluidized layer unit),  $\rho_{fl}$  is the fluidized layer density. From here we find,

$$m_0 = S_c * h_{fl} * \rho_{fl} \quad (2.4.)$$

For the conical part  $m_0 = S_c * h_{fl} * \rho_{fl} / 3 \quad (2.5.)$

Consumption of  $g_{10}$  incoming into the device component of the powdered grinded target product is determined by the product of total material consumption (calcium carbonate) of chalk in device  $G_0$  at its  $a_{10}$  grinded target product concentration.

$$g_{10} = G_0 * a_{10} \quad (2.6.)$$

The flow rate of component  $g_{11}$  from the device is determined by the product of material  $G_1$  to its concentration  $a_{11}$ .

$$g_{11} = G_1 * a_{11} \quad (2.7.)$$

Where  $G_0$ -value of material consumption in the machine;  $G_1$ -value of output material;

Here  $k_1$  is the conversion factor of the target product into a shredded product.

By substituting the values of these parameters, we obtain the following differential equation for the selected quasi-apparatus:

$$\frac{da_{i1}}{d\tau} = \frac{1}{S_c * h_{nc} * \rho_{nc}} (G_0 * a_{i0} - G_1 * a_{i1} - K * (1 - a_{i1})) \quad (2.8.)$$

Coefficient "k" depends on the flow and temperature of gas and material, thickness and porosity of the fluidized layer, particle diameter, density, size of the apparatus, etc.

$$k = f(G_g; T_g; T_m; h_{fl}; \varepsilon; d_p; \rho_p; \text{и т.д.})$$

It is an independent task to determine that coefficient for all cases. In periodic mode we can determine the value of the coefficient "k" by conducting experiments. We're identifying private solutions for the case under consideration. A laboratory device was designed for this purpose, and the experimental results were analyzed.

It is accepted that part of the non-crushed target product remains in pseudo-liquidated volume for the construction of the mathematical model of the continuous process of pneumatic separation of the crushed target product, and the other part should pass completely to the gas phase. The material equilibrium for the material component of the crushed target product has the form:

$$G_0(1 - a_0) = G_1(1 - a_1) \quad (2.9.)$$

Where,  $G_0$  — the flow rate of the initial crushed target product at a concentration of  $\alpha_{10}$ , corresponding to the initial concentration of the light component;  $G_1$  — the flow rate of the crushed target product from the pneumatic separation process in the fluidized fountain volume with light component concentration  $\alpha_{11}$ .

Solving this equation with regard to  $G_1$ , we obtain:

$$G_1 = G_0 * \frac{1 - a_{10}}{1 - a_{11}} \quad (2.10.)$$

By substituting values  $\alpha_{10}$  and  $\alpha_{11}$  in this equation we can calculate  $G_1$ .

If  $a_{10} = 0$

$$G_1 = G_0 * \frac{1}{1 - a_{11}} \quad (2.11.)$$

By substituting (2.11.) in the equation (2.8.), we obtain a differential equation similar to that in [98]:

$$\frac{da_1}{d\tau} = \frac{1}{S_c * h_{nc} * \rho_{nc}} \left( G_0 * a_0 - G_0 \left( \frac{1 - a_0}{1 - a_1} \right) a_1 - k_1 * (1 - a_1) \right), \quad (2.12.)$$

or for the i-th quasi-device in a gushing fluidized layer volume

$$\frac{da_{i1}}{d\tau} = \frac{1}{S_c * h_{nc} * \rho_{nc}} \left( G_0 * a_{i0} - G_0 * \left( \frac{1 - a_0}{1 - a_1} \right) * a_{i1} - K * (1 - a_{i1}) \right) \quad (2.13.)$$

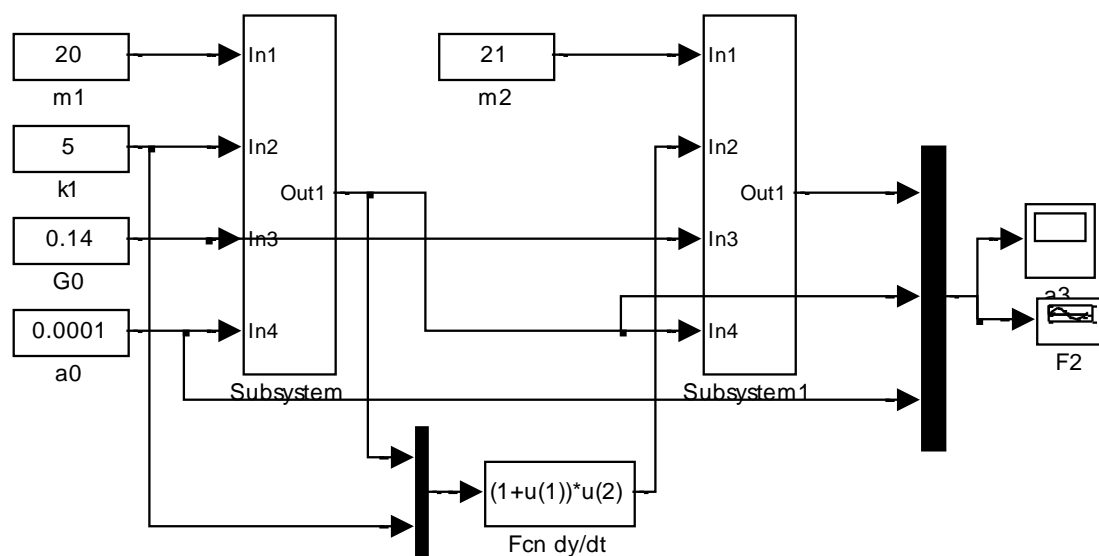
Computer recording on MATLAB

$$(u(4) * u(5) - (u(4) * (1 - u(1)) / (1 - u(5))) * u(1) - (u(3) * (1 - u(1)))) / u(2)$$

The above equation makes it possible to determine the concentration of volatile component (calcium carbonate) of chalk  $a_1$  in the selected  $i$ -th quasi-device of dried material (calcium carbonate) of chalk. Now you can build a mathematical and computer model for the entire process of drying with simultaneous grinding (calcium carbonate) of chalk in a gushing fluidized volume.

Based on the mathematical model obtained, computer models were received for continuous process on the basis of works using MATLAB software package [5 ].

The following input parameters are entered into the computer models input unit: total material mass  $m_0$ , grinding factor  $k$ , and initial light part concentration of the total  $a_0=0$  mixtures. The performance parameter is the shift in luminous component concentration in the total mixture.



**Fig.2.4. Computer model for continuous process of pneumatic separation of dried crushed target product in the first conical part with flowing fluidized layer volume.**

The computer model consists of the following blocks:

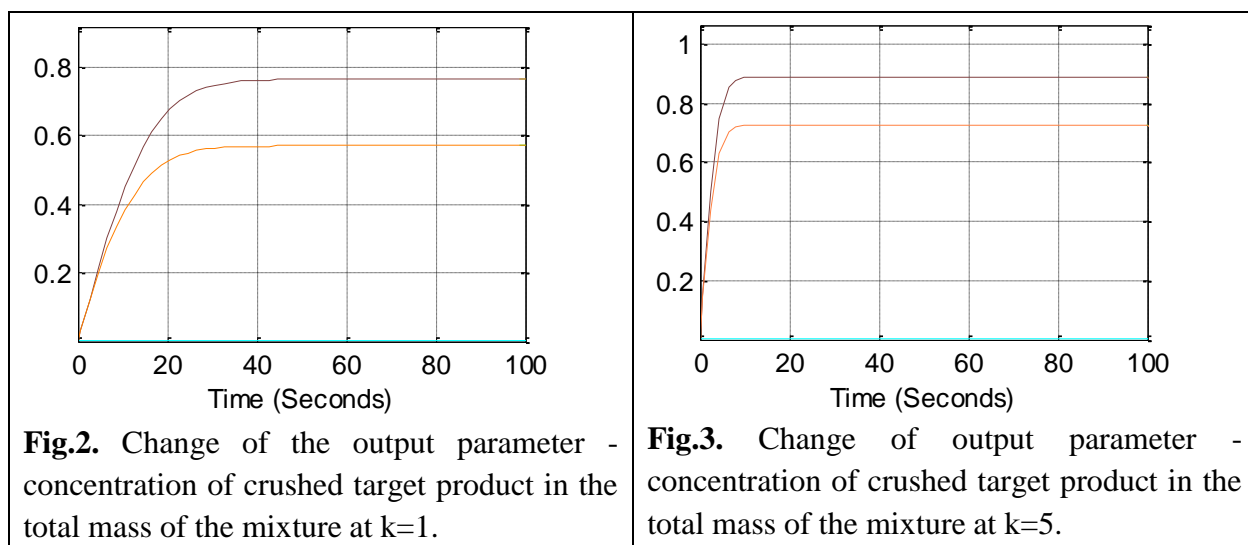
The single cell computer model consists of the following blocks:

1. Input blocks 1, 2, 3, 4;
2. Block 5, at the bottom, calculates a new value for the grinding ratio;
3. Blocks of 6, 7 grinded target product in quasi-devices;
4. Blocks of calculated concentrations in the form of time charts 8, 9, 10.

The following input parameters are entered into the input unit of single cell computer models: content of heavy components in the mixture, for example,  $G=500/3600=0.14$  kg/s, concentration of light component  $a_{10}$ , total mass of material  $m$ . The output parameter is the concentration of the light component of the crushed target product at the output of two quasi-components.

The computer model operates as follows: These signals are obtained in the signal acquisition unit after entering the input parameters in the input blocks 1,2,3,4 and then the parameter values in blocks 6,7 are determined. The results of the calculation are displayed in signal acquisition unit 7 as time charts. In blocks 8.9 9 in the time graph we get the effects of a change in the concentration of light portion of the crushed target product.

Results of calculations can be observed on the digital screen and in the form of time diagrams in the devices of Fig. 2. For values of a target product conversion factor in the crushed product  $k=1$ , and in Fig. 3. For values the destination product conversion factor in the crushed product  $k=5$ .



The diagram in Fig. 2 displays a change in the output parameter - change in the concentration of the crushed target product of the mixture's total mass at  $k=1$ . In the time diagram Fig. 3. Changes in concentration graphs of the crushed target product-light component after the first and second quasi-groups in relation to the total mass of the material at  $k=5$ ; Provided that there is a noticeable rise in quasi-apparatus quantities and the coefficient of transformation of the target product into a crushed product should facilitate the acceptance of a higher quality product.

### References

1. Artikov A. Introduction to systematic analysis. Certificate of Deposit of Copyright objects № 000300. Agency for Intellectual Property of the Republic of Uzbekistan, 10.11.2016.
2. Artykov A. Computer methods of analysis and synthesis of chemical and technological systems. Tashkent "Voriz Nashriet" 2010.. 160 p.
3. System analysis of pneumoseparation of bulk materials in fluidized layer. / Yunusov B.I., Karabaev D.T., Artikov A.A. TashGTU // Kimeviy Tekhnologii. Nazat va Boshkaruv. 1/2011.
4. Formalization of mathematical model of process of air separation of loose materials / D.T. Karabaev, B.I. Yunusov, A.Artikov... // Sixth World Conference on Intelligent Systems for Industrial Automation.- Tashkent, Uzbekistan November 25-27, 2010.

5. The Mathworks Inc., "MATLAB - The Language of Technical Computing", <http://www.mathworks.com/products/matlab>.
6. Lees, F. P. (1984). Process computer alarm and disturbance analysis: Outline of methods for systematic synthesis of the fault propagation structure. // Computers and Chemical Engineering, 8(2), 91-103. doi:10.1016/0098-1354(84)87016-7
7. Tula, A. K., Eden, M. R., & Gani, R. (2019). ProCAFD: Computer-aided tool for sustainable process synthesis, intensification and hybrid solutions doi:10.1016/B978-0-12-818634-3.50081-3 Retrieved from [www.scopus.com](http://www.scopus.com)
8. Tula, A. K., Babi, D. K., Bottlaender, J., Eden, M. R., & Gani, R. (2017). A computer-aided software-tool for sustainable process synthesis-intensification. // Computers and Chemical Engineering, 105, 74-95. doi:10.1016/j.compchemeng, 2017.01.001
9. Rakhmonov, I.U.; Niyozov, N.N.; and Ли, К. (2019) DEVELOPMENT OF CORRELATION AND REGRESSION MODELS OF ELECTRIC ENERGY INDICATORS OF THE EQUIPMENT WITH CONTINUOUS NATURE OF PRODUCTION, // Technical science and innovation: Vol. 2019 : Iss. 4 , Article 2. Available at: <https://uzjournals.edu.uz/btstu/vol2019/iss4/2>
10. Usmonov, N.O.; Sanayev, Sh.S.; and Yusupov, Z. (2019) CALCULATION OF TEMPERATURE OF ROUTINE WATER COOLED IN IRRIGATED LAYERS, // Technical science and innovation: Vol. 2019 : Iss. 3 , Article 7. Available at: <https://uzjournals.edu.uz/btstu/vol2019/iss3/7>
11. Misrikhanov, M. Sh. and Khamidov, Sh.V. (2019) MATHEMATICAL POWER FLOW MODEL IN AN ELECTRICAL SYSTEM CONTAINING A SERIAL COMPENSATOR THYRISTOR CONTROLLED REACTIVE COMPONENT, // Technical science and innovation: Vol. 2019 : Iss. 3 , Article 5. Available at: <https://uzjournals.edu.uz/btstu/vol2019/iss3/5>
12. Eshbaeva, U.J.; Shin, I.G.; and Djalilov, A.A. (2019) OPTIMIZATION OF COLOR PERCEPTION PROCESS IN THE PRINT PRODUCT BY THE STEEP CLIMBING METHOD BY BOX-WILSON, Technical science and innovation: Vol. 2018 : Iss. 4 , Article 2. Available at: <https://uzjournals.edu.uz/btstu/vol2018/iss4/2>
13. Bobomurodov, Q.H.; Razakov, J.Kh.; Bobomurodov, S.Q.; and Shokirov, R.A. (2019) METHODS FOR RESEARCHING THE LOCALIZATION AND DELOCALIZATION OF CARRIERS IN YBA2CU3O6+X FILMS, // Technical science and innovation: Vol. 2019 : Iss. 4 , Article 7. Available at: <https://uzjournals.edu.uz/btstu/vol2019/iss4/7>.

UDC 662.7.622.342(575.3)

## DEVELOPMENT OF AN OPTIMAL TECHNOLOGICAL SCHEME FOR COLLECTIVE FLOTATION OF REFRACTORY GOLD ORES

**D.B. Makhmarejabov<sup>1</sup>, N.Z. Sharifova<sup>1</sup>**

<sup>1</sup>Tashkent State Technical University

*Annotatsiya. Maqolada boyitilishi qiyin bo'lgan oltin rudalarini bosqichma-bosqich flotatsiyalashning maqbul texnologik sxemasi ishlab chiqilgan. Laboratoriya sharoitida O'zbekistondagi polimetal konlaridan boyitilishi qiyin bo'lgan oltin-mishyak ruda namunasidan*

*bir nechta tajriba asosida, yanchishning turli yirikligida boyitishning flotatsiya usulini bosqichma - bosqich qo'llab, reagentlarning sarfini o'zgartirmagan holda, sifatli oltin boyitmasini olishning maqbul texnologik shemasi ishlab chi?ilgan. O'tkazilgan laboratoriya ishlarining asosiy mohiyati chiqindiga foydali komponentning ajralishini maksimal darajasida kamaytirish va sifatli oltin boyitma ko'zda tutilgan. O'tkazilgan laboratoriya ishlaridan olingan eng maqbul natijasiga ko'ra rudani selektiv sxema bo'yicha boyitishda tarkibida 45,38 % g/t oltin, 3,97 % mishyak, 23,83 % oltingugurt, 4,74 % glinozem saqllovchi boyitma olish mumkin bo'lib, bunda oltinning boyitmaga ajralishi 93,5 %, yo'ldosh element sifatida kumushning boyitmaga ajralishi 34,6 % ni tashkil etadi. Olingan natijalar faqatgina laboratoriya sharoitida erishilgan, agar hozirgi kunda Respublikamizdagi oltin saralash fabrikasida oltin - mishyak rudalari uchun, maqolada tavsiya qilingan texnologik sxema joriy qilinsa oltinning boyitmaga ajralishini 90 % dan yuqoriroq va zararli qo'shimchalar miqdorini pasaytirishga erishish mumkin.*

**Abstract.** *The article deals with the development of a technological scheme of a phased technology of flotation of hard-to-rich and persistent gold-bearing ores. The results of laboratory tests of difficult-to-enrich gold-arsenic polymetallic ores of Uzbekistan deposits are substantiated. On the basis of the taken several crushed samples of different size, used in the enrichment by flotation method, without changing the reagent flow rate, the optimal technological scheme for the extraction of high-quality gold-containing concentrate. As a result of the laboratory work, a composition of unreacted useful components sent to the tails was obtained and more qualitative gold-containing concentrate was drained. The most acceptable result of the laboratory work was the enrichment of ore according to the selective scheme to obtain 45.38% g / t of gold, 3.97% arsenic, 23.83% sulfur, 4.74% alumina preserving enrichment, while the breakdown of gold for enrichment is 93.5%, and the breakdown of silver for enrichment as a satellite element-34.6%. The obtained results were achieved only in laboratory conditions, if at present the technological scheme recommended in the article for gold extraction ore is implemented at the gold recovery plant in our Republic, then it is possible to achieve the separation of gold for enrichment above 90% and reduce the number of harmful additives.*

**Keywords:** *flotation, copper sulphate, arsenic, stepwise, silver, concentrate, iron sulfide, pyrite, antimony, lime, method of enrichment, antimonite, process, gold.*

**Аннотация.** *В статье рассматривается разработка технологической схемы поэтапной технологии флотации труднообогатимых и упорных золотосодержащих руд. Обоснованы результаты лабораторных испытаний труднообогатимых золото-мышьяковых полиметаллических руд месторождений Узбекистана. На основе взятых нескольких измельченных образцов различных по крупности, применяемых при обогащении флотационным методом, не меняя реагентного расхода, разработана оптимальная технологическая схема по извлечению качественного золотосодержащего концентрата. В результате проведенных лабораторных работ получен состав из непрореагировавших полезных компонентов, направляемых в хвосты и более качественный золотосодержащий концентрат. Наиболее приемлемым результатом проведенных лабораторных работ стало обогащение руды по селективной схеме с получением 45,38% г/т золота, 3,97% мышьяка, 23,83% серы, 4,74% глинозема консервирующего обогащения,*



при этом доля золота при обогащении составляет 93,5%, а доля серебра при обогащении в качестве спутникового элемента – 34,6%. Полученные результаты были достигнуты только в лабораторных условиях, если в настоящее время на золотоизвлекательной фабрике в нашей республике внедрена технологическая схема, рекомендованная в статье для золотоизвлекательной руды, то можно добиться разделения золота при обогащении выше 90% и снижения количества вредных добавок.

The purpose of the research work is to develop an optimal technological scheme of gradual flotation of gold – mishyak containing ores, which is difficult to enrich, and in laboratory conditions for the separation of high - quality gold, along with the gradual flotation of pyrite and arsenopyrite - containing gold – mishyak ores - grinding at a different value of 0,074 mm li class, as well as

In laboratory conditions, the following reagents were used, which corresponded to a good separation of gold from gold-containing ore: copper clipping as a preservative reagent, potassium butyl xanthogenate as a repellent, soda as a repellent and T-80 as a foaming reagent. The amount of reagents was used in flotation stages at different values, and in experiments at the same value. Flotation was carried out between 7-15 minutes.

In order to develop an optimal technological scheme of gradual flotation of gold-bearing ores, which is difficult to enrich, the following analyzes and experiments were conducted.

a) general chemical analysis of gold-bearing ores with pyrite and arsenopyrite content, difficult to enrich. The results of the chemical analysis of the average sample of Ruda are presented in Table 1.

**Table. 1.**

**Results of the chemical analysis of the average sample of ore**

Components	Quantity, %	Components	Quantity,%
Kremnezyom	42,0	Sodium oxide	0,44
Iron (+3) oxide	13,0	Phosphor (+5) oxide	0,16
Iron (+2) oxide	1,98	Sulphur	9,79
Titanium oxide	0,7	Sulfur (+6) oxide	1,08
Manganese oxide	0,04	Uglerod oxide	3,52
Glinosome	13,86	N2O gidroscopic	0,72
Kaltsium oxide	2,3	Arsenic)	0,26
Magnesium oxide	2,4	Gold, g/t	3,34
Potassium oxide	2,89		2,52

According to the data presented in Table 1, the main industrial component of ore is gold and silver, the amount of which, respectively, is 3,34 and 2,52 g/t.

b) two-stage flotation experiments of gold-bearing ore.

To improve the technological performance of gold flotation, experiments were conducted on two-stage flotation schemes of ore. In the first stage, ruda was milled up to 56-82% – 0,074 mm li class, in the second stage up to 85,0-100% – 0,074 mm li class. The results of the milling process and the experiment on the ore in stages are presented in Table 2.

**Table.2.**

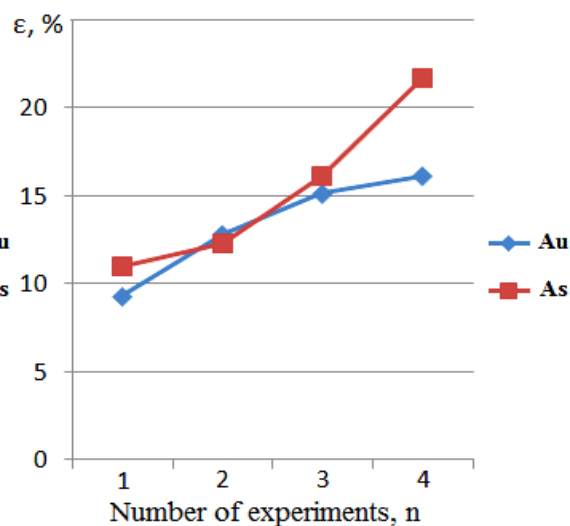
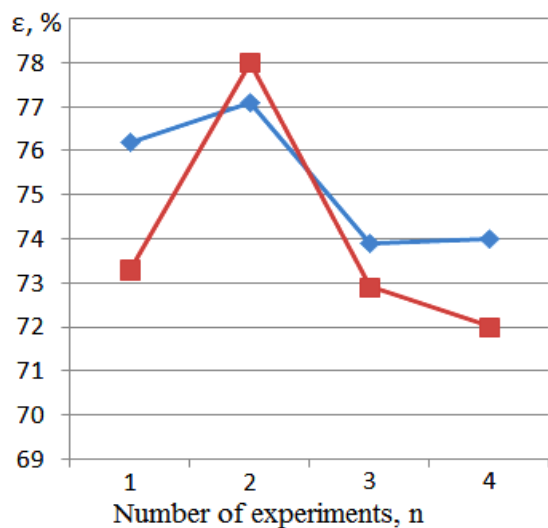
**Results of flotation of ore in two stages**

Products	Output, %	Quantity,		Divorce, %		Milling pus %, -07074 mm	
		Au,g/t	As, %	Au	As	Stage I	Stage II

### Chemistry and chemical technology

1	2	3	4	5	6	7	8
I-longitudinal II-staining Intermed.prod. Garbage Ore	10,80 6,80 5,50 76,90 100,00	23,92 4,64 3,33 0,40 3,39	1,97 0,47 0,27 0,04 0,29	76,20 9,30 5,40 9,10 100,00	73,30 11,00 5,20 10,50 100,00	56	85
I-longitudinal II-staining Intermed.prod. Garbage Ore	12,50 7,80 4,70 75,00 100,00	19,74 5,25 2,66 0,26 3,20	1,87 0,47 0,15 0,03 0,30	77,10 12,80 3,90 6,20 100,00	78,00 12,30 2,30 7,40 100,00	56	90
I-longitudinal II-staining Intermed.prod. Garbage Ore	14,80 6,30 5,10 73,80 100,00	16,98 8,15 3,40 0,27 3,40	1,33 0,69 0,30 0,02 0,27	73,90 15,10 5,10 5,90 100,00	72,90 16,10 5,60 5,40 100,00	82	90
I-longitudinal II-staining Intermed.prod. Garbage Ore	15,30 8,00 5,10 71,60 100,00	16,06 6,68 2,67 0,27 3,32	1,32 0,76 0,20 0,01 0,28	74,00 16,10 4,10 5,80 100,00	72,00 21,70 3,70 2,60 100,00	82	100

According to the data presented in Table 2, the introduction of the control flotation allows to obtain 0,26-0,4 g/t gold preservative (in the second phase 90-100% – 0,074 mm li class) and 0,01 - 0,04% mishyak preservative waste in the composition of grinding and flotation of ore in two stages. The minimum loss of gold in the waste composition can be seen from the results of experiments in Table 2 as well as from the graph that the ore can be milled in two stages up to 100% – 0,074 mm li grade (Figure 1) [1,2,5].



**1-picture. Based on the conducted experience of gold ores, which are difficult to enrich, a graphical view of the interconnected cleavage of the main components in its composition**

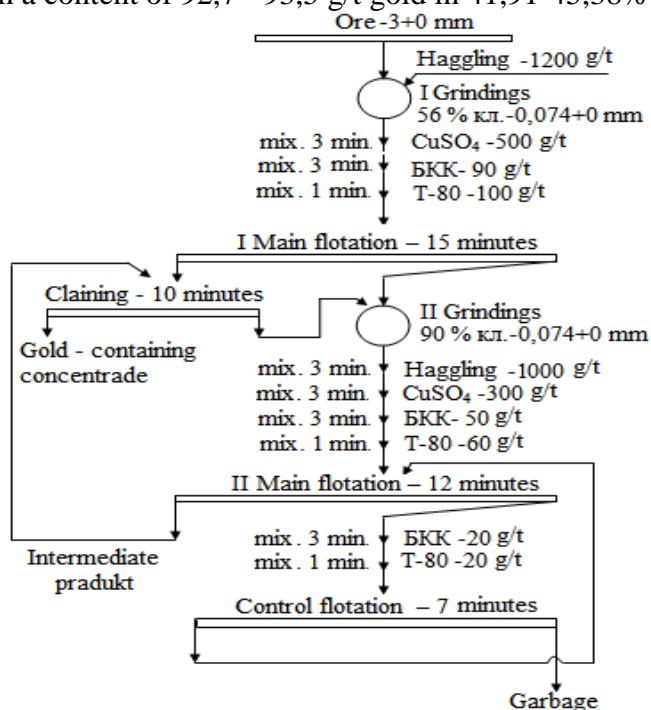
Experiments on continuous process printing were conducted in a closed cycle to determine the technological parameters of the products obtained under the scheme of two-stage flotation. The results of these experiments are presented in Table 3.

**Table. 3.**

**Results of flotation of ore by continuous process printing in two stages**

Products	Output, %	Quantity,			Divorce, %		
		g/t		%			
		Au	Ag	As	Au	Ag	As
Longitudinal	6,80	45,38	12,21	3,97	93,50	34,60	87,00
Intermed.prod.	9,10	0,54	5,41	0,08	1,50	20,50	2,30
Garbage	84,10	0,20	1,28	0,04	5,00	44,90	10,70
Ore	100,00	3,30	2,40	0,31	100,0	100,0	100,0
Longitudinal	7,30	41,91	11,51	3,60	92,70	40,00	87,60
Garbage	92,70	0,26	1,36	0,04	7,30	60,00	12,40
Ore	100,00	3,30	2,10	0,30	100,00	100,00	100,00

As can be seen in Table 3, the scheme of two-stage flotation of ore allowed to obtain a sulfide enrichment with a content of 92,7 - 93,5 g/t gold in 41,91-45,38% decomposition of gold.



**2-picture. The scheme of collating gold ores, which are difficult to enrich by continuous process printing**

As a result of the return of the intermediate product flotation to the control flotation operation, the separation of the Surma into the gold-bearing enrichment increased by 10% compared to other experiments. Therefore, we considered it more expedient to combine the product of the flotation of the intermediate with the total waste in the chamber. The amount of silver in gold-bearing enrichment was 34,6-40 g/t in 11,51-12,21% separation from ore [3,4].

It should be noted that the enrichment of intermediate products in a separate cycle according to the two-stage scheme allows to obtain a waste with a low content of gold, as well as the optimal technological scheme of gradual flotation of gold-containing ores, which is difficult to enrich based on the results of the above experiments, the scheme is recommended

Thus, in the enrichment of ore according to the selektiv scheme, it is possible to obtain 45,38 g/t gold, 3,97% mishyak, 23,83% sulfur, 4,74% glinosem preservative enrichment, in which the separation of gold into the enrichment is 93,5%, the separation of silver into the enrichment is 34,6%.

Silver is associated with surma minerals in the ruda sample, so the selectivity of the gold-bearing minerals goes to the waste. Separation of gold into flotation waste, that is, its loss is 6,5% [6].

In order to enrich the collection of gold-bearing ores, which are difficult to enrich, the ores were milled in different output sizes of 0,074 mm, 6 experiments were carried out and the following conclusion was made according to the results obtained. In order to obtain a quality gold enrichment, it is necessary to add a sample at the first stage on a continuous process print – 2000 g/t soda, 56% KL.- Milled up to 0,074+0 mm and fluted by adding CuSO<sub>4</sub> – 500 g/t, BKK – 90 g/t, T-80 – 100 g/t. At the second stage, the output from the first flotation is 90 % kl. By adding milled soda – 1000 g/t, Siso<sub>4</sub> – 300 g/t, BKK – 50 g/t and T-80 – 60 g/t up to -0,074+0 mm, flotation was repeated, and the resulting products were cleaned of enrichment, while the waste BKK – 20 g/t, T-80 – 20 g/t, transferred to the controlled flotation, the gold content of the enrichment was observed to rise.

**References**

1. Sergo E.E. Droblenie, izmelcheniei groxochenie poleznix iskopayemix. – Moskva: «Nedra», 1985. - 162s.
2. Glembotskiy V.A., Klassen V.I. Flotatsiya. – Moskva: «Nedra», 1973. - 264 s.
3. Rossovskiy S.N., Nizamutdinova R.A. i dr. Razvitiye novix metodov obogasheniya upornix uglisto – mishyakovix zolotosoderjashix rud. – Moskva: «Nedra», 2009. - 302 s.
4. Umarova I.K. Foydali qazilmalarni boyitish texnologiyasi – Toshkent: “TOSHKENT-MOLIYA”, 2015. - 95b.
5. Umarova I.K. Boyitish fabrikalarini loyihalash – Toshkent: “TOSHKENT - MOLIYA”, 2015. - 95b.
6. Maxmarejabov D.B., Umirzoqov A.A. Flotatsiya usulini qo'llab qaysar oltin-mishyakli rudalarini olingan optimal boyitishning tajribaviy natijalari. - Toshkent: ToshDTU: // ToshDTU xabarlari, 2017. № 2. - 202 b.
7. Fridman I.D., Savari E.E., Demina N.N. Vliyaniye uglerodistix veshestv, soderrjashixsya v rudax, na protsess sianirovaniya. Svetniye metalli, 1999; №9; S. 104- 106.
8. Ignateva K.D. Ratsionalnoye ispolzovaniye zolotosoderjashix rud. -M.: «Nedra», 2003; 213 s.
9. Odilxonov K. «Mineralogiya». - Toshkent, 2011 y.
10. Golikov A.A. Povedeniye zolota pri flotatsii. - «Svetniye metalli», 1996; №6; S. 71-74.
11. Petrovskaya N.V. Samarodnoye zoloto. Obshaya xarakteristika, tipomorfizm, voprosi genezisa. -M.; «Nauka», 1973. -347 s.
12. Guchetl I.S., Druker E.A., Barishnikov I.F. Pererabotka upornix zolotosoderjashix rud i konsentratov. -M.; «Svetmetinformatsiya», 1992.- 60 s.

13. Juravlyov B.N., Moskvicheva G.I., Gudrzibeev T.D. i dr. Izvlecheniye zolota iz mishyakovistix zolotosoderjashix rud. Gidrometallurgiya zolota. - M.: «Nauka», 1980; S. 19-22.
14. Permyakov V.V. i dr. Texnika I texnologiya izvlecheniya zolota iz rud za rubejom. - M.:Metallurgiya, 2003; -237 s.
15. KarimovI.A.. Po puti bezopasnosti i stabilnogo razvitiya.-T.: «Uzbekistan», 1998.- 158 s.
16. KarimovI.A..Miravoy finansoviy krizis, puti i meri po ego preodeleniyu v usloviyax Uzbekistana.-Tashkent: Uzbekistan, 2009. -87 s..
17. [http:// www.rsl.ru](http://www.rsl.ru) – rossiyskaya gosudarstvennaya biblioteka.
18. [http:// www.elibrary.ru](http://www.elibrary.ru) – nauchno – elektronnyaya biblioteka.
19. [http:// www.geolge.ru](http://www.geolge.ru)
20. [http:// www.rambler.ru](http://www.rambler.ru)

UDC.547.458.81

### NEW COMPOSITION MATERIAL FOR WINE FILTRATION

E. A. Egamberdiev<sup>1</sup>, M. K. Abdumavlyanova<sup>1</sup>, G. R. Rakhmonberdiev<sup>1</sup>  
Tashkent chemical-technological institute

**Annotatsiya.** Jahon miqyosida sellyuloza asosidagi kompozitsion materiallar jumladan, turli maqsadlarda qo'llaniladigan qog'oz va qog'oz maxsulotlari kimyo, oziq-ovqat, neft va gazni qayta ishlash sanoatida, tibbiyot va farmatsevtika sohasida mahsulot tozaligini ta'minlash maqsadida fil'trlovchi materiallar sifatida, kundalik turmushda o'ram qog'ozlari, qimmatbaho qog'ozlar sifatida keng qo'llanilib kelinmoqda. Sellyuloza asosida kompozitsion materiallar olish, kompozitsiya tarkibidagi chetdan keltiriladigan ingradientlarni mahalliy lashtirish, ularni qo'llanilish sohaslarini o'rganish, texnologiyalarini yaratish hamda amaliyotga qo'llash muhim vazifalardan biri bo'lib qolmoqda.

Jahonda tabiiy polimerlar asosida yangi qog'oz kompozitsiyalarni yaratish, ular assortimentlarni faqatgina o'simlik tolalaridan emas balki, noan'anaviy tolalardan foydalangan xolda noyob va kamyob xususiyatga ega bo'lgan mahsulotlarni qo'llash sohaslarini kengaytirish, bu sohadagi ustuvor yo'nalishlarga an'anaviy qog'oz ishlab chiqarish va mavjud uskunalarni asosidagi mineral tolalardan qog'oz kabi materiallar ishlab chiqarish bo'yicha intensiv rivojlantirish borasida yaratishga bag'ishlangan ilmiy-tadqiqotlar olib borilmoqda.

Respublikamizda oxirgi yillarda kimyo sanoatidagi aniq va keng ko'lamli chora-tadbirlar natijasida mavjud va yangi ishlab chiqarish korxonalarini modernizatsiya qilish, raqobatbardosh maxsulotlar turlari va hajmining kengayishiga katta e'tibor qaratilmoqda, shu jumladan, mahalliy xomashyolar asosida qog'oz va qog'oz mahsulotlari ishlab chiqarish texnologiyalarini rivojlantirish bo'yicha ilmiy va amaliy natijalarga erishilmoqda.

Tadqiqot natijalarining ilmiy ahamiyati turli tolali bir yillik o'simliklar poyalaridan olingan sellyulozalarning fizik, fizik-kimyoviy va struktura xossalari ulardan olingan mahsulotlar sifat ko'rsatkichlari o'rtasida korrelatsiyani amalga oshirilganligi bilan izohlanadi. Tadqiqot natijalarining amaliy ahamiyati respublikada mavjud xom ashyolar asosida kompozitsion materiallar olish usulini ishlab chiqish va olingan materiallarni yengil sanoat, oziq-ovqat va qishloq xo'jaligi sohaslarida foydalanishni tavsifa etishdan iborat

**Abstract.** Currently, cellulose-based composites, including paper, paper-like materials and paper products, are widely used in the chemical, oil refining, pharmaceutical, medicine, household and other industries as filter, wrapping paper and packaging material.

*The preparation of cellulose-based composite material, the development of technologies for their preparation, the study of their properties and applications, the localization of imported ingredients in composites, the study of their fields of application is an urgent problem.*

*All over the world, scientific research is being conducted aimed at creating new effective composite materials based on natural fibers. It is advisable to expand the range of paper-like materials not only from plant fibers, but also from non-traditional fibers, which makes it possible to create composite paper-like materials with rare and unique properties. As a result of specific and large-scale measures taken in the chemical industry of our republic, much attention is paid to the modernization of new production enterprises, the expansion of the types and volumes of competitive products, including scientific and practical results on the development of paper technology and paper products based on local raw materials.*

*The scientific significance of the research results is to establish the influence of the physical, chemical and structural characteristics of cellulose obtained from various one- and perennial cellulose-containing plants on the properties of the obtained composite materials based on them.*

*The practical significance of the research results lies in the fact that the obtained paper-like composite materials can be used as filtering and packaging materials in the oil and gas, automotive, food and agricultural sectors.*

**Key words:** *Cellulose from topinabour, cellulose from cotton linters, basalt fiber, strength, composite filter paper, fiber hydration, filler.*

**Аннотация.** *В настоящее время композиционные материалы на основе целлюлозы, в том числе бумага, бумагоподобные материалы и бумажные продукты широко используются в химической, нефтеперерабатывающей, фармацевтической промышленности, медицине, быту и других отраслях в качестве фильтровальной, оберточной бумаги и упаковочного материала.*

*Получение композиционного материала на основе целлюлозы, разработка технологий их получения, изучения их свойств и областей применения, локализация импортных ингредиентов в композитах, изучение областей их применения является актуальной проблемой.*

*Во всем мире ведутся научные исследования, направленные на создание новых эффективных композиционных материалов на основе натуральных волокон. Целесообразным является расширение ассортимента бумагоподобных материалов не только из растительных волокон, но и из нетрадиционных волокон, что даст возможность создать композиционные бумагоподобные материалы с редкими и уникальными свойствами. В результате конкретных и масштабных мер, принятых в химической промышленности нашей республики, большое внимание уделяется модернизации новых производственных предприятий, расширению видов и объемов конкурентоспособной продукции, включая научные и практические результаты по разработке технологии бумаги и изделий из бумаги на основе местного сырья.*

*Научная значимость результатов исследования заключается в установлении влияния физических, химических и структурных характеристик целлюлозы полученной из различных одно- и многолетних целлюлозосодержащих растений на свойства полученных композиционных материалов на их основе.*

*Практическая значимость результатов исследования заключается в том, что полученные бумагоподобные композиционные материалы могут быть использованы в качестве фильтрующих и упаковочных материалов в нефтегазовой, автомобильной, пищевой и сельскохозяйственной сферах.*

**Introduction.** One of the main indicators characterizing the quality of the finished wine is its transparency, which must meet the concepts of "crystal transparency" and "transparent with brilliance." The filtration process plays a crucial role in the further transparency of the wine. Filtration is widely used in the processing of primary and secondary products of winemaking, in particular, yeast sediments, grape juice and wine materials. However, the task of increasing the transparency and biological stability of the filtrates remains relevant. [1-3].

**Methods and materials.** Different concepts are meant by the words "transparency" and "purity" of a liquid. A transparent liquid is a liquid that contains particles not exceeding 1  $\mu$  and which transmits light well. [2]. A pure liquid is a completely homogeneous medium that does not contain suspended particles, in other words, its particles do not exceed 1  $m\mu$ . An ordinary microscope does not pick up particles having a size less than 0.2  $\mu$ , but these particles are detected using an ultramicroscope, which makes it possible to see the reflection of light from particles suspended in a liquid, the sizes of which are very small (3-10  $\mu\mu$ ).

Filtration is the operation of separating the solid phase of a substance from the liquid using a porous septum.

The filtration process is based on the retention of solid suspended particles by porous partitions capable of passing liquid and retaining solid particles on its surface. [4-6].

As a result of direct contact of the suspension with the surface of the porous septum, the liquid phase, due to the pressure difference on one or the other side of the septum, passes through the pores of the septum and collects in the form of a filtrate free from solid particles; solid particles are retained on the surface of the septum, forming a layer of sediment.

The filtration technique uses this layer of sludge on the septum, which increases as the process proceeds, as a filter medium, while striving to minimize the hydraulic resistance of the sludge. The precipitate deposited on the surface of the filtering septum is one of the most important factors, in most cases deciding the success of the filtration process. The filter performance and the energy consumption for moving the liquid through the filter layer depend on the nature of the precipitate and the thickness of its layer. [1-7].

The diameter of the pores or tubules through which the liquid penetrates must be in accordance with the degree of dispersion of the turbidity particles. The filter should have the finer the latter, the smaller the pores. The size of the suspended particles can vary within a very wide range - from fractions of a millimeter to very small sizes, expressed in a few nanometers.

After fermentation, particles of wort, yeast and tartar remain in the wine, making the drink cloudy. [5]. This problem is solved by processing the finished wine with special substances that absorb unwanted particles, causing them to precipitate. In the language of professionals, wine clarification is called "fining". The most effective method of clarifying wine is long aging. But this natural process takes too much time (3-6 years) and is suitable only for expensive branded drinks.

In production conditions, wine passes through a filter. The filter layer is formed by the filter baffle and sediment deposited in it. As partitions in the filters, cotton, linen and woolen (rarely) fabrics are used, as well as rigid metal nets, asbestos, cellulose, asbestos and cellulose plates, as well as collodion plates on cellulose, and more recently, perlon films.

The material for the formation of the filter layer must pass through all the substances dissolved in the wine and keep it in suspension. Semi-permeable membranes and membranes preventing the passage of colloids contained in wine in a dissolved state cannot be used as filter material. [9-10].

Thus, wine filtration during production is a multi-stage production process in which mechanical suspensions, turbidity, yeast sediment, sizing agents, tartar, microorganisms and other substances affecting the durability and organoleptic characteristics of the finished product are separated from the wine material. Depending on the technological scheme, type of wine, method of microbiological stabilization, productivity (capacity of the enterprise), various types of filtration are used at certain stages of wine production. Any filtration should ensure on the one

## Chemistry and chemical technology

hand a high purity of the product, and on the other hand, not affect its dissolved components. Properly selected filtering has a positive effect on the quality of the wine, while thoughtless filtering can cause permanent damage. [8-9].

**Results and discussion.** We have obtained filtering material obtained on the basis of cellulose of various origins, including waste from the pulp and paper industry, basalt fiber and mineral fillers.

The composition of the filter material obtained by us is shown in table №1.

**Table 1**

**Composition of the filter material**

№	Ingridients	Content of composition, amount, in %				
		№1	№2	№3	№4	№5
1.	Cellulose from annual plant (topinambour)	45	-	-	30	-
2.	Cellulose from annual plant (cotton lint)	-	40	-	30	-
3.	Wastes from cellulose-paper manufacture	-	-	-	-	40
4.	Basalt fiber	20	20	25	-	20
5.	Waste paper MC6-MC7	5	10	45	40	10
6.	Solution from sulgate aliminium	30	30	30	-	30

Sample preparation for filtration was carried out according to the technology introduced into the enterprise.

Using a polymer-composite (paper-like) material as a filter in the production sites of HAMKOR LLC, an experimental batch of wines of various brands such as “Versal”, “Noterdam”, “Kadimiy-Bukhara”, “Jador”, “Samarkand”, “Khiva”, “Muskat” and “Kabernet” obtained from grapes of various varieties was filtered.

The results are shown in table 2.

**Table 2**

**Material quality indicators**

Name of quality indicator	Plant filter	Accordind to one-time technological regulations					Factual on the standard
		Sample №1	Sample №2	Sample №3	Sample №4	Sample №5	
“Versal”	transparent	transparent	transparent	weak opacity	possesses opacity	possesses opacity	transparent
“Noterdam”	red	red	red	possesses settlement of grapes parts	possesses settlement of grapes parts	possesses settlement of grapes parts	red
“Kadimiy-”	ruby	ruby	light brown	unclarified	possesses settlement of	possesses settlement	ruby



Bukh ara” №1					grapes parts	of grapes parts	
“Kadi miy-Bukh ara” №2	light yellow	transparent yellow	light yellow	light yellow	possesses settlement of grapes parts	possesses settlement of grapes parts	transparent yellow
“Jador”	pink	pink	pink	weak opacity	possesses settlement of grapes parts	possesses settlement of grapes parts	pink
“Sam arkan d” №1	crimson	crimson	crimson	crimson	possesses settlement of grapes parts	possesses settlement of grapes parts	crimson
“Sam arkan d” №2	unclarified	unclarified	unclarified	unclarified	possesses settlement of grapes parts	possesses settlement of grapes parts	transparent
“Khiv a” №1	crimson	crimson	crimson	crimson	possesses settlement of grapes parts	possesses settlement of grapes parts	crimson
“Khiv a” №2	unclarified	unclarified	unclarified	unclarified	possesses settlement of grapes parts	possesses settlement of grapes parts	transparent
“Mus kat” №1	crimson	crimson	crimson	unclarified	possesses settlement of grapes parts	possesses settlement of grapes parts	crimson
“Mus kat” №2	unclarified	unclarified	unclarified	unclarified	possesses settlement of grapes parts	possesses settlement of grapes parts	unclarified
“Kab ernet”	ruby	ruby	ruby	light brown	light brown	light brown	ruby

Studies have shown that the use of filtering material made it possible to filter wine with the required quality indicators, which made it possible to replace the filtering material used at the plant based on wood pulp, which is purchased abroad. The best indicators are criteria for assessing the quality of wine, such as transparency, turbidity, showed sample №1 and sample №2, obtained on the basis of cellulose from annual plants (topinambour, cotton fiber), basalt fiber, inorganic filler and waste paper. [11-13].

The use of cellulose obtained from perennial and perennial plants, the cost of which is several times lower than the materials currently used in the filter, has reduced the cost of the product. [12-14]. Also, during the filtration of the production of an experimental batch of wine, it was found that up to 10-25% of waste paper can be found in the composition of such materials without a significant reduction in their basic properties, which also lead to a reduction in the cost of the material.

Most bottling lines use a two-stage wine filtering scheme. The combination of "prefilter - finish filter" increases the reliability of the process, provides maximum performance and efficiency of the filtration system. The filtering action consists of two processes: screening and adsorption. If the particles are larger than the pores of the filter layer, they are screened. But the suspension of suspended particles of turbidity during filtration occurs not only from the outside as a result of screening, but also inside the filter layer as a result of adsorption. During filtration, suspended particles, the diameter of which does not allow them to pass through the inlet openings, remain on the surface of the filter layer. [15]. Accumulating at the inlet openings and cluttering them, the particles form a second filter layer through which the liquid must pass. As the liquid is filtered, the layer thickens and the rate of liquid penetration through the layer progressively decreases until the filtration completely stops.

**Conclusion.** In the case of using a filter, this effect developed by us is achieved by a single-stage wine filtration scheme, which leads to a reduction in this technological cycle. This is due to the presence of basalt fiber, which is present in the composition of the composite material, which has led to an increase in the time of use of the filter material.

The results of production tests fully confirmed the laboratory results, which indicates good on their performance.

The results obtained make it possible to replace the imported filter material with a domestic one and save currency.

### References

1. Egamberdiev E., Rakhmanberdiev G., Mardonova A. "Study of the sorption rate of composition paper samples obtained on the bases of cellulose-bearing plants cellulose and basalt fiber" Austrian Journal of Technical and Natural Sciences 1-2, 2018.56-62p
2. Mirkamilov T.M. Issledovanie vliyaniya usloviy ximicheskoy ochistki siklonnogo puxa i korotkoshtapelnogo linta na ximicheskoy sostav poluchennoy sellulozi i ee prigodnost dlya pererabotki na atsetilsellyulozu i visokokachestvennyu bumagu. Avtoref.dis.kand.tex.nauk. Leningrad, 1966, S.26.
3. Zaripova A.M., Golovnikova M.V., Tishaboev U., Igamberdiev I.I., Usmanov X.U. Vozmozhnost ispolzovaniya zasorennix sortov linta dlya polucheniya visokokachestvennoy xlopkovoy sellulozi. V kn.: Struktura i modifikatsiya xlopkovoy sellulozi. Vip. 3, izd. "Fan", UzSSR, Tashkent, 1966, S.11-15.
4. Akim L.E., Mirkamilov T.M., Usmanov X.U. Poluchenie visokokachestvennoy xlopkovoy sellulozi iz siklonnogo puxa dlya pererabotki ee na atsetilsellyulozu i bumagu. - V kn.: Struktura i modifikatsiya xlopkovoy sellulozi. Tashkent, 1966, izd. "Fan", S.32-37.
5. Tishaboev U. Vliyanie texnologicheskogo rejima polucheniya sellulozi iz xlopkovogo linta na ee ximicheskoy sostav i reaktivnuyu sposobnost k viskozobrazovaniyu. Avtoref.diss.kand.tex. nauk. Tashkent, 1970, S.22.
6. Eroxin N.G. Razrabotka texnologii polucheniya visokooblagorajennoy xlopkovoy sellulozi dlya proizvodstva atsetatov sellulozi. V kn.: Ximiya i texnologiya proizvodnix sellulozi. VNIIS, g. Vladimir, 1964, S.141.
7. Zaripova A.M., Plovnikova M.V., Tishabaev U., Igamberdiev I.I., Usmanov X.U. Vozmozhnost ispolzovaniya zasorennix sortov linta dlya polucheniya visokokachestvennoy xlopkovoy sellulozi. V kn. Struktura i modifikatsiya xlopkovoy sellulozi. Vip. 3, izd. "Fan", UzSSR, Tashkent, 1966, S.11-15.

8. Sayfutdinov R.S. Razrabotka ximicheskoy texnologii ispolzovaniya otxodov xlopkovodstva dlya proizvodstva drevesno-strujchnix plit i sellulozi. Avtoref.dis.dokt.tex.nauk. Tashkent, 1998, S.49.
9. Primkulov M.T., Ismoilov S.N., Umarova V.K. // Bugʻdoy somoni, sholi va gʻoʻza poyalaridan selluloza olish va ularning strukturasini suvda boʻkishi usuli orqali oʻrganish. // Kompozitsion materiallar. 2015g. №1. Toshkent. – 62-64 betlar.
10. Nikitin N.I., Klenkova N.I. Vliyanie slabogo alkilirovaniya na svoystva selluloznogo volokna. Jurnal prikladnoy ximii, 24, 296, 1951.
11. Klenkova N.I., Kulakova N.A. Deystvie pervichnix aminov jirnogo ryada na strukturu i reaksionnuyu sposobnost xlopkovix selluloznix volokon. Jurnal prikladnoy ximii. 38, 4, 1965, S.919.
12. A.S.573524 SSSR Sposob polucheniya xlopkovoy sellulozi. Mirkamilov T.M., Turaev E., Djalilov A.T., Askarov M.A./- Opubl.v BI N 35, 1977.
13. A.S.604888 SSSR Sposob polucheniya xlopkovoy sellulozi. Mirkamilov T.M., Turaev E., Djalilov A.T., Askarov M.A./-Opubl.v BI N 16, 1978.
14. Bozorov O.N. Texnologiya pererabotki nizkosortnogo linta dlya polucheniya efirov sellulozi. Avtoref.diss. kandidata texnicheskix nauk. – Tashkent, 2005, S.22.
15. Akim E.L., Mirkomilov T.M., Akim L.E., Usmanov X.U. Vliyanie chastichnogo oksietilirovaniya na skorost atsetilirovaniya xlopkovoy sellulozi, poluchennoy iz siklonnogo puxa i korotkoshtapelnogo linta. V kn. Trudi Leningradskogo instituta SBP. vip. 23, M., 1970, izd.Lesnaya promishlennost, S.59-62.