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Engineering and Technology

Potash Ore Processing: Technology Research and Physicochemical Properties

***Urazkeldiyeva D.A., Kadirbayeva A.A., Koshkarbayeva Sh.T.**

M. Auezov South Kazakhstan Research University, Shymkent, Kazakhstan

** Corresponding author email: urazkeldieva.97@list.ru*

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ABSTRACT

The Republic of Kazakhstan is endowed with a distinctive endowment of potash ores, concentrated in the West Kazakhstan and Aktobe regions. These reserves are regarded as one of the largest in the world. The proven reserves are estimated at approximately 6 billion tons and are distributed across four major deposits: These are the Zhilyanskoye, Satimola, Inderskoye and Chelkar deposits. The article provides a concise overview of the major potassium salt deposits and the chemical composition of the associated minerals. Notwithstanding the existence of these deposits, the production of potash salts in the country, for which there is an ever-increasing demand, is yet to be established, resulting in a high level of demand. In light of this, it is imperative to conduct a comprehensive investigation into the mineralogical and chemical composition of these promising potash ores, to identify viable methods for processing natural salt systems into products that meet the high demand both within the domestic fertiliser and salt market and abroad. The Satimola deposit represents one of the largest silvinite basins yet to be sufficiently studied, and its industrial development has yet to commence. The analysis of raw materials and products was conducted using a combination of spectral microscopy, X-ray analysis, and differential thermal analysis. A comprehensive study of the composition of the silvinite ore from the Satimola deposit has been conducted. The elemental composition and the ratio of potassium and sodium salts in the mineral were established. The ore was found to have a heterogeneous composition, with sodium chloride representing the dominant component.

Keywords: potassium chloride, potassium, halurgic method, silvinite, Satimola deposite.

	Information about authors:
Urazkeldiyeva Dilbar Abdikhmidovna	*PhD doctoral student, The Higher School of Chemical Engineering and Biotechnology, M. Auezov South Kazakhstan University, Shymkent, Kazakhstan. Email: urazkeldieva.97@list.ru; ORCID ID:https://orcid.org/0000-0001-7825-6995*
Kadirbayeva Almagul Akkopeykyzy	*Candidate of technical sciences. Assistant Professor. The higher School of chemical engineering and biotechnology, M. Auezov South Kazakhstan University, Shymkent, Kazakhstan. Email: diac_2003@mail.ru; ORCID ID: https://orcid.org/0000-0003-0702-1114*
Koshkarbayeva Shaizada Tortaevna	*Candidate of chemical sciences. Assistant Professor. The higher School of chemical engineering and biotechnology, M. Auezov South Kazakhstan Research University, Shymkent, Kazakhstan. Email: shayzada-1968@mail.ru; ORCID ID: https://orcid.org/0000-0001-8753-3245*

Introduction

Potassium salts represent a significant and highly sought-after resource in the contemporary economic landscape. The principal product resulting from the processing of potash salts is potash fertilisers. Since the middle of the 20th century, the global population has continued to grow at a steady rate, which has increased food shortages. In the contemporary era, the intensification of food production, including the introduction of fertilisers, is a prerequisite for global agricultural practices. Consequently, at the beginning of the 21st century, there was a notable surge in demand for potash fertilisers. For the first time since the 1970s, projects for the expansion of existing production facilities, as well as the study of new fields, and the design and construction of new enterprises, have commenced. In addition to the well-known potassium basins of Western Europe, Canada, Russia and Belarus, new deposits of potassium salts are currently being explored in Central and Southeast Asia, South America, West and East Africa, as well as previously undeveloped euporite deposits in North America (Rauche, 2015; Cocker, 2016). The global total of potash salt reserves is estimated at 40 billion tons. According to reference [1], the leading producers of potassium are Canada, Russia, Belarus, and Germany (Figure 1).

Nevertheless, the intensification of production inevitably conflicts with the objective of preserving and developing the natural environment of the regions in which it is situated, while simultaneously minimising anthropogenic impact. The potash industry represents a complex economic sector, encompassing both mining and chemical production [2].

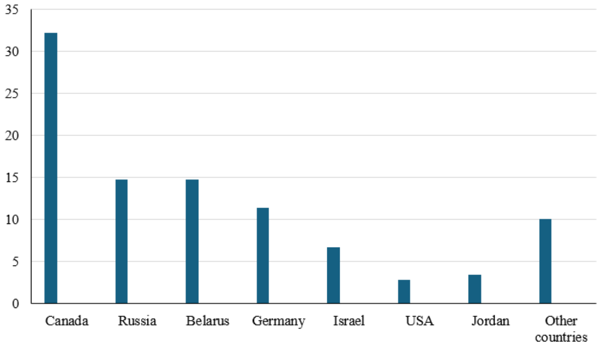

Figure 1 - Distribution of potash production by country, % [1]

Potassium is a relatively common chemical element. In terms of its concentration in the Earth's crust (approximately 2%), potassium is the second most abundant element after oxygen, with silicon, aluminium, iron, calcium and sodium also present in significant quantities [3]. Potassium is present in several naturally occurring minerals, including feldspar, granites, leucites, gneiss, and solid fossil salt deposits. It is also found in salt waters of marine and continental origin. The most common minerals containing potassium are as follows: sylvine KCl, carnallite $KCl\bullet MgCl_2\bullet 6H2O$, langbeinite $K_2SO_4\bullet 2\ MgSO_4$, cainite KCl- $MgSO_4\bullet 3H_2O$, shenite $K_2SO_4\bullet MgSO_4\bullet 6H_2O$, glazerite $K_2SO_4\bullet XNa_2SO_4$, polygalite $K_2SO_4\bullet MgSO_4\bullet 2CaSO4\bullet 2H_2O$, and alunite $K_2SO_4\bullet A1_2(SO_4)_3\bullet 4Al(OH)_3$. The potassium-containing aluminosilicates are as follows: $K_2O\bullet Al_2O_3\bullet 6SiO_2$; muscovite $K_2O\bullet 3Al_2O_3\bullet 4SiO_2\bullet 2H_2O$; nepheline $(K, Na)_2O\bullet Al_2O_3\bullet 2SiO_2$; leucite $K_2O\bullet Al_2O_3\bullet 4SiO_2$ [4].

The main sources of potassium compounds are soluble potassium salts such as sylvinite, carnallite, langbeinite and kainite [[5], [6]]. In Kazakhstan, the main potassium salt reserves are located near the North Caspian Sea (potassium chloride salts) and the Ural Mountains in Aktobe (potassium sulphate salts). In the North Caspian region, the balance reserves are 8 million tonnes, with inferred resources of about 500 million tonnes. In the vicinity of the Ural Mountains (Aktobe), balance reserves are estimated at 100 million tonnes and inferred reserves at 300 million tonnes. The inferred potash resource in Western Kazakhstan in the 1980s was 1 billion tonnes, including 217 million tonnes of sulphate salts.

The Inder salt dome deposit has proven reserves of 709 million tonnes, of which 40 million tonnes are in commercial categories. Lake Inder in Inder district contains about 1.5 billion tonnes of salt, of which 647 million tonnes have been explored since 1993 [7].

Potassium salts are an important commercial product of Kazakhstan. Studies of their raw material base began in the 1950s [8]. The availability of reserves and the demand for potash fertilisers require the development of new processing technologies. The potash industry development strategy includes increasing explored reserves and introducing innovative technologies [[9], [10]]. It is also important to develop resource-saving potash processing technologies and improve the qualifications of labour resources for the efficient operation of the mining and processing complex [11].

The high demand for potassium salts in Kazakhstan emphasises the relevance of developing technologies for the production of chlorides, sulphates, phosphates, and other products from local chloride salts [12].

The Republic has significant reserves of natural salts containing sodium and potassium chloride. These salts are found in different regions of the country, including Atyrau (deposits of Inder, Chelkar, Satimola), and Zhambyl regions [[13], [14]].

The Satimola, Inder, Shalkar and Zhylandy deposits have some of the largest potash reserves. A mining and processing complex is currently under construction at the Satimola deposit to produce potassium chloride. The mine is expected to have a capacity of 25 million tonnes of potash ore per year and the reserves will take more than 50 years to develop. Geological studies began as early as the 1960s, and in 2010 a potash fertiliser production technology was developed [15].

The Satimola deposit, located in the West Kazakhstan region, contains potassium and boron salts. The potassium content of the ore characterises ores for medium, high and priority recovery. The salt and mineral composition of the samples Satimola deposit is shown in Table 1,2 [[16], [17]].

Table 1- Salt content of the ore [16]

№	Mass fraction, %					
	KCl	$NaCl$	$CaSO_4$	$MgSO_4$	K_2SO_4	i/r
1	27.2	65.1	14.4	1.44	1.13	0.48
2	42.8	52.44	1.83	1.47	0.96	0.33
3	32.9	60.78	2.65	2.33	0.86	0.48

Table 2 - Mineral composition of the ore [16]

№	Mass fraction, %					
	Sylvin	Halite	Anhydrite	Polyhalite	Kieserite	i\r
1	27.21	65.1	2.63	3.91	0.76	0.48
2	42.76	52.4	0.33	3.32	0.93	0.33
3	32.86	60.8	1.31	2.98	2	0.48

When studying the mineralogy of halide deposits of the Satimola structure, it is important to consider the minerals of the salt beds. The salt beds contain more than 30 minerals including halogens, sulphates, borates and carbonates due to the presence of clayey material and differences in their quantities and textures.

Lake Inder is a large salt lake in the northern part of the Atyrau region of Kazakhstan. Since 1932, potash salts have been prospected in the pre-Caspian lowland. From 1939 to 1945, drilling operations on the Inder Upland revealed deposits of polyhalite rocks and sylvinites, and further research was carried out by the Inder Geological Exploration Expedition and the Research Institute of Galurgy [13].

The Zhylandy deposit in the Ural region, 5-10 km from Aktobe, has potash reserves of 500 to 600 million tonnes of crude ore [18]. The rocks of the deposit contain polyhalite (65%) and sylvinite with anhydrite and calcite. Polyhalite deposits are located at a depth of 235-770 metres and are separated by layers of rock salt. The Shalkarskoye potash deposit is a salt dome with commercial layers of potassium-magnesium salts at a depth of 300-1000 metres [[20], [21]].

From the analytical review, it can be concluded that potash processing is of high relevance due to the significant resources and long-term development prospects. The Satimola deposit, with more than 50 years of reserves, is of strategic importance for agriculture, as potash is an important fertiliser. The development of efficient processing technologies will support the region's economic growth, creating jobs and improving the environmental situation. The introduction of resource-saving technologies will ensure sustainable development of the industry and meet the growing demand for potash fertilisers.

Experimental part

Methods of analysis. The analysis of raw materials and products was conducted using a combination of spectral microscopy, X-ray analysis, and differential thermal studies. X-ray identification was conducted on a stationary installation, designated DRON-4, which employs a tube with a cobalt anode. The voltage applied to the tube was 40 kV, and the current was 40 mA. The decoding of samples and the search for phases were conducted using the Search/Match program with the 2003-2023 CRYSTAL IMPACT powder diffractometric database, Bonn, Germany. A raster electron microscope (REM) JSM 6490l V (Jeol, Japan) was employed for the microscopic spectral analysis of raw materials. The REM is based on scanning the surface of the sample with an electronic probe, whereby a wide range of radiation is recognised during this process. The ore was subjected to differential thermal analysis using a Q-1500D derivatograph. A derivatograph is a device for complex thermal analysis that allows for the simultaneous measurement of changes in temperature and mass, the rate of temperature change and the change in enthalpy of the substance under study.

Experimental methodology. A pre-prepared solution containing 12.5% potassium chloride (KCl) and 18.5% sodium chloride (NaCl) is placed in the reactor, which corresponds to the composition of the mother liquor obtained after crystallization of potassium chloride (KCl). This solution serves as the basis for further leaching and separation of the components. The reactor is placed in a thermostat, where it is heated to a temperature of 100 °C. After reaching the set temperature, a set amount of sylvinite is added to the reactor. Leaching of potassium chloride (KCl) is carried out within 1- 1.5 hours. During this period, it is important to maintain a stable temperature (100°C) and continue mixing the solution to speed up the dissolution process and ensure complete potassium extraction. At the end of the leaching time, the process is interrupted, and the mass from the reactor is transferred to the filter for hot filtration. For this purpose, a cylinder and a funnel are used, which are pre-weighed, which allows you to accurately take into account the mass of the filtrate. The filtrate is collected in pre-weighed vessels (cylinder or glass), after which the solution and the wet NaCl precipitate remaining after filtration are weighed. After collecting the filtrate, the solution is cooled under running water to 25 ° C. As a result of cooling, a precipitate of CL precipitates on the surface of the solution. This precipitate is further filtered and then weighed to determine the mass of the extracted potassium. After all operations, the data obtained is analyzed, calculating the effectiveness of leaching of potassium oxide and determining the potassium content in the sediment.

The discussion of the results

The objective of this research was to obtain a natural mineral potassium from the Satimola ore. The resulting mineral sample is illustrated in Figure 2.

Figure 2 - Natural Potassium Mineral from Satimola Ore

In order to fully study the mineral composition, a series of physicochemical analysis methods were employed, including raster electron microscopy, differential thermal analysis and X-ray phase analysis. The results of the XRF (Fig.3) indicated that the mineral is composed of 81.2% NaCl and 13.1% KCl, with a further 5.7% constituted by okenite mineral.

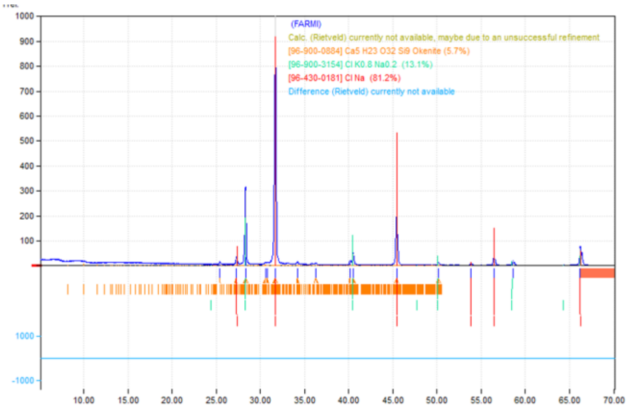

Figure 3 – XRF of potassium mineral from Satimola ore

The results of the REM analysis (Fig.4., Tab.3.) indicate that the ore contains significant quantities of sodium, potassium and chlorine elements. Upon calculation of the elemental composition of the salts, it was determined that the mineral comprises 68% NaCl and 32% KCl.

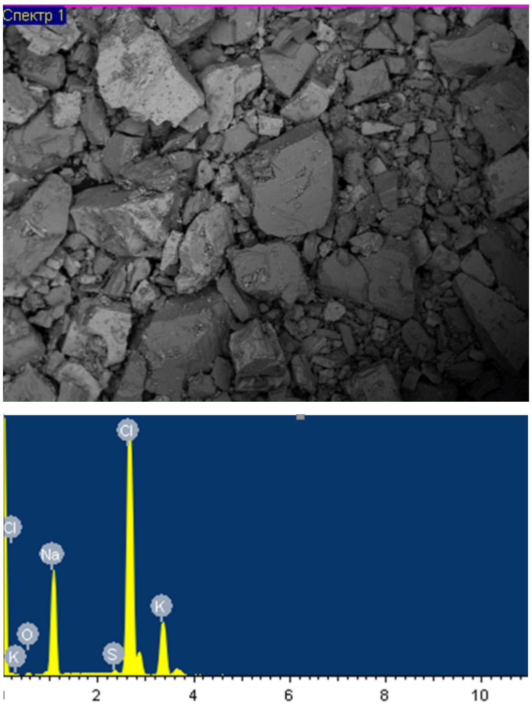

Figure 4 – Microstructure image and spectrogram of potassium mineral from Satimola ore

Table 3 - Elemental composition of potassium mineral from Satimola ore

Element	O	Na	Cl	S	K	Total
Mass. %	3.39	26.78	52.29	0.33	17.21	100.0

The DTA curve (Fig.5) of the natural salt of potassium chloride and sodium with a twofold predominance exhibits four stepwise exothermic effects and two intense endothermic effects.

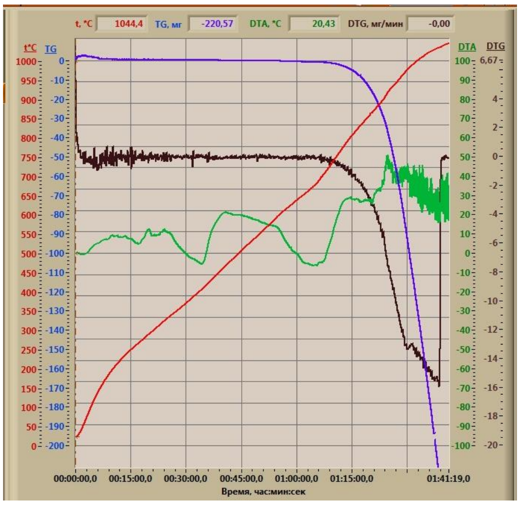

Figure 5 - Differential-thermal analysis of potassium mineral from Satimola ore

The stepwise exoeffects observed in the region of 230-330°C are indicative of the molecular-bound moisture. At 470°C, the exothermic effect indicates the removal of crystallohydrate moisture. An intensified endoeffect at 670°C is linked to the decomposition of potassium chloride salt. The stepwise curve of the endoeffect in the region of 950°C is indicative of the decomposition of the complex of inorganic salts.

The discrepancy in salt content between the REM and XRF results can be attributed to the unequal distribution of elements in the resulting sample. As illustrated in Figure 2, the sample comprises a mixture of pink, white-yellow, and maroon crystals. To comprehensively examine the mineral's composition, a detailed physical and chemical analysis was conducted on each individual crystal. The elemental analysis revealed that the red-coloured composition of the mineral (Fig.6., Tab.4.) is predominantly composed of potassium chloride, with a range of 90-98%.

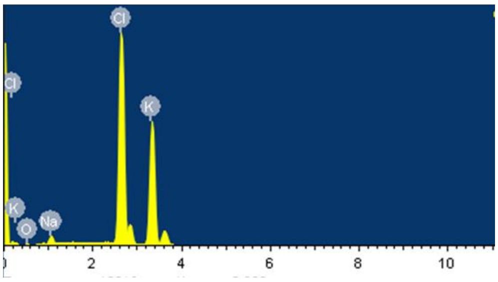

Figure 6 - Spectrogram of red crystals of potassium mineral from Satimola ore

Table 4 - Elemental composition of red crystals of potassium mineral from Satimola ore

Element	O	Na	Cl	K	Total
Mass. %	3.01	3.21	48.52	45.26	100.0

The results of the elemental analysis of the white crystals (Fig.7., Tab.5.) of the natural mineral from the Satimola deposit demonstrated that the sample contains 97-99% sodium chloride, which is distinguished by markedly intense picts of sodium and chlorine elements.

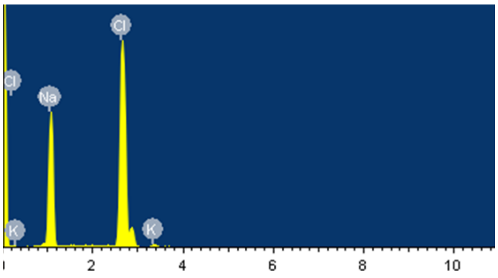

Figure 7 - Spectrogram of white crystals of potassium mineral from Satimola ore

Table 5 - Elemental composition of white crystals of potassium mineral from Satimola ore

Element	Na	Cl	K	Total
Mass. %	39.15	59.75	1.10	100.0

According to the results of physico-chemical analyses, the sample contains 81.2% sodium chloride (NaCl) and 13.1% potassium chloride (KCl), which indicates the presence of two main components in the mineral — halite (NaCl) and sylvinite (KCl). A high level of sodium chloride indicates the predominance of halite, which is typical for minerals formed under conditions of evaporation of water, such as in salt deposits. The sample also contains 5.7% okenite, a calcium silicate mineral that is an insoluble residual. This indicates that calcium compounds could be involved in the formation of the mineral, forming okenite as a by-product. Okenite is insoluble in water and is part of the insoluble residue, which indicates its role as a mineral that cannot be dissolved during processing. The sample is a mixture of halite and silvinite with the presence of okenite. This composition is typical for potash deposits or evaporative deposits, where sodium and potassium chlorides, as well as insoluble minerals such as okenite, can coexist. This confirms that the sample is potentially promising for the extraction of potassium and sodium salts, as well as for further research for possible processing.

The halurgical method was chosen for processing the potassium salt of the Satimol deposit. The halurgic method of mineral processing has several key advantages over flotation, including higher separation efficiency, less dependence on chemical reagents, a high degree of extraction of target components, and the ability to process complex mineral compositions. These aspects make the halurgy method preferable in several cases, especially in the processing of potassium and sodium salts, where the accuracy and purity of the final product play an important role. The processing of this sample by halurgy involves the effective extraction of sodium chloride (NaCl) and potassium chloride (KCl) by dissolution and crystallization, as well as the processing of the insoluble mineral okenite to obtain additional products. This approach will make it possible to obtain high—quality products - potassium and sodium salts, which can be used in industry, as well as calcium compounds, if necessary**.**

By the method of metallurgy described earlier, the technological scheme is shown in Figure 8.

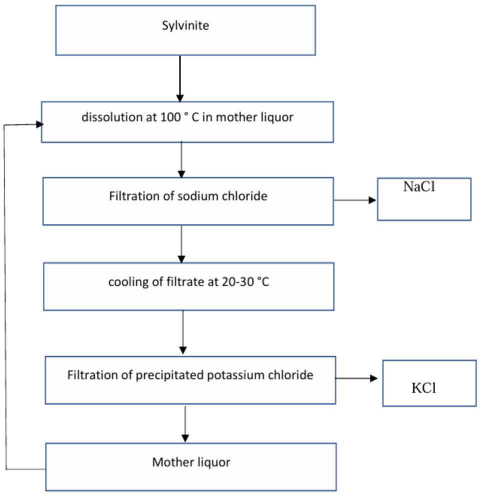

Figure 8 - Technological scheme of potassium chloride processing by the halurgic method

Processing of potash ore allowed to obtain high—quality products - potassium chloride (KCl) and sodium chloride (NaCl). According to the experimental results, the yield of the potassium chloride product was 95-98% of the theoretically possible, which indicates a high efficiency of leaching and separation of components during processing.

Elemental analyses of the obtained potassium and sodium chlorides, carried out using spectroscopy methods, showed that the products have a high degree of purity and meet the standards for further industrial use. The spectrograms obtained during the analysis, as well as the results of the elemental analysis, are shown in Figures 9,10 and Tables 6,7, which reflect the content of potassium and sodium in the obtained substances.

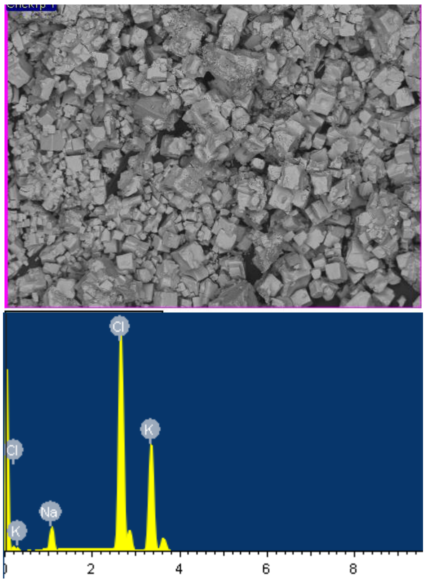

Figure 9 – Microstructure image and spectrogram of potassium chloride obtained by the halurgic method

Table 6 - The elemental composition of potassium chloride obtained by the halurgic method

Element	Na	Cl	K	Total
Mass. %	3.17	50.66	46.17	100.0

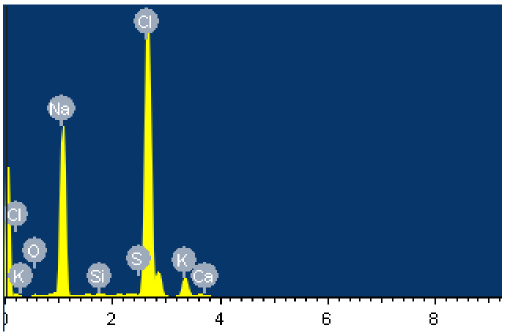

Figure 10 – Spectrogram of sodium chloride obtained by the halurgic method

Table 7 - The elemental composition of sodium chloride obtained by the halurgic method

Element	Na	Cl	O	S	Total
Mass. %	3.49	55.47	1.98	0.16	100,0
Element	Ca	K	Si		
Mass. %	0.30	2.95	0.18		

These data confirm the success of the applied technology of halurgy for the separation of potassium and sodium salts, and also demonstrate the stability and high degree of extraction of useful components from the source ore.

The elemental analysis of the obtained products, including potassium and sodium chloride, confirmed the high degree of purity of these compounds, which makes them suitable for further use in agriculture and the chemical industry. The spectrograms obtained during the analysis showed compliance of the potassium and sodium content with the established standards, which confirms the success of the chosen technology. Thus, the results of the study confirm the prospects of the halurgy method for processing potash ores, ensuring effective separation of components and obtaining high-quality products. This technology has potential for industrial applications, especially in the context of the need to increase the efficiency of processing potassium and sodium salts, as well as improve the environmental safety of the process.

Conclusions

The article offers a concise overview of the occurrence of large deposits of potassium salts, as well as an analysis of the chemical composition of minerals.

A comprehensive study of the chemical composition of the silvinite ore from the Satimola deposit has been conducted. The elemental composition and the ratio of potassium and sodium salts in the mineral were established. Additionally, it was established that the naturally occurring mineral potassium in the Satimola deposit exhibits a minor insoluble residue. The ore was found to have a heterogeneous composition, with sodium chloride representing the dominant component.

According to the results of physico-chemical analyses, the sample contains 81.2% sodium chloride (NaCl) and 13.1% potassium chloride (KCl), which indicates the presence of two main components in the mineral — halite (NaCl) and sylvinite (KCl). A high level of sodium chloride indicates the predominance of halite, which is typical for minerals formed under conditions of evaporation of water, such as in salt deposits. The sample also contains 5.7% okenite, a calcium silicate mineral that is an insoluble residual.

In the course of the study, the technology of processing potash ore by the method of halurgy was considered, which demonstrated high efficiency in the extraction of potassium oxide and sodium chloride. The technique, including leaching in solution followed by filtration and precipitation, made it possible to obtain potassium chloride with a yield of 95 to 98%, which indicates the high productivity of the process.

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Conflicts of Interest. On behalf of all authors, the correspondent author declares that there is no conflict of interest.

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Калий кенін өңдеу: технологиялары мен физика-химиялық қасиеттерін зерттеу

Уразкелдиева Д.А., Кадирбаева А.А., Қошқарбаева Ш.Т.

М. Әуезов атындағы Оңтүстік Қазақстан Зерттеу Университеті, Шымкент, Қазақстан

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Қабылданды: *30 қаңтар 2025*

ТҮЙІНДЕМЕ

Қазақстан Республикасының Батыс Қазақстан және Ақтөбе облыстарында калий кендерінің бірегей қорлары шоғырланған. Бұл қорлар әлемдегі ең ірі қорлардың бірі болып саналады. Дәлелденген қорлар шамамен 6 миллиард тоннаға бағаланады және төрт ірі кен орындарына бөлінеді: олар Жилянское, Сатимола, Индер және Челкар кен орындары. Мақалада калий тұздарының негізгі кен орындары мен онымен байланысты минералдардың химиялық құрамы туралы қысқаша шолу берілген. Бұл кен орындарының болуына қарамастан, елімізде сұраныс үнемі өсіп келе жатқан калий тұздарын өндіру әлі жолға қойылмаған. Осыны ескере отырып, табиғи тұз жүйелерін тыңайтқыштар мен тұздардың ішкі нарығында да, шетелде де жоғары сұранысты қанағаттандыратын өнімдерге айналдырудың өміршең әдістерін анықтау мақсатында осы перспективалы калий кендерінің минералогиялық және химиялық құрамын жан-жақты зерттеу өте маңызды. Сатимола кен орны әлі жеткілікті зерттелмеген ең ірі сильвинит бассейндерінің бірі болып табылады және оның өнеркәсіптік дамуы әлі басталған жоқ. Шикізат пен өнімге талдау спектрлік микроскопия, рентгендік талдау және дифференциалды термиялық талдаудың көмегімен жүргізілді. Сатимола кен орнындағы сильвинит кенінің құрамына жан-жақты кешенді зерттеу жүргізілді. Элементтік құрамы және минералдағы калий мен натрий тұздарының қатынасы анықталды. Сонымен қатар, Сатимола кен орнының табиғи калий минералында ерімейтін қалдықтардың аздаған мөлшері болатыны анықталды. Кеннің құрамы әртекті екені және оның құрамында натрий хлориді басым екендігі дәлелденді.

Түйін сөздер: калий хлориді, калий, галургиялық әдіс, сильвинит, Сатимола кен орны.

Уразкелдиева Дилбар Абдихамидовна	***Авторлар туралы ақпарат:*** *PhD докторант, Химиялық инженерия және биотехнология жоғарғы мектебі, М. Әуезов атындағы Оңтүстік Қазақстан зерттеу университеті, Шымкент, Қазақстан. E-mail: urazkeldieva.97@list.ru; ORCID ID: https://orcid.org/0000-0001-7825-6995*
Қадірбаева Алмагүл Аққөпейқызы	*Техника ғылымдарының кандидаты, қауымдастырылған профессор, Химиялық инженерия және биотехнология жоғарғы мектебі, М. Әуезов атындағы Оңтүстік Қазақстан зерттеу университеті, Шымкент, Қазақстан. E-mail: diac_2003@mail.ru; ORCID ID: https://orcid.org/0000-0003-0702-1114*
Қошқарбаева Шайзада Тортаевна	*Техника ғылымдарының кандидаты, қауымдастырылған профессор, Химиялық инженерия және биотехнология жоғарғы мектебі, М. Әуезов атындағы Оңтүстік Қазақстан зерттеу университеті, Шымкент, Қазақстан. Email: shayzada-1968@mail.ru; ORCID ID: https://orcid.org/0000-0001-8753-3245*

Переработка калийной руды: исследование технологий и физико-химических свойств

Уразкелдиева Д.А., Кадирбаева А.А., Кошкарбаева Ш.Т.

Южно-Казахстанский Исследовательский Университет имени М. Ауезова, Шымкент, Казахстан

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АННОТАЦИЯ

Республика Казахстан обладает уникальными запасами калийных руд, сосредоточенных в Западно-Казахстанской и Актюбинской областях. Эти запасы считаются одними из крупнейших в мире. Доказанные запасы оцениваются примерно в 6 миллиардов тонн и распределены по четырем крупным месторождениям: Жилянскому, Сатимольскому, Индерскому и Челкарскому. В статье представлен краткий обзор основных месторождений калийных солей и химического состава сопутствующих минералов. Несмотря на наличие этих месторождений, производство калийных солей в стране, спрос на которые постоянно растет, еще предстоит наладить. В свете этого крайне важно провести всестороннее исследование минералогического и химического состава этих перспективных калийных руд с целью определения эффективных методов переработки природных солевых систем в продукты, которые удовлетворяют высокому спросу как на внутреннем рынке удобрений и соли, так и за рубежом. Месторождение Сатимола представляет собой один из крупнейших сильвинитовых бассейнов, который еще недостаточно изучен, и его промышленное освоение только начинается. Анализ сырья и продуктов был проведен с использованием комбинации методов спектральной микроскопии, рентгеноструктурного анализа и дифференциального термического анализа. Проведено комплексное исследование состава сильвинитовой руды месторождения Сатимола. Установлен элементный состав и соотношение солей калия и натрия в минерале. Кроме того, было установлено, что природный минерал калия месторождения Сатимола содержит незначительное количество нерастворимых остатков. Было установлено, что руда имеет неоднородный состав, при этом преобладающим компонентом является хлорид натрия.

Ключевые слова: хлорид калия, калий, галургический метод, сильвинит, месторождение Сатимола.

Уразкелдиева Дилбар Абдихамидовна	***Информация об авторах:*** *PhD докторант, Высшая школа химической инженерии и биотехнологии, Южно-Казахстанский исследовательский университет имени М. Ауезова, Шымкент, Казахстан. E-mail: urazkeldieva.97@list.ru; ORCID ID: https://orcid.org/0000-0001-7825-6995*
Кадирбаева Алмагул Аккопейкызы	*Кандидат технических наук, ассоцированный профессор, Высшая школа химической инженерии и биотехнологии, Южно-Казахстанский исследовательский университет имени М. Ауезова, Шымкент, Казахстан. E-mail: diac_2003@mail.ru; ORCID ID: https://orcid.org/0000-0003-0702-1114*
Кошкарбаева Шайзада Тортаевна	*Кандидат технических наук, ассоцированный профессор, Высшая школа химической инженерии и биотехнологии, Южно-Казахстанский исследовательский университет имени М. Ауезова, Шымкент, Казахстан. Email: shayzada-1968@mail.ru; ORCID ID: https://orcid.org/0000-0001-8753-3245*

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Structure of turbulent non-isothermal flow in a pipe with a sudden expansion

Zhapbasbayev U.K., *Bossinov D.Zh.

Satbayev University, Almaty, Kazakhstan

** Corresponding author email: dansho.91@mail.ru*

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ABSTRACT

The article studies a mathematical model of turbulent non-isothermal flow of non-Newtonian fluid. At the inlet, the fluid is Newtonian and, due to a decrease in temperature, it becomes non-Newtonian due to increased viscosity and yield strength. The system of turbulent motion and heat transfer equations is solved by the numerical control volume method in variables of the velocity and pressure components. The calculations yielded average and pulsation characteristics of the non-isothermal motion of non-Newtonian fluid in a pipe with sudden expansion. The calculations show a sharp reduction in the structure of the recirculation zone and a decrease in its parameters with an increase in the Bingham number Bn. In this zone, the maximum negative value of the average velocity, equal to–Umax/Um1 ≈ 0.2 for a Newtonian fluid, decreases to –Umax/Um1 ≈ 0.1 at the Bingham number Bn = 17. A decrease in the turbulent characteristics of the non-Newtonian fluid flow is also observed with an increase in the Bingham number. Heat exchange characteristics in the flow region of turbulent non-Newtonian and Newtonian fluids are qualitatively similar. The location of the flow attachment and maximum heat exchange of non-Newtonian fluid does not exceed 10%. The length of the recirculation zone of viscoplastic fluid is shorter by up to 66% compared to Newtonian fluid.

Keywords: non-isothermal turbulent flow, viscoplastic fluid, recirculation zone of pipe flow with sudden expansion.

Information about authors:

Zhapbasbayev Uzak Kairbekovich — *Doctor of Technical Sciences, Professor, Head of the Modeling in Energy Scientific and Production Laboratory, Satbayev University, Almaty, Kazakhstan. E-mail: uzak.zh@mail.ru; ORCID: https://orcid.org/0000-0001-5973-5149*

Bossinov Daniyar Zhumadilovich — *Master of natural sciences, researcher in laboratory Modeling in Energy, Satbayev University, Almaty, Kazakhstan. Email: dansho.91@mail.ru; ORCID ID: https://orcid.org/0000-0003-3757-6460*

Introduction

Turbulent fluid flow with sudden expansion in pipes is used to intensify heat exchange in various technical devices. Such a design of pipe connections takes place in centralized heating systems. This primitive geometry was used at a fundamental level to study the flow division pattern. With the sudden expansion of the flow in the pipe, the static pressure increases and the kinetic energy of the fluid decreases, a recirculation zone is formed, which divides the flow into two parts. Behind the recirculation zone, the flow fills the entire cross-section of the pipe with a flat velocity profile. The separation surface becomes unstable at moderate and high Reynolds numbers (Re) and is the source of vortex generation in both parts of the flow. Moreover, the vortices develop and gradually disappear. This is because the liquid flows against the increase in static pressure with the formation of a recirculation zone. The flow after a sudden expansion of the pipe cross-section can be considered as a jet in an annular recirculation region. This jet expands radially in the expansion region of the pipe until it reattaches to the pipe wall. As experimental studies of fully developed turbulent flow in pipes show [[1], [2]], reattachment of the flow occurs at lengths 6-9 times greater than the height of the expansion stage of the pipe.

In [3] the separation and reattachment of a turbulent flow in a pipe with sudden expansion were also studied experimentally. Experimental measurements of the velocity field were carried out by several authors [[4], [5], [6]] at moderate and high Reynolds numbers of the flow in a pipe with sudden expansion. These measurements showed that the separation region contains large velocity gradients and high shear in combination with an unfavourable pressure gradient. Whereas the reverse flow velocities in the recirculation region are of the order of 10% of the average velocity in the expanding part. The presence of the pipe wall prevents fluid entrainment so that an unstable vortex structure is established further downstream. As experiments [[4], [5], [6]] show, it is difficult to obtain accurate quantitative measurements of turbulence in the recirculation zone, since the average velocities are usually small and the turbulence intensity is high. In this paper, the calculated data of a turbulent non-isothermal flow of a non-Newtonian fluid in a pipe with sudden expansion are presented.

Statement of the problem. Figure 1 shows the flow diagram.

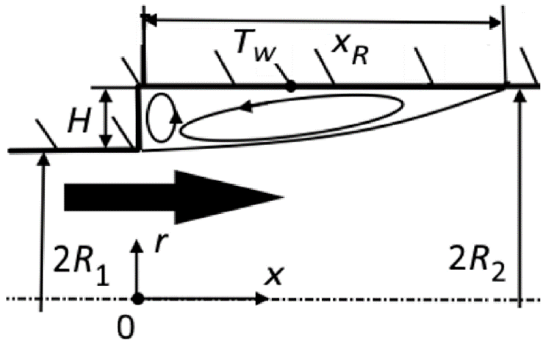

Figure 1 - Flow diagram in a pipe with a sudden expansion

A Newtonian fluid with a temperature T_1 = 303 K and an average velocity u_1 flows into a pipe. The ambient temperature T_w = 273 K is less than the temperature of the incoming fluid. Heat exchange with the environment leads to cooling and a decrease in the temperature of the fluid. A decrease in temperature increases the viscosity and yield point, causing the transition of the Newtonian fluid to non-Newtonian. It is required to find the patterns of average and pulsation characteristics of turbulent non-isothermal flow of a non-Newtonian fluid in a pipe with a sharp expansion.

The system of equations of non-isothermal turbulent motion and heat transfer of a viscoplastic fluid is written in the form [[7], [8], [9]]:

$$\nabla \cdot \mathbf{U} = 0 \tag{1}$$

$$\nabla \cdot (\rho \mathbf{U}\mathbf{U}) = -\nabla P + \nabla \cdot \left(2\mu_{eff}\mathbf{S}\right) + \nabla \cdot \left(-\rho\left\langle \mathbf{u}'\mathbf{u}'\right\rangle\right) + \nabla \cdot \left\langle 2\mu'_{eff}\mathbf{S}'\right\rangle \tag{2}$$

$$\nabla \cdot \left(\rho C_p T \mathrm{U}\right) = \nabla \cdot \left(\lambda \nabla T\right) + \nabla \cdot \left(-\rho C_p \left\langle \mathbf{u}' t' \right\rangle\right) + \boldsymbol{\tau} : \nabla \mathbf{U} \tag{3}$$

The coefficient of effective molecular viscosity μ_{eff} of a viscoplastic liquid is found from the expression [[10], [11], [12], [13], [14]]:

$$\mu_{eff} = \begin{cases} \mu_p + \tau_0 \left|\dot{\gamma}\right|^{-1}, & |\tau| \quad \tau_0 \\ \infty, & |\tau| \le \tau_0 \end{cases} \tag{4}$$

The singular property $|\tau| \le \tau_0$ of formula (4) can be regularized using the approach [15] and written as:

$$\mu_{eff} = \mu_p + \frac{{}_0\left[1 - \exp\left(-m|\dot{\gamma}|\right)\right]}{|\dot{\gamma}|} \tag{5}$$

Where the regularization parameter is m=1000 s [15].

The turbulent stress model (RSM model) [[9], [16]] is used to close the system of equations of motion and heat transfer (1-5):

$$\frac{\partial}{\partial x_j}\left(\rho U_j \left\langle u_i' u_j' \right\rangle\right) = \rho\left(P_{ij} + \phi_{ij} - \varepsilon_{ij}\right) + \frac{\partial}{\partial x_l}\left[\rho \nu_{eff}\delta_{lm} + \rho\frac{C_\mu T_T}{\sigma_k}\left\langle u_l' u_m' \right\rangle\right]\frac{\partial}{\partial x_m}\left\langle u_i' u_j' \right\rangle + D_{NNF}$$
$$\frac{\partial}{\partial x_j}\left(\rho U_j \varepsilon\right) = \frac{1}{T_T}\left(C_\varepsilon \tilde{P} - C_{\varepsilon 2}\varepsilon\right) + \frac{\partial}{\partial x_l}\left[\rho \nu_{eff}\delta_{lm} + \rho\frac{C_\mu T_T}{\sigma_\varepsilon}\left\langle u_l' u_m' \right\rangle\right]\frac{\partial \varepsilon}{\partial x_m} + \varepsilon_{NNF}$$
$$\chi - L_T^2 \nabla^2 \chi = 1. \tag{6}$$

In [[9], [16]] the constants and closing functions of the system of equations (6) are given.

Boundary conditions. The flow diagram is shown in Figure 1.

On the pipe wall before and after expansion:

$$U = V = \left\langle \mathbf{u}'\mathbf{u}' \right\rangle = 0;\ T = T_W = \text{const};$$
$$\varepsilon = 2\nu_W \frac{k}{y^2};\ \chi = 0 \tag{7}$$

On the pipe axis:

$$\frac{\partial U}{\partial r} = V = \frac{\partial T}{\partial r} = \frac{\partial \left\langle \mathbf{u}'\mathbf{u}' \right\rangle}{\partial r} = \frac{\partial \varepsilon}{\partial r} = \frac{\partial \chi}{\partial r} = 0 \tag{8}$$

Constant values of variables are set at the pipe inlet, and soft boundary conditions are set at the outlet.

Numerical method for solving the problem. The numerical solution is obtained using a control volume method on a staggered grid. The algorithm for solving the system of equations (1)-(6) in the variables “velocity-pressure components” is described in detail in the work [[7], [8], [9]].

Numerical calculations are obtained using our software.

Discussion of the calculated data

Flow structure in a pipe with a sudden expansion. Figure 2 shows the contour lines of turbulent kinetic energy for Newtonian (NF) and viscoplastic fluids (NNF). The contour lines show the structure of the mixing layer and the development of turbulent kinetic energy in the flow region. From the edge of the sharp expansion area, the mixing layer separates and develops to the point of re-attachment of the flow (see Figure 2). The separation zone for the Newtonian fluid reaches x/H=9.3, and for the viscoplastic fluid - x/H=6.7, i.e. the separation zone is reduced in the viscoplastic fluid. The maximum level of turbulent kinetic energy is achieved in the mixing layer (k / U_{m1}^2 = 0.06). The region with maximum TKE values (k / U_{m1}^2 = 0.06) is located in the downstream position (x/H ≈ 5, y/H ≈ 0.6) for Newtonian fluid and (k / U_{m1}^2 = 0.045) for viscoplastic fluid in the position (x/H ≈ 4, y/H ≈ 0.5) (see Figure 2).

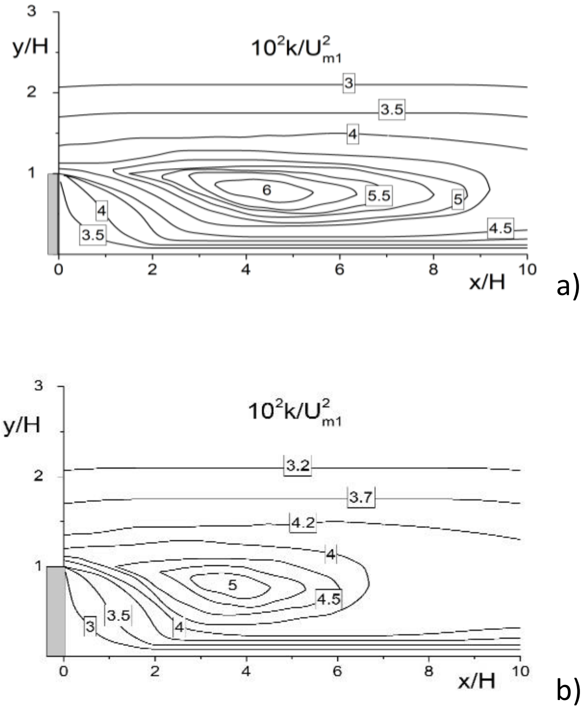

Figure 2 - Plots of the kinetic energy of turbulence in NF (a) and NNF (b) fluids behind a sudden expansion of a pipe. Re = 10^4, Re_H =2600, Pr = 42, Bn_1 = 0.007

Fig. 3 shows the change in the maximum axial velocity along the longitudinal coordinate in the recirculation zone of the flow behind a sudden expansion of the pipe.

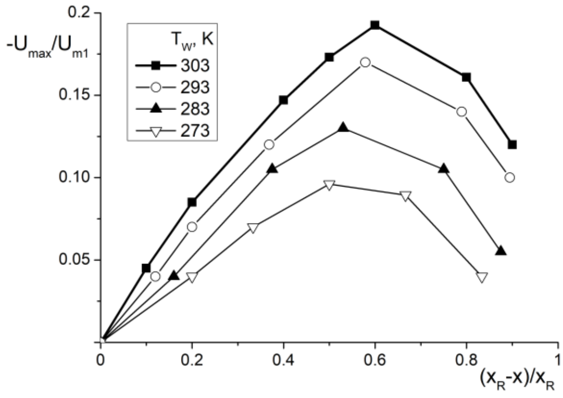

Figure 3 - Change in axial velocity along the length of the recirculation zone in a pipe with a sudden expansion. Re = 10^4, Re_H =2600, Pr = 42, Bn_1 = 0.007

The calculations were performed at the inlet temperature T_1=303 K and different values of the wall temperature T_w. The calculated data at T_w=303 K correspond to the turbulent flow of a Newtonian fluid (NF). In this mode, the maximum value of the velocity is $-U_{max}/U_{m1} \approx 0.2$. For a viscoplastic fluid at T_w=273 K, the maximum value of the velocity is $-U_{max}/U_{m1} \approx 0.1$. It can be said that the appearance of the viscoplastic property of the fluid suppresses the intensity of the circulation flow compared to a Newtonian fluid (see Figure 3).

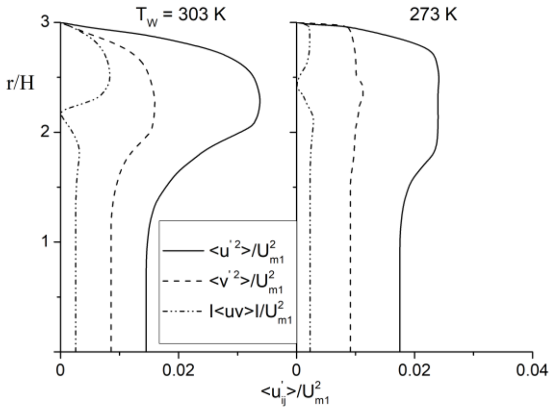

Figure 4 - Reynolds stress profiles of NF (T_w = 303 K) and NNF (T_w = 273 K) fluids at x/H = 4. Re = 10^4, Re_H =2600, Pr = 42, Bn_1 = 0.007

Figure 4 shows the distributions of Reynolds stresses for Newtonian (Tw = 303 K) and viscoplastic (Tw = 273 K) fluids at x/H = 4. The highest value of Reynolds stresses is achieved in the shear layer of mixing of Newtonian and non-Newtonian fluids. These data are in agreement with the results of separated flows of Newtonian fluid after a step [6]. The highest value of the ratio of axial velocity fluctuations to radial ones for NF fluid is up to 2 times and for NNF fluid up to 2.6 times.

The length of the circulation zone characterizes the intensity of the vortex in a pipe with a sharp expansion (see Figure 5). The Reynolds number of the flow has little effect on the position of the zone attachment point and the maximum heat transfer for Newtonian and non-Newtonian fluids for all calculations of wall temperatures and flow rates (up to 7%). The position of the flow reattachment point and the maximum heat transfer for Newtonian and non-Newtonian fluids are slightly affected by the Reynolds number (up to 7%) for all calculations of wall temperatures and flow rates. The difference between the flow reattachment point and the maximum heat transfer between Newtonian and non-Newtonian fluids does not exceed 5%. The flow reattachment point and the position of maximum heat transfer are located upstream for a non-Newtonian fluid compared to the position of the reattachment point in Newtonian fluids [[6], [17], [18], [19]]. The maximum difference reaches 10% for a non-Newtonian fluid at Tw = 273 K.

The length of the separation zone and the position of the maximum heat transfer are affected by the wall temperature. For a non-Newtonian fluid, the length of the recirculation zone is reduced to 66% compared to the length of a Newtonian fluid and is xR/H ≈ 6.5–6.8 at Tw = 273 K.

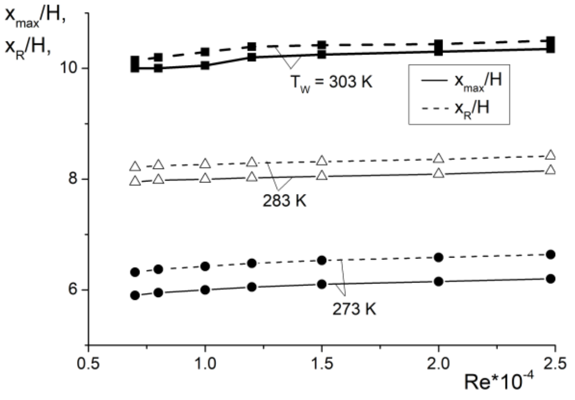

Figure 5 - Recirculation length x_R and maximum heat transfer point x_{max} as a function of wall temperature. Re = 10^4, Re_H =2600, Pr = 42, Bn_1 = 0.007

Heat transfer. Figure 6 shows the changes in the Nusselt number Nu = hH/λ_{W1} along the axial coordinate (a) and the effect of the Reynolds number Re on the maximum heat transfer (b). Here, h is the heat transfer coefficient, Numax is the maximum Nusselt number, and Nu_{fd} is the Nusselt number for a fully developed Newtonian flow in a pipe without expansion.

The Nusselt number is found from the step height $\mathrm{Nu} = -(\partial T/\partial y)_W H/(T_W - T_m)$. The flow reattachment points are indicated by arrows in Fig. 6a. As can be seen from Fig. 6a, the changes in heat transfer along the pipe length for NF (line 1) and NNF (lines 2–4) are qualitatively similar. There is no local minimum for NNF in the corner part of the step. At Tw = 293 K, the change in heat transfer (curve 2) and the length of the recirculation zone of NNF are practically the same as the NF data. The faster manifestation of non-Newtonian properties of the NNF fluid causes a decrease in the wall temperature and confirms the above-mentioned calculated data. A decrease in the intensity of heat transfer of turbulent flow in a pipe with sudden expansion can be noted for NNF. The maximum heat transfer values coincide with the location of the flow reattachment point. For NNF at Tw = 273 K, the maximum heat transfer is shifted by up to 70% (line 4).

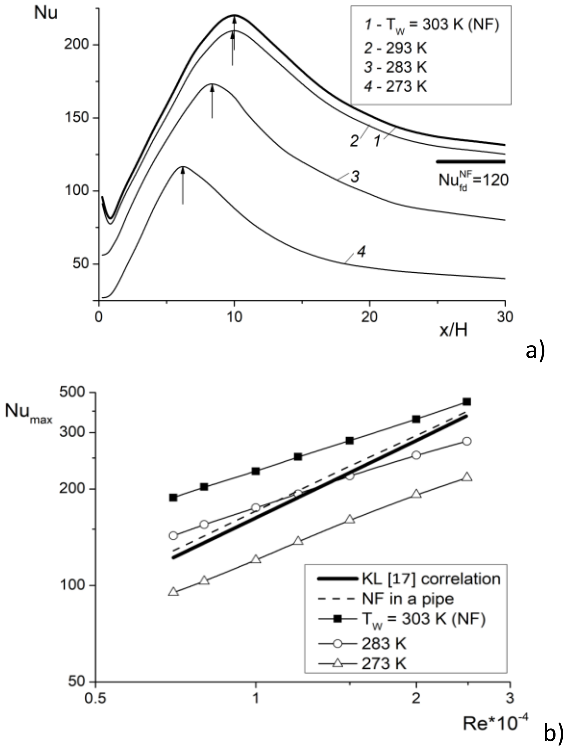

Figure 6 - Dependence of maximum heat transfer on Nusselt (a) and Reynolds (b) numbers in NF and NNF turbulent flows.
Re = 10^4, Re_H =2600, Pr_1 = 42, Bn_1 = 0.007

Similar data were obtained for the maximum values of the Nusselt number depending on the Reynolds number (see Fig. 6b). The bold line is the calculated data using the formula $\mathrm{Nu} = 0.023\mathrm{Re}^{0.8}\mathrm{Pr}^{0.4}$ [20] for a stabilized turbulent flow of a Newtonian fluid in a pipe without expansion.

The authors' calculated data for a fully developed turbulent Newtonian fluid in a pipe are shown as a dashed line. An increase in the Reynolds number leads to an increase in heat transfer for turbulent NF, as well as for NNF. For a turbulent flow of a Newtonian fluid, the maximum value of heat transfer is higher (dashed line) than for turbulent flows of a non-Newtonian fluid (T_w = 283 and 273 K). It can be noted that the slope of the well-known experimental heat transfer formula [20] (bold line) and the authors' calculated data for a pipe without expansion differ from the data for NF and NNF after a sudden expansion of the pipe.

Conclusion

Calculated data are obtained for the turbulent non-isothermal flow of non-Newtonian fluid in a pipe with abrupt expansion. An elliptic model of Reynolds stress relaxation is used to simulate turbulent kinetic energy. The calculations show the occurrence of completely stagnant flow in the near-wall region of a pipe for non-Newtonian fluid, where the value of $U \approx 0$ ($y/R \leq 0.4$ at $x/H = 15$). The stagnation zone occurs at $x/H > 8$ with the Bingham number Bm=17. In the shear mixing layer of the isothermal non-Newtonian fluid, a significant decrease in the turbulence level (up to 50%) is obtained. In the core zone, the turbulence level of the non-Newtonian fluid is up to 15% higher than that of the Newtonian fluid. The Bingham number affects the value of the maximum negative velocity in the separation region ($-U_{max}/U_{m1} \approx 0.075$ with Bm=17).

For a non-Newtonian fluid, the vortices in the recirculation zone after a sudden expansion are less intense than for a Newtonian fluid.

For turbulent flows of non-Newtonian and Newtonian fluids, the heat transfer distributions are qualitatively similar. The values of the location of the flow attachment point and the maximum heat transfer are also close, the difference does not exceed 10%. The length of the recirculation zone and the position of the maximum heat transfer are strongly affected by the wall temperature. For a non-Newtonian fluid, the length of the recirculation zone is shorter by up to 66% than for a Newtonian fluid. The flow attachment point of a non-Newtonian fluid is located higher than that of a Newtonian fluid. Heat transfer in the recirculation zone of a non-Newtonian fluid is two times lower than for a Newtonian fluid. The length of the recirculation zone is shorter by up to 40% for a non-Newtonian fluid, compared to a Newtonian fluid.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement. U. Zhapbasbayev: contributed to the conceptualization, investigation, methodology, and original draft preparation; **D. Bossinov:** contributed to the methodology, visualization, and writing—review and editing.

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Құбырдың кенеттен кеңейетін аймағындағы турбуленттік изотермиялық емес ағынның құрылымы

Жапбасбаев Ұ.Қ., Босинов Д.Ж.

Satbayev University, Алматы, Қазақстан

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ТҮЙІНДЕМЕ

Мақалада Ньютондық емес сұйықтықтың турбулентті изотермиялық емес ағынының математикалық моделі қарастырылады. Құбырға кіргенде сұйықтық Ньютондық болып табылады және температураның төмендеуіне байланысты тұтқырлық пен аққыштық беріктігінің жоғарылауына байланысты ол Ньютондық емес сұйықтыққа айналады. Турбулентті қозғалыс пен жылу алмасу теңдеулер жүйесі жылдамдық пен қысымның айнымалы құрамдас бөліктерінде сандық басқару көлемі әдісімен шешіледі. Есептеулер нәтижесінде кенет кеңеюі бар құбырдағы Ньютондық емес сұйықтықтың изотермиялық емес қозғалысының орташа және пульсациялық сипаттамалары алынды. Есептеулер рециркуляция аймағының құрылымының күрт төмендеуін және Бингам санының Bn ұлғаюымен оның параметрлерінің төмендеуін көрсетеді. Бұл аймақта Ньютон сұйықтығы үшін –Umax/Um1 ≈ 0,2-ге тең орташа жылдамдықтың максималды теріс мәні Bingham санында Bn = 17 –Umax/Um1 ≈ 0,1-ге дейін төмендейді. Ньютондық емес сұйықтық ағынының турбуленттік сипаттамаларының төмендеуі Бингем санының жоғарылауымен де байқалады. Турбуленттік Ньютондық емес және Ньютондық сұйықтықтардың ағыс аймағындағы жылу алмасу сипаттамалары сапалық жағынан ұқсас. Ньютондық емес сұйықтықтың ағындық қосылысының орны және максималды жылу беруі 10% аспайды. Ньютондық емес сұйықтықтың рециркуляция аймағының ұзындығы Ньютон сұйықтығымен салыстырғанда 66%-ға дейін қысқа.

Түйін сөздер: изотермиялық емес турбулентті ағын, тұтқыр пластикалық сұйықтық, кенеттен кеңеюі бар құбыр ағысының рециркуляция аймағы.

Авторлар туралы ақпарат:

Жапбасбаев Ұзақ Қайырбекұлы — *Техника ғылымдарының докторы, профессор, Энергетикадағы модельдеу ғылыми-өндірістік зертханасының меңгерушісі, Сәтбаев университеті, Алматы, Қазақстан. Email: uzak.zh@mail.ru; ORCID: https://orcid.org/0000-0001-5973-5149*

Босинов Данияр Жұмаділлаұлы — *Жаратылыстану ғылымдарының магистрі, Энергетикадағы модельдеу зертханасында ғылыми қызметкер, Алматы, Қазақстан. Email: dansho.91@mail.ru; ORCID ID: https://orcid.org/0000-0003-3757-6460*

Структура турбулентного неизотермического течения в трубе с внезапным расширением

Жапбасбаев У.К., Боссинов Д.Ж.

Satbayev University, Алматы, Казахстан

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АННОТАЦИЯ

В статье рассматривается математическая модель турбулентного неизотермического течения неньютоновской жидкости. На входе жидкость является ньютоновской и из-за понижения температуры становится неньютоновской за счет увеличения вязкости и предела текучести. Система уравнений турбулентного движения и теплопереноса решается численным методом контрольного объема в переменных компонентах скорости и давления. В результате расчетов получены средние и пульсационные характеристики неизотермического движения неньютоновской жидкости в трубе с внезапным расширением. Расчеты показывают резкое сокращение структуры зоны рециркуляции и уменьшение ее параметров с ростом числа Бингама Bn. В этой зоне максимальное отрицательное значение средней скорости, равное –Umax/Um1 ≈ 0,2 для ньютоновской жидкости, уменьшается до –Umax/Um1 ≈ 0,1 при числе Бингама Bn = 17. Также наблюдается уменьшение турбулентных характеристик течения неньютоновской жидкости с ростом числа Бингама. Характеристики теплообмена в области течения турбулентных неньютоновской и ньютоновской жидкостей качественно подобны. Расположение присоединения потока и максимального теплообмена неньютоновской жидкости не превышает 10%. Длина зоны рециркуляции неньютоновской жидкости короче до 66% по сравнению с ньютоновской жидкостью.

Ключевые слова: неизотермическое турбулентное течение, вископластическая жидкость, зона рециркуляции потока в трубе с резким расширением.

Информация об авторах:

Жапбасбаев Узак Кайрбекович — *Доктор технических наук, профессор, заведующий научно-производственной лаборатории Моделирование в энергетике, Satbayev University, Алматы, Казахстан. E-mail: uzak.zh@mail.ru; ORCID ID: https://orcid.org/0000-0001-5973-5149*

Босинов Данияр Жумадилович — *Магистр естественных наук, научный сотрудник лаборатории Моделирование в энергетике, Satbayev University, Алматы, Казахстан. Email: dansho.91@mail.ru; ORCID ID: https://orcid.org/0000-0003-3757-6460.*

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Using ion modification methods for targeted change of strength properties of near-surface layers of composite ceramics

[1]Borgekov D.B., [1,2*]Kozlovskiy A.L., [1,2]Shlimas D.I., [2]Shakirziyanov R.I., [3]Popov A.I., [3]Konuhova M.

[1] The Institute of Nuclear Physics, Almaty, Kazakhstan
[2] L.N. Gumilyov Eurasian National University, Astana, Kazakhstan
[3] Institute of Solid State Physics University of Latvia, Riga, Latvia

** Corresponding author email: kozlovskiy.a@inp.kz*

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ABSTRACT

The use of ionic modification methods is one of the promising methods of directed change of strength properties of near-surface layers of materials along with such methods as magnetron sputtering and mechanically induced deformation. Interest in this area of research is primarily due to the possibility of enhanced resistance of materials to external mechanical and thermal influences, as well as improved wear resistance of refractory ceramics, which have great prospects in industrial use and metallurgy and reactor building. This paper presents the assessment results of the possibility of using the ion modification method by irradiating the near-surface layer of $ZrO_2 – Al_2O_3$ ceramics with low-energy Kr^{15+} and Xe^{22+} ions with energies of 300 and 440 keV to create a radiation-modified layer in the surface layer that is highly resistant to external influences. During the studies, it was found that irradiation with fluences of 10^{14} - 5×10^{14} ion/cm^2 for Xe^{22+} ions and 10^{15} ion/cm^2 for Kr^{15+} ions are optimal conditions for modifying the surface layer, as a result of which growth in wear resistance by 2.0 – 2.5 times and hardening by more than 15 – 20 % is observed compared to non-irradiated ceramics.

Keywords: hardening, ion modification, hardness and wear resistance enhancement, $ZrO_2 – Al_2O_3$ ceramics, low-energy ions.

	Information about authors:
Daryn B. Borgekov	*PhD, The Institute of Nuclear Physics, 050032, Almaty, Kazakhstan. Email: d.borgekov@inp.kz; ORCID ID: https://orcid.org/0000-0002-9727-0511*
Artem L. Kozlovskiy	*PhD, The Institute of Nuclear Physics, 050032, Almaty, Kazakhstan. Email: kozlovskiy.a@inp.kz; ORCID ID: https://orcid.org/0000-0001-8832-7443*
Dmitriy I. Shlimas	*PhD, L.N. Gumilyov Eurasian National University, 010008, Astana, Kazakhstan. E-mail: shlimas@inp.kz; ORCID ID: https://orcid.org/0000-0003-2454-7177*
Rafael I. Shakirziyanov	*PhD, L.N. Gumilyov Eurasian National University, 010008, Astana, Kazakhstan. E-mail: halfraf@mail.ru; ORCID ID: https://orcid.org/0000-0001-9908-3034*
Anatoliy I. Popov	*Dr., Institute of Solid State Physics University of Latvia, LV-1063, 8 Kengaraga Str, Riga, Latvia. E-mail: popov@latnet.lv; ORCID ID: https://orcid.org/0000-0003-2795-9361*
Marina Konuhova	*PhD, Institute of Solid State Physics University of Latvia, LV-1063, 8 Kengaraga Str, Riga, Latvia. E-mail: marina.konuhova@cfi.lu.lv; ORCID ID: https://orcid.org/0000-0003-0743-5915*

Introduction

Modern technological processes in metallurgy are accompanied by the operation of materials at high temperatures (1000 – 1300 °C), as well as thermal cycling processes (rapid heating and cooling), which can lead to thermal shocks, as well as accelerated degradation of strength properties, including due to temperature changes and exposure to aggressive environments [[1], [2]]. In this regard, the materials used in these technological processes must withstand heavy mechanical loads, and thermal shocks, and also be resistant to high-temperature corrosion and degradation processes that occur during long-term operation [[3], [4]]. To improve the productivity of technological processes, the use of composite materials, including high-entropy alloys based on refractory compounds or ceramic materials, has recently been proposed [[5], [6], [7]]. Interest in composite ceramics based on oxide or nitride compounds in the field of metallurgy and modern mechanical engineering is due to the possibility of creating materials that have a combination of properties such as a high melting point, allowing them to be used in extreme conditions (at high temperatures), high strength and wear resistance, increasing resistance to external influences, as well as insulating properties, including dielectric characteristics and thermal insulation (low thermal conductivity) [[8], [9], [10]]. At the same time, the combination of these properties allows to expand the capabilities of technological lines, as well as to increase the productivity of technological processes, which allows to reduce the cost of manufactured products. In addition to replacing traditional steels and alloys with refractory alloys and ceramics, various options are offered to increase the resistance of materials to external influences.

Thus, among the currently known methods for improving resistance to external influences, including mechanical damage caused by impacts or friction, one can distinguish methods based on the creation of highly deformed near-surface layers that have higher resistance to external influences [[11], [12], [13]]. The main hardening mechanism, in this case, is an increase in dislocation density in a small near-surface layer, the presence of which, together with small grain sizes, results in the creation of barrier boundaries that prevent the propagation of cracks and chips under external influence, and also increases resistance to corrosion and degradation. The mechanisms for creating such layers are different; for example, for steels and alloys, as a rule, they use deformation rolling methods, which, by acting on the near-surface layer, create additional structural defects in it that prevent the propagation of cracks or corrosion [[14], [15]]. Also, one of the promising methods for increasing stability is the method of laser processing of near-surface layers, which consists of surfacing protective coatings on the surface of materials by direct exposure of the surface to laser radiation [[16], [17]]. This method is based on sintering the surface with the applied material, thereby creating a protective layer with higher resistance to external influences. When using the method of magnetron sputtering of protective coatings, much attention is paid to the materials applied to the surface used as protective coatings or layers, due to the need to create good adhesive properties between the applied coating and the surface of the protected material, since the loss of adhesion of the protective coating to the surface of the samples can lead to their peeling or rapid destruction [[18], [19], [20]].

However, in the last few years, the use of ion irradiation methods for targeted modification of near-surface layers has been actively discussed. The method of ion modification or ion implantation itself has been known for quite a long time since it is based on the possibility of introducing impurity atoms into the near-surface layer of a material, the technology of introducing which is actively used to create semiconductor materials [[21], [22], [23], [24], [25]]. The main goal of ion implantation in this direction is to determine the possibilities of increasing the optical, conductive or luminescent properties of modified materials, which have great potential for use in modern materials science. At the same time, the use of ion modification methods, including irradiation with low-energy heavy ions (O, Ar, Kr, Xe with energies of the order of 200 – 500 keV) has recently been actively used to increase the resistance of materials to external influences, due to the creation of dislocation strengthening effects in the near-surface layer of the irradiated material, associated with the processes of deformation distortion and recrystallization (grain crushing) [[26], [27], [28]]. At the same time, the selection of irradiation conditions is important for carrying out such studies, since high-dose irradiation can initiate processes not only of hardening while maintaining a certain balance of deformation distortions but also in the case of accumulation of a large concentration of deformation stresses and distortions in the near-surface layer, it can initiate processes of surface peeling or partial sputtering. Similar effects are usually observed with high-dose irradiation and high concentrations of implanted atoms [[29], [30]].

The purpose of this study is to conduct experiments aimed at determining the possibilities of increasing the resistance to external influences of the surface layer of ZrO_2 – Al_2O_3 ceramics due to ionic modification by creating deformation distortions and recrystallization in the surface layer, leading to the initialization of dislocation hardening effects. Interest in this type of ceramics is primarily due to the possibility of combining high strength indicators and low thermal expansion of zirconium dioxide, as well as good thermophysical parameters of aluminum oxide. Moreover, the use of an equal stoichiometric ratio of these oxides during mechanochemical solid-phase synthesis makes it possible to obtain ceramics of the «Al_2O_3 matrix with embedded ZrO_2 grains» type [31], which creates additional strengthening due to the presence of interphase boundary effects [32].

The difference between this work and previously conducted studies is the possibility of using low-energy irradiation with heavy ions for targeted modification of composite ceramics in order to increase their resistance to external influences. At the same time, the emphasis in the study is on determining the effect of irradiation on changes in the strength characteristics of ceramics.

Experimental part

To assess the prospects of using the method of ion modification of the near-surface layer in order to increase the strength parameters, as well as resistance to external influences, including mechanical friction, pressure, and thermal effects, the following experiments were carried out, described below.

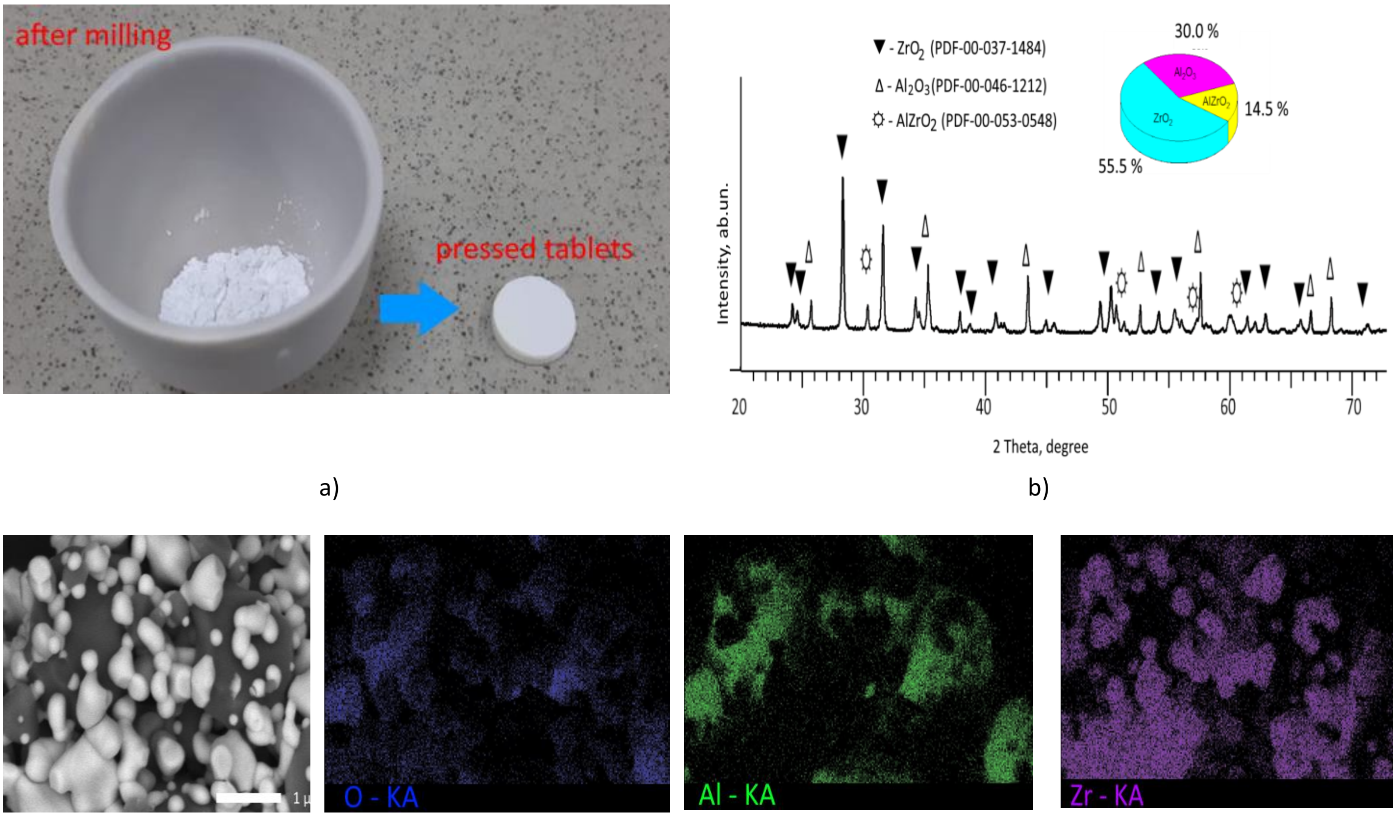
The objects of study were $ZrO_2 – Al_2O_3$ ceramics samples obtained by mechanochemical solid-phase mixing of initial oxides in an equal weight ratio (50: 50) followed by high-temperature sintering in a muffle furnace. Mechanochemical grinding of the initial zirconium and aluminum oxides was carried out in a planetary mill PULVERISETTE 6 (Fritsch, Berlin, Germany) at a grinding speed of 250 rpm and a time of 30 minutes. The choice of mixing conditions is determined by the need to obtain a homogeneous composition of ceramics that is isotropic by volume, without initializing processes associated with phase transformations as a result of the deformation mechanical action of the grinding media. The samples were sintered in a PM-1700 muffle furnace (Rusuniverstal, Chelyabinsk, Russia) at a heating rate of 10 °C/min. Upon reaching the set temperature of 1500 °C, the samples were kept for 8 hours, after which the samples cooled along with the furnace for 24 hours until they cooled completely and reached room temperature. After thermal annealing, the resulting powders were pressed into tablets with a diameter of about 10 mm and a thickness of 1 mm. The tablets were pressed in a special cylindrical mold under a pressure of 250 MPa for 30 minutes. After pressing, the resulting tablets were annealed at a temperature of 700 °C for 5 hours to remove the deformation distortions in the structure resulting from pressing.

Characterization of the initial samples was carried out using X-ray phase analysis and scanning electron microscopy. X-ray phase analysis was used to determine the degree of structural ordering, the change in which as a result of ionic modification indicates the proportion of the defective fraction in the surface layer, as well as to determine the phase composition of ceramics synthesized using the thermal sintering method. A D8 ADVANCE ECO powder diffractometer (Bruker, Karlsruhe, Germany) was used to take X-ray diffraction patterns. The survey was carried out in the Bragg – Brentano geometry, in the angular range $2θ=20-75°$. The determination of the phase composition of ceramics was carried out using the method of determining the weight contributions of diffraction reflections of each established phase with subsequent calculation of their share in the total diffraction pattern. To determine the phases, the PDF-2 (2016) database was used, from which the reference values of each phase and the positions of their main diffraction lines were taken.

The morphological features of the synthesized $ZrO_2 – Al_2O_3$ ceramics, in order to determine the grain distribution features or the mechanisms by which the ceramic structure is formed, were studied by obtaining images and element distribution maps, which were made using a Phenom™ ProX scanning electron microscope (Thermo Fisher Scientific, Eindhoven, the Netherlands).

Figure 1 reveals the results of characterization of the studied samples using X-ray phase analysis and scanning electron microscopy combined with mapping results. As can be seen from the presented X-ray diffraction pattern of the sample under study, the phase composition of ceramics is represented by a combination of three phases: the monoclinic ZrO_2 phase, the weight contribution of which is more than 55 %, the rhombohedral Al_2O_3 phase, the content of which is no more than 30 %, and the tetragonal $AlZrO_2$ phase, the weight contribution of which is about 15 %. The formation of the $AlZrO_2$ phase is associated with the initialization of polymorphic transformation processes in zirconium dioxide, the result of which is the formation of the tetragonal phase t-ZrO_2 with partial replacement of zirconium with aluminum. Moreover, a general analysis of the presented X-ray diffraction pattern indicates a fairly good structural ordering degree (more than 89%), and the presence of a tetragonal phase indicates that during thermal annealing in a given mode, the process of partial replacement of aluminum with zirconium is initiated, resulting in the formation of a new phase near grain boundaries.

As can be seen from the data presented in Figures 1c-d, which reflect the morphological features of the resulting ceramics, the choice of an equal weight ratio of ZrO_2 and Al_2O_3 oxides during their mechanochemical grinding and subsequent thermal annealing leads to the formation of ceramics of the «Al_2O_3 matrix with embedded ZrO_2 grains» type. It should be noted that ZrO_2 has a fairly large spread in size (the size range varies from 200 – 300 nm to 1.2 – 1.5 μm), and these grains form agglomerates consisting of three to four ZrO_2 grains embedded in an aluminum oxide matrix.

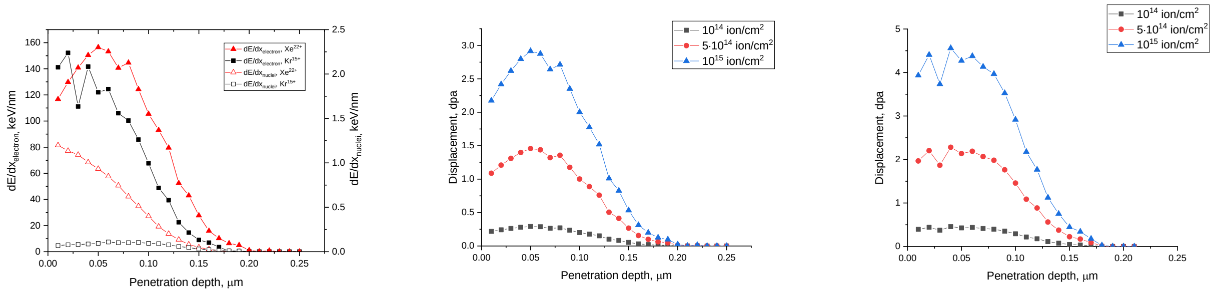


a) b)

c) d)

Figure 1 - a) Appearance of samples after thermal annealing in the form of powder and in the form of tablets after pressing; b) The results of X-ray phase analysis of the studied ZrO_2 – Al_2O_3 ceramics, presented in the form of a diffraction pattern and a diagram of the relationship of the established phases; c) SEM image of ZrO_2 – Al_2O_3 ceramics, indicating the formation of a structure of the «Al_2O_3 matrix with embedded ZrO_2 grains» type; d) Mapping results reflecting the distribution of elements in ceramics

The ionic modification was carried out by irradiating the studied samples of ZrO_2 – Al_2O_3 ceramics with low-energy Kr^{15+} and Xe^{22+} ions with energies of 300 and 440 keV. The choice of ions for directional modification is determined by their sizes (ionic radii), as well as the magnitude of ionization losses (see data in Figure 2). Irradiation was carried out at three fluences: 10^{14}, 5×10^{14} and 10^{15} ion/cm^2. Irradiation fluences were selected to simulate the processes of deformation distortion of a near-surface layer with a thickness of about 200 – 250 nm, while the selection of irradiation fluences was chosen in such a way as to avoid the effect of sputtering of the near-surface layer as a result of the accumulation of structural changes in it during high-dose irradiation. Irradiation was carried out at the DC-60 heavy ion accelerator (Institute of Nuclear Physics of the Ministry of Energy of the Republic of Kazakhstan, Almaty, Kazakhstan). As can be seen from the presented data, the results of modeling the magnitudes of ionization losses of incident ions along the trajectory of motion, presented in Figure 2, the main contribution to the change in structural features in the surface layer is made by the ionization losses of ions during interaction with electron shells, thereby initiating ionization processes (changes in the distribution of electron density), and as a consequence the occurrence of athermal processes associated with deformation distortion of the structure. At the same time, the differences in the ionization losses of ions in interaction with electron shells are of the order of 15 – 20 keV/nm, while the difference in ionization losses in interaction with nuclei is about 1 order of magnitude, which indicates that during irradiation with Kr^{15+} ions, the main contribution to the changes is made by the interaction of ions with electron shells, while during irradiation with Xe^{22+} ions, the effects due to the interaction of ions with nuclei, especially near the surface, should be taken into account.



a) b) c)

Figure 2 – a) Simulation results of ionization loss values of Kr^{15+} and Xe^{22+} ions in the near-surface layer of ZrO_2 – Al_2O_3 ceramics based on data from the SRIM Pro 2013 calculation code;
b) Simulation results of the values of atomic displacement along the trajectory of ion movement in the near-surface layer of ceramic at irradiation with Kr^{15+} ions; c) Simulation results of the values of atomic displacement along the ion trajectory in the near-surface ceramic layer at Xe^{22+}

Figure 2b-c presents the results of calculations of the magnitudes of atomic displacements caused by the interaction of incident ions with the crystal structure of the near-surface layer and reflects the degree of structural disorder associated with knocking atoms out of positions, as well as deformation distortions arising from athermal effects. The calculation of the magnitude of atomic displacements was performed according to the method specified in [33]. As can be seen from the data presented, the most pronounced changes in the values of atomic displacements are observed upon irradiation with Xe^{22+} ions, the value of which is approximately 1.5 times higher than the similar values of atomic displacements arising during irradiation with Kr^{15+} ions. This difference can be explained not only by differences in the initial energy of the incident ions (for Xe^{22+} ions the initial energy is 440 keV), but also by the fact that during irradiation with Xe^{22+} ions, rather large values of ionization losses associated with interactions with nuclei, resulting in atomic displacement effects, as well as the formation of vacancy defects, are observed. For further description of the observed effects of changes in strength parameters, the maximum values of atomic displacements arising during irradiation with heavy ions will be used.

The study of the effect of ionic modification on the strengthening of the near-surface layer of ceramics was carried out using the method of nanoindentation in depth, to establish changes in the hardness values of the samples along the trajectory of ion movement with variations in the irradiation fluence. To carry out the research, a technique was used to determine the hardness of samples at different loads on the indenter, which makes it possible to carry out measurements at different depths of the sample. The load value was selected a priori in the range from 1 to 100 N. Measurements were carried out in several parallels to determine the isotropy of the strength characteristics, as well as determine the standard deviation and measurement error. Transverse sections were made by pre-pressing the samples in a mold, followed by cutting off the edges of the sample and polishing them.

Tribological tests to determine wear resistance under mechanical friction were carried out using a Unitest framework SKU UT-750 (Unitest, USA). As a method for determining the coefficient of dry friction, the “ball on disk” method was used, the load on the ball was 20 N, the sliding speed was 0.25 m/s, the number of cycles in the tests was about 15000 and was chosen to take into account the tests of the original sample, for which, after 10000 cycles, a sharp deterioration in wear resistance was observed (an increase in the dry friction coefficient was recorded). The silicon carbide ball was used as a counterweight during wear tests, and no wear of the ball was observed. The wear value was determined by measuring the wear profile obtained using the visualization method and then measuring the profile with a profilometer to determine the length of the path travelled. These alterations in the dry friction coefficient were recorded during each test, and the graphs show points after every 1000 tests, clearly demonstrating the change in wear resistance. The growth in wear resistance was assessed by comparing the values of the wear rate determined based on changes in the values of the dry friction coefficient at the beginning and at the end of the tests, and the resulting values were compared with each other.

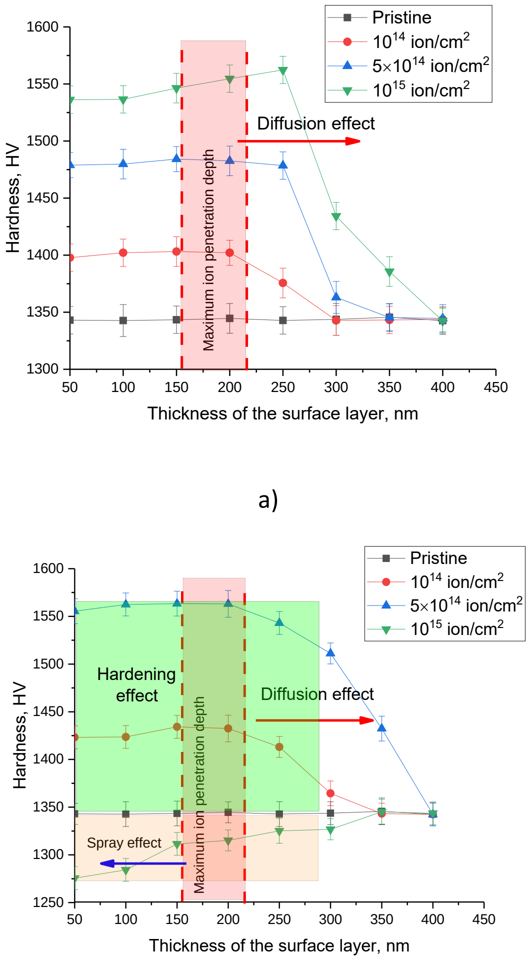
Determination of the resistance of modified ZrO_2 – Al_2O_3 ceramics to thermal cycling, which includes rapid heating of samples to high temperatures and subsequent sharp cooling, was carried out by conducting the following experiments. Samples of ZrO_2 – Al_2O_3 ceramics in the initial (non-irradiated) and irradiated state were subjected to rapid heating (heating rate 50 °C/min) to temperatures of 700, 1000 and 1200 °C, after which the samples were kept at a given test temperature for 1 hour, then the samples were removed from the muffle furnace into the air to initiate thermal shock processes. This procedure was carried out with the samples in the form of a sequence of tests (5 cycles); after each cycle, the hardness values of the surface layer were measured to determine resistance to thermal effects.

Results and Discussion

The most indicative parameters reflecting the influence of ionic modification on the strengthening of the near-surface layer are the values of hardness and wear resistance of ceramics, measured depending on the irradiation fluence. Moreover, in the case of hardness measurements, it is important to understand not only the general trend of changes in hardness but also the thickness of the modified layer, in which the hardness parameters differ from the base material.

Figure 3 shows the results of hardness measurements (Vickers) along the ion penetration depth (with a step of 50 nm), reflecting changes in the strength characteristics of ceramics depending on the irradiation fluence. Also, for comparison, the results of hardness measurements for the original (non-irradiated sample) are given, which reflect the isotropy of the strength parameters in depth.

The general appearance of the presented dependences of the change in hardness with depth indicates several effects associated with irradiation with heavy ions, which depend on both the type of ions and the irradiation fluence. The observed hardening (rise in sample hardness depending on the irradiation fluence) is in good agreement with the results of [[33], [34], [35]], in which the observed strengthening is explained by an elevation in dislocation density and the formation of dislocation loops, as well as, as a consequence, the formation of deformation distortions in the damaged layer caused by the accumulation of implanted ions. In this case, the observed strengthening can be explained by the fact that during the interaction of incident ions with the structure of the near-surface layer, due to the high values of ionization losses in the damaged layer, recrystallization processes can be initiated, accompanied by a change in crystallite sizes or orientation, which, as a consequence, result in an alteration in the dislocation density (its increase in the case of observed decreases in crystallite sizes, due to the inverse square dependence of the dislocation density on the crystallite sizes).

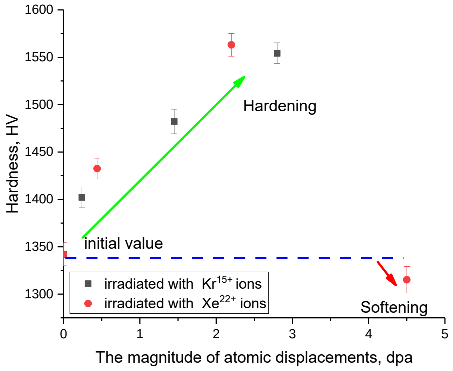


a)

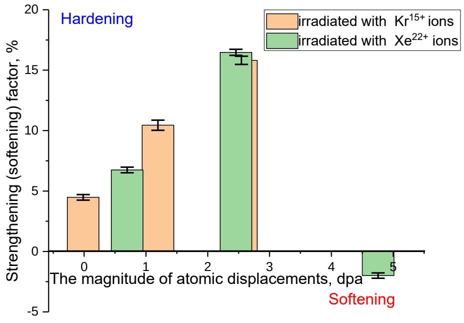
b)

Figure 3 - Results of evaluation of change in hardness value of ZrO_2 – Al_2O_3 ceramics by depth in case of heavy ion irradiation: a) when irradiated with Kr^{15+} ions; b) when irradiated with Xe^{22+} ions (the dotted lines indicate the region of maximum ion penetration depth in ceramics)

In the case of irradiation with Kr^{15+} ions, two effects can be distinguished in changes in hardness values, associated with the strengthening of the damaged layer, as well as diffusion effects, which lead to an increase in the thickness of the modified layer. Analysis of changes in hardness by depth demonstrated that an elevation in irradiation fluence leads to an expansion of the depth of the modified layer, which has higher hardness values than the base material. Such effects are caused by diffusion processes of ion penetration and consequences associated with their interactions in the form of cascade effects that can penetrate to a greater depth than the maximum ion penetration depth. In this case, an increase in the irradiation fluence, and, as a consequence, the magnitude of atomic displacements, leads to the fact that the number of structural changes occurring in the surface layer can have an impact at a deeper level as a result of cascade effects, which is in good agreement with the results of [36]. A general analysis of the observed changes in hardness values by depth showed that irradiation fluence growth from 10^{14} ion/cm^2 to 10^{15} ion/cm^2 in the case of irradiation with Kr^{15+} ions leads to an increase in the thickness of the modified layer by approximately 70 – 100 nm, while the hardening value decreases as the depth increases, which also confirms the diffusion nature of the propagation of structural damage deep into the material with increasing irradiation fluence [[37], [38]].



a)



b)

Figure 4 - a) Results of a comparative analysis of changes in hardness values at the maximum (at a depth of 150 - 200 nm) upon irradiation with heavy ions Kr^{15+} and Xe^{22+}; b) Results of assessment of the factors of hardening (softening) of the surface layer of $ZrO_2 – Al_2O_3$ ceramics depending on the value of atomic displacements caused by irradiation

In the case of irradiation with Xe^{22+} ions, depending on the irradiation fluence, not only hardening effects are observed (at a fluence of 10^{14} – 5×10^{14} ion/cm^2), but also reverse effects associated with a decline in hardness in the damaged surface layer. Such a reduction can be explained by the effects of overstresses as a result of the accumulation of a large number of structural distortions in the near-surface layer, which can lead to deformation embrittlement and peeling of the surface under external mechanical influences, as well as partial sputtering of the near-surface damaged layer, an effect that is characteristic of high-dose irradiation [[39], [40]]. Moreover, in the case of irradiation with Xe^{22+} ions, the magnitude of the change in hardness at the same irradiation fluence in comparison with samples irradiated with Kr^{15+} ions is slightly larger, which indicates a more intense modification of the surface layer when irradiated with Xe^{22+} ions. This effect can be explained by differences in the values of ionization losses, which are significantly higher in the case of irradiation with Xe^{22+} ions. At the same time, the observed softening at an irradiation fluence of 10^{15} ion/cm^2 can be explained by the contribution to the structural changes from the effects of more intense accumulation of atomic displacements due to higher values of ionization losses of Xe^{22+} ions during interaction with nuclei.

Results of a comparative analysis of changes in the maximum hardness values of the samples depending on the value of atomic displacements are presented in Figure 4a.

According to the presented comparative analysis data, it is clear that the change in the hardness of the near-surface damaged layer of ZrO_2 – Al_2O_3 ceramic samples has a clear dependence on the value of atomic displacements, which is different for the two types of ions. In this case, we can conclude that the most effective way to modify the surface layer is irradiation with heavy Kr^{15+} and Xe^{22+} ions with fluences at which the value of atomic displacements is no more than 3 dpa. At the same time, for Kr^{15+} ions, this value is achieved at a fluence of the order of 10^{15} ion/cm^2, while in the case of irradiation with Xe^{22+} ions, the achievement of atomic displacements of the order of 3 dpa can be achieved at fluences of the order of 5 – 6×10^{14} ion/cm^2. It should also be noted that the use of Xe^{22+} ions with fluences below 10^{15} ion/cm^2 allows modification of the damaged layer at a greater depth (the depth of the modified layer in ZrO_2 – Al_2O_3 ceramics when irradiated with Xe^{22+} is about

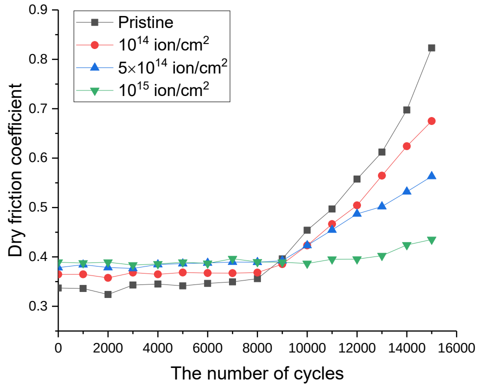
300 – 350 nm, taking into account diffusion effects).

Figure 4b reveals the results of the hardening (softening) value determined by comparative analysis of the hardness values in the initial (non-irradiated) state and the values obtained during irradiation with different irradiation fluences. The data is presented as a dependence of the change in the hardening factor as a percentage on the magnitude of atomic displacements, determined from the data presented in Figure 2b-c. The results presented in Figure 4b confirm the above assumption about the influence of the magnitude of atomic displacements on the strengthening of the near-surface layer of ceramics, according to which the most effective is irradiation with Xe^{22+} ions with fluences of 5 – 6×10^{14} ion/cm^2.

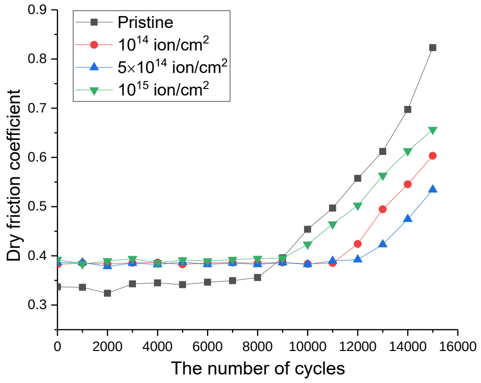
Figure 5 demonstrates the results of tribological tests of ZrO_2 – Al_2O_3 ceramics depending on the type of modification of the surface layer (in the case of varying the type of ion irradiation and irradiation fluence). The observed changes in the dry friction coefficient in the initial state when comparing the values for non-irradiated and irradiated ceramics indicate that ionic modification leads to a slight increase in the dry friction coefficient (no more than 3 – 5 %), which can be explained by the effects of changes in the morphological features of the surface of ceramics as a result of ion modification. As a rule, during low-energy irradiation, a change in morphology is caused by deformation extrusion of the damaged volume in the near-surface layer onto the surface in the form of hillock-like inclusions, which in turn can lead to an increase in friction and resistance during tribological tests.

The results of tribological tests revealed that the main changes for ZrO_2 – Al_2O_3 ceramics in the initial (non-irradiated) state are observed after 8000 – 10000 cycles, and consist in a sharp increase in the dry friction coefficient, which indicates surface degradation and a decline in wear resistance. At the same time, for samples modified with Kr^{15+} ions, irradiation fluence growth from 10^{14} ion/cm^2 to 5×10^{14} ion/cm^2 results in a decrease in the trend in the dry friction coefficient value, which indicates an increase in resistance to surface degradation under mechanical influences, and in the case of modification of fluences of 10^{15} ion/cm^2, in addition to reducing the trend of deterioration of the dry friction coefficient, there is also an increase in the number of test cycles in which changes in the dry friction coefficient are within the measurement error.

For ZrO_2 – Al_2O_3 ceramics modified with Xe^{22+} ions at fluences of 10^{14} – 5×10^{14} ion/cm^2, a similar decrease in the trend of changes in the dry friction coefficient is observed depending on the number of test cycles. Moreover, it should be noted that with an irradiation fluence of 5×10^{14} ion/cm^2, and in the case of irradiation with Kr^{15+} ions with an irradiation fluence of 10^{15} ion/cm^2, an increase in surface resistance to wear is observed over a greater number of cycles than under other irradiation conditions. At the same time, samples of ZrO_2 – Al_2O_3 ceramics irradiated with Xe^{22+} ions with a fluence of 10^{15} ion/cm^2, as in the case of hardness determination, during tribological tests show a negative trend due to the lower stability of the ceramic surface associated with a destructive change in structural properties at a high concentration of atomic displacements.



a)



b)

Figure 5 - Results of tribological tests of ZrO_2 – Al_2O_3 ceramics: a) when irradiated with Kr^{15+} ions; b) when irradiated with Xe^{22+} ions

Figure 6 shows the results of the specific volumetric wear of the surface of ZrO_2 – Al_2O_3 ceramics as a result of tribological tests, determined based on changes in the dry friction coefficient before and after cyclic tests.

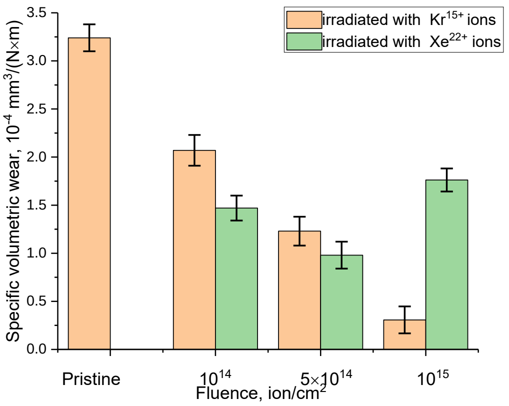
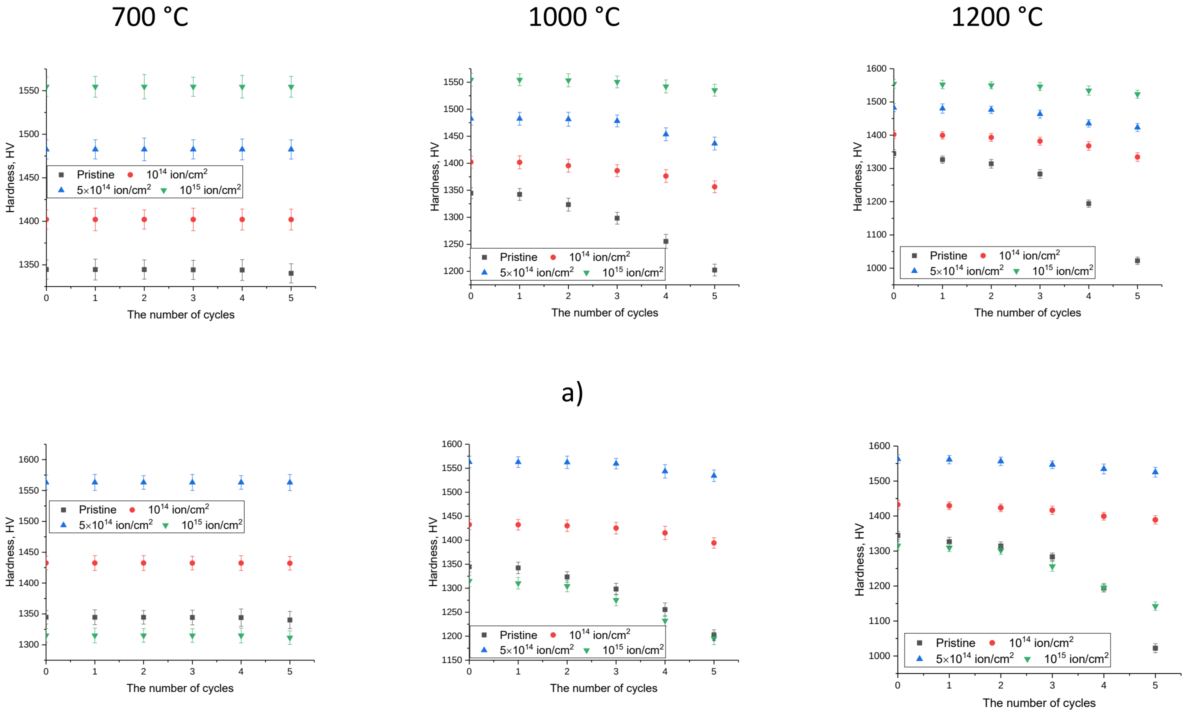


Figure 6 - Results of a comparative analysis of the specific volumetric wear of the surface of $ZrO_2 - Al_2O_3$ ceramics as a result of tribological tests



a)

b)

Figure 7 - Results of experiments on thermal cycling of ceramics depending on irradiation conditions: a) when irradiated with Kr^{15+} ions; b) when irradiated with Xe^{22+} ions

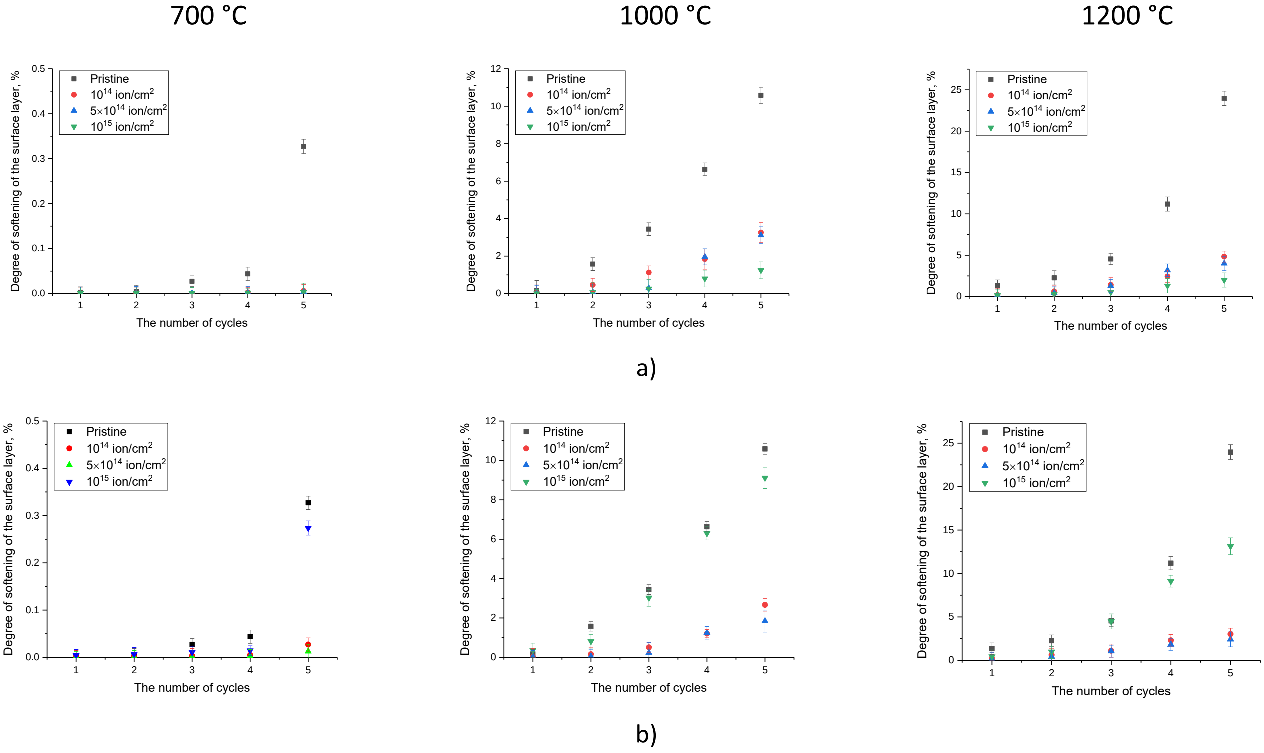
Analysis of the specific wear of the surface as a result of tribological tests showed that ionic modification with fluences of $10^{14} - 5\times10^{14}$ ion/cm^2 leads to an increase in wear resistance, which is due to dislocation strengthening of the near-surface layer. Moreover, in the case of samples of $ZrO_2 - Al_2O_3$ ceramics irradiated with Kr^{15+} ions, the reduction in the specific wear volume is more than 2 – 2.5 times compared to the original (non-irradiated) ceramics, which indicates the high prospects of using ion irradiation to increase the strength and wear resistance of the ceramic surface.

Figure 7 demonstrates the assessment results of changes in the hardness of $ZrO_2 - Al_2O_3$ ceramics as a result of thermal cycling at different test temperatures (700, 1000 and 1200 °C) depending on the number of test cycles.

As can be seen from the data presented, there are practically no changes in hardness depending on the number of thermal cycling cycles, there are practically no significant changes at a temperature of 700 °C for all studied samples, regardless of the type of ion exposure, which indicates a fairly high resistance of ceramics to thermal shocks at this temperature. It is worth to note that a slight decrease after 4-5 test cycles at a given temperature can be explained by a low degradation degree of the surface layer, but this decrease is less than 0.5 %, which is within the permissible error. Growth in the temperature of thermal cycling tests to 1000 – 1200 °C in the case of original (non-irradiated) ceramics results in a decline in hardness values after 2-3 test cycles and a sharp deterioration in strength characteristics after 5 test cycles. So, after 5 cycles of tests at a temperature of 1200 °C for the initial samples, the decrease in hardness is more than 20 %, which indicates degradation of the resistance of ceramics to mechanical stress. Moreover, in the case of ion modification, a less pronounced trend of decrease in hardness is observed during thermal cycling tests, which indicates the positive effect of the presence of a radiation-deformed layer, which prevents oxidation processes of the surface layer as a result of temperature changes, as well as during interaction with the atmosphere in the case of rapid removal of samples from the furnace chamber into the air.

Figure 8 shows the results of a comparative analysis of changes in the softening degree of ceramics in the original (non-irradiated) and irradiated ceramics, reflecting a decrease in the stability of the strength properties of ceramics with changes in thermal cycling conditions (with an increase in the number of cycles and temperature). The softening degree was assessed by comparing the hardness values of the ceramics in the initial state (before thermal cycling) with the hardness value after each test cycle.

As can be seen from the results of the degree of softening to thermal cycling processes and the accompanying oxidation processes of the surface layer when interacting with air, ceramics are least resistant to high-temperature thermal shocks at 1200 °C, at which successive thermal cycling leads to accelerated degradation of the hardness of the surface layer. In the case of modified ceramics, the decrease in hardness after 5 consecutive cycles of thermal cycling at temperatures of 1000 °C and 1200 °C is about 2-5 % (with the exception of ceramic samples irradiated with Xe^{22+} ions), which indicates fairly high resistance of ceramics to external influences and thermal shocks that may occur during operation.



a)

b)

Figure 8 - Assessment results of the surface layer softening degree (changes in surface hardness) depending on the amount of thermal cycling of ceramic samples: a) when irradiated with Kr^{15+} ions; b) when irradiated with Xe^{22+} ions

Conclusions

The paper presents the assessment results of the influence of low-energy ion irradiation (with Kr^{15+} and Xe^{22+} ions) on changes in the near-surface layer of composite ZrO_2 – Al_2O_3 ceramics, which have great prospects for use as structural materials capable of withstanding high temperatures and large mechanical loads during operation. The method of low-energy ion irradiation with Kr^{15+} and Xe^{22+} ions with energies of 300 and 440 keV and irradiation fluences from 10^{14} to 10^{15} ion/cm^2 was chosen as a method for modification of the near-surface layer. The choice of ions for irradiation is determined by the possibility of carrying out targeted modification of a near-surface layer with a thickness of about 200 – 250 nm.

During the studies, it was found that the use of Xe^{22+} ions with fluences below 10^{15} ion/cm^2 makes it possible to modify the surface layer at greater depths. The depth of the modified layer in ZrO_2 – Al_2O_3 ceramics when irradiated with Xe^{22+} is about 300 – 350 nm, taking into account diffusion effects, while when irradiated with Kr^{15+} ions, the thickness of the modified layer is no more than 300 nm.

The results of tribological tests showed that for unmodified ZrO_2 – Al_2O_3 ceramics, a decrease in wear resistance appears after 8000 – 10000 cycles, while for ceramics modified with Kr^{15+} ions, an elevation in irradiation fluence from 10^{14} ion/cm^2 to 5×10^{14} ion/cm^2 results in a decrease in the trend in the value of the dry friction coefficient, which indicates an increase in resistance to surface degradation under mechanical influences. It should also be noted that in the case of modification of fluences of 10^{15} ion/cm^2, in addition to reducing the trend of deterioration of the dry friction coefficient, there is also an increase in the number of test cycles in which changes in the dry friction coefficient are within the measurement error.

The results of thermal cycling (determining resistance to thermal shock) revealed that modification by ion irradiation results in a rise in degradation resistance and a reduction in hardness. Moreover, in the case of an exposure temperature of 700 °C, no changes in hardness are observed during cyclic tests, which indicates the high resistance of ceramics to external influences at this temperature regime.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **D.Borgekov:** Conceptualization, Writing - Original Draft, Methodology, Formal analysis; **A. Kozlovskiy:** Conceptualization, Writing - Original Draft, Writing - Review & Editing, Funding acquisition, Supervision, Methodology; **D.Shlimas:** Writing - Review & Editing, Supervision, Methodology; **R. Shakirziyanov:** Original Draft, Methodology, Formal analysis, Writing - Review & Editing; **A. Popov:** Conceptualization, Writing - Review & Editing; **M. Konuhova:** Conceptualization, Writing - Review & Editing.

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Композиттік керамиканың беткі қабаттарының беріктік қасиеттерін мақсатты өзгерту үшін ионды модификациялау әдістерін қолдану

[1]Боргеков Д.Б., [1,2]Козловский А.Л., [1,2]Шлимас Д.И., [2]Шакирзянов Р.И., [3]Попов А.И., [3]Конухова М.

[1] ҚР ЭМ Ядролық физика институты, Алматы, Қазақстан
[2] Л.Н. Гумилев атындағы Еуразия ұлттық университеті, Астана, Қазақстан
[3] Латвия Университетінің Қатты дене физикасы институты, Рига, Латвия

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Қабылданды: *10 ақпан 2025*

ТҮЙІНДЕМЕ

Магнетронды шашырату және механикалық индукцияланған деформациялық әсер сияқты әдістермен қатар материалдардың беткі қабаттарының беріктік қасиеттерін мақсатты өзгертудің перспективалы әдістерінің бірі ионды модификациялау әдістері болып табылады Зерттеудің осы бағытына қызығушылық, ең алдымен, материалдардың сыртқы механикалық және темтературалық әсерлерге төзімділігін арттыру, сонымен қатар өнеркәсіпте пайдалану, металлургия саласы мен реактор жасауда үлкен перспективалары бар баяу балқитын керамикалардың тозуға төзімділігін арттыру мүмкіндігімен байланысты. Бұл жұмыста беттік қабатта сыртқы әсерге төзімділігі жоғары радиациялық модификацияланған қабат құру мақсатында ZrO_2 – Al_2O_3 керамикасының беткі қабатын энергиясы 300 және 440 кеВ төмен энергиялы Kr15+ және Xe22+ иондарымен сәулелендіру арқылы ионды модификациялау әдісін қолдану мүмкіндігін бағалау нәтижелері ұсынылған. Жүргізілген зерттеулер барысында Xe^{22+} иондары үшін 10^{14} - 5×10^{14} ион/см^2 және Kr^{15+} иондары үшін 10^{15} ион/см^2 флюенсімен сәулелендіру беткі қабатты модификациялаудың оңтайлы шарттары болып табылатыны анықталды, нәтижесінде сәулелендірілмеген керамикалармен салыстырғанда, тозуға төзімділік 2.0 - 2.5 есе артты және беріктік 15 - 20%-ға дейін күшейді.

Түйінді сөздер: беріктену, иондық модификация, қаттылық пен тозуға төзімділікті арттыру, ZrO_2 – Al_2O_3 керамикалары, төмен энергиялы иондар.

Авторлар туралы ақпарат:

Дарын Б. Боргеков	*PhD, Ядролық физика институты, 050032, Алматы, Қазақстан. Email: d.borgekov@inp.kz; ORCID ID: https://orcid.org/0000-0002-9727-0511*
Артем Л. Козловский	*PhD, Ядролық физика институты, 050032, Алматы, Қазақстан. Email: kozlovskiy.a@inp.kz; ORCID ID: https://orcid.org/0000-0001-8832-7443*
Дмитрий И. Шлимас	*PhD, Л.Н. Гумилев атындағы Еуразия ұлттық университеті, 010008, Астана, Қазақстан. E-mail: shlimas@inp.kz; ORCID ID: https://orcid.org/0000-0003-2454-7177*
Рафаэль И. Шакирзянов	*PhD, Л.Н. Гумилев атындағы Еуразия ұлттық университеті, 010008, Астана, Қазақстан. E-mail: halfraf@mail.ru; ORCID ID: https://orcid.org/0000-0001-9908-3034*
Анатолий И. Попов	*Доктор, Латвия Университетінің Қатты дене физикасы институты, LV-1063, 8 Кенгарага, Рига, Латвия. E-mail: popov@latnet.lv; ORCID ID: https://orcid.org/0000-0003-2795-9361*
Марина Конухова	*PhD, Латвия Университетінің Қатты дене физикасы институты, LV-1063, 8 Кенгарага, Рига, Латвия. E-mail: marina.konuhova@cfi.lu.lv; ORCID ID: https://orcid.org/0000-0003-0743-5915*

Использование методов ионной модификации для направленного изменения прочностных свойств приповерхностных слоев композитных керамик

[1]Боргеков Д.Б., [1,2]Козловский А.Л., [1,2]Шлимас Д.И., [2]Шакирзянов Р.И., [3]Попов А.И., [3]Конухова М.

[1] Институт ядерной физики МЭ РК, Алматы, Казахстан
[2] Евразийский национальный университет им. Л.Н. Гумилева, Астана, Казахстан
[3] Институт физики твердого тела Университета Латвии, Рига, Латвия

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АННОТАЦИЯ

Использование методов ионной модификации является одним из перспективным методов направленного изменения прочностных свойств приповерхностных слоев материалов наравне с такими методами как магнетронное напыление и механически индуцированное деформационное воздействие. Интерес к данному направлению исследований обусловлен в первую очередь возможностью повышения устойчивости материалов к внешним механическим и термическим воздействиям, а также увеличению износостойкости тугоплавких керамик, обладающих большими перспективами в промышленном использовании и металлургии и реакторостроении. В данной работе представлены результаты оценки возможности применения метода ионной модификации путем облучения приповерхностного слоя ZrO_2 – Al_2O_3 керамик низкоэнергетическими ионами Kr^{15+} и Xe^{22+} с энергиями 300 и 440 кэВ с целью создания в приповерхностном слое радиационно-модифицированного слоя, обладающего высокой устойчивостью к внешним воздействиям. В ходе проведенных исследований было установлено, что облучение с флюенсами 10^{14} - 5×10^{14} ион/см^2 для ионов Xe^{22+} и 10^{15} ион/см^2 для ионов Kr^{15+} являются оптимальными условиями модификации приповерхностного слоя, в результате которых наблюдается увеличение износостойкости в 2.0 – 2.5 раза и упрочнение более чем на 15 – 20 % по сравнению с необлученными керамиками.

Ключевые слова: упрочнение, ионная модификация, повышение твердости и износостойкости, ZrO_2 – Al_2O_3 керамики, низкоэнергетические ионы.

Дарын Б. Боргеков	***Информация об авторах:*** *PhD, Институт ядерной физики, 050032, Алматы, Казахстан. Email: d.borgekov@inp.kz; ORCID ID: https://orcid.org/0000-0002-9727-0511*
Артем Л. Козловский	*PhD, Институт ядерной физики, 050032, Алматы, Казахстан. Email: kozlovskiy.a@inp.kz; ORCID ID: https://orcid.org/0000-0001-8832-7443*
Дмитрий И. Шлимас	*PhD, Евразийский национальный университет им. Л.Н.Гумилева, 010008, Астана, Казахстан. E-mail: shlimas@inp.kz; ORCID ID: https://orcid.org/0000-0003-2454-7177*
Рафаэль И. Шакирзянов	*PhD, Евразийский национальный университет им. Л.Н.Гумилева, 010008, Астана, Казахстан. E-mail: halfraf@mail.ru; ORCID ID: https://orcid.org/0000-0001-9908-3034*
Анатолий И. Попов	*Доктор, Институт физики твердого тела Университета Латвии, LV-1063, 8 Кенгарага, Рига, Латвия. E-mail: popov@latnet.lv; ORCID ID: https://orcid.org/0000-0003-2795-9361*
Марина Конухова	*PhD, Институт физики твердого тела Университета Латвии, LV-1063, 8 Кенгарага, Рига, Латвия. E-mail: marina.konuhova@cfi.lu.lv; ORCID ID: https://orcid.org/0000-0003-0743-5915*

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Engineering and Technology

Epoxy coatings for anticorrosion applications: a review

[1,2*]Bekbayeva L.,[2] Negim E.-S., [2]Zhanibekov R., [2]Sharipov R., [2]Maldybayev G.,
[3]Puzikova D.S., [3]Kenzin N.R., [4]Maridan A.A.B.

[1]Al-Faraby Kazakh National University, al-Farabi av., Almaty, Kazakhstan
[2]Kazakh British Technical University, Almaty, Kazakhstan
[3]D.V. Sokolsky Institute of Fuel, Catalysis and Electrochemistry, Kazakh British Technical University, Almaty, Kazakhstan
[4]Universiti Teknologi PETRONAS, 32610 Bandar Seri Iskandar, Perak, Malaysia

**Corresponding author: Bekbayeva Lyazzat, lyazzat_bk2019@mail.ru*

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ABSTRACT
Epoxy resins are among the most commonly used materials for anticorrosion applications due to their excellent adhesion, mechanical strength, and chemical resistance. However, conventional epoxy coatings face significant limitations in providing durable, long-term protection, especially under harsh environmental conditions. As a result, extensive research has been conducted worldwide to enhance the anticorrosion performance of epoxy coatings. This review summarizes the latest advancements in the field, categorizing current developments into three primary approaches: modification of the epoxy resin structure, incorporation of functional fillers, and the development of multifunctional composite coatings. Structural modifications focus on improving the intrinsic properties of epoxy resins to enhance their barrier effect. The inclusion of functional fillers introduces additional protective mechanisms, including self-healing, superhydrophobicity and corrosion inhibition. Multifunctional composite coatings combine the benefits of several approaches, integrating advanced materials and techniques to achieve high performance. By analyzing recent studies and innovations, this review highlights the strengths of each approach, providing insights into future directions for developing high-performance epoxy-based anti-corrosion coatings.

Keywords: composites, epoxy resin, anticorrosive coating, corrosion, corrosion inhibition, anti-corrosion protection.

	Information about authors:
Lyazzat Bekbayeva	*National Nanotechnology Open Laboratory, Al-Faraby Kazakh National University, al-Farabi av., 050040, Almaty, Republic of Kazakhstan. Email: lyazzat_bk2019@mail.ru*
El-Sayed Negim	*School of Materials Science and Green Technologies, Kazakh British Technical University, St. Tole bi, 59, 050000, Almaty, Republic of Kazakhstan. Email: elashmawi5@yahoo.com*
Rinat Zhanibekov	*School of Materials Science and Green Technologies, Kazakh British Technical University, St. Tole bi, 59, 050000, Almaty, Republic of Kazakhstan. Email: rin_zhanibekov@kbtu.kz*
Rustam Sharipov	*School of Materials Science and Green Technologies, Kazakh British Technical University, St. Tole bi, 59, 050000, Almaty, Republic of Kazakhstan. Email: r.sharipov@kbtu.kz*
Galymzhan Maldybayev	*School of Materials Science and Green Technologies, Kazakh British Technical University, St. Tole bi, 59, 050000, Almaty, Republic of Kazakhstan. Email: g.maldybaev@kbtu.kz*
Puzikova Darya Sergeevna	*D.V. Sokolsky Institute of Fuel, Catalysis and Electrochemistry, Kazakh British Technical University, 050010, Kunaev st., 142, Almaty, Republic of Kazakhstan. Email: d.puzikova@ifce.kz*
Kenzin Nail Rashidovich	*D.V. Sokolsky Institute of Fuel, Catalysis and Electrochemistry, Kazakh British Technical University, 050010, Kunaev st., 142, Almaty, Republic of Kazakhstan. Email: n.kenzin@ifce.kz*
Anis Aeshah Binti Maridan	*CO_2 Research Centre (CO_2RES), Universiti Teknologi PETRONAS, 32610 Bandar Seri Iskandar, Perak, Malaysia. Email: ad.aeshah@gmail.com*

Introduction

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A natural process known as corrosion occurs when metal interacts with environmental elements including oxygen, moisture, and chemical agents. This results in structural deterioration, loss of functioning, and eventually large financial losses. A study by the National Association of Corrosion Engineers (NACE) estimates that the yearly cost of corrosion worldwide exceeds $2.5 trillion, or around 3-4% of the world's gross domestic product [1]. Due to the extremely corrosive conditions that are present during the extraction and transportation of hydrocarbons, the oil and gas industry, for example, suffers significant corrosion-related damage to pipes and infrastructure. Likewise, offshore platforms and marine boats are particularly vulnerable to corrosion brought on by saltwater, which speeds up the rate of metal deterioration [2]. Especially in the petroleum industry, corrosion poses a safety risk and results in significant financial losses [3]. The hardened mixture's structure is destroyed by both internal and external forces acting on concrete and the depletion of quality attributes like density, strength, and so forth.

Anticorrosion coatings have been used extensively to address these problems as an affordable way to shield metal surfaces from deterioration, increasing longevity and preserving the structural soundness of important parts. Corrosive substances like oxygen, water, and salts are kept from coming into direct contact with the metal substrate by these coatings, which serve as a physical and chemical barrier. Epoxy resins stand out as one of the most adaptable and efficient choices among the variety of materials used in anticorrosion coatings [3]. Epichlorohydrin reacts with bisphenol-A or other phenolic chemicals to form epoxy resins, which are thermosetting polymers. Epoxy resins, or ERs, are a unique class of organic macromolecules with a wide range of industrial applications. Compared to basic organic corrosion inhibitors, ERs offer superior surface coverage and anticorrosive properties due to their macromolecular nature. During metal-inhibitor interactions, the polar functional groups on the periphery of ERs serve as adsorption sites. Particularly for carbon steel in acidic and sodium chloride (3% and 3.5%) solutions, a few ERs in their pure and cured forms have been employed as anti-corrosive coating materials. Most of the ER's function as mixed-type and interface corrosion inhibitors [4].

The anticorrosive properties and adsorption behaviours of ERs on metallic surfaces have been demonstrated through a variety of computer simulations. Since most ERs are not very soluble, they work best as coating materials for anticorrosive applications. A review of the literature revealed that numerous ER-based coatings are developed and successfully employed for carbon steel and aluminium in brine solution . They are especially good at shielding metal surfaces from corrosion because of their remarkable qualities, which include great mechanical strength, superior adherence to metal surfaces, superior chemical resistance, and thermal stability [5]. They are especially good at shielding metal surfaces from harsh external elements because of their remarkable qualities, which include great mechanical strength, strong chemical resistance, excellent adherence to metal surfaces, and thermal stability [6]. Epoxy coatings are widely used in industrial applications because they form a tight bond with substrates, which lowers the risk of delamination and corrosion undercutting. Their efficacy in reducing corrosion-related damage is further enhanced by their low permeability to oxygen and moisture. Because of their high degree of cross-linking, these resins are stiff and impervious to deterioration in the environment [7].

Benefits of epoxy resins

Epoxy resins, as thermoset polymers, have distinct manufacturing properties such as low pressure, minimal cure shrinkage, and low residual stresses, allowing the creation of precise and durable products. These resins can be used at a wide temperature range by adjusting the cross-linking levels with appropriate curing agents. They are suitable for electrical insulation, surface coatings, engineered composites, and structural adhesives, and are available in both low-viscosity liquid and powder forms. Furthermore, their efficient manufacturing processes contribute to faster production and lower industrial costs, making them highly practical in various industries [8].

In terms of mechanical properties, epoxy resins offer modified fatigue resistance and mechanical strength, as well as superior chemical and heat resistance. These properties are due to their thermosetting polymer nature, which occurs when polyoxides react with hardeners. Their structural integrity allows them to endure severe chemicals, high temperatures, and mechanical strain, making them indispensable for applications that require durable and robust materials. Further, epoxy resins also have excellent chemical resistance, which increases their adaptability to a wide range of industries, including maritime, construction, and automotive. Their ability to form strong protective layers results in excellent bonding with substrates such as concrete, wood, and metals. This makes them ideal for waterproofing, protecting surfaces, and extending the life of materials. Epoxy composites, known for their strength and lightweight characteristics, support advanced manufacturing processes, while epoxy coatings effectively combat corrosion and maintain the integrity of metal surfaces [[9], [10]].

One major advantage of epoxy resins is their ability to improve the mechanical properties of

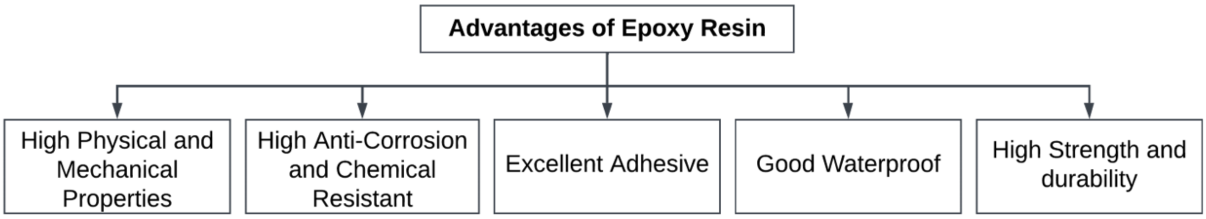

Figure 1 - Advatages of epoxy resins

materials used in industrial manufacturing. Epoxy resins help to produce goods that can withstand harsh industrial conditions by increasing their strength, resilience, and overall performance. Their role includes reinforcing concrete structures, creating high- performance components, and ensuring the dependability and durability of industrial products [[8], [9]]. Another significant advantage of epoxy resins is their ability to address environmental concerns while still providing exceptional performance. These resins have superior mechanical and thermal resistance because they contain cross-linking co-reactants such as polyfunctional amines and acids. Their durability and efficiency make them ideal for applications such as metal coatings and composites. Researchers are developing sustainable alternatives to replace harmful chemicals in epoxy resins, contributing to greener industrial practices [10]. Main advantages of epoxy resins demonstrated in Figure 1.

In recent years, there has been a significant increase in the number of works related to anticorrosive epoxy coatings and composites. The current development of anti-corrosion epoxy coatings can be grouped into 3 main approaches: epoxy composites based on nanofillers, epoxy structure modifications, and multifunctional coatings (Figure 2). Enhancing the corrosion resistance of coatings is achieved by barrier or sacrificial protection or corrosion inhibition [11].

Epoxy composites and systems

One of the common and most affordable methods for improving the anticorrosive properties of epoxy coatings is the introduction of modified mineral nanofillers. Surface modification of the nanoparticles makes them stable and provides a high degree of dispersion, which prevents their aggregation caused by high specific surface area and surface energy [12]. Such nanoparticles are evenly distributed in the coating matrix, reducing its porosity, closing microcracks decreasing coating affinity with corrosion medium, and significantly increasing corrosion resistance. Ou et al. [13] conducted surface modification of TiO_2 nanoparticles by toluene diisocyanate (TDI). The TDI molecules were covalently bonded to the nanoparticle surface, significantly enhancing their dispersibility in toluene. This functionalization approach effectively mitigated the agglomeration tendency of TiO_2 nanoparticles, ensuring a more stable dispersion. In a separate study by Situ et al. [14], polyaniline-titanium nitride (PANI-TiN) nanocomposites were developed through a low-temperature chemical oxidation polymerization process. Adding TiN nanoparticles to PANI nanorods minimized their tendency to aggregate and improved their uniform distribution in the epoxy matrix. The resulting PANI-TiN/epoxy coatings demonstrated significantly enhanced corrosion protection, with impedance modulus values increasing by 115 times compared to pure epoxy coatings after two days of exposure to a 3.5 wt.% NaCl solution. This improvement was primarily due to the combined effects of PANI's passivation properties and creating winding paths by TiN nanoparticles for the electrolyte, slowing down the corrosion process and increasing the durability of the coating. In a study by Jlassi et al. [15], magnetite-clay nanocomposites modified with polyaniline (PANI) and diazonium salts (B-DPA-PANI@Fe_3O_4) were synthesized and integrated into epoxy coatings. The inclusion of 3 wt.% of these fillers significantly enhanced the anticorrosion performance, increasing the charge transfer resistance up to 110×10^6 Ω·cm² compared to 0.35×10^6 Ω·cm² for pure epoxy. This improvement was attributed to the uniform dispersion of the nanofillers, reduced coating porosity, and the synergistic interaction between the filler and the epoxy matrix, which decreased the penetration of chloride ions in a corrosive environments.

A large number of studies are devoted to the use of graphene to improve coating properties. Graphene, being a two-dimensional nanomaterial, consists of a single layer of atoms and has exceptional barrier properties that prevent the

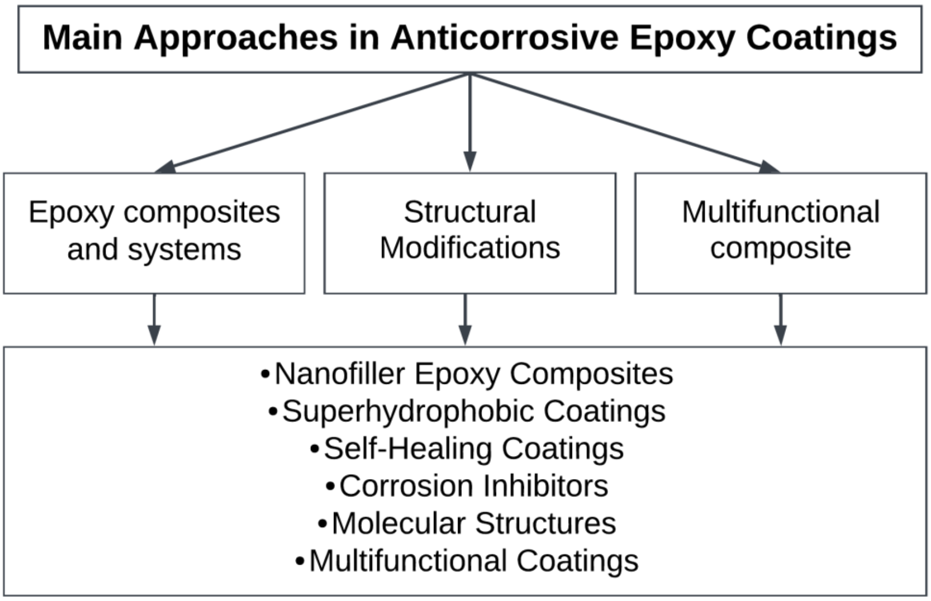

Figure 2 - Main Approaches in Anticorrosive Epoxy Coatings

penetration of almost any molecules or ion. Even small additions of graphene to the coating composition can significantly improve its physical characteristics and enhance protection from aggressive media [16]. In [17], the corrosion behavior of epoxy coatings reinforced with reduced graphene oxide (RGO) on N80 steel was studied under conditions of high salinity (10% NaCl) and a temperature of 80 °C. Coatings with different RGO content (0%, 0.5%, 1%, 2% and 4%) were characterized by SEM, EIS, and Tafel polarization methods. It was found that the addition of RGO reduces the number and size of pores in the coating, improves its adhesion and strength. The coating with 1% RGO showed the best anticorrosive properties, at which the charge transfer resistance (Rct) reached 17×10^6 Ω·cm2, while with an increase in the RGO content above 2%, particle aggregation and deterioration of protective properties were observed. In [18], the effect of an ionic liquid based on 1-butyl-3-methylimidazolium chloride (BMIM-Cl) on the dispersion of graphene oxide (GO) in epoxy resin and the anticorrosive properties of the obtained composites was investigated. The results showed that the adsorption of BMIM-Cl on the GO surface improved the compatibility of nanoparticles with the epoxy matrix, preventing aggregation. Coatings with GO-BMIM demonstrated higher protective properties compared to pure epoxy resin, which was confirmed by EIS, salt spraying and polarization methods. The best barrier effect was achieved with a GO-BMIM content of 0.12%.

It can be functionalized to reduce the aggregation of graphene particles. Ziat et al. [19] dispersed single-layer graphene in pure ethanol, and then created nanocomposites based on epoxy resin with a graphene content of 2 wt.% using the in-situ polymerization method. The functionalized graphene in the coating showed excellent dispersibility. Potentiodynamic polarization and electrochemical impedance spectroscopy have confirmed a significant improvement in the corrosion resistance of the coating on the copper substrate in a NaCl solution with a concentration of 3%. Scanning electron microscopy (SEM) analysis confirmed the effectiveness of the coating as a barrier, and measurements of the wetting angle indicated the hydrophobic properties of the coating, emphasizing its high mechanical strength and barrier characteristics.

Xie et al. [20], in turn, modified graphene oxide (GO) with polyacrylate using radical copolymerization technology. First, the GO surface was treated with silane (KH-570) to create active centers, then polymerization of acrylate monomers was performed in the presence of an initiator (AIBN) at 80 °C. This improved the dispersibility of GO in the epoxy matrix and the interaction at the interface, which was confirmed by scanning electron microscopy. The coatings obtained demonstrated a noticeable increase in anticorrosive properties.

The so-called superhydrophobic surfaces have recently gained particular interest. Such coatings are determined by their ability to minimize interaction with water: the water contact angle (WCA) on their surface exceeds 150°, and the rolling angle (RA) is less than 10°. This gives the coatings unique properties such as self-cleaning, water resistance, and anti-icing, resulting in a wide range of applications potential in various fields [[21], [22]].

Superhydrophobic coatings based on epoxy resins have been developed. A superhydrophobic coating [23] based on hydrophobic silica nanoparticles modified with paraffin wax synthesized by alkaline hydrolysis of silane precursors such as γ-aminopropyltriethoxysilane (APS) and tetraethoxysilane (TEOS) in the presence of paraffin wax was developed. The resulting nanoparticles were incorporated into epoxy matrices, with a 1 wt.% of the modified nanoparticles coating demonstrating improved dispersing of nanoparticles in epoxy coatings and anticorrosive properties, withstanding up to 1,500 hours in a salt mist. In addition, the water-wetting angles reached 100-110° when blended with 3Wt% of nanoparticles, which confirms their hydrophobicity. These results confirm that the modification of silica with paraffin wax increases corrosion resistance, improves mechanical properties, and reduces the wettability of coatings. In another work [24], a superhydrophobic coating based on epoxy resin was created using modified TiO_2 and ZnO nanoparticles synthesized using the "one pot" method. The process involved the preliminary modification of nanoparticles, and then their mixing with epoxy resin cured with HHPA hardener in a ratio of 5:3 by weight. The finished coating had a wetting angle of up to 155.6° and demonstrated high resistance to corrosion and abrasives. Additionally, experiments have shown that the degree of crosslinking of the epoxy matrix and adhesion to the metal substrate plays a key role in increasing the service life of the coating.

In the study [25], a superhydrophobic coating was developed by incorporating modified sepiolite powder into pure epoxy resin. The coating demonstrated improved corrosion resistance compared to pure epoxy, with a contact angle of 154.6° and a sliding angle of 3° when 5 wt% of the modified powder was added. Characterization techniques, including CA, SEM, FT-IR, and XRD, revealed its enhanced wettability and composition. Corrosion tests using EIS, salt-spray, and SKP confirmed that the composite coating effectively restricted corrosion in damaged regions, delaying substrate degradation and significantly enhancing corrosion protection.

In order to increase long-term corrosion protection, self-healing coatings or coatings with corrosion inhibitors can be used. The principle of the first type of coatings is their ability to restore integrity and anti-corrosion properties after damage. This is achieved either by releasing encapsulated active substances that react with the damaged surface and form a protective layer or by reactions triggered by external factors such as temperature or light [[26], [27]]. As for inhibitory protection, its essence lies in the release of inhibitory compounds from the coating matrix into damaged areas, which slows down or prevents corrosion processes [28].

Attaei et al. [29] developed a self-healing epoxy coating with microcapsules containing isophorone diisocyanate (IPDI). Microcapsules were synthesized by interphase polymerization in oil-in-water emulsions, ensuring their thermochemical stability and a diameter of 5-20 microns. Experiments using EIS, SVET, and LEIS have shown that when the capsule coating is damaged, IPDI is released, which forms a polymer layer that effectively suppresses corrosion. The destroyed microcapsules promote polymerization, restoring the protective properties of the coating. A similar study on self-healing coatings by microcapsules is presented in [30]. In another study [31], N-isopropylacrylamide-co-vinyltrimetho-xysilane (NIPAM-VTS) was used as a self-healing agent, with hydrolyzed microgel combined with tetraethoxysilane (TEOS) and 3-aminopropyltriethoxysilane (APTS) to create amino-functionalized smart silica composites and nanoparticles.

The direct addition of corrosion inhibitors to epoxy coatings often results in limited long-term protection and can negatively impact the coating's performance, causing issues such as blistering, reduced corrosion resistance, and weakened mechanical properties [32]. To overcome these drawbacks, an effective alternative approach involves incorporating inhibitors into micro- or nanostructures, which encapsulate or fix the inhibitors within the epoxy resin system, thereby avoiding harm to the coating's overall performance [33].

Structural Modifications of Epoxy Resins

As mentioned above, basic epoxy coatings are not capable of providing long-term corrosion protection. This is due to their limited mechanical and barrier properties, which deteriorate under the influence of aggressive environmental factors. Therefore, the approach discussed below aims to increase the wear resistance and corrosion resistance of epoxy coatings by developing new formulations or synthesizing epoxy resins with different molecular structures. In one study, Dagdag et al. [34] formulated a novel epoxy resin coating (TGEDA-MDA) by curing tetraglycidyl ether of diaminodiphenylmethane (TGEDA) with methylene dianiline (MDA). This coating demonstrated a protective efficiency of approximately 93% in a 3 wt% NaCl solution, as confirmed by potentiodynamic polarization (PDP) and electrochemical impedance spectroscopy (EIS). Additionally, computational modeling revealed the strong adhesion of TGEDA-MDA to carbon steel surfaces, forming a stable and effective protective layer. In another study [35], the same group developed a macromolecular epoxy coating (DGEDDS-MDA) based on bisphenol S diglycidyl ether (DGEDDS) cured with methylene dianiline. This molecular structure, rich in hydroxyl and amino groups, enhances adsorption of corrosive substances such as water and chloride ions. Even after 180 days of exposure to ultraviolet radiation, the coating exhibited high durability, with an impedance value of 2.12 kΩ cm², indicating sustained protective performance.

Researchers [36] studied the effect of curing agents with different molecular structures—diethylenetriamine (DETA), isophorone diamine (IPDA), and m-phenylenediamine (m-PDA)—on the tribological and anticorrosion properties of epoxy coatings. The molecular structure of these curing agents significantly influenced the cross-linking network and internal structure of the coatings, impacting their performance. Electrochemical tests in 3.5 wt% sodium chloride solution demonstrated that IPDA-cured coatings exhibited the best corrosion resistance due to their compact structure, which effectively blocked corrosive agents. In contrast, m-PDA-cured coatings showed the poorest performance, attributed to a looser internal structure that allowed easier penetration of the corrosive medium.

In recent years, there has been a growing demand for bio-based epoxy resins as sustainable alternatives to petroleum-based counterparts. This shift is driven by concerns over the depletion of fossil fuel resources, greenhouse gas emissions, and the toxicological impacts of conventional materials like bisphenol A (BPA), which is widely used in epoxy resins [37]. The itaconic acid-based epoxy resin (EIA) synthesized by Ma et al. [38] through an esterification reaction between itaconic acid and epichlorohydrin, yielding a resin with a high epoxy value of 0.625 and excellent curing reactivity. When cured with methyl hexahydrophthalic anhydride (MHHPA), EIA demonstrated superior thermal and mechanical properties, including a glass transition temperature of 130.4 °C and a tensile strength of 87.5 MPa, comparable to or exceeding traditional diglycidyl ether of bisphenol A (DGEBA) resins. Furthermore, the properties of EIA were enhanced by incorporating comonomers such as divinyl benzene (DVB) and acrylated epoxidized soybean oil (AESO), underscoring its potential as a sustainable replacement for petroleum-based thermosetting resins.

A bio-based epoxy resin synthesized from cashew nutshell liquid was developed for a one-component anticorrosive coating using a solvent-free process with in situ generated performic acid [39]. The curing behavior of the epoxy-phenol system with 1-methylimidazole (1-MIM) as an accelerator demonstrated efficient catalysis with only 5% by weight of 1-MIM, achieving a curing temperature of 150 °C. The coating exhibited high crosslink density, thermal stability (decomposition at 200 °C), and a glass transition temperature of 30 °C. Electrochemical impedance spectroscopy, along with adhesion and visual assessments, confirmed its excellent anticorrosive performance under salt spray and immersion in 3.5% NaCl solution. The material also maintained high adhesive strength and low delamination during humidity exposure tests. These results highlight the potential of this bio-based epoxy coating for corrosion protection applications.

Multifunctional composite coating

Epoxy resin coatings relying on a single anticorrosion approach often fall short in addressing the challenges posed by complex real-world conditions. The development of materials that integrate multiple protective approaches offers innovative opportunities to enhance the effectiveness and durability of epoxy coatings in practical applications. Dagdag et al. [40] developed an anticorrosive epoxy coating by synthesizing DGEDDS with epichlorohydrin, 4,4′-dihydroxy diphenyl sulfone, and sodium hydroxide, using 4,4′-methylene dianiline (MDA) as a hardener and zinc phosphate tetrahydrate (ZPH) as an anticorrosion pigment, resulting in a highly stable and corrosion-resistant coating. Liang et al. [41] developed nanocomposite epoxy coatings by incorporating 5% nano-Al through a two-stage process, ensuring high concentration and low viscosity. The nano-Al particles initially corrode to protect the substrate and subsequently form aluminum oxide and hydroxide, which act as barriers to prevent the penetration of corrosive liquids. Characterization through immersion, salt spray tests, and EIS confirmed a significant improvement in the coating's corrosion resistance. In the study by Lakouraj et al. [42], a high-performance epoxy resin was developed by curing diglycidyl ether of bisphenol-A (DGEBA) with diaminoxanthone (DAX) and incorporating functionalized Fe_3O_4 nanoparticles. Corrosion resistance tests, including potentiodynamic polarization and immersion in HCl solution, showed that the addition of 10% Fe_3O_4 nanoparticles significantly enhanced the anticorrosion performance compared to the neat DGEBA/DAX system.

In one study [43], α-Fe_2O_3@TA@GO composites were prepared by modifying mica iron oxide with tannic acid and graphene oxide and then incorporating them into an epoxy-acrylic resin. This hybrid system significantly improved corrosion resistance, achieving a low corrosion current density of 1.459 $\mu A/cm^2$ and a high charge transfer resistance of 14,350 $\Omega \cdot cm^2$. The addition of 5 wt% of the composite provided optimal performance, showcasing excellent compatibility and improved dispersion stability. In another study [44], epoxy resins were combined with functionalized materials, and similar processes involving surface modifications and composite incorporation were employed. The results indicated enhanced mechanical strength, thermal stability, and anticorrosion properties, demonstrating the potential of hybrid systems to overcome the inherent limitations of pure epoxy coatings.

Advancement of epoxy resin as anticorrosion coatings

Over the years, the field of epoxy resins has continuously evolved due to their unique properties such as high mechanical strength, temperature and solvent resistance, and strong adhesion to metals and composites. These developments have made epoxy resins crucial across aerospace, automotive, construction, and biomedical applications. Supplementing these commercial coatings are some of the newer developments such as the incorporation of nanomaterials into epoxy-based nanocomposite coatings to design coatings with a combination of unique properties of both epoxy and nanomaterials, providing a new range of advanced coatings with enhanced functionalities.

The development of superhydrophobic epoxy-based nanocomposite coatings; however, is a game-changer in this aspect, as they exhibit exceptional wetting resistance, chemical stability, and mechanical robustness. Incorporation of nanomaterials, such as silica nanoparticles, carbon nanotubes, and graphene, further enhances the properties of these coatings, resulting in high-performance coatings for applications such as anti-corrosion and self-cleaning surface [[45], [46]]. As epoxy is comparatively strong, durable and can bond with any material such as metal and glass, it is being used as a matrix material in these nanocomposites. These coatings show super hydrophobicity with water contact angles over 150° and very high corrosion and mechanical stress resistance and are very effective in extreme environments such as aerospace and marine industries [47]. In addition to performance enhancements, the establishment of superhydrophobic coatings matches sustainability objectives. Researchers are developing eco-friendly formulations to replace volatile organic compounds (VOCs), substances that have a negative environmental impact. This trend is part of a larger paradigm shift in materials science, to create smart materials that are high- performing but responsible at the same time [48].

Furthermore, the introduction of nanomaterials such as silica nanoparticles and graphene into epoxy resins has made considerable contributions to their corrosion resistance, hydrophobicity, and mechanical properties. By mimicking the arrangement of hydrophobic structures found in nature (e.g. lotus leaves), such nanocomposites repel water as well as prevent the buildup of corrosive agents on the composite surface, while also enabling other properties for example self-cleaning ability [[48], [49]]. These advanced coatings are usually made by spray coating, drop-coating and 3D bioprinting [45], while spray coating has received the most attention for large-scale usage. Additionally, continuous developments in environmentally friendly formulations are rendering these coatings sustainable, maintaining high performance and low environmental impacts [50]. These developments not only enhance the durability and effectiveness of epoxy coatings but also establish them as a cornerstone in modern industrial applications, ensuring long-term protection and sustainability.

The molecular weight of epoxy oligomers has a noticeable effect on the anti-corrosion properties of coatings [[51], [52], [53], [54], [55], [56]]. Low molecular-weight epoxy resins provide a higher density of crosslinks, thereby reducing free volume and segmental mobility, which makes it more difficult for corrosive molecules to penetrate the coating. However, excessive crosslink density can lead to the formation of microcracks and pores in the coating, compromising its protective performance [[52], [55], [56], [57], [58], [59]]. In contrast, high molecular-weight epoxy resins, with their higher hydroxyl functionality, offer improved wetting and adhesion to metal substrates but result in coatings with lower crosslink density. This reduces hardness and chemical resistance while enhancing flexibility and impact resistance. Therefore, optimizing the molecular weight of epoxy resins requires balancing the desired protective properties with mechanical and structural integrity.

Conclusions

In summary, epoxy resin coatings have established themselves as a reliable method of preventing corrosion in a wide range of sectors, such as the infrastructure, marine, and oil and gas industries. They are extremely effective at protecting metal surfaces from the damaging effects of oxygen, water, and salts as the main causes of corrosion due to their exceptional mechanical strength, chemical resistance, and superior adhesion. The performance of epoxy- based coatings has been greatly improved by developments, especially the addition of nanomaterials like carbon nanotubes, graphene, and silica nanoparticles. Even in the harshest conditions, these developments have increased the coatings' lifespan in addition to improving their hydrophobicity and durability. Epoxy resins cross-linked molecular structure adds to their durability by making them resistant to physical and chemical stresses like impact and abrasion. Additionally, a major advancement in water repellence and maintenance ease has been the development of superhydrophobic qualities. The continuous transition to more environmentally friendly formulations, which seek to lessen the environmental impact while preserving peak performance levels, complements this development. The future of protective coatings is being reshaped by epoxy-based anticorrosion coatings, particularly those enhanced with nanocomposites, in a variety of demanding industries, including automotive and aerospace. These coatings provide an economical and environmentally friendly way to combat corrosion, greatly lowering maintenance expenses while boosting the dependability and safety of vital infrastructure. Epoxy coatings are expected to become even more important in maintaining the longevity and integrity of industrial assets for many years to come because of ongoing advances in material science.

Conflicts of interest. Authors declare no conflict of interest.

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Коррозиядан қорғайтын эпоксидті жабындар: шолу

[1,2*]Бекбаева Л.К., [2] Negim E.-S., [2]Жәнібеков Р., [2] Шәріпов Р., [2] Малдыбаев Ғ.,
[3]Пузикова Д.С., [3]Кензин Н.Р., [4]Maridan A.A.B.

[1]Әл-Фараби атындағы ҚазҰУ, Алматы, Қазақстан
[2]Қазақстан-Британ техникалық университеті, Алматы, Қазақстан
[3]Д.В. Сокольский атындағы Жанармай, Катализ және Электрохимия Институты,
Қазақстан-Британ техникалық университеті, Алматы, Қазақстан
[4]PETRONAS технологиялық университеті, 32610 Бандар Сери Искандар, Перак, Малайзия

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ТҮЙІНДЕМЕ

Эпоксидті шайырлар тамаша адгезиясы, механикалық беріктігі және химиялық төзімділігі арқасында коррозияға қарсы қолдануда ең көп пайдаланылатын материалдардың бірі болып табылады. Алайда, дәстүрлі эпоксидті жабындар агрессивті орта жағдайларында ұзақ мерзімді және сенімді қорғауды қамтамасыз етуде айтарлықтай шектеулерге ие. Осыған байланысты, бүкіл әлемде эпоксидті жабындардың коррозияға қарсы қасиеттерін жақсартуға бағытталған ауқымды зерттеулер жүргізілуде. Бұл шолуда осы саладағы соңғы жетістіктер жинақталып, үш негізгі бағытқа бөлінді: эпоксидті шайыр құрылымын өзгерту, функционалды толтырғыштарды қосу және көпфункционалды композиттік жабындарды әзірлеу. Құрылымды өзгерту эпоксидті шайырлардың тосқауылдық әсерін жақсарту үшін олардың ішкі қасиеттерін жетілдіруге бағытталған. Функционалды толтырғыштарды қолдану өзін-өзі қалпына келтіру, супергидрофобтық қасиеттер және коррозияны тежеу сияқты қосымша қорғаныс механизмдерін қамтамасыз етеді. Көпфункционалды композиттік жабындар бірнеше тәсілдердің артықшылықтарын біріктіріп, жоғары тиімділікке қол жеткізу үшін алдыңғы қатарлы материалдар мен технологияларды біріктіреді. Соңғы зерттеулер мен инновацияларды талдау арқылы бұл шолу әрбір тәсілдің артықшылықтары мен кемшіліктерін көрсетіп, жоғары тиімді эпоксидті коррозияға қарсы жабындарды әзірлеудің болашақ бағыттары туралы түсінік береді.

Түйін сөздер: Композиттер, эпоксидті шайыр, коррозияға қарсы жабын, коррозия, коррозияны тежеу, коррозияға қарсы қорғаныс.

	Авторлар туралы ақпарат:
Бекбаева Ляззат Кайратовна	*Ашық Түрдегі Нанотехнологиялық Зертхана, әл-Фараби атындағы ҚазҰУ 71, әл-Фараби даңғылы, 050040, Алматы, Қазақстан. Email: lyazzat_bk2019@mail.ru*
Negim El-Sayed	*Перспективті материалдар мен технологиялар зертханасы, Қазақстан-Британ техникалық университеті, 050000, Төле би көшесі, 59, Алматы, Қазақстан. Email: elashmawi5@yahoo.com*
Ринат Жәнібеков	*Перспективті материалдар мен технологиялар зертханасы, Қазақстан-Британ техникалық университеті, 050000, Төле би көшесі, 59, Алматы, Қазақстан. Email: rin_zhanibekov@kbtu.kz*
Рустам Шәріпов	*Перспективті материалдар мен технологиялар зертханасы, Қазақстан-Британ техникалық университеті, 050000, Төле би көшесі, 59, Алматы, Қазақстан. Email: r.sharipov@kbtu.kz*
Ғалымжан Малдыбаев	*Перспективті материалдар мен технологиялар зертханасы, Қазақстан-Британ техникалық университеті, 050000, Төле би көшесі, 59, Алматы, Қазақстан. Email: g.maldybaev@kbtu.kz*
Пузикова Дарья Сергеевна	*Д.В. Сокольский атындағы Жанармай, Катализ және Электрохимия Институты, Қазақстан-Британ техникалық университеті, 050010, Кунаев көшесі, 142, Алматы, Қазақстан. Email: d.puzikova@ifce.kz*
Кензин Наиль Рашидович	*Д.В. Сокольский атындағы Жанармай, Катализ және Электрохимия Институты, Қазақстан-Британ техникалық университеті, 050010, Кунаев көшесі, 142, Алматы, Қазақстан. Email: n.kenzin@ifce.kz*
Anis Aeshah Binti Maridan	*CO2 зерттеу орталығы (CO2RES), PETRONAS технологиялық университеті, 32610 Бандар Сери Искандар, Перак, Малайзия. Email: ad.aesah@gmail.com*

Эпоксидные покрытия для защиты от коррозии: обзор

[1,2*]Бекбаева Л.К., [2] Negim E.-S., [2]Жанибеков Р., [2] Шарипов Р., [2] Малдыбаев Г.,
[3]Пузикова Д.С., [3]Кензин Н.Р., [4]Maridan A.A.B.

[1]КазНУ им. Аль-Фараби, проспект Аль-Фараби, Алматы, Казахстан
[2] Казахстанско-Британский технический университет, Алматы, Казахстан
[3]АО Институт топлива, катализа и электрохимии им. Д.В. Сокольского,
Казахстанско-Британский технический университет, Алматы, Казахстан
[4]Технологический университет ПЕТРОНАС, 32610 Бандар-Сери-Искандар, Перак, Малайзия

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АННОТАЦИЯ

Эпоксидные смолы являются одними из самых широко используемых материалов для антикоррозионного применения благодаря их отличной адгезии, механической прочности и химической стойкости. Однако традиционные эпоксидные покрытия имеют существенные ограничения в обеспечении долговечной и надежной защиты, особенно в условиях агрессивной окружающей среды. В связи с этим во всем мире ведутся масштабные исследования, направленные на улучшение антикоррозионных свойств эпоксидных покрытий. В данном обзоре обобщены последние достижения в этой области, которые можно разделить на три основные направления: модификация структуры эпоксидной смолы, внедрение функциональных наполнителей и разработка многофункциональных композитных покрытий. Модификация структуры направлена на улучшение внутренних свойств эпоксидных смол для повышения их барьерного эффекта. Использование функциональных наполнителей обеспечивает дополнительные защитные механизмы, включая самовосстановление, супергидрофобность и ингибирование коррозии. Многофункциональные композитные покрытия объединяют преимущества нескольких подходов, интегрируя передовые материалы и технологии для достижения высокой эффективности. Анализируя недавние исследования и инновации, данный обзор подчеркивает сильные стороны каждого подхода, а также дает представление о перспективах разработки высокоэффективных эпоксидных антикоррозионных покрытий.

Ключевые слова: Композиты, эпоксидная смола, антикоррозионное покрытие, коррозия, ингибирование коррозии, антикоррозионная защита.

Информация об авторах:

Бекбаева Ляззат Кайратовна	*Открытая нанотехнологическая лаборатория КазНУ им. Аль-Фараби, проспект Аль-Фараби, 71, 050040, Алматы, Казахстан. Email: lyazzat_bk2019@mail.ru*
Negim El-Sayed	*Лаборатория перспективных материалов и технологий, Казахстанско-Британский технический университет, ул. Толе би, 59, 050000, Алматы, Казахстан. Email: elashmawi5@yahoo.com*
Ринат Жәнібеков	*Факультет материаловедения и зеленых технологий, Казахстанско-Британский технический университет, ул. Толе би, 59, 050000. Алматы, Казахстан. Email: rin_zhanibekov@kbtu.kz*
Рустам Шарипов	*Лаборатория перспективных материалов и технологий, Казахстанско-Британский технический университет, ул. Толе би, 59, 050000, Алматы, Казахстан. Email: r.sharipov@kbtu.kz*
Галымжан Малдыбаев	*Лаборатория перспективных материалов и технологий, Казахстанско-Британский технический университет, ул. Толе би, 59, 050000, Алматы, Казахстан. Email: g.maldybaev@kbtu.kz*
Пузикова Дарья Сергеевна	*АО Институт топлива, катализа и электрохимии им. Д.В. Сокольского, Казахстанско-Британский технический университет, ул. Кунаева, 142, 050010, Алматы, Казахстан. Email: d.puzikova@ifce.kz*
Кензин Наиль Рашидович	*АО Институт топлива, катализа и электрохимии им. Д.В. Сокольского, Казахстанско-Британский технический университет, ул. Кунаева, 142, 050010, Алматы, Казахстан. Email: n.kenzin@ifce.kz*
Anis Aeshah Binti Maridan	*Центр исследований CO2 имени (CO2RES), Технологический университет ПЕТРОНАС, 32610 Бандар-Сери-Искандар, Перак, Малайзия. Email: ad.aesah@gmail.com*

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Earth sciences

Influence of Radiation and Magnetic Pulse Treatment on The Wear Resistance of Carbide Tools

[1] Toshov J.B., [2*] Rabatuly M., [2] Bogzhanova Zh.K., [2] Zheldikbayeva A.T.,
[3] Malikov Sh.R., [4] Toshov B.R., [1] Ergashev O.S.

[1] Tashkent state technical university, Tashkent, Uzbekistan
[2] Abylkas Saginov Karaganda Technical University, Karaganda, Kazakhstan
[3] Institute of Nuclear Physics of the Academy of Sciences, Tashkent, Uzbekistan
[4] Navoi state mining and technology university, Navoi, Uzbekistan

** Corresponding author email: mukhammedrakhym@mail.ru*

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ABSTRACT
In the mining industry, hard alloy tools with high wear resistance are essential for drilling operations. This study introduces a combined magnetic-pulse treatment method, integrating preliminary gamma irradiation and pulsed magnetic field exposure, to extend the service life of VK8 hard alloy drilling tools. Gamma irradiation utilized ^{60}Co sources with doses from $3.2×10^4$ to $5.0×10^8$ R, followed by magnetic-pulse treatment using a custom installation with electromagnetic coils, achieving magnetic induction levels of 0.2–0.4 Tesla and pulse durations of 3 µs. The VK8 alloy, comprising 8% cobalt and 92% tungsten carbide, was tested on DZL Ø118 mm blade bits across ten batches. Results showed a 1.7–3.2-fold increase in wear resistance, influenced by treatment parameters, alloy composition, and operating conditions. The hardening effect persisted for 5–6 months after gamma irradiation and over a year after magnetic-pulse treatment. This method offers significant potential to enhance tool performance and durability in rock-destroying equipment.

Keywords: drilling tools, drilling, gamma radiation, magnetic pulse hardening, carbide.

	Information about authors:
Toshov Javokhir Buriewicz	*Doctor of Technical Sciences, Professor of Islam Karim Tashkent State Technical University, 100095 Republic of Uzbekistan, Tashkent, Almazar district, Universitetskaya street 2. E-mail: j.toshov@tdtu.uz; ORCID ID: https://orcid.org/0000-0003-4278-1557*
Rabatuly Mukhammedrakhym	*Ph.D., Acting Associate Professor of the Department of Development of Mineral Deposits of Abylkas Saginov Karaganda Technical University, 100027, The Republic of Kazakhstan, Karaganda, Ave. Nursultan Nazarbayev, 56. E-mail: mukhammedrakhym@mail.ru; ORCID ID: https://orcid.org/0000-0002-7558-128X*
Bogzhanova Zhanbota Kizatovna	*Master of Technical Sciences, Senior Lecturer of the Department of Development of Mineral Deposits of Abylkas Saginov Karaganda Technical University, 100027, The Republic of Kazakhstan, Karaganda, Ave. Nursultan Nazarbayev, 56. E-mail: botikum@mail.ru; https://orcid.org/0000-0001-8713-4981*
Zheldikbayeva Aisaule Takenovna	*PhD student of the department of the Department of Automation of manufacturing processes of Abylkas Saginov Karaganda Technical University, 100027, The Republic of Kazakhstan, Karaganda, Ave. Nursultan Nazarbayev, 56. E-mail: aisaule89@mail.ru; ORCID ID: https://orcid.org/0009-0005-1325-5576*
Malikov Shavkat Rustamovich	*Candidate of Physical and Mathematical Sciences, Associate Professor, Researcher of the Institute of Nuclear Physics Academy of Sciences of the Republic of Uzbekistan, Tashkent, 100214, st. Khurasan 1. E-mail: malikov@inp.uz; ORCID ID: https://orcid.org/0000-0001-6561-5362*
Toshev Buri Radjabovich	*Candidate of Physical and Mathematical Sciences, Professor, Head of the Department of Engineering Mechanics Navoi State University of Mining and Technology 210100 Republic of Uzbekistan, Navoi, Galaba St 27. E-mail: toshov b@mail.ru; ORCID ID: https://orcid.org/0009-0006-4058-1702*
Ergashev Otabek Sanokulovich	*PhD student of the department Mining electro mechanics of the Islam Karim Tashkent State Technical University, 100095 Republic of Uzbekistan, Tashkent, Almazar district, Universitetskaya street 2. E-mail: Otabekergashev1979@yandex.ru; ORCID ID: https://orcid.org/0000-0003-3628-7958*

Introduction

In the mining industry, a variety of hard alloy tools with high wear resistance are extensively used for drilling operations. The efficiency of drilling wells or boreholes, particularly in hard and abrasive rock formations, is largely determined by the operational characteristics of these hard alloy tools. Their service life is significantly influenced by design features, drilling parameters, physical and mechanical properties of the rocks being drilled and by the used type and composition of the hard alloy.

Enhancing the wear resistance of hard alloy tools can be achieved by creating new or modifying existing composite materials. Various methods are used to increase the wear resistance of hard alloy tools, including nitriding, boriding, electro-spark alloying, plasma spraying, rolling, shot peening, isothermal quenching, thermomechanical treatment, and others.

Strengthening of hard alloys is achieved by using the methods of surface and bulk strengthening. However, numerous methods of surface strengthening of metals and hard alloys were not widely used for strengthening hard alloy tools [[1], [2], [3]]. While the positive effect of hardening the surface layer of the working elements of hard alloy tools is undeniable, the efficiency of surface hardening is low, making its practical application impractical.

Some last studies [4] have shown that results in the cryogenic treatment of VK8 hard alloy samples (a mixture of tungsten carbide grains and cobalt, acting as a binder) with a dislocation density 4.8×109 cm-2 results in the during a short-term (20 minutes) strengthening of these samples occurs due to plastic deformation.

Recently researches [[5], [6]] were observed an increase in wear resistance during the investigation of strengthening of TSI (presumably a material or alloy) through prolonged (24 to 36 hours) cryogenic treatment in liquid nitrogen vapors. The hardening effect during thermal shock (T=-196 °C) is explained by changes in the fine crystalline structure of the hard alloy. However, immersing the products in liquid nitrogen significantly increases the brittleness of the metal, since under the cold shock, tensile residual thermal stresses arise in the body of the hard alloy tools, leading in many cases to cracking and tool breakage [[7], [8]].

The modification mechanism of the physical and mechanical properties of drilling crowns during cryogenic treatment is primarily based on substructural hardening due to plastic deformation of the cobalt binder, owing to the significant difference in the thermal coefficient of linear expansion between tungsten carbide and cobalt and due to an increase in dislocation density in the hard alloy [9]. High-temperature treatment of the manufacturing cemented carbides can also significantly alter the properties of hard alloys, since it considerably increases the internal stress in the carbide [10]. For instance, as the temperature exceeds 500 °C, a decrease in the hardness of the VK8 alloy accompanied by an increase in the alloy's ductility and a change in its resistance to cyclic micro-contact loads. Therefore, it is more prudent to perform the strengthening of the finished tool, rather than its individual working elements before the product is manufactured.

The literature analysis has revealed that radiation treatment using gamma rays, which possess high penetrating ability and do not lead to residual radioactivity, is a more promising method [[11], [12]]. When hard alloys are irradiated, the strengthening of the material's structure is determined by the absorbed dose of ionizing radiation. Research results on the effects of gamma irradiation and electron irradiation, as reported in [[12], [13]], showed that with increasing the doses of irradiation, the bending strength limit and deformation increase up to the irradiation dose range from 8×104 - 5×105 R, after which a sharp decrease is observed [13]. The increase in defect density, especially dislocations, according to general views on the nature of substructural strengthening in metals and alloys, leads to changes in their physico-mechanical characteristics. This results in the increase of the wear resistance and strength properties of the material, but at the same time, an excessively high defect density leads to the increasing brittleness, stiffness of products, and consequently, to the decreasing their operational indicators, primarily durability and reliability.

The method of magnetic-pulse treatment is the most effective way for bulk strengthening of cemented carbide tools. The feasibility of using magnetic-pulse treatment for cemented carbide tools is due to the presence of cobalt in the tool composition – a ferromagnetic material with high magnetic permeability. The improvement in the properties of ferromagnetic materials that have undergone magnetic-pulse treatment is achieved through the directed orientation of the free electrons of the hard alloy by an external field, which consequently increases its thermal and electrical conductivity. The interaction between the pulsed magnetic field and the ferromagnetic material is more intense with higher structural and energetic heterogeneity. After processing cemented carbide tools with this method, an increase in their fatigue strength and overall strength, and a reduction in residual thermal stress occur [14]. Utilizing magnetic-pulse treatment notably diminishes the surplus energy in the material, which is linked to the accumulation of internal and surface stresses. Consequently, it was decided to combine two methods of enhancing the wear resistance of cemented carbide materials: radiation and magnetic-pulse treatments.

The goal of this work is to develop a method and optimal regime for the bulk strengthening of cemented carbide tools through the combined application of penetrating gamma radiation and pulsed magnetic field.

Experimental part

For gamma radiation exposure of the samples, a pool-type Gamma Facility of the institute Nuclear Physics, Uzbek Academy of Science (INP UzAS) was used (Figures 1). In so doing, we have used the ^{60}Co sources with average gamma quantum energy of 1.25 MeV and a dose rate of 130 R/s, with an exposure dose ranging from 3.2×10^4 to 5.0×10^8 R. The choice of gamma irradiation is determined by its technological efficiency and high penetrating ability.

Figure 1 - Pool-type Gamma Facility [14]

For the implementation of magnetic-pulse treatment (MPT), a magnetic-pulse facility was constructed (see Figures 2 and 3), featuring a magnetic inductor 2, consisting of two pairs of electromagnetic coils, and a magnetic core 1 on which the electromagnetic coils are mounted.

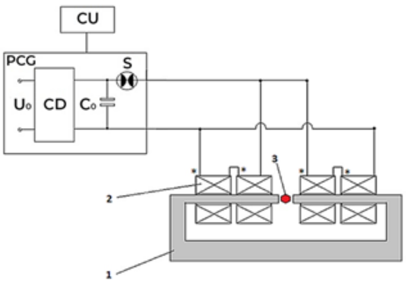

Figure 2 - Block Diagram of the Magnetic-Pulse Installation:
1 - Magnetic core; 2 - Solenoid inductors; 3 - Drill bit; CU - Control unit; PCG - Pulse current generator; CD - Charging device; S - Switching device; C0 - Capacitor bank; U0 - Mains voltage [14]

This design allows carrying out the treatment of entire drill bits and chisels with a pulsed magnetic field, which is a necessary condition for the method of enhancing the wear resistance of finished cemented carbide tools.

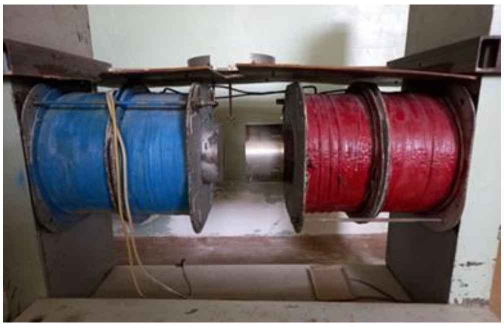

Figure 3 - Magnetic Field Inductors Mounted on a Magnetic Core

The use of cylindrical inductors with a magnetic core is associated with the fact that such a configuration possesses high efficiency and a high-power factor, with the efficiency of an inductor with a magnetic core reaching at least 70-80 %. During magnetic-pulse hardening, as the pulsed electric current flows through the inductor, the magnetic field lines through the magnetic core are concentrated on the hardened sample (drilling tool), which is positioned in the gap of the magnetic core. This induces an eddy current on the surface of the sample, proportional to the changing rate of the magnetic flux through the cross-sectional area of the work piece.

If we take the current strength $I_1 = 10^3$ A, with a coil having N = 10 turns and a length of $l_0 = 0.136$ m, and coil wire thickness a = 0.01 m, we do the calculation using formula

$$H_{1b} = NI_1/l, \qquad (1)$$

where N - the number of turns of the inductor coil; I_1 – the strength of the discharge current in the inductor; l - length of the contour, for points located within the inductor in immediate proximity to the turns (length $l=l_0+2a=0{,}156$ m) yields a value for the modulus of the magnetic field strength denoted as H1b, equal to 6.41×10^4 A/m. To study the effect of the magnetic field on the hardening of solid alloys, the treatment of samples of drill bits was conducted using a magnetic-impulse approach with a magnetic induction of 0.2-0.4 tesla and pulse durations of 3 ms. The magnetic-pulse installation allows us to apply the fields with intensities ranging from 10^4 to 10^{11} A/m to the parts being processed, with a pulse duration from 1 to 0.1 ms.

In experiments, the technology of volumetric hardening of rock-destroying tools was applied, using a combination of two types of physical effects: gamma-ray irradiation and treatment in a pulsed magnetic field.

Industrial samples of three-blade drill bits DZLØ118mm, used in the mining enterprises of the Republic of Uzbekistan, were used as test objects. For the rock-destroying assembly of drilling bits, the hard alloy material VK8, which is characterized by strength and wear resistance [15] with an average hardness of 88 HRA (Rockwell), is primarily used. The mentioned hard alloy VK8, used for DZL Ø118mm drill bits, is a composite material belonging to the tungsten-cobalt group with a composition of 8 % cobalt and 92 % tungsten carbide. The chemical composition of the tungsten-cobalt mixture VK8 [16] (mass percentage, %) is: cobalt - 7.5-8.1, oxygen, not more than – 0.5, total carbon - 5.30-5.65, free carbon, not more than – 0.1, iron-0.3. By altering the chemical composition ratio of the alloy, its physic-mechanical properties are regulated. The increasing of the cobalt content in the alloy leads to an enhancement in strength, wear resistance, and a reduction in brittleness (the more cobalt in the alloy, the softer and stronger it becomes). The microstructure of the VK8 alloy is two-phase, consisting of tungsten carbide and cobalt crystals with an uneven distribution throughout the volume of the hard alloy.

The samples were subjected to preliminary treatment with gamma radiation of ^{60}Co in a dose range from $3 \cdot 10^4$ to $9 \cdot 10^6$ R and subsequent exposure to magnetic induction of 0.4 Tesla. Ten batches of nine DZLØ118mm blade drill bits each were processed for the purpose of testing nine batches during drilling, and one batch is used to determine the interval and rate of reduction in strength limit during storage.

Discussion of the results

The measurement of hardness using the Rockwell scale (HRA) showed us an increase in the samples with an increase in the dose of gamma-ray irradiation within the range of $2.5 \cdot 10^4$ to $5.5 \cdot 10^5$ R, and a decrease with a further increase in the irradiation dose.

It was found that processing samples in a magnetic-pulse field increases wear resistance by an average of two times compared to samples without magnetic-pulse processing, which is in good agreement with research data in recent studies [[17], [18]].

The results of drilling with hard alloy bits with VK8 plates on the surface chisels after combined gamma magnetic treatment are presented in Table 1 and Figure 4.

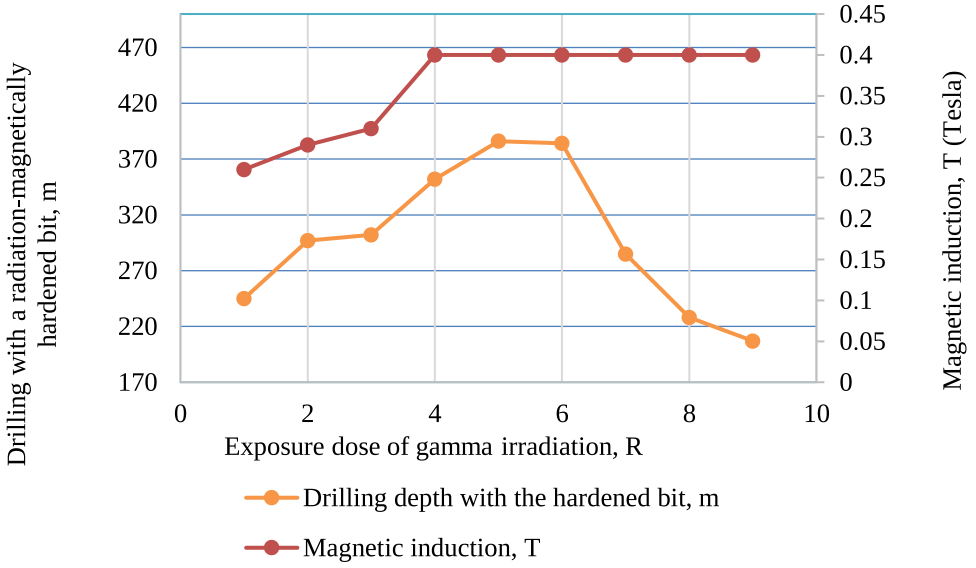

Figure 4 - Test results of drill bits hardened by gamma irradiation and magnetic-pulse treatment

Table 1 - Main quality criteria for core samples

Serial №.	Gamma Radiation Exposure Dose, R	Magnetic Induction, T	Average Penetration per Unhardened Bit, m	Penetration per Bit with Radiation-Magnetic Hardening, m	Increase in Penetration, % / times
1	$3.2 \cdot 10^4$	0.26	120	245	104 / 2
2	$6.5 \cdot 10^4$	0.29	120	297	148 / 2,4
3	$8.9 \cdot 10^4$	0.31	120	302	152 / 2,5
4	$2.4 \cdot 10^5$	0.4	120	352	193 / 2,9
5	$5.5 \cdot 10^5$	0.4	120	386	222 / 2,7
6	$5.6 \cdot 10^5$	0.4	120	384	284 / 3,2
7	$8.9 \cdot 10^5$	0.4	120	285	185 / 2,4
8	$2.2 \cdot 10^6$	0.4	120	228	90 / 1,9
9	$8.9 \cdot 10^6$	0.4	120	207	72.5 / 1.7

The analysis of the data obtained suggests that the use of combined processing of three-blade DZL bits leads to an increase in their durability. Treatment of hard alloy tools within a radiation-magnetic-impulse environment yields a substantial enhancement of the wear resistance, ranging from 1.7 to 3.2 times when contrasted with the untreated counterparts. The increase in the hardening degree of the samples from the absorbed dose of ^{60}Co gamma radiation under the combined effect of the magnetic field has a non-monotonic character (Table 1). From the results of the drilling tests using three-blade DZL drill bits, it is evident that combined radiation and magnetic-pulse treatment significantly enhances the resource of hard alloy tools, and the hardening effect is determined by the dose of gamma irradiation, reaching its maximum value within the absorbed dose range from 2.5×10^4 to 5.5×10^5 R. To monitor the reduction in the hardening magnitude during storage, hardness measurements were conducted after irradiation and after the combined radiation and magnetic-pulse treatment of the samples. [[19], [20]]. It was found that the hardening effect persists after gamma irradiation during 5-6 months and in the case of combined treatment for this effect persists during about 1 year.

Conclusion

A new method of combined magnetic-pulse treatment has been proposed, incorporating preliminary gamma irradiation and the influence of a pulsed magnetic field. This method allows to enhance the service life of drilling tools made from various types of hard alloys.

Using the gamma irradiation of ^{60} Co on hard alloys employed in DZL drilling bits results in the enhancement of the strength of these alloys within the absorbed dose interval ranging from 2.5×10^4 to 5.5×10^5 R.

The degree of hardening of hard alloys for drilling bits is determined by the dose of gamma irradiation and reaches its maximum within the absorbed dose range from 2.5×10^4 to 5.5×10^5 R. Exceeding this dose range results in a decrease in strength, wear resistance, and an increase in the brittleness of hard alloy tools.

Gamma irradiation by using gamma quanta of ^{60}Co within the absorbed dose range from 2.5×10^4 to 5.5×10^5 R, followed by magnetic-pulse treatment with a magnetic induction of 0.2-0.4 Tesla and pulse durations of 3 µs, would increase wear resistance by 1.7 to 3.2 times, depending on the treatment regime, the composition of the hard alloy, and operating conditions.

The hardening effect is retained during 5–6 months after gamma irradiation, and during about 1 year after magnetic-pulse treatment.

Conflict of interest. On behalf of all the authors, the correspondent author declares that there is no conflict of interest.

CRediT author statement: **J. Toshov, M. Rabatuly:** Conceptualization, Methodology, Software; Zh**. Bogzhanova, A. Zheldikbayeva**: Data curation, Writing- Original draft preparation; **J. Malikov, B. Toshov:** Visualization, Investigation; **O. Ergashev**: Software, Validation.

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Қатты қорытпалы құралдардың тозуға төзімділігіне радиациялық және магниттік импульстік өңдеудің әсері

[1] Тошов Ж.Б., [2] Рабатұлы М., [2] Богжанова Ж.К., [2] Желдикбаева А.Т.,
[3] Маликов Ж.М., [4] Тошов Б.Р., [1] Эргашев О.С.

[1] Ислам Кәрім атындағы Ташкент мемлекеттік техникалық университеті, Ташкент, Өзбекстан
[2]Ә. Сағынов атындағы Қарағанды техникалық университеті, Қарағанды, Қазақстан
[3] Ғылым академиясының ядролық физика институты, Ташкент, Өзбекстан
[4] Навои мемлекеттік тау-кен технологиялық университеті, Навои, Өзбекстан

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Қабылданды: *17 ақпан 2025*

ТҮЙІНДЕМЕ

Кен өндіру өнеркәсібінде жоғары тозуға төзімді қатты қорытпалардан жасалған құралдар бұрғылау операциялары үшін маңызды болып табылады. Бұл зерттеу VK8 қатты қорытпасы негізінде бұрғылау құралдарының жұмыс мерзімін ұлғайту мақсатында гамма-сәулелендіру мен импульстік магниттік өріс әсерін біріктіретін магнитті-импульстік өңдеу әдісін ұсынады. Гамма- сәулелендіру ^{60}Co көздерін пайдалана отырып, мөлшерлері $3{,}2\times10^4$-ден $5{,}0\times10^8$ R дейін болды, одан кейін магнитті-импульстік өңдеу арнайы орнатылған электромагниттік катушкалармен жүзеге асырылып, магниттік индукция деңгейі 0,2–0,4 Тесла және импульс ұзақтығы 3 мкс болды. VK8 қорытпасы, құрамында 8% кобальт және 92% вольфрам карбиді бар, DZL Ø118 мм ұстаралы бұрғыларда он партияда сыналды. Нәтижелер өңдеу параметрлері, қорытпа құрамы және жұмыс жағдайларының әсерінен тозуға төзімділіктің 1,7–3,2 есеге артатынын көрсетті. Қатыру әсері гамма- сәулелендіруден кейін 5–6 айға дейін және магнитті-импульстік өңдеуден кейін бір жылдан астам уақыт бойы сақталды. Бұл әдіс тау-кен құралдарының жұмыс көрсеткіштері мен ұзақ мерзімділігін арттыру үшін үлкен әлеуетке ие.

Түйін сөздер: бұрғылау құралдары, бұрғылау, гамма-сәулелену, магниттік импульстің қатаюы, карбид.

Авторлар туралы ақпарат:

Тошов Жавохир Буриевич	*Техника ғылымдарының докторы, Ислам Кәрім атындағы Ташкент мемлекеттік техникалық университетінің профессоры, 100095, Алмазар ауданы Университетская көшесі 2, Ташкент, Өзбекстан. E-mail: j.toshov@tdtu.uz; ORCID ID: https://orcid.org/0000-0003-4278-1557*
Рабатұлы Мұхаммедрахым	*PhD докторы, Әбілқас Сағынов атындағы Қарағанды техникалық университетінің Пайдалы қазбалар кенорындарын өндіру кафедрасының доцент м.а., 100027, Нұрсұлтан Назарбаев даңғ. 56, Қарағанды, Қазақстан. E-mail: mukhammedrakhym@mail.ru; ORCID ID: https://orcid.org/0000-0002-7558-128X*
Богжанова Жанбота Кизатовна	*Техника ғылымдарының магистрі, Әбілқас Сағынов атындағы Қарағанды техникалық университетінің Пайдалы қазбалар кенорындарын өндіру кафедрасының аға оқытушысы, 100027, Нұрсұлтан Назарбаев даңғ. 56, Қарағанды, Қазақстан. E-mail: botikum@mail.ru; https://orcid.org/0000-0001-8713-4981*
Желдикбаева Айсауле Такеновна	*Әбілқас Сағынов атындағы Қарағанды техникалық университетінің Өндірістік процестерді автоматтандыру кафедрасының PhD докторанты, 100027, Нұрсұлтан Назарбаев даңғ. 56, Қарағанды, Қазақстан. E-mail: aisaule89@mail.ru; ORCID ID: https://orcid.org/0009-0005-1325-5576*
Маликов Шавкат Рустамович	*Физика-математика ғылымдарының кандидаты, Ғылым Академиясының ядролық физика институтының доценті, 100214, ст. Хорасан 1, Ташкент, Өзбекстан. E-mail: malikov@inp.uz; ORCID ID: https://orcid.org/0000-0001-6561-5362*
Тошов Бури Раджабович	*Физика-математика ғылымдарының кандидаты, профессор, Инженерлік Механика кафедрасының меңгерушісі, Навои мемлекеттік тау-кен технологиялық университеті, 210100, Галаб көшесі, 27, Навои, Өзбекстан. E-mail: toshov b@mail.ru; ORCID ID: https://orcid.org/0009-0006-4058-1702*
Эргашев Отабек Санакулович	*Ислам Кәрім атындағы Ташкент мемлекеттік техникалық университетінің Тау-кен электромеханикасы кафедрасының PhD докторанты, 100095, Алмазар ауданы Университетская көшесі 2, Ташкент, Өзбекстан. E-mail: Otabekergashev1979@yandex.ru; ORCID ID: https://orcid.org/0000-0003-3628-7958*

Влияние радиационной и магнитно-импульсной обработки на износостойкость твердосплавных инструментов

[1] Тошов Ж.Б., [2] Рабатулы М., [2] Богжанова Ж.К., [2] Желдикбаева А.Т.,
[3] Маликов Ж.М., [4] Тошов Б.Р., [1] Эргашев О.С.

[1] Ташкентский государственный технический университет имени Ислама Карима, Ташкент, Узбекистан
[2]Карагандинский технический университет имени А. Сагинова, Караганда, Казахстан
[3] Институт ядерной физики академии наук, Ташкент, Узбекистан
[4] Навойский государственный горно-технологический университет, Навои, Узбекистан

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АННОТАЦИЯ
В горной промышленности инструменты из твердых сплавов с высокой износостойкостью необходимы для буровых операций. В данном исследовании представлен комбинированный метод магнитно-импульсной обработки, интегрирующий предварительное, гамма-облучение и воздействие импульсного магнитного поля, с целью увеличения срока службы буровых инструментов из твердого сплава VK8. Для гамма-облучения использовались источники ^{60}Co с дозами от 3,2×10^4 до 5,0×10^8 R, после чего проводилась магнитно-импульсная обработка с использованием специализированной установки с электромагнитными катушками, обеспечивающими уровни магнитной индукции 0,2–0,4 Тесла и длительность импульсов 3 мкс. Сплав VK8, состоящий из 8% кобальта и 92% вольфрамового карбида, испытывался на лезвийных сверлах DZL диаметром 118 мм в десяти партиях. Результаты показали увеличение износостойкости в 1,7–3,2 раза, что зависело от параметров обработки, состава сплава и условий эксплуатации. Эффект упрочнения сохранялся в течение 5–6 месяцев после гамма-облучения и более года после магнитно-импульсной обработки. Этот метод представляет собой значительный потенциал для повышения эксплуатационных характеристик и долговечности инструмента в горнодобывающем оборудовании.

Ключевые слова: буровые инструменты, бурение, гамма-излучение, магнитно-импульсное упрочнение, карбид.

Информация об авторах:

Тошов Жавохир Буриевич	*Доктор технических наук, профессор Ташкентского государственного технического университета имени Ислама Карима, 100095, Алмазарский р-н, ул. Университетская 2, Ташкент, Узбекистан. E-mail: j.toshov@tdtu.uz; ORCID ID: https://orcid.org/0000-0003-4278-1557*
Рабатулы Мухаммедрахым	*Доктор PhD, и.о. доцента кафедры Разработки месторождений полезных ископаемых Карагандинского технического университета имени Абылкаса Сагинова, 100027, пр. Нурсултана Назарбаева, 56, Караганда, Казахстан. E-mail: mukhammedrakhym@mail.ru; ORCID ID: https://orcid.org/0000-0002-7558-128X*
Богжанова Жанбота Кизатовна	*Доктор PhD, и.о. доцента кафедры Разработки месторождений полезных ископаемых Карагандинского технического университета имени Абылкаса Сагинова, 100027, пр. Нурсултана Назарбаева, 56, Караганда, Казахстан. -mail: botikum@mail.ru; https://orcid.org/0000-0001-8713-4981*
Желдикбаева Айсауле Такеновна	*PhD докторант кафедры Автоматизации производственных процессов Карагандинского технического университета имени Абылкаса Сагинова, 100027, пр. Нурсултана Назарбаева, 56, Караганда, Казахстан. E-mail: aisaule89@mail.ru; ORCID ID: https://orcid.org/0009-0005-1325-5576*
Маликов Шавкат Рустамович	*Кандидат физико-математических наук, доцент Института ядерной физики Академии наук, 100214, ст. Хурасан 1, Ташкент, Узбекистан. E-mail: malikov@inp.uz; ORCID ID: https://orcid.org/0000-0001-6561-5362*
Тошов Бури Раджабович	*Кандидат физико-математических наук, Профессор, заведующий кафедрой Инженерная Механика Навойский государственный горно-технологический университет, 210100, улица Галаба, 27, Навои, Узбекистан. E-mail: toshov b@mail.ru; ORCID ID: https:// orcid.org/0009-0006-4058-1702*
Эргашев Отабек Санакулович	*PhD докторант кафедры Горная электромеханика Ташкентского государственного технического университета Ислам Каримов, 100095, Алмазарский р-н, ул. Университетская, 2, Ташкент, Узбекистан. E-mail: Otabekergashev1979@yandex.ru; ORCID ID: https://orcid.org/0000-0003-3628-7958*

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Land Suitability for Wind Farm Development in Pandeglang Regency, Banten Province, Indonesia

[1] ***Suci F.R.Z.***, [2] ***Muhamad I.B.***

[1]College of Industrial Technology STTIND Padang, West Sumatra, Indonesia
[2]State University of Padang, Indonesia

** Corresponding author email: sucifitria1228@gmail.com*

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ABSTRACT

The use of wind as the main energy in power plants cannot be separated from the ability of windmills to produce energy to meet needs. Based on a literature study, the Ministry of Energy and Mineral Resources 2018 released data related to the total potential of wind power in Banten Province of 300 MW spread across two areas, namely Lebak Regency and Pandeglang Regency with the potential in each area of 150 MW, thus it is necessary to conduct a spatial study related to the appropriate location for the construction of a new wind farm. Topographic factors (elevation, viewshed, slope, aspect direction, and area), technical (wind speed and power grid proximity), environmental (distance from lakes, rivers, reservoirs, dams, and land cover), and socioeconomic (settlement and transportation) are parameters that determine the success and potential of placing windmills as environmentally friendly renewable energy. This study aimed to determine the potential for wind farm development areas in Pandeglang Regency, Banten Province. The method used to analyze these parameters is the Multi-Weighted Criteria Modeler, by giving weight to each parameter in raster format and classified using the deterministic logic method and selecting areas with a large area coverage (≥ 10,000 m^2) using boolean. The results of this study indicate that there is an area of 350.71 km^2 that is suitable for the construction of a new wind farm in Pandeglang Regency. The final suitability map can be used as a guide for more detailed wind farm location exploration. This study concludes that Indonesia has great potential for the development of Wind Power Plants, especially in Pandeglang Regency. However, a wider research area coverage is needed to find out which areas have the potential for the development of Wind Power Plants in Indonesia.

Keywords: Renewable Energy, Wind Farm, Wind Power Plant.

Information about authors:

Suci Fitria Rahmadhani Z
Master in Geological Engineering, Padjadjaran University, Bandung City, West Java Province; Lecturer of Mining Engineering Study Program at the College of Industrial Technology (STTIND) Padang City. West Sumatra. Email: sucifitria1228@gmail.com; ORCID ID: https://orcid.org/0000-0003-0714-3672

Muhamad Ikhwan Bagus
Bachelor of Geography Study Program at State University of Padang, West Sumatera. Email: Muhamadikhwanbagus@gmail.com; ORCID ID: https://orcid.org/0009-0001-0361-1072

Introduction

When conditions are limited in energy resources, at the same time the need for world energy also increases, as well as demands to be able to maintain and protect the earth from global warming events and environmental pollution. So humans are required to be able to realize the latest technology for renewable energy sources. Fossil energy sources have contributed 87.7% of all the world's total energy needs which are expected to decrease due to reduced availability of reserve energy sources [1].

Wind Power Plant is a series of generating systems that utilize wind as a source that will be converted into electrical energy, wind power is an alternative energy source that has great potential to be utilized and clean energy that does not damage the environment [2]. The use of wind as an energy source can be carried out in areas with any landscape with high wind energy potential, but it needs to be identified so that it is more optimal [3]. Wind Power Plants are the main choice as an energy source for areas with good wind potential [4].

The use of wind as an energy source has been used by the Netherlands first and is referred to as the country of wind turbines. Starting in the 1970s the use of fossil energy has begun to be reduced because of the impact it has on the environment. then it is replaced by energy sources that are friendlier to the environment and are abundantly available. Entering the 21st century the use of wind energy is increasingly being followed by developing countries with increasing capacities [5].

Wind is air that moves due to high pressure to low pressure or from low temperature to high temperature caused by radiation from the sun. The conditions of the earth's regions that are not the same cause differences in pressure and temperature in each region and cause an air flow [6].

As an archipelago that has a long coastline, Indonesia is also a country that has great wind energy potential. Based on the analysis of wind energy potential and mapping of wind energy potential that has been carried out, areas with considerable potential include Sukabumi with 170 MW, Garut with 150 MW, Lebak with 150 MW, Pandeglang with 150 MW and Lombok with 100 MW. The development and utilization of new renewable energy including wind energy as the backbone of national energy will continue to be pursued by the government in order to achieve the national energy mix target of 23% derived from renewable energy in 2025 [7].

Currently, Indonesia already has several Wind Power Plants, including those on the island of South Sulawesi, namely the Sidrap Wind Power Plants and the Jeneponto Wind Power Plants. According to the Ministry of Energy and Mineral Resources, apart from the Sidrap 1 Wind Power Plants, which will soon be put into operation, the Sidrap Phase 2 Wind Power Plants will soon be operational, then the Jeneponto Wind Power Plant in Jeneponto Regency, South Sulawesi Province and the Tanah Laut Wind Power Plants which is located in Tanah Laut Regency, South Kalimantan Province [7].

Based on the data above, there are 4 Wind Power Plants that are already operating and which will operate in Indonesia in stages. This is also a serious step for Indonesia in transitioning the use of fossil energy to renewable energy, especially in the utilization of wind potential which is quite potential in Indonesia [7].

Wind power generation is a renewable energy that is more environmentally friendly and has good work efficiency [8]. Wind energy offers several advantages such as providing continuous electrical energy from morning to night, does not produce greenhouse gas emissions, this energy system can be used in remote areas, and is not covered by the State Electricity Company's electricity grid [9].

The potential for the development of renewable energy sources depends on the geographical position and existing infrastructure [10]. Knowledge of the right location for optimal utilization of energy sources is very important. But in fact, determining the location to generate wind power is not easy. This determination requires in-depth research to get the right location [11].

Optimization of the development of wind power energy in Indonesia is still relatively low. Academics and the government are expected to be able to make a major contribution in supporting this renewable energy as a source in the development of electrical energy. Until 2004, the utilization of wind energy only reached 0.5 MW installed from the total existing potential [12].

Wind turbines as a source of electrical energy are expected to be able to work and operate continuously so that the wind supply can always be available. In order to obtain sufficient wind energy, research activities are needed to monitor wind availability. The monitoring process requires effort and data analysis. [13]. Monitoring and mapping of potential wind energy areas is carried out so that energy utilization can occur optimally. The selection of suitable locations for wind power plants is a complex problem and requires careful analysis of many criteria. Making a land suitability map is useful in order to show the exact location and description of the area where the Wind Power Plant will be established [14].

Experimental part

This type of research is quantitative research. Source of data used in the form of secondary data obtained from agencies, libraries, archives, and individuals. The secondary data used are elevation, viewshed, slope, aspect direction, wind speed, shapefiles of lakes, rivers, reservoirs or dams, land cover, settlements, and transportation. The methods used in this research are Multi - Weighted Criteria Method Modeler and Boolean logic.

Multi - Weighted Criteria Method Modeler is a tool used for decision-making with various spatial parameters to be combined [15]. The analysis will be carried out using the Multi - Weighted Criteria method the modeler uses several scripts. Decision making is carried out using Boolean logic which will be applied to the factors used in the analysis of wind power development based on the research of Idrizi. B, et.al in 2018 which will be explained further in the research results.

Boolean logic is a form of algebra whose variable values are true and false values, usually denoted by the numbers 1 and 0 for each parameter, simplifying the parameters of the boolean being able to search in other functions with fewer operations or terms [16]. The Boolean method is used in this study to select areas in Pandeglang Regency for the suitability of PLTB land with a large area coverage (> 100.000 m^2) as well as other factors as determinants in wind farm development. In its application to the Multi-Weighted Criteria method, areas with factors and criteria that are met will be denoted by the number 1, and areas that are not met will be denoted by the number 0.

Geographic Area Pandeglang Regency is geographically located between $6^0$21'-$7^0$10' South Latitude and $104^0$48' - $106^0$11' East Longitude with a total outer area of around 2.746,89 km^2 or 28.43% of the total area of Banten Province. In the northern part of this area is bordered by Serang Regency and in the east by Lebak Regency. Astronomically, the Pandeglang region is a district located in Banten Province. This regency is bordered on the north by Serang Regency, on the east by Lebak Regency and by the Indian Ocean on the west and south [17].

Pandeglang Regency consists of 35 sub-districts and 339 sub-districts/villages with additional villages, namely Bojenwetan, Ganggaeng, Simpang Tiga and Ramaya. The topography of the central and southern parts of Pandeglang Regency is dominated by highlands with hills that are not too high, namely around 320 - 480 meters. The area of the hills comprises around 85% of the total area of Pandeglang [17].

The Geology and Geomorphology Area of the research was conducted in the administrative area of Pandeglang Regency. In the northern part of this area is bordered by Serang Regency and in the east by Lebak Regency. Geologically Pandeglang Regency is included in the Bogor area zone which consists of hilly paths. Most of this area forms lowlands in the center to the south.

Stratigraphically, in the highlands of the eastern part of the Pandeglang region, there are holocene volcanoes which are the result of volcanic deposits, these deposits consist of alternations of lava, breccias, tuffs and lava deposits. There are also volcanic rocks of Pleistocene age which are composed of basalt-andesite originating from old volcanoes. In the western part of the coast of the Pandeglang area, alluvium (Qa) is found, coastal terrace deposits in the form of coral limestone (Qc), Cimapag formations (Tmc) and Bojongmanik formations (Tmb1) in the form of alternating sandstone and flaky claystone interspersed with claystone, conglomerate, tuff and agglomerates [18].

Identification of potential areas to become wind farms uses the Multi-Weighted Criteria Modeler method. The Multi-Weighted Criteria Modeler method is a tool used to decide from a variety of special parameters to be combined [15]. This method uses weighting for each parameter in the form of elevation data, viewshed, slope, aspect direction, wind speed, shapefiles of lakes, rivers, reservoirs or dams, land cover, settlements and transportation. In detail the description and geology of the Pandeglang area can be seen in Figure 1.

Then classified by statistical methods by selecting areas with an area of ≥10,000 m2 in boolean. Boolean logic is a form of algebra whose variable values are true and false. Usually denoted by the numbers 1 and 0 for each parameter. Boolean is able to find other functions with fewer operations or terms [16].

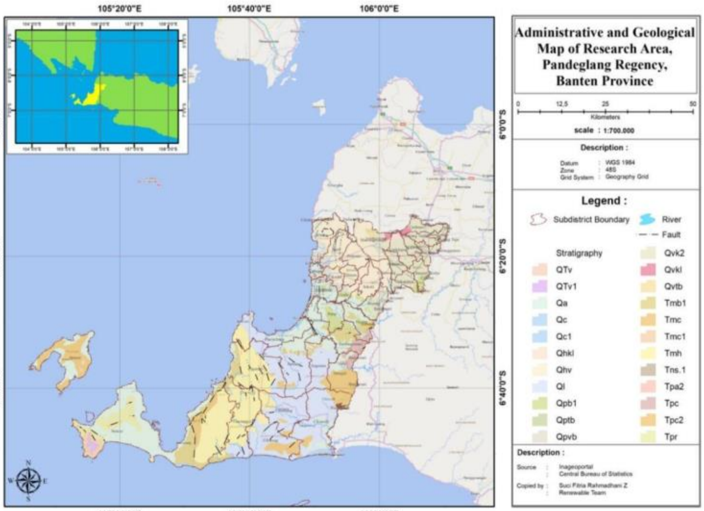

Figure 1 - Administrative Map of Pandeglang Regency, Banten Province

This type of research is quantitative research. Source of data used in the form of secondary data obtained from agencies, libraries, archives and individuals. The secondary data used are elevation, viewshed, slope, aspect direction, wind speed, shapefiles of lakes, rivers, reservoirs or dams, land cover, settlements and transportation.

Results and Discussion

The spatial analysis that has been carried out from the dataset of factors for the suitability of Wind Power Plant areas in Pandeglang Regency includes topographical factors, technical, environmental and socio-economic criteria.

Topographic factors include elevation on the height factor, slope, aspects of the slope direction and area with each criterion. Areas that are too high are generally not suitable for placing wind turbines, steep slopes are also considered unsuitable for placing wind turbines because they can pose a danger to wind turbines and the area around where wind turbines are placed. The details of the criteria for topographic factors can be seen in Table 1.

Height was obtained that the Pandeglang Regency area was dominated by areas with an altitude of less than 1500 meters above sea level. So that almost the entire area of Pandeglang Regency is included in the "appropriate" category. The suitability map for all topographical factor parameters can be seen in Figure 2.

Table 1 - Topographic Factors and Criteria

Factor	Criteria
Height	<1.500 m
Slope	<15%
Aspect of Slope	315<value>360
Direction	>1.000 m^2
Area	>100.000 m^2

Source: Idrizi, 2018

Slopes that have a role as a determining factor in carrying out spatial planning to assess whether the observation area is suitable can be used as a Wind Power Plant. The Pandeglang Regency area has a slope that is included in the "appropriate" category because it has an area with a slope of less than 15°. The suitability map for all topographical factor parameters can be seen in Figure 3.

The direction of the slope of the slopes in the Pandeglang Regency area is dominated by Northwest to Southeastern directions, which is also influenced by factors from the direction of the Monsoon winds in the Banten region. The suitability map for all topographical factor parameters can be seen in Figure 4.

Technical factors only observe one parameter, namely the wind speed in the Pandeglang Regency area. In determining the area to be used as a wind farm, namely the success of a wind turbine turbine can produce energy obtained from the wind which will rotate the turbine. Suitable and unsuitable wind speed can be seen in Figure 5.

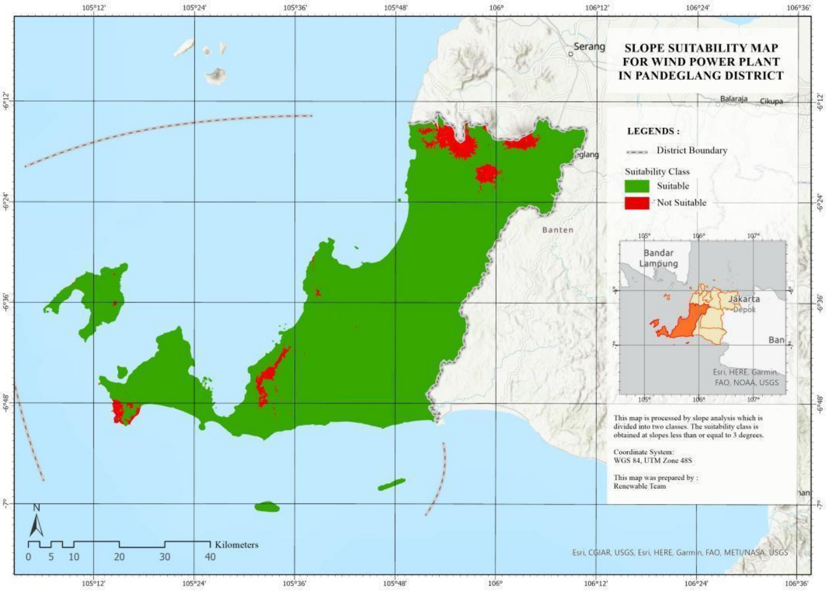

Figure 2 - Altitude Parameters on Topographic Factors

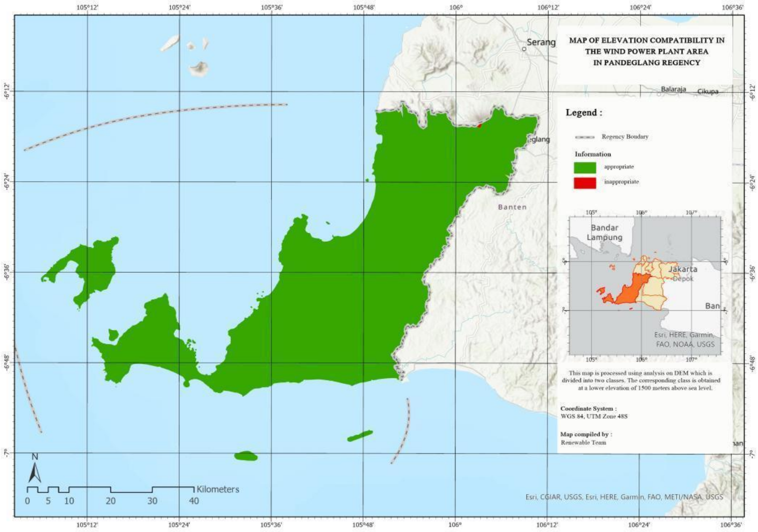

Figure 3 - Slope Height Parameters on Topographic Factors

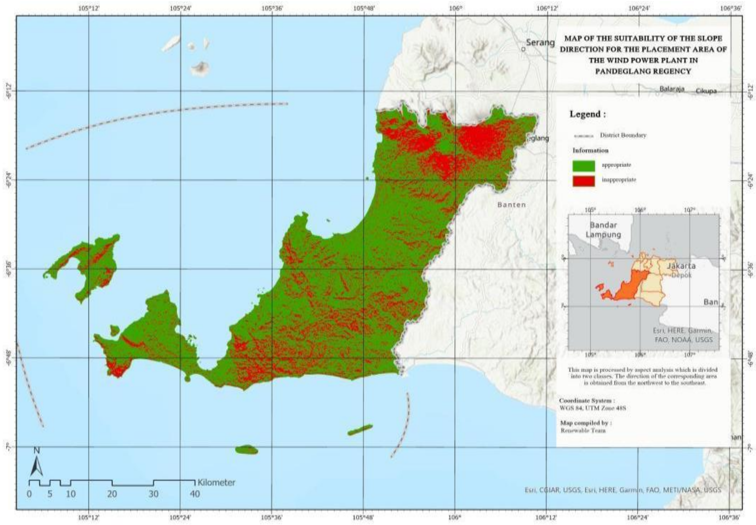

Figure 4 - Slope Direction Aspect Parameters on Topographic Factors

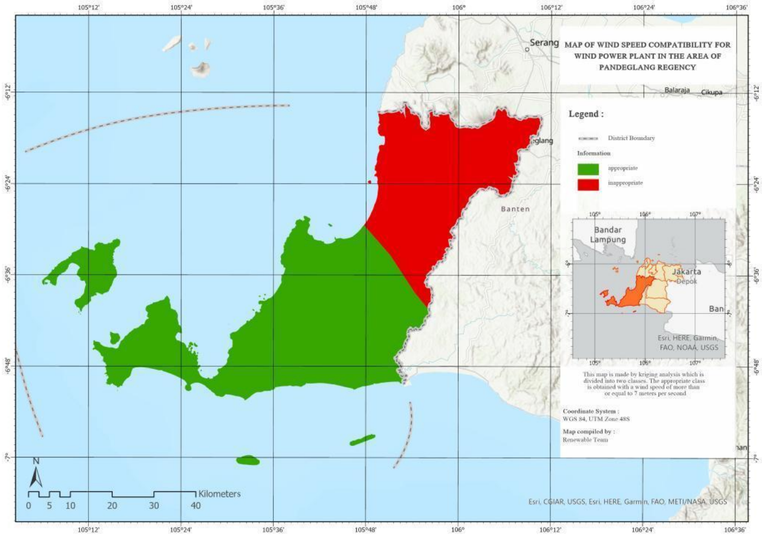

Figure 5 - Parameters on Technical Factors namely Wind Speed

Wind speed is a technical factor as well as a key holder in connecting renewable energy sources and producing as much energy as possible into the national energy network. This is a very important factor and must be considered. The details of technical factors can be seen in Table 2.

Table 2 - Technical Factors and Criteria

Factor	Criteria
Wind velocity	>5 meters/second

Source: Idrizi, 2018

Environmental factors include the area's proximity to rivers, lakes, dams and land cover. This is as a precaution so that the foundation of the wind turbine avoids flooding, because it will cause damage to the wind turbine. Land cover in the form of forest must also be considered in making wind farms and placing wind turbines, because vegetation in the forest can interfere and hinder the performance of wind turbines. The details on the environmental factors for placing wind turbines can be seen in Table 3.

Table 3 - Environmental Factors and Criteria

Factor	Criteria
River *Proximities*	>300 m
Proximity Lake	>300 m
Land Cover Soil Surface	>300 m
Dam *Proximity*	>500 m
River *Proximities*	>300 m

Source: Idrizi, 2018

Proximity Analysis or proximity factor analysis is an analysis of geospatial information systems that are usually used in determining land for strategic purposes. Buffering is another form of proximity analysis from a proximity analysis which is carried out by identifying the relationship between a point or area with other areas around it [[19], [20]].

The western and eastern parts of the Pandeglang Regency are dominated by jungle, only a few swamp areas, namely around the coast of Ujung Kulon National Park. Throughout Pandeglang Regency there are many rivers flowing, such as the Cipunten Agung, Caringin and Ciliman rivers. In this area there are also lakes such as Tegal Paku Lake, Gonggong Lake and Cikendal Lake. The proximity of rivers, lakes, forests and swamps can be seen in Figure 6.

The appropriate area in Figure 6 is an area that is not too close to rivers, lakes, forests and swamps. The Wind Power Plant area must have a distance of more than 300 meters from rivers, lakes, forests and swamps. The distance between the Wind Power Plant area and rivers and lakes is to prevent flooding. The Wind Power Plant area should not be too close to the swamp because the strength of the soil in the swamp area is very low to support the windmill. Meanwhile, the Wind Power Plant area must be far from the forest to avoid equipment accidents that can have a negative impact on the forest.

The distance from the dam to the Wind Power Plant construction site is an environmental factor that should be taken into account because the existence of a dam increases the risk of hydrometeorological disasters, especially floods and flash floods, so that a considerable distance from the dam is needed to build a Wind Power Plant. The proximity of the dam can be seen in Figure 7.

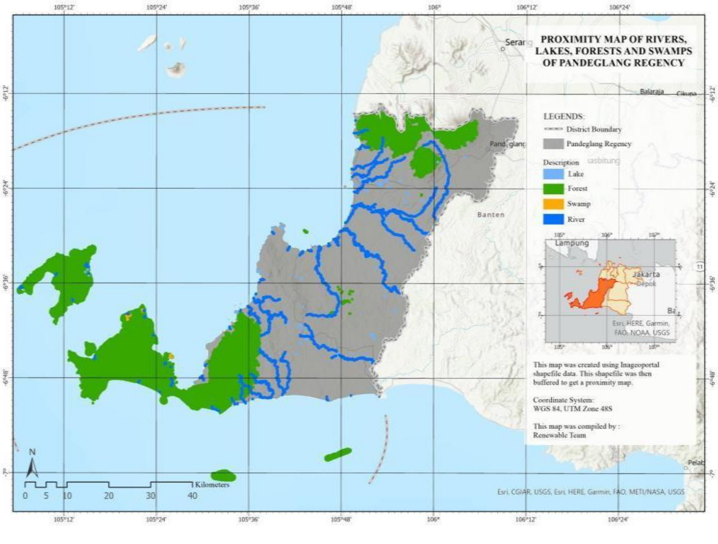

Figure 6 - Proximity to Rivers, Lakes, Forests and Swamps

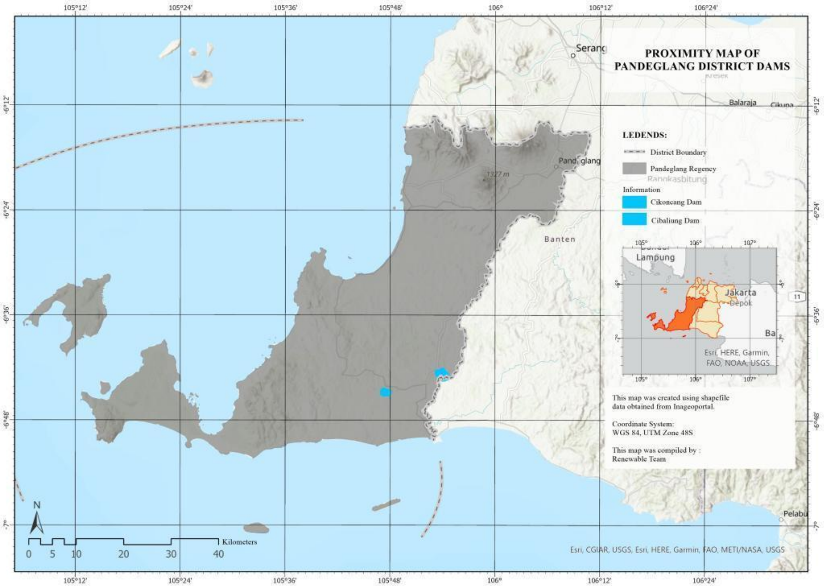

Figure 7 - Proximity to Dams

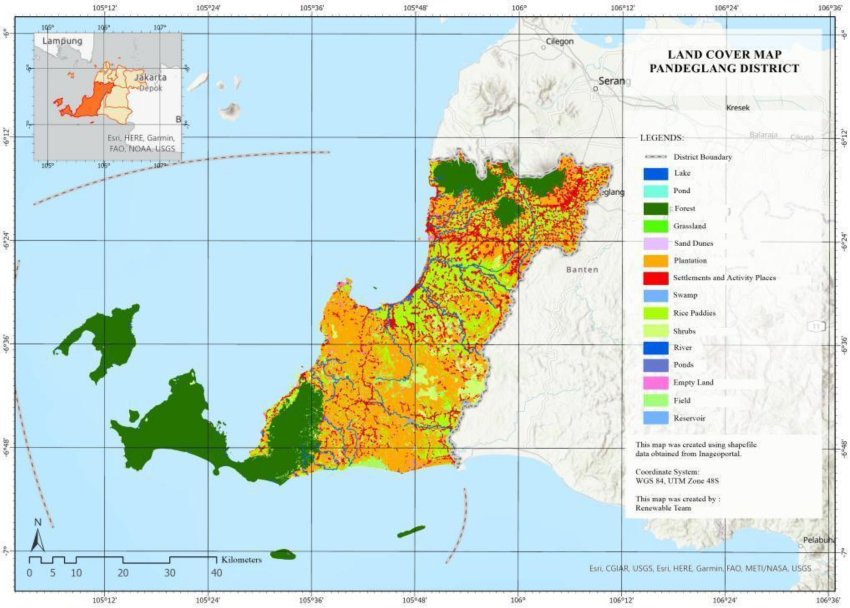

Figure 8 - Land Cover in Pandeglang Regency

The potential distance for establishing a PLTB is >500 meters from the dam. Pandeglang Regency itself has 2 dams, namely the Cikoncang Dam and the Cibaliung Dam. However, in terms of the reach of the dam, it does not really dominate the Pandeglang Regency area, so there are still many potential locations for establishing PLTB in Pandeglang Regency.

Data and information related to land cover in Pandeglang Regency was obtained based on Sentinel-2 image processing. Sentinel image results monitor land cover in the form of lakes, ponds, jungle, grasslands, sand dunes, plantations, settlements, swamps, rice fields, shrubs, rivers, ponds, vacant land, fields, and reservoirs. Based on the processing results, the land cover of Pandeglang Regency was obtained as can be seen in Figure 8.

The area of Pandeglang Regency is dominated by land cover in the form of plantations covering an area of 1,188 km², jungle forest covering an area of 770 km², rice fields covering an area of 570 km², settlements covering an area of 110 km², shrubs covering an area of 55 km² and fields covering an area of 53 km².

The rest of this area is filled with land cover in the form of lakes, ponds, swamps, rivers, ponds and reservoirs. The Wind Power Plant development area in Pandeglang Regency is covered in scrub land and vacant land. This is because the Wind Power Plant construction is unlikely to disturb the land cover that has been used by the community, such as settlements, plantations, rice fields, and others.

Socio-Economic Factors are only influenced by the proximity of the road, because this is useful for the safety and comfort of road users caused by wind turbines at Wind Power Plant.

The ideal distance criterion between the Wind Power Plant area and the main road is around >250 meters. The proximity of roads in the Pandeglang Regency area can be seen in Figure 9 and the details on the socio-economic factors for placing wind turbines can be seen in Table 4.

Table 4 - Socioeconomic Factors and Criteria

Factor	Criteria
Distance to Highway	>250 m

Source: Idrizi, 2018

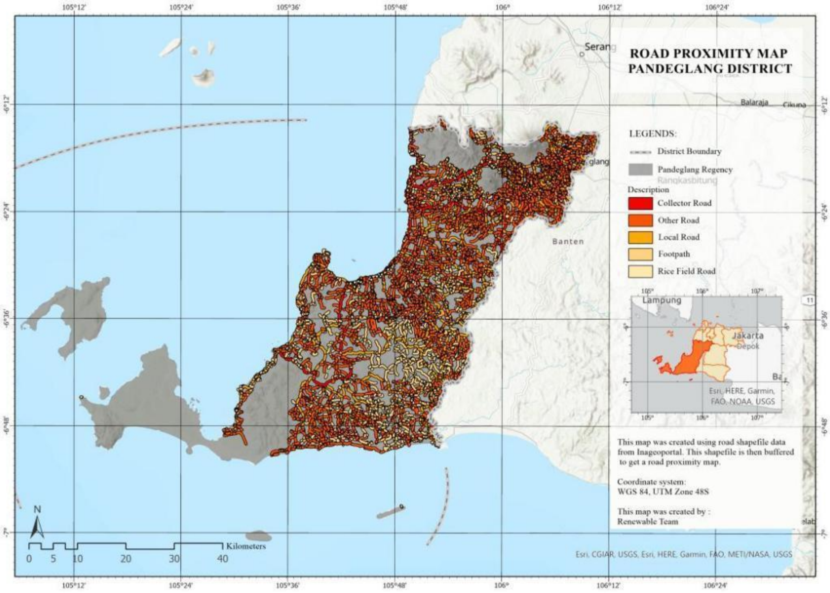

Figure 9 - Proximity to the Road

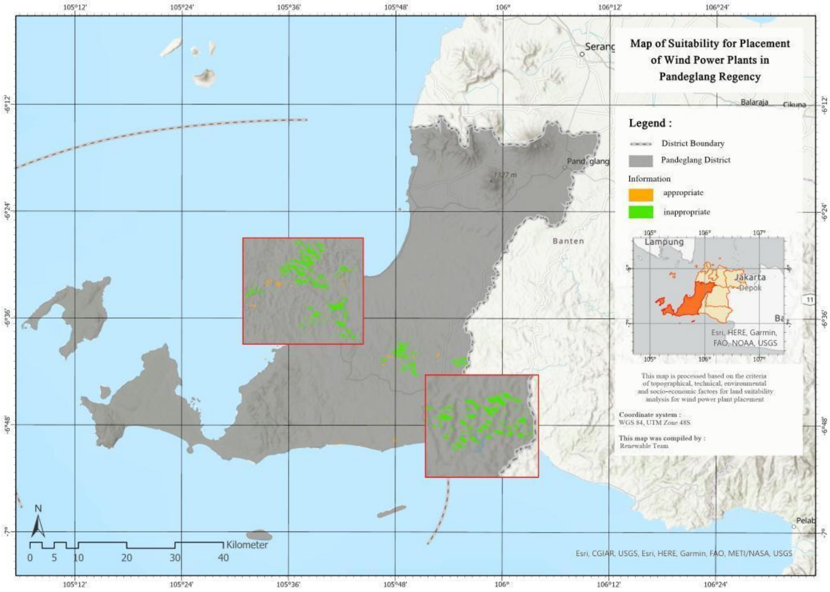

Figure 10 - Wind Power Plant Area Suitability Map

Potential Wind Farm Areas. The suitability of the area obtained for Wind Power Plant placement is based on factors and parameters consisting of categories, suitable and not suitable.

The areas suitable for Wind Power Plant are Sobang Sub-District, Leuwibalang Sub-District, Kadubadak Sub-District, Cihanjuang Sub-District, Cikadongdong Sub-District, and Pancaran Sub-District. As for the less suitable areas, there are Sindangkerta Village and Tanjungan Village. From the total area of Pandeglang Regency of approximately 2,746.89 km^2, the area suitable for wind farms is 350.71 km^2 and the area less suitable for wind farms is 30.49 km^2 but can still be considered for utilization. As for the appropriate and less suitable areas can be seen in Figure 10.

Conclusions

The construction of the Wind Power Plant in Pandeglang Regency can be carried out in the Leuwimalang, Kadubadak, Cihanjuang, Cikadongdong and Pancaran Sub-Districts. The total area suitable for the construction of a Wind Power Plant is 350.71 km^2. Areas suitable for the construction of Wind Power Plants are obtained from the Multi - Weighted Criteria analysis Modeler and Boolean with factors namely: topography, technical, environmental, and socio-economic.

The Geographic Information system-based method in this study provides a quantitative evaluation and assessment of factors and constraints that must be considered in determining land suitability for the wind power plant area in Pandeglang Regency. The final suitability map can be used as a guide towards a detailed search of the Wind Power Plant site. Additional surveys and measurements should be carried out before making a final decision for wind development. In addition, the final results of the final map must be additionally validated by wind speed measurements as well as by observing other important ecological variables in each specific area for the placement of a potential Wind Power Plant.

This study concludes that Indonesia has the potential to develop Wind Power Plant, which can meet Indonesia's target in the transition from fossil fuel use to the use of renewable energy in total, in addition to solar, water and micro-hydro power. However, an analysis with a wider coverage area is needed to find out other areas that have a high level of suitability for the development of Wind Power Plant. Based on these findings, it is expected to be a basis for the government and related institutions in the development of renewable energy, especially in the development of wind power in the construction of power plants.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **F.Suci**: Conceptualization, Methodology, Writing draft preparation, Editing, Investigation; **I. Muhamad**: Data curation, Writing draft preparation, Software, Visualization, Investigation, Editing.

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[1] Suci F.R.Z., [2] Muhamad I.B.

[1] STTIND Паданг өнеркәсіптік технологиялар колледжі, Батыс Суматра, Индонезия
[2] Паданг мемлекеттік университеті, Индонезия

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Сараптамадан өтті: *26 желтоқсан 2024*
Қабылданды: *18 ақпан 2025*

ТҮЙІНДЕМЕ

Қарастырылып отырған зерттеу Индонезияның Бантен провинциясы Пандегланг-Редженсидегі жел электр станциясын дамытудың әлеуетін ашады және мұндай нысандарды орналастыру орнын таңдауға әсер ететін әртүрлі факторларды жан-жақты талдаудың маңыздылығын көрсетеді. Қарастырылған параметрлер топографиялықтан әлеуметтік-экономикалыққа дейін жел диірменің орнату және энергия өндіру жобасының сәтті болуына әсер етеді. Зерттеуде қолданылатын «Көп салмақты критерийлерді модельдеу» әдісі аумақтың әртүрлі сипаттамаларын тиімді бағалауға және құрылыс үшін ең қолайлы жерлерді таңдауға мүмкіндік береді. Бұл тәсіл сандық және сапалық деректерді біріктіреді, нәтижесінде дәлірек және дұрыс нәтижелер алынады. Зерттеу нәтижелері Пандегланг округінде жаңа жел электр станциясын құру критерийлеріне сәйкес келетін 350,71 км² аумақ бар екенін көрсетеді. Бұл ақпарат аймақтағы жаңартылатын энергия жобаларын одан әрі зерттеуге және егжей-тегжейлі әзірлеуге негіз бола алады. Осылайша, зерттеу нәтижелері Индонезияның жел энергетикасын дамыту үшін үлкен әлеуеті бар екенін көрсетеді және қосымша қолайлы аймақтарды анықтау үшін елдің басқа аймақтарын зерттеуді жалғастырудың маңыздылығын көрсетеді. Осылайша, Индонезиядағы жел энергетикасы әлеуетінің зор екендігіне қарамастан, елдегі жел энергиясын тиімді пайдаланудың барлық ықтимал бағыттарын анықтау үшін зерттеулер кеңірек болуы керек.

Түйін сөздер: жаңартылатын энергия, жел электр станциясы, жел диірмені.

Авторлар туралы ақпарат:

Suci Fitria Rahmadhani Z – *Геологиялық инженерия магистрі, Паджаджаран университеті, Бандунг қаласы, Батыс Ява провинциясы; Паданг қаласындағы өнеркәсіптік технологиялар колледжінің (STTIND) тау-кен инженериясын оқу бағдарламасының оқытушысы, Батыс Суматра. Email: sucifitria1228@gmail.com; ORCID ID: https://orcid.org/0000-0003-0714-3672*

Muhamad Ikhwan Bagus – *Паданг мемлекеттік университетінің географиялық оқу бағдарламасының бакалавры, Батыс Суматра. Email: Muhamadikhwanbagus@gmail.com; ORCID ID: https://orcid.org/0009-0001-0361-1072*

Оценка пригодности земель для строительства ветряной электростанции в районе Пандегланг, провинция Бантен, Индонезия

[1] Suci F.R.Z., [2] Muhamad I.B.

[1] STTIND Колледж промышленных технологий Паданга, Западная Суматра, Индонезия
[2] Государственный университет Паданга, Индонезия

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АННОТАЦИЯ

Исследование, о котором идет речь, раскрывает потенциал развития ветряных электростанций в округе Пандегланг провинции Бантен, Индонезия, и подчеркивает важность комплексного анализа различных факторов, влияющих на выбор местоположения для таких объектов. Рассмотренные параметры — от топографических до социально-экономических — оказывают влияние на успех реализации проекта по установке ветряных мельниц и производству энергии. Метод Multi-Weighted Criteria Modeler, использованный в исследовании, позволяет эффективно оценить различные характеристики территории и выбрать наиболее подходящие участки для строительства. Этот подход сочетает количественные и качественные данные, что обеспечивает более точные и обоснованные результаты. Выводы исследования говорят о наличии области площадью 350,71 км², которая соответствует критериям для создания новой ветряной электростанции в округе Пандегланг. Эта информация может стать основой для дальнейших исследований и детальной разработки проектов в области возобновляемой энергетики в регионе. Таким образом, результаты исследования свидетельствуют о большом потенциале Индонезии для развития ветряных электростанций, и подчеркивается важность продолжения исследования других регионов страны для выявления дополнительных подходящих территорий. Таким образом, несмотря на потенциал ветряной энергии в Индонезии, исследования должны быть более широкими, чтобы выявить все возможные районы для эффективного использования ветряной энергии в стране.

Ключевые слова: возобновляемая энергия, ветряная электростанция, ветряная мельница.

Информация об авторах:

Suci Fitria Rahmadhani Z – *Магистр геологического инжиниринга, Университет Паджаджаран, Бандунг, провинция Западная Ява; Преподаватель программы обучения горному делу в Колледже промышленных технологий (STTIND) города Паданг, Западная Суматра. Email: sucifitria1228@gmail.com; ORCID ID: https://orcid.org/0000-0003-0714-3672*

Muhamad Ikhwan Bagus – *Бакалавр программного обучения географии в Государственном университете Паданга, Западная Суматра. Email: Muhamadikhwanbagus@gmail.com; ORCID ID: https://orcid.org/0009-0001-0361-1072*

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Earth sciences

Monitoring of the earth's surface and mining facilities by radar interferometry

[1*]Altayeva A. A., [2]Sadykov B.B., [1]Umirbayeva A.B., [3]Darkenbaeva A.B., [1]Dastan A.

[1] International Educational Corporation LLP, Almaty, Kazakhstan
[2] Leica Geosystems Казахстан LLP, Almaty, Kazakhstan
[3]Kazakh National Agrarian Research University, Almaty, Kazakhstan

** Corresponding author email: a.aselya_92@mail.ru*

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ABSTRACT

Safe and efficient development of mineral deposits by underground method, occurring at great depths, is complicated by the fact that with an increase in the depth of mining, the nature of the course of deformation processes in the rock mass and the degree of their impact on the environment change significantly. Studies of deformation processes, their control and forecast in many cases determine the efficiency and safety of the development of deposits of solid minerals. A practical forecast can be made as a result of continuous tracking in space and time of deformation processes. This article presents modern satellite radars and their main characteristics. The features of the radar interferometry method are described, and the advantages and disadvantages of various methods of interferometric processing of radar images are considered. The experience of using space radar for monitoring mineral deposits in the Republic of Kazakhstan is analyzed. The question was raised about the possibility of intensifying the use of radar interferometry in the mining industry. Regular field observations are provided throughout the mining site, including hard-to-reach and dangerous areas, regardless of weather conditions. It is confirmed that the use of the radar interferometry method determines the displacement of the earth's surface with high accuracy. When using this method, data is received and processed remotely and generally does not require presence on the site. In addition, this article provides examples of successful application of the radar interferometry method in foreign countries at mining and oil facilities. Also, the use of radar interferometry allows you to quickly determine the zones of possible deformations of the earth's surface and organize high-precision surveying and geodetic observations in these zones. In this article, radar interferometry has been used to monitor the surface movement of the Annensky field with high accuracy since 2016 using Sentinel radar images, and as a result, a displacement map of the earth's surface has been generated.

Keywords: radar interferometry, radar sensing of the Earth, synthesized aperture radar, interferometric processing methods, radar sensors, surface deformation, mining industry.

	Information about authors:
Altayeva Assel Abdikerimkyzy	*PhD, Associate Professor of the Department of Geodesy and Cartography, Cadastre at the International Educational Corporation LLP, Almaty, Kazakhstan. Email: a.aselya_92@mail.ru; ORCID ID: https://orcid.org/0000-0002-1675-6828*
Sadykov Batyrkhan Bolatuly	*PhD, Associate Professor, Leica Geosystems Казахстан, Almaty, Kazakhstan. Email: batyrkhan_sadykov@mail.ru; ORCID ID: https://orcid.org/0000-0003-2037-247X*
Umirbayeva Aliya Batukhanovna	*PhD, Associate Professor of the Department of Geodesy and Cartography, Cadastre at the International Educational Corporation, Almaty, Kazakhstan. Email:* a_umirbayeva@mail.ru; *ORCID ID: https://orcid.org/0000-0002-4746-3411*
Darkenbaeva Assel Berikovna	*Doctoral student of the Department of Land Resources and Cadastre, Kazakh National Agrarian Research University, Almaty, Kazakhstan. Email: darkenbayevaasel@gmail.com; ORCID ID: https://orcid.org/0009-0001-7115-8951*
Dastan Anel	*Student of the Department of Geodesy and Cartography, Cadastre at the International Educational Corporation, Almaty, Kazakhstan. Email: dastan_a@mail.ru*

Introduction

Among the most important problems of monitoring the Earth's surface at mining facilities is the observation of hard-to-reach and dangerous areas with a high risk of Geodynamic phenomena. In this regard, non-contact monitoring methods, in particular space radar sensing, are actively used.

Radar shooting has its advantages in comparison with the classic methods of ground observation, laser scanning, aerial photography, space sensing in the optical range. A key point is the ability to conduct systematic field observations over the entire area of the object, including hard-to-reach and dangerous areas and regardless of weather conditions.

The use of the radar interferometry method makes it possible to determine the displacement of the Earth's surface with high accuracy. When using this method, data acquisition and processing is carried out remotely and in general does not require a presence at the object.

In recent years, radar sensing has been developing rapidly: the number of satellites, the quality and diversity of the received data is increasing, the technical characteristics of the shooting system and the methods of processing the received data are being improved, and new progressive software for processing data from satellite radar is constantly being updated and developed. In addition, the share of space radar data provided in free access is growing, which expands the possibilities of their use for scientific purposes. Successful scientific research, expansion of areas of implementation, improvement of processing technologies contribute to an increase in interest in the commercial use of these technologies [1].

Methods

Radar capture is an active method of sensing: the radar antenna emits a radio beam, and then, with the help of recording equipment, a reflected signal is obtained from the Earth's Surface [2]. Space radar sensing is carried out in the L-, C - and X-bands and provides the receipt of the necessary information in any weather conditions, regardless of the illumination of the surface, the state of the atmosphere. Radar shooting is carried out using a radar with a synthesized aperture (RSA). The use of radars with a synthesized aperture makes it possible to achieve a high spatial resolution in a wide review bar [3].

Currently, various radar sensor — projects of leading space agencies are working, among them: TerraSAR-X/TanDEM-X (DLR, Germany), Cosmo-SkyMED (ASI, Italy), Sentinel-1, T2 (ESA, European Space Agency), Radarsat-2 (MDA/CSA, Canada), ALOS-2 (JAXA, Japan), Kompsat-5 (KARI, Republic of Korea). In addition, there is a large amount of archived radar data from the satellites ERS, JERS, ENVISAT, RADARSAT-1, ALOS, etc. Currently, for example, New tandem-l shooting systems are being developed.

The radio waves used in the RSA differ in range, space resolution, width of the review bar, frequency of shooting, set of shooting modes (Table 1). These features must be taken into account when planning the study and selecting the initial data in accordance with the characteristics of the object under study.

Table 1 - The main characteristics of modern radar satellites

	TerraSAR-X TanDEM-X	Cosmo-SkyMED	Kompsat-5	Sentinel-1	Radarsat-2	ALOS-2
Radio Wave range	X	X	X	C	C	L
Wavelength (sm)	3.1	3.1	3.2	5.5	5.6	22.9
spatial resolution (m)	0.25—40	1—100	0.85—20	5—40	1—100	1—100

The method of radar interferometry

It uses the effect of electromagnetic wave interference to make measurements with radar interferometry. To do this, several coherent measurements of a certain area of the Earth's surface are carried out with a change in the position of the radar in Space [4]. Interferometric processing involves combining 2 different time PSA-images and creating an interferogram that allows you to measure the phase difference of two images, on the basis of which in the future the calculation of height marks and displacement will be performed [1]. Interferometric pairs of radar images are used as input data. The phase component is involved in processing, the amplitude of radar images is used in large-scale mapping [[4], [5]].

An important role is played by the characteristics of the initial images: spatial resolution, spatial and temporal basis, parameters of the orbit from which the data were obtained. These parameters determine the possibility of using images, in particular, the accuracy of detecting deformations and the scale of displacement maps obtained in Geodynamic, geomechanical monitoring.

Today, the term radar interferometry refers to various ways of processing radar images. When using the classical method of differential interferometry (DInSAR), the analysis of the Earth's surface displacement is carried out according to two capture cycles. If there is a Geodynamic, geomechanical observation of the Earth's surface, then the survey is carried out before and after the movements, respectively, and the analysis of the processes is limited to the period between the two surveys. A joint analysis of a series of radar images is more effective. For this, advanced processing methods are used — the small base distance method (Small Baseline — SBas) and the permanent reflector method (Persistent / Permanent Scatterers-PS). The SBas method uses interferograms with a minimal spatial basis, which helps to reduce geometric decorativeness. When using constant reflector interferometry, points are selected in the images that are constant reflectors of the radar signal, and this is used by them to analyze the phase change. Anthropogenic objects: buildings and structures, objects of the road network, bridges, etc. are often used as permanent sprinklers. In this regard, this method is most suitable for monitoring on built-in sites. Artificial angle reflectors are used for monitoring specific areas and objects [[1], [4], [6]].

Research results and their discussion

The main areas of use of this method in mining are the creation of digital models of the terrain and the calculation of the displacement of the Earth's surface. The problems of storage and visualization of the received information products are solved with the help of a temporary data model and Geoinformation technologies that ensure the correct presentation of monitoring data, their effective use, viewing of "historical" data [[7], [8], [9]].

Radar images are a source of information for solving the following problems in the mining industry:

- determination of deformation zones on the Earth's surface;
- control of subsidence and displacement of the Earth's surface in the cultivated areas;
- control of changes in buildings and structures;
- monitoring the stability of quarries, cemeteries and dump boards;
- monitoring the condition of hydro-reservoirs and tailings, including protective structures;
- monitoring the condition of mining transport equipment in quarries;
- environmental monitoring of reclaimed land.

Interferometric technologies, as well as amplitude information processing, are used to obtain hydrogeological characteristics of the area of deposits, in particular, to assess wetting [[10], [11], [12]]. Modern information on the state and level of groundwater allows for intensive and systematic, uniform development, with the exception of a sudden stop in the development process as a result of unexpected behavior of the Water Horizon [13].

Despite a number of successful projects in the field of radar interferometry [[14]], the use of radar technologies in Kazakhstan fields is currently limited, which is due to a number of factors. High-precision measurements are required to monitor mining facilities and infrastructure. To achieve the required accuracy, large spatial resolution data and the help of specialized software with a fairly high cost are required [15]. In addition, not all radar satellites provide the research frequency required for surface observation [[16]]. Analysis of vertical and horizontal shifts is possible in the presence of data from different orbits. Processing large series of radar images is a lengthy process and imposes hardware requirements, the necessary computing resources are not always available [17].

A significant part of the Earth's surface of Kazakhstan deposits is characterized by heterogeneity: the presence of snow cover, wetlands, seasonal changes in vegetation negatively affect the compatibility of the data obtained, limit the measurement period and the size of the images used.

There are different points of view on the need for additional measurements of the Earth when conducting radar observations of the Earth's surface. When using radar interferometry at Kazakhstani fields, it is usually necessary to carry out underground measurements: for a preliminary assessment of the possibilities of using radar surveys at a specific object, monitoring and checking the results, calculating absolute altitude values. For open-pit mines, the use of the sprinkler method involves the installation and control of additional artificial corner reflectors.

The determination of deformation of the Earth's surface depends on the capture parameters, the characteristics of the lower layer of the Earth's surface, the characteristics of the existing radar data, the availability of additional information products (reference DEM, etc.). The correct and realistic assessment of the determination of the displacement is still an undeveloped area. Due to all of the above factors, space radar is increasingly used to obtain a field idea of the dynamics of the Earth's surface and is used as an addition to classical methods to identify areas where high-precision monitoring is required.

Review of scientific research carried out using radar interferometry

As you know, natural and man-made displacement of the Earth's surface in the area of developing deposits is a potentially dangerous process, therefore, monitoring of the state of mining divisions by geodetic methods is provided, which, as a rule, requires significant costs and is not always carried out in the mode of operational monitoring. Currently, many mining companies have begun to use the radar interferometry method to measure the displacement of the Earth's surface with high accuracy, clarify the displacement values, promptly assess the situation and dynamics of processes, as well as predict their development.

An example of the successful application of the radar interferometry method is the case that occurred in 2007 on the territory of the city of Berezniki as a result of emergency flooding of a mine. The research was carried out by the Technical University of Claustal. The Department of SGGIS conducted independent studies of sedimentation processes in the territory of the city of Berezniki on the basis of satellite data from the Envisat spacecraft. In this area, very impressive data were obtained on the sedimentation detected in the period from 2007 to 2008, which corresponded to the actual processes of sedimentation (Figure 1), [18].

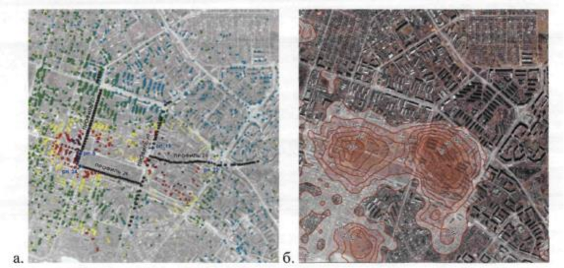

Figure 1 - (A) based on point interferometric analysis, the sedimentation area in the city of Berezniki and (B) the detected sedimentation is indicated by isoslines

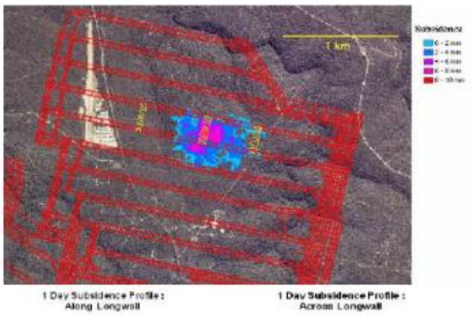

Figure 2 - Comparison of DInSAR interferogram with mine plan

Since 2007, Australia has been using satellite radar interferometry to monitor surface deformations of the Carmichael coal field located in new Wales. An interesting result was obtained using a tandem pair of ERS SAR. During the 24-hour period, the maximum amplitude of 1 cm shrinkage was determined by a resolution of 2 mm (see Figure 2). This was independently confirmed by the Geodetic service of the mining company. This example shows the possibility of temporary high-resolution (i.e. one-day) monitoring of the displacement of underground production using the DINSAR technique.

Since 2017, radar interferometry technology has been used at the Sishen field (South Africa) to monitor surface displacement based on data from the TerraSAR-X satellite. At the Sishen field, the area of dangerous subsidence was determined using radar interferometry. Due to the results obtained, work in this territory was suspended. The development of subsidence of the Earth's surface according to the results of radar interferometry is shown in Figure 3.

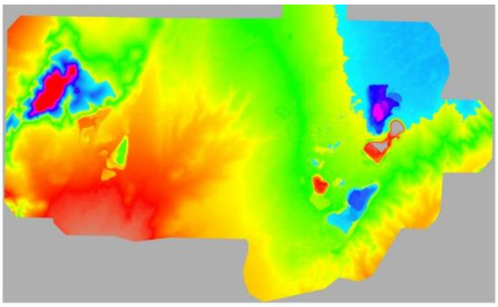

Figure 3 - Subsidence areas of the Earth's surface of the Sishen mine according to TerraSAR-X

Observation of the deformation of the Earth's surface of the Annensk field using radar interferometry:

The Zhezkazgan copper deposit is located in the central part of Kazakhstan, in the Kara-Kengir River Basin, in the south-east of the Ulytau mountains, in the west of the city of Zhezkazgan and the kengir reservoir, near the settlements of Satpayev. It includes the S mines and covers an area of 10 x 7 km. Active underground mining of the deposit has been carried out since the middle of the 20th century. Currently, the field is being developed by the Kazakhmys Corporation.

During the development period, a significant part of the reserves of the field was withdrawn. The field is mined by a chamber-column system. The empty excavated underground space is supported by pillar posts between tens of thousands of stones.

Of course, such a volume of underground cavities causes displacement and deformation of the Earth's surface and structures along the amplitude. Since the mid-1990s, individual settlement areas have been merged into larger areas that have been weakened. Underground and surface rock collapses began to occur, including leading to the destruction of buildings, structures, and infrastructure elements. Taking into account the location of settlements near the field (Satpayev. Rudnik, etc.), these processes pose a serious threat to human life.

In 2011, Kazakhmys Corporation organized monitoring of surface displacement at the entire site of the Zhezkazgan field using the methods of space radar interferometry conducted by the Russian company Sovzond [19].

The initial control was carried out on 17.01.11. During the observation period (9 months), the greatest subsidence of the Earth's surface was recorded at the combined mulda shift in the zone of Block 4-4yug along the Ann-4-I field of about 35 mm, at the Annensk field in the south-eastern part.

According to the results of observations, it was found that the rate of constant subsidence of the Earth's surface is on average 4 mm per month.

In accordance with the requirements of the" methodological guidelines for the identification of weakened sites and the operational assessment of the state of the excavated space in mines according to Zhezkazgan, 2011 " (Zhezkazgan, 2011), the Earth's surface is considered to be more than 35 mm of subsidence and sedimentation rate of more than 1 mm/month. The state of the mined space of block 4-4 south at the Ann-4-I field should be assessed as unstable.

Figure 4 shows the dynamics of the development of subsidence mulda from February to July 2012 according to the space radar interferometry.

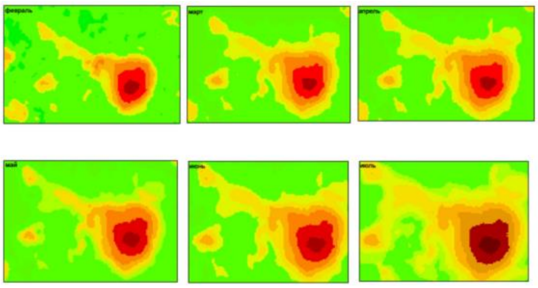

Figure 4 - Plan of subsidence of the Annensk field from February to July 2012

In addition, since 2016, with the help of Sentinel radar images, we have tracked the subsidence of the Earth's surface of the Annensk field. This scientific work was carried out at the Department of Surveying and geodesy of Satbayev University within the framework of the grant funding project "development of a system and methods for predicting the displacement of mountain mass in dangerous areas of the Earth's surface during subsoil development based on innovative methods of GIS technology" in 2018-2020. As a result of complex processing and interpretation of radar images, a vertical map of the Earth's surface displacement of the Annensky mine was obtained (Figure 5) [[20], [21]]. For a comparative analysis of the obtained interferometric data, the site where the control reps are located was selected. On this site, instrumental monitoring of surface deformations is carried out annually by the surveying service of Kazakhmys Corporation LLP. High-precision leveling is carried out along the profile lines, which are a network of soil and supporting rappers.

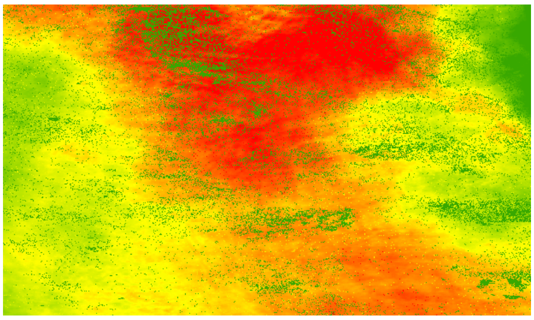

Figure 5 - Map of the Earth's surface displacement of the Annensk mine

We continued our research and continued monitoring the subsidence of the Annensk field surface using Sentinel radar images from 2021 to 2023 (Fig. 6).

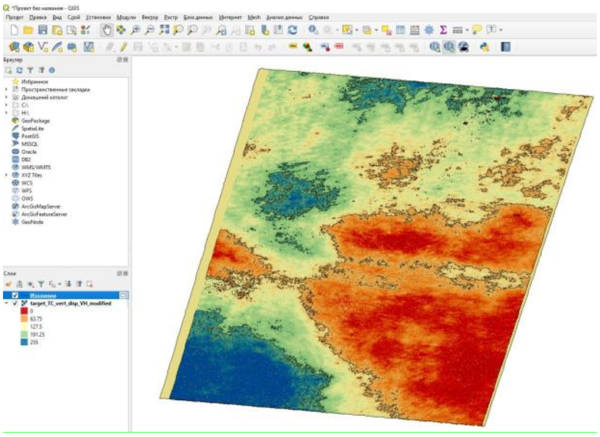

Figure 6 - Earth's surface shift map of the Annensk field obtained using Sentinel radar images

The study, conducted between 2021 and 2023, showed that due to the heating of the Annenskoye field, there are deposits characteristic of the Central and South-Eastern parts of the earth's surface. At the same time, the maximum absolute value of the subsidence of the earth's surface within the shrinkage of the Annenskoye field was 0.8 cm

For a more complete and detailed assessment of deformations in the deposit area, it is necessary to analyze monthly mining plans and space data. During the research, some areas were processed and secured, which made them inaccessible for the corresponding analysis.

This feature of the processes of collapse and displacement of rocks in the real conditions of the Annensk highlands leads to the following practical conclusions:

- Mining operations in the collapse zone are permitted only after the process of displacement and stabilization of the geomechanical environment has been completed, which can be determined by comprehensive observation of the massif. Based on the results of visual observations of the state of mining operations and the development of manifestations of rock pressure, simple beacons, signs, gates, as well as the weakening of the seismic activity of the massif and the shift of the earth's surface;
- for further monitoring of the development of rock deformations it is necessary to continue geomechanical monitoring based on an expanded model of geomechanical monitoring of the territory of the Annensky mine using topographic and geodetic methods and space radar interferometry technologies;
- Use permanent reflector techniques to improve the accuracy of geomechanical observations using space radar interferometry technologies.

Conclusion

Space radar interferometry is an important part of an integrated system for monitoring the condition and creating a continuous situational map of deformations on the earth's surface. We chose to use Sentinel spacecraft data and SAR interferometry methods to monitor the drift of the Annensk field's earth's surface.

To properly use SAR interferometry methods, it is necessary to use a tandem pair of radar satellite images with a minimum value of the perpendicular baseline (the distance between the spacecraft) and a minimum consistency value to determine the absolute values of the Earth's surface displacement generated during the studied period of time. To apply SAR interferometry methods, a coherent analysis of tandem pairs of radar satellite images was carried out to determine their optimal parameters.

As a result of the work carried out, a land management map of the Annensk mine territory was compiled. In addition, the maximum absolute value of the subsidence of the earth's surface within the subsidence of the Annenskoye deposit was 0.8 cm. Shifts occurred both in the direction of increase during the formation of rock dumps, and in the direction of decrease as a result of subsidence of the earth's surface.

The obtained results have proven that they correspond to the data of radar interferometry, conducted by surface measurements, as well as by the limited liability company "Sovzond".

Thus, radar technologies have great potential, their use in the mining industry is very promising. This is facilitated by the development and improvement of technologies for obtaining and processing radar data. Also, the use of radar interferometry allows you to quickly determine the zones of possible deformations of the earth's surface and organize high-precision mine surveying and geodetic observations in these zones.

Conflict of interest. On behalf of all authors, we declare that there is no conflict of interest.

Credit statement of the authors: **A. Altayeva:** processing of space data, writing of the article; **B.Sadykov:** analysis and processing of space data; **A.Umirbaeva:** performing a review of domestic and foreign information; **A. Dastan:** performing a translation of the article summary.

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Радарлық интерферометрия әдісінің көмегімен жер беті мен тау-кен нысандарын бақылау

[1*]Алтаева А.А., [2]Садықов Б. Б., [1] Умирбаева А.Б., [3]Даркенбаева А.Б., [1]Дастан А.

[1]Халықаралық білім беру корпорациясы ЖШС, Алматы, Қазақстан
[3] ТОО Leica Geosystems Казахстан, Алматы, Қазақстан
[3]Қазақ ұлттық аграрлық зерттеу университеті, Алматы, Қазақстан

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ТҮЙІНДЕМЕ

Үлкен тереңдікте орналасқан пайдалы қазбалардың кен орындарын жерасты әдістерімен қауіпсіз және тиімді игеру тау-кен жұмыстарын жүргізу тереңдігінің ұлғаюымен тау-кен массасындағы деформация процестерінің сипаты мен олардың қоршаған ортаға әсер ету дәрежесінің айтарлықтай өзгеруімен қиындайды. Деформациялық процестерді зерттеу, оларды бақылау және болжау көп жағдайда қатты пайдалы қазбалардың кен орындарын игерудің тиімділігі мен қауіпсіздігін анықтайды. Деформация процестерін кеңістікте және уақыт аралығында үздіксіз бақылау нәтижесінде практикалық болжам жасауға болады. Бұл мақалада қазіргі заманғы спутниктік радарлар және олардың негізгі сипаттамалары ұсынылған. Радиолокациялық интерферометрия әдісінің ерекшеліктері сипатталып, радиолокациялық кескіндерді интерферометриялық өңдеудің әртүрлі әдістерінің артықшылықтары мен кемшіліктері қарастырылады. Қазақстан Республикасындағы пайдалы қазбалар кен орындарын бақылау үшін ғарыштық радиолокацияны қолдану тәжірибесі талданды. Тау-кен өнеркәсібінде радиолокациялық интерферометрия әдісін қолдануды жандандыру мүмкіндігі туралы мәселе көтерілді. Тау-кен кешенінің бүкіл аумағында, оның ішінде жетуі қиын және қауіпті аймақтарда, ауа райы жағдайларына қарамастан тұрақты далалық бақылаулар жүргізуге болатындығы дәлелденді. Радар интерферометриясы әдісін қолдану нәтижесінде жер бетінің жылжуын жоғары дәлдікпен анықтауға болатыны расталды. Бұл әдісті қолданғанда деректерді алу және өңдеу қашықтықтан жүргізіледі және жалпы жағдайда объектіде болуды талап етпейді. Сонымен қатар, бұл мақалада шет елдерде радар интерферометрия әдісін тау-кен, мұнай объектілерінде сәтті қолданудың мысалдары келтірілген. Сондай-ақ, радар интерферометриясын қолдану жер бетінің мүмкін болатын деформация аймақтарын жедел анықтауға және осы аймақтарда дәлдігі жоғары маркшейдерлік-геодезиялық бақылауларды ұйымдастыруға мүмкіндік береді. Бұл мақалада 2016 жылғы Sentinel радиолокациялық суреттерін және радиолокациялық интерферометрия әдісін пайдалана отырып, Анненское кен орнының жер бетіндегі жылжулары жоғары дәлдікпен анықталып, орын ауыстыру картасы жасалды.

Түйін сөздер: радар интерферометриясы, жерді радиолокациялық зондтау, синтезделген апертурлық радиолокатор, интерферометрлік өңдеу әдістері, радар сенсорлары, жер бетінің деформациясы, тау-кен өнеркәсібі.

	Авторлар туралы ақпарат:
Алтаева Асель Абдикеримкызы	*PhD, Халықаралық білім беру корпорациясының Геодезия және картография, кадастр кафедрасының қауымдастырылған профессоры, Алматы, Қазақстан. Email: a.aselya_92@mail.ru; ORCID ID: https://orcid.org/0000-0002-1675-6828*
Садыков Батырхан Болатұлы	*PhD, Leica Geosystems Қазақстан ЖШС қауымдастырылған профессоры, Алматы, Қазақстан. Email: batyrkhan_sadykov@mail.ru; ORCID ID: https://orcid.org/0000-0003-2037-247X*
Умирбаева Алия Батухановна	*PhD, Халықаралық білім беру корпорациясының Геодезия және картография, кадастр кафедрасының қауымдастырылған профессоры, Алматы, Қазақстан. Email: a_umirbayeva@mail.ru; ORCID ID: https://orcid.org/0000-0002-4746-3411*
Даркенбаева Асель Бериковна	*Жер ресурстары және кадастр кафедрасының докторанты, Қазақ ұлттық аграрлық зерттеу университеті, Алматы, Қазақстан. Email: darkenbayevaasel@gmail.com; ORCID ID: https://orcid.org/0009-0001-7115-8951*
Дастан Анель	*Халықаралық білім беру корпорациясының Геодезия және картография, кадастр кафедрасының студенті, Алматы, Қазақстан. Email: dastan_a@mail.ru*

Мониторинг земной поверхности и горнодобывающих объектов методом радарной интерферометрии

[1*]Алтаева А.А., [2]Садыков Б.Б., [1]Умирбаева А.Б., [3]Даркенбаева А.Б., [1]Дастан А.

[1] ТОО Международная образовательная корпорация, Алматы, Казахстан
[2] ТОО Leica Geosystems Казахстан, Алматы, Казахстан
[3]Казахский национальный аграрный исследовательский университет, Алматы, Казахстан

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АННОТАЦИЯ

Безопасное и эффективное освоение месторождений полезных ископаемых подземным способом, залегающих на больших глубинах, осложняется тем, что с увеличением глубины ведения горных работ существенно изменяется характер протекания деформационных процессов в массиве пород и степень их влияния на окружающую среду. Исследования деформационных процессов, их контроль и прогноз определяют во многих случаях эффективность и безопасность разработки месторождений твердых полезных ископаемых. Практический прогноз может быть осуществлен в результате непрерывного слежения в пространстве и во времени за деформационными процессами. В данной статье представлены современные спутниковые радары и их основные характеристики. Описаны особенности метода радиолокационной интерферометрии, рассмотрены преимущества и недостатки различных методов интерферометрической обработки радиолокационных изображений. Проанализирован опыт использования космической радиолокации в целях мониторинга месторождений полезных ископаемых в Республике Казахстан. Был поднят вопрос о возможности интенсификации применения метода радиолокационной интерферометрии в горнодобывающей отрасли. Доказана возможность проведения регулярных полевых наблюдений на всей территории горнорудного комплекса, включая труднодоступные и опасные участки, независимо от погодных условий. Подтверждено, что применение метода радиолокационной интерферометрии определяет смещение земной поверхности с высокой точностью. При использовании этого метода получение и обработка данных производится дистанционно и в общем случае не требует присутствия на объекте. Кроме того, в данной статье приведены примеры успешного применения метода радиолокационной интерферометрии в зарубежных странах на горных, нефтяных объектах. Также применение радиолокационной интерферометрии позволяет оперативно определять зоны возможных деформаций земной поверхности и организовывать высокоточные маркшейдерско-геодезические наблюдения в этих зонах. В данной статье, используя радарные снимки Sentinel от 2016 года и метод радарной интерферометрии были определены сдвижения земной поверхности Анненского месторождения с высокой точностью и построена карта смещения.

Ключевые слова: радиолокационная интерферометрия, радиолокационное зондирование Земли, синтезированный апертурный радиолокатор, методы интерферометрической обработки, радарные датчики, деформация земной поверхности, горнодобывающая промышленность.

Информация об авторах:

Алтаева Асель Абдикеримкызы	*PhD, ассоциированный профессор кафедры Геодезия и картография, кадастр Международной образовательной корпорации, Алматы, Казахстан. Email: a.aselya_92@mail.ru; ORCID ID: https://orcid.org/0000-0002-1675-6828*
Садыков Батырхан Болатұлы	*PhD, ассоциированный профессор ТОО Leica Geosystems Казахстан, Алматы, Казахстан. Email: batyrkhan_sadykov@mail.ru; ORCID ID: https://orcid.org/0000-0003-2037-247X*
Умирбаева Алия Батухановна	*PhD, ассоциированный профессор кафедры Геодезия и картография, кадастр Международной образовательной корпорации, Алматы, Казахстан. Email: a_umirbayeva@mail.ru; ORCID ID: https://orcid.org/0000-0002-4746-3411*
Даркенбаева Асель Бериковна	*Докторант кафедры Земельных ресурсов и кадастра, Казахский национальный аграрный исследовательский университет, Алматы, Казахстан. Email: darkenbayevaasel@gmail.com; ORCID ID: https://orcid.org/0009-0001-7115-8951*
Дастан Анель	*Студент кафедры Геодезия и картография, кадастр Международной образовательной корпорации, Алматы, Казахстан. Email: dastan_a@mail.ru*

Reference

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Metallurgy

Enlarged tests on the processing of copper-lead mattes obtained after reductive smelting of balanced feed charge

[1]Dosmukhamedov N.K., [2*]Zholdasbay E.E., [2]Argyn A.A., [2]Icheva Yu.B., [1]Kurmanseitov M.B.

[1] Satbayev University, Almaty, Kazakhstan
[2] O.A. Baikonurov Zhezkazgan University, Zhezkazgan, Kazakhstan

* Corresponding author email: zhte@mail.ru

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ABSTRACT

The paper examines the behavior of copper, lead, zinc and arsenic during the oxidative blowing of intermediate copper-lead matte, which represents the second stage of the general technology for processing balanced raw materials for copper and lead production. The optimal parameters for the oxidative blowing of intermediate matte have been established: the time of blowing the melt with oxygen is 20 min; the oxygen consumption is 1.4 times higher than its consumption from the stoichiometrically required amount for the oxidation of zinc and iron sulfide; the temperature is 1250 °C. High indicators have been achieved for the complex selective extraction of metals into targeted products: lead into rough lead – 97.6%; copper into matte – 98.6%; zinc into slag – 56.8%, into matte – 1.7, into dust and gases – 41.5; arsenic and antimony into dust – up to 97.4% and 90%, respectively. A general process flowsheet has been developed for separate processing of balanced charges consisting of intermediate products of copper and lead production. The technology can be used for separate processing of multi-component raw materials of copper smelters and lead production of various types and compositions.

Keywords: copper-lead matte, copper, lead, zinc, arsenic, oxidizing blowing, melting, distribution.

	Information about authors:
Dosmukhamedov Nurlan Kalievich	*Candidate of Technical Sciences, Professor, Satbayev University, 050013, 22 Satbayev St., Almaty, Kazakhstan. E-mail: n.dosmukhamedov@satbayev.university; ORCID ID: https://orcid.org/0000-0002-1210-4363*
Zoldasbay Erzhan Esenbaiuly	*PhD, O.A. Baikonurov Zhezkazgan University, 100600, 1b Alashahan st., Zhezkazgan, Kazakhstan. E-mail: zhte@mail.ru; ORCID ID: https://orcid.org/0000-0002-9925-4435*
Argyn Aidar Abdilmalikuly	*PhD, O.A. Baikonurov Zhezkazgan University, 100600, 1b Alashahan st., Zhezkazgan, Kazakhstan. E-mail: aidarargyn@gmail.com; ORCID ID: https://orcid.org/0000-0001-5001-4687*
Icheva Yulianna Borisovna	*Candidate of Technical Sciences, O.A. Baikonurov Zhezkazgan University, 100600, 1b Alashahan St., Zhezkazgan, Kazakhstan. E-mail: isheva1967@mail.ru; ORCID ID: https://orcid.org/0000-0001-5914-9772*
Kurmanseitov Murat Bayrzhanuly	*PhD, Satbayev University, 050013, 22 Satbayev St., Almaty, Kazakhstan. E-mail: murat.kmb@mail.ru; ORCID ID: https://orcid.org/0000-0001-5008-2866*

Introduction

The growth of volumes of substandard intermediate products and recycled materials of lead production necessitates the search for new technologies for their processing. At present, in lead and copper production, in connection with the involvement of complex multi-component raw materials in the processing, the yield of intermediate products with a high content of impurity metals, such as lead, zinc, arsenic, etc., is sharply increasing [[1], [2], [3], [4], [5], [6]]. The use of this type of raw material in processing, particularly in lead production plants, has resulted in an increase in the yield of intermediate products and recycled materials containing higher levels of arsenic, antimony, and their toxic compounds. Currently, none of the processes in the lead production technological chain achieve a sufficiently high level of sublimation of arsenic and antimony into dust. This shortcoming limits their removal from the main production cycle, leading to a significant accumulation within the facility. The existing technologies are no longer capable of effectively processing these materials, resulting in arsenic buildup, a notable rise in material costs, deterioration of technological performance, and an increase in health issues for communities living near metallurgical plants.

Special attention is drawn to the process of converting copper-lead matte, aimed at obtaining rough copper. The technology of converting copper matte is well developed in practice and is sufficiently fully covered in the technical literature. Nevertheless, the issues of increasing the extraction of copper into rough copper and improving the quality of copper-lead matte conversion products still remain open.

We did not have the task of conducting a detailed analysis of the conversion process of copper-lead matte produced at the Ust-Kamenogorsk Metallurgical Complex (UK MC) of Kazzinc LLP. The interest in considering the issue is caused by the fact that conversion can be considered as the final stage of processing an intermediate product – matte, obtained by separate processing of intermediates and recycled materials.

The processing of copper-lead matte at the UK MC is accompanied by a number of technological features, which we reported in previously published works. The conducted studies have established that the low extraction of copper into rough copper (~ 80%) is accompanied by an increased (up to 15%) distribution of copper into converter slag, and an insignificant (up to 5%) transition of it into dust.

In the process of converting copper-lead matte into rough copper, up to 1.5% of lead passes. The extraction of lead into dust is at a low level and is 40%. Up to 60% of the total amount of lead is concentrated in the converter slag. During conversion, galena present in matte is easily oxidized to form lead oxide and sulfurous anhydride. Lead oxide binds to silica to form lead silicate.

Zinc is distributed mainly between converter slag and dust: up to 80% of zinc is concentrated in converter slag.

The situation is somewhat different with the distribution of arsenic and antimony. During conversion, these impurities are distributed between converter slag, dust and rough copper. The main part of arsenic – up to 70%, passes into dust. 22% of arsenic is concentrated in the converter slag and ~ 7% of it is distributed into rough copper.

26.2% of antimony passes into rough copper, which is four times higher than that of arsenic. As a result of the low sublimation of antimony during conversion, its distribution into dust is insignificant, and amounts to only 40%. The remaining part of antimony - up to 36%, is concentrated in the converter slag.

From the results obtained, the following can be concluded. When converting copper-lead matte having a complex chemical and phase composition, it is not possible to achieve an optimal distribution of non-ferrous metals and related impurities (As, Sb) between the conversion products. In order to achieve optimal technological indicators for the distribution of metals during conversion, it is necessary either to significantly improve the quality of the matte before conversion, or already in the conditions of conducting the conversion process, to provide additional technological measures to improve the quality of products. In our opinion, the first option seems to be the most promising.

When organizing the technology of separate processing of intermediates and recycled materials, it is necessary to strive to obtain high-quality copper matte, ensuring a minimum content of Pb, Zn, As, Sb in it. In this regard, the existing technology at UK MC cannot be considered as a prospect for the future, and requires drastic changes.

One of the solutions to this problem is the development of technology for separate processing of copper- and lead-containing products, where various pyro- and hydrometallurgical processes are used [[7], [8], [9], [10], [11], [12], [13], [14], [15], [16], [17], [18]].

At KazNITU named after K.I. Satbayev, under the supervision of Professor Dosmukhamedov N.K., researchers have worked for several years with scientists from the Moscow Institute of Steel and Alloys (MISiS) and the Weizmann Institute of Science in Israel, Rehovot. They are conducting thorough research to develop new technology for processing intermediate products and recycled materials from copper and lead production. Based on the positive results of fundamental research, a comprehensive technology has been developed aimed at processing multi-component balanced raw materials and selective extraction of valuable metals into targeted products [[19], [20]].

The core *methodological principle* of the developed technology is a thermodynamic approach to modeling bubbling metallurgical processes. This approach ensures the accurate determination of the qualitative and quantitative characteristics of the resulting complex condensed (liquid) and vapor-gas phases, based on specific input parameters of the pyrometallurgical bubbling process. These parameters include the compositions and quantities of the initial materials, temperature conditions, oxidation-reduction potentials, and others.

The thermodynamic approach is grounded in the concept of reaching thermodynamic equilibrium (or a state near equilibrium) between the smelting products. This enables the application of chemical thermodynamics laws to predict the behavior of both the main and impurity components during the melting process. The basis for the possibility of using the equilibrium approach to describe processes occurring under conditions of intensive mixing of a multicomponent melt of complex composition are high rates of mass and heat transfer in them, and the conditions for the formation and separation of phases.

The basis for the developing of the general concept of the new bubbling technology is the separate **two-stage processing** of a balanced feed charge consisting of intermediate products and recycled materials from non-ferrous metallurgy.

At the first stage, selective separation of metals into targeted products is carried out in an electric furnace: copper into copper-lead matte; lead into rough lead; zinc into slag, and partial sublimation of rare, rare earth and dispersed metals into dust.

At the second stage, in an oxidation-reduction furnace with submerged tuyeres, a sufficiently deep extraction of lead (up to 98%) is carried out from copper-lead matte into rough lead, and copper is extracted into commercial copper matte with a high copper content (up to 70%). Zinc is concentrated in slag, and rare, rare earth and scattered metals pass into dust.

Studies on the behavior of non-ferrous and associated impurity metals during the smelting of a balanced charge in an electric furnace are covered in detail in [[19], [20]].

The aim of the present research is to study the distribution of copper, lead, zinc and arsenic between the products of oxidative blowing of intermediate copper-lead matte obtained after the reduction smelting of a balanced batch based on copper- and lead-containing products in an electric furnace.

Research methods

The object of the study is copper-lead matte obtained after reductive smelting of a balanced charge with natural gas of the following composition, % by weight: 54.6 Cu; 2.23 Pb; 12.4 Zn; 6.2 Fe; 0.19 As; 22.2 S.

Copper-lead matte was subjected to air blowing at different air consumption and time. Air consumption was calculated based on the condition of its consumption from the stoichiometric required amount (SRA) for complete oxidation of lead, zinc and iron sulfides.

In the experiments, the oxygen consumption varied within the range from 0.9 to 1.5 of the SRA, the melt blowing time was changed from 5 to 20 minutes. Based on the obtained results, the optimal process parameters were determined, ensuring high technological indicators for the extraction of non-ferrous metals into targeted commercial products.

The methodology for conducting the experiments was as follows.

A crucible containing an initial sample of 500 g was placed into a quartz reactor, which was then loaded into a furnace. Once the specified temperature of 1523 K was reached, the melt was held for 10 minutes to ensure homogeneity. Afterward, the melt was subjected to oxygen blowing for a specified duration. Oxygen consumption varied between 1 and 1.4 fractions of SRA for the oxidation of zinc and iron sulfides. The blowing time was set at 5, 10, 15, and 20 minutes. Once the oxygen blowing was completed, the tube was raised above the melt, and the furnace was allowed to cool. After cooling, the crucible with the sample was removed from the quartz reactor. The resulting smelting products, matte and slag, were separated and analyzed for metal content. The elemental composition of the melt products was determined using a scanning electron microscope equipped with a JED-2300 (JEOL) energy-dispersive X-ray spectrometer. The obtained diffraction data were processed, and the interplanar distances were calculated using the EVA software. Sample decoding and phase identification were performed with the Search/Match program, utilizing the PDF-2 powder diffractometric database for reference.

In order to obtain accurate data on the elemental composition of dust, an additional analysis was carried out using a D8 Advance spectrometer.

The dust yield in all experiments was determined by calculating the difference between the initial sample weight and the combined weight of the obtained smelting products.

Each experiment was repeated three times for reproducibility. The results of parallel experiments on the content of metals in the smelting products showed good convergence (error +/- 0.5% abs.).

Based on the average results of product yield and metal content in them, the material balance of the oxidative smelting of copper-lead matte was calculated.

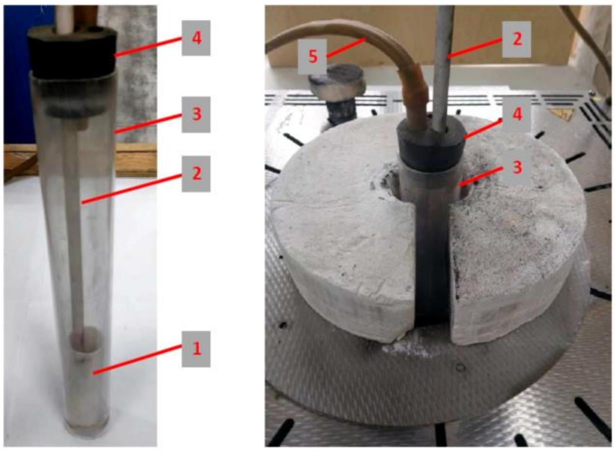
Results and discussion

The general view of the enlarged laboratory setup is shown in Fig. 1.

Table 1 shows the calculated material balance of the oxidative smelting of copper-lead matte with oxygen.

During oxidative smelting of matte with air, the yield of products was, % (of the total charge): matte – 49.8; slag – 12.1; dust, gases – 38.1.

The patterns of distribution of copper, lead, zinc and arsenic between smelting products under conditions of oxidative blowing of matte are shown in Fig. 2-5.



1 – crucible with charge; 2 – alundum tube for blowing the melt;
3 – quartz reactor; 4 – plug; 5 – gas outlet hose

Figure 1 - General view of the installation for reaching final copper-lead matte

Table 1 – Material balance of oxidative smelting of intermediate matte with air at optimal parameters: oxygen consumption – 1.4 times exceeding its consumption from the SRA for oxidation of lead, iron and zinc sulfides; blowing time – 20 min.; T = 1250 °C

Products name	**Quantity**		**Cu**			**Pb**			**Zn**			**Fe**			**As**		
	g.	**%**	**I**	**II**	**III**	**I**	**II**	**III**	**I**	**II**	**III**	**I**	**II**	**III**	**I**	**II**	**III**
Loaded:																	
Matte	115.83	68.9	64.0	55.3	100.0	2.6	2.3	100.0	14.5	12.5	100.0	6.3	5.5	100.0	0.15	0.1	100.0
Air	44.53	26.5															
Flux	7.81	4.6															
Total:	**168.18**	**100.0**	**64.0**		**100.0**	**2.6**		**100.0**	**14.5**		**100.0**	**6.3**		**100.0**	**0.15**		**100.0**
Received:																	
Matte	83.84	49.8	63.9	76.3	99.9	0.8	0.9	30.0	0.4	0.5	3.0	0.2	0.2	3.0	0.05	0.1	30.0
Slag	20.34	12.1	0.1	0.1	0.1				2.8	13.6	19.0	6.2	30.3	97.0			
Dust. gases	64.00	38.1				1.8	2.9	70.0	11.3	17.7	78.0				0.10	0.2	70.0
Total:	**168.18**	**100.0**	**64.0**		**100.0**	**2.6**		**100.0**	**14.5**		**100.0**	**6.3**		**100.0**	**0.15**		**100.0**

Products name	**Sb**			**S**			**O**			**N_2**			**SiO_2**			**Others**			**Total:**
	I	**II**	**III**	**I**	**II**	**III**	**I**	**II**	**III**	**I**	**II**	**III**	**I**	**II**	**III**	**I**	**II**	**III**	
Loaded:																			
Matte	0.03	0.0	100.0	25.5	22.0	100.0										2.7	2.3	100.0	115.8
Air							9.4	21.0	100.0	35.2	79.0	100.0							44.5
Flux													5.8	74.8	100.0	2.0	25.2		7.8
Total:	**0.03**		**100.0**	**25.5**		**100.0**	**9.4**		**100.0**	**35.2**		**100.0**	**5.8**		**100.0**	**4.7**		**100.0**	**168.18**
Received:																			
Matte	0.03	0.03	100.0	17.1	20.4	67.0										1.3	1.6	28.9	83.8
Slag							2.9	14.1	30.6				5.8	28.7	100.0	2.7	13.1	57.0	20.3
Dust. gases				8.4	13.1	33.0	6.5	10.1	69.4	35.2	55.0	100.0				0.7	1.0	14.1	64.0
Total:	**0.03**		**100.0**	**25.5**		**100.0**	**9.4**		**100.0**	**35.2**		**100.0**	**5.8**		**100.0**	**4.7**		**100.0**	**168.18**

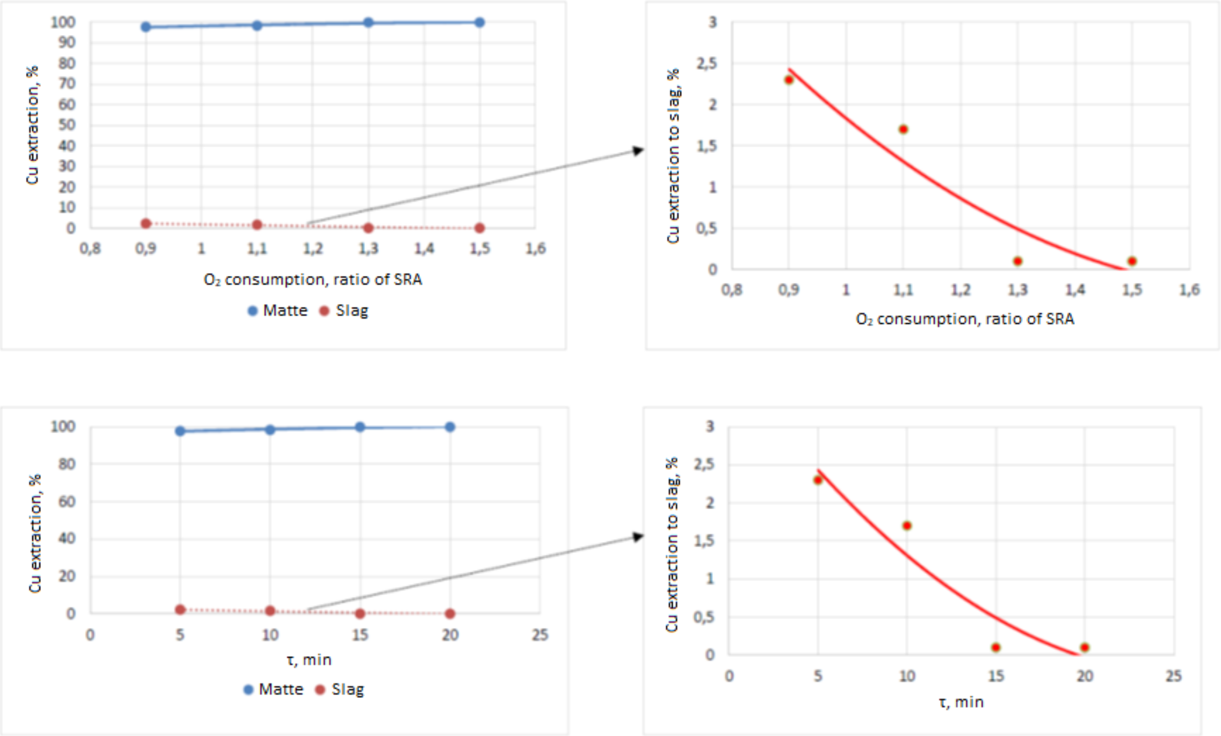


Figure 2 – Effect of oxygen consumption (ratio of SRA for oxidation of Pb, Zn, Fe sulfides) and the melt blowing time (τ, min) for copper extraction into smelting products

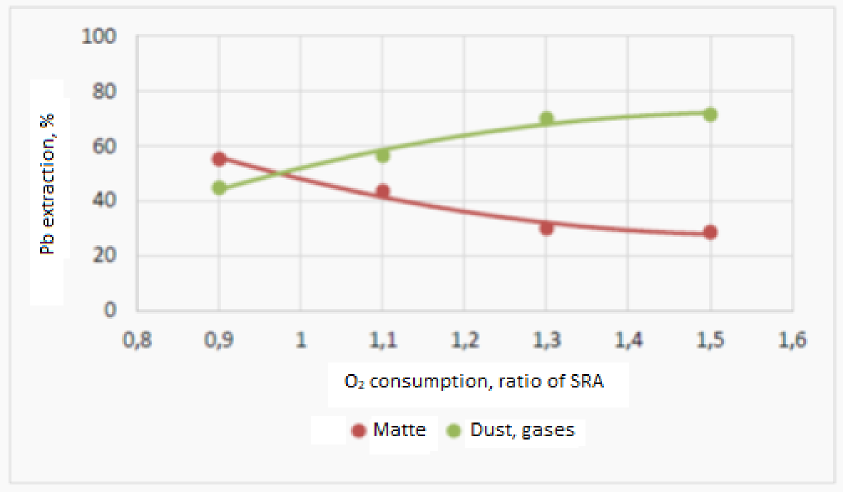


Figure 3 – Effect of oxygen consumption (ratio of SRA for oxidation of Pb, Zn, Fe sulfides) for the extraction of lead into smelting products

The best results for the complex selective extraction of copper, lead, zinc, and arsenic into the targeted smelting products were achieved with an oxygen consumption rate 1.4 times higher than the consumption from the SRA for the oxidation of zinc and iron sulfides, followed by their transfer as oxides into the slag. The matte blowing duration was set to 20 minutes. Under optimal process conditions, copper extraction into matte exceeded 98%, lead extraction into matte and dust was 30% and 70%, respectively, and zinc extraction into matte, slag, and dust was 3%, 19%, and 78%, respectively.

The results of the studies demonstrated the fundamental feasibility of separately processing copper-lead matte by intensively blowing it with air to produce commercial copper matte with a copper content of 76.3% (Table 1). These findings represent the final second stage in the overall technology for processing a balanced charge, which consists of copper-lead-containing intermediate products and recycled materials from copper and lead production. This process allows for the selective extraction of non-ferrous metals into targeted commercial products.

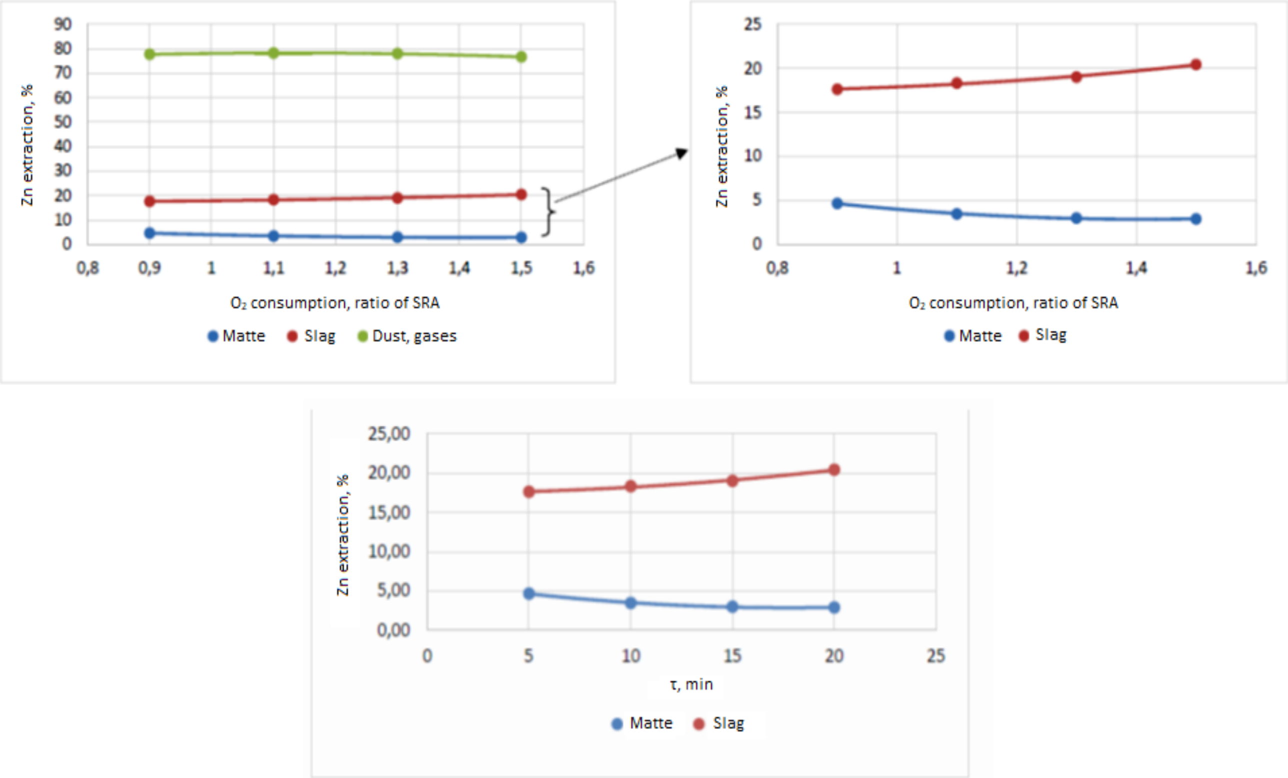


Figure 4 – Effect of oxygen consumption (ratio of SRA for oxidation of PbS, ZnS, FeS) and the melt blowing time (τ, min) for the extraction of zinc into the smelting products

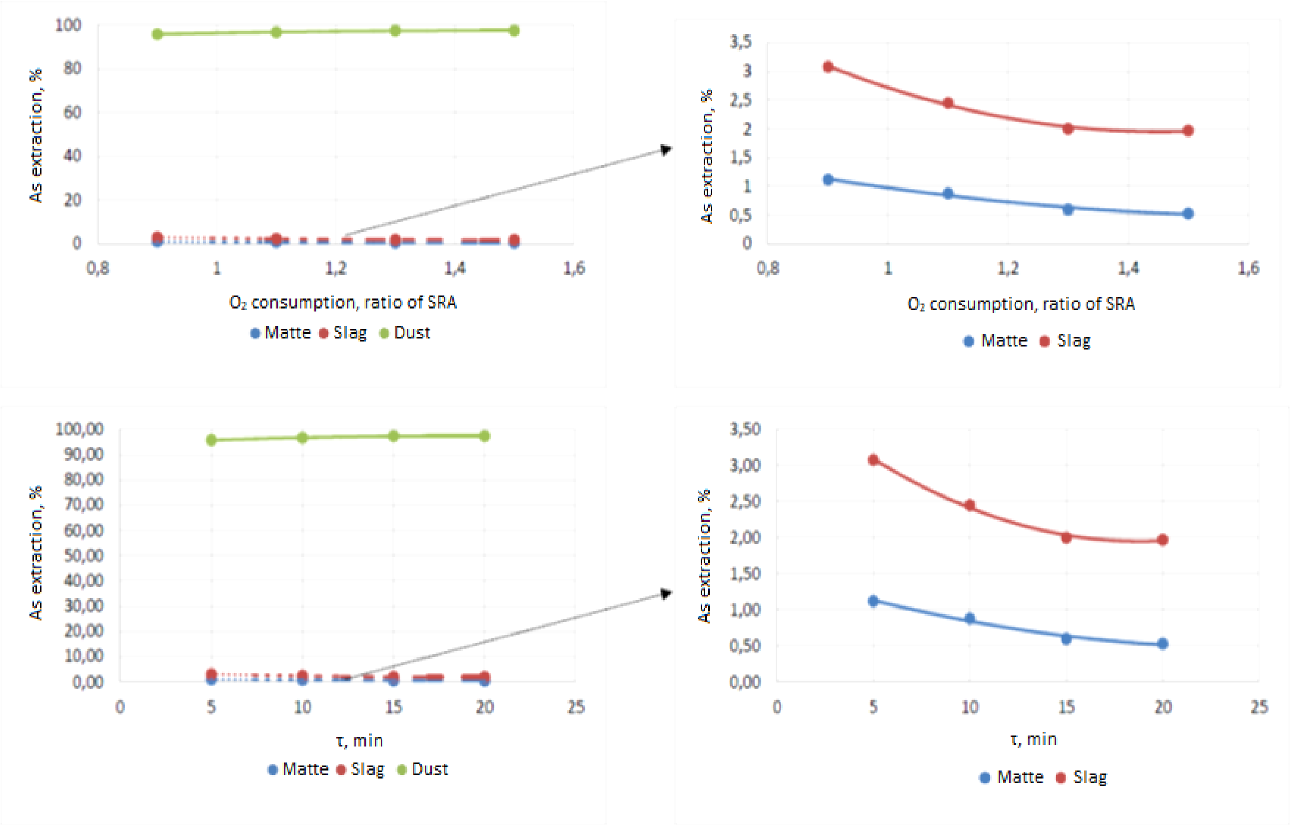


Figure 5 – Effect of oxygen consumption (ratio of SRA for oxidation of PbS, ZnS, FeS) and the melt blowing time (τ, min) for the extraction of arsenic into the melt products

For industrial implementation of the general reduction-oxidation technology for processing balanced charge the following parameters are recommended.

For the first stage – the recovery smelting of the balanced charge:

– melt blowing time:

- natural gas – 20 min;
- consumption of CH_4 is 1.7 times higher than its consumption from the stoichiometric required amount for the reduction of lead compounds;
- melting temperature – 1250 °C.

For the second stage – oxidative blowing of intermediate copper-lead matte:

- melt blowing time – 20 minutes;
- oxygen consumption is 1.4 times higher than its consumption from the stoichiometric required amount for the oxidation of zinc and iron sulfide;
- melting temperature – 1250 °C.

The implementation of the technology ensures the production of high-quality targeted products of the following compositions at the first stage:

- rough lead. % by weight: 99.34 Pb; 0.18 Cu; 0.08 Sb; other.
- Copper-lead matte, wt.%: 55.25 Cu; 2.25 Pb; 12.54 Zn; 5.48 Fe; 22.0 S; 0.13 As; 0.03 Sb; other.
- Slag, wt.%: 20.45 Fe; 18.58 SiO_2; 4.0 CaO; 8.33 Zn; 0.16 Cu; 1.11 Pb; others.

In the second stage:

- Copper -lead matte, wt.%: 76.3 Cu; 0.93 Pb; 0.52 Zn; 0.23 Fe; 20.4 S; 0.05 As; 0.03 Sb; other.
- Slag, wt.%: 30.3 Fe; 28.7 SiO_2; 6.0 CaO; 13.6 Zn; 0.19 Cu; others.

Total recovery of metals into targeted products:

– lead in rough lead – 97.6%;

– copper in matte – 98.6%;

- zinc:

into slag – 56.8%;

in matte – 1.7%;

into dust and gases – 41.5%;

– arsenic and antimony in dust – 97.4% and 90%, respectively.

The proposed general process flow diagram for the reduction-oxidation smelting of a balanced charge with the complex extraction of copper, lead and zinc into targeted commercial products is presented in Fig. 6.

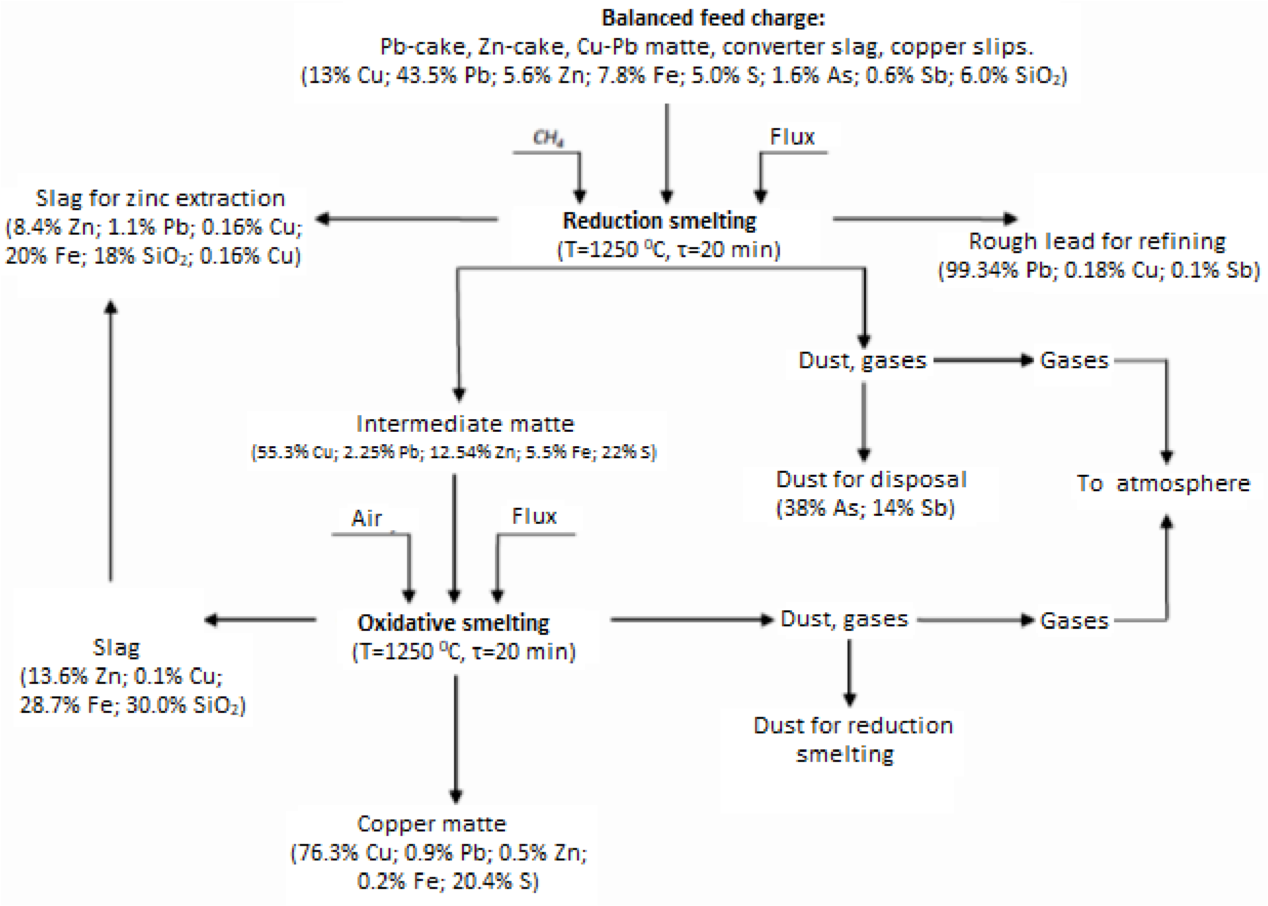


Figure 6 – Flow diagram of the technology for processing balanced charge, compiled on the basis of intermediate products and recycled materials of copper and lead production

The developed technology can be used for separate processing of balanced feed charge, composed on the basis of multi-component intermediate products and recycled materials of non-ferrous metallurgy of different types and compositions.

Conclusions

1. The feasibility of implementing reduction-oxidation smelting of a balanced charge has been demonstrated. The optimal technological parameters for oxidative blowing of intermediate matte have been established: the melt is blown with oxygen for 20 minutes, oxygen consumption is 1.4 times higher than its consumption from the SRA for the oxidation of zinc and iron sulfides, and the temperature is maintained at 1250 °C.

2. Under optimal process parameters, the following product yields were obtained as a percentage of the total charge: rough lead – 38.3%, copper matte – 15%, slag – 31.2%, and dust/gases – 15.5%. The copper matte produced had a high copper content (over 76%) with minimal impurities, including 0.93% Pb, 0.52% Zn, 0.23% Fe, 0.05% As, and 0.03% Sb.

3. High efficiency has been achieved in the complex selective extraction of metals into targeted products, with the following extraction rates: lead into rough lead – 97.6%; copper into matte – 98.6%; zinc into slag – 56.8%, into matte – 1.7%, and into dust and gases – 41.5%; arsenic and antimony into dust – up to 97.4% and 90%, respectively.

4. Based on the collected data, a basic technological scheme for processing a balanced batch composed of a complex conglomerate of a mixture of intermediate products from copper and lead production was developed.

Conflict of interest. On behalf of all authors, the corresponding author confirms that there is no conflict of interest.

CRediT author statement: **N. Dosmukhamedov:** Supervision, Conceptualization; **E. Zoldasbay:** Investigation, Data curation; **A. Argyn:** Methodology, Writing-Original draft preparation; **Y. Icheva:** Investigation, Software, Validation; M**. Kurmanseitov:** Writing- Reviewing.

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Теңдестірілген шикіқұрамды тотықсыздандырып балқытқаннан кейін алынған мыс-қорғасын штейндерін қайта өңдеу бойынша ірілендірілген сынақтар

[1]Досмухамедов Н.К., [2]Жолдасбай Е.Е., [2]Арғын А.Ә., [2]Ичева Ю.Б., [1]Құрмансейтов М.Б.

[1]Сәтбаев университеті, Алматы, Қазақстан
[2]Ө.А. Байқоңыров атындағы Жезқазған университеті, Жезқазған, Қазақстан

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ТҮЙІНДЕМЕ
Жұмыста мыс және қорғасын өндірісінің теңдестірілген шикізатын өңдеуге арналған жалпы технологияның екінші сатысындағы аралық мыс-қорғасын штейнін тотықтырып үрлеу кезінде мыс, қорғасын, мырыш және мышьяктың бөлініп таралуы зерттелді. Аралық штейнді тотықтырып үрлеудің оңтайлы параметрлері белгіленді: балқыманы оттегімен үрлеу уақыты – 20 мин; оттегі шығыны – мырыш пен темір сульфидін тотықтыру үшін СҚМ-рі шығыннан 1,4 есе артық; температура-1250 °С. Мақсатты өнімдерге металдарды кешенді селективті бөліп алу бойынша: қорғасынды тазартылмаған қорғасынға – 97,6%; мысты штейнге – 98,6%; мырышты қожға – 56,8%, штейнге – 1,7%, шаңға, газдарға – 41,5%; мышьяк пен сурьманы шаңға – сәйкес 97,4% және 90% дейінгі жоғары көрсеткіштерге қол жеткізілді. Мыс және қорғасын өндірісінің жартылай өнімдерінен тұратын теңдестірілген шикіқұрамды бөлек өңдеуге арналған жалпы технологияның сұлбасы әзірленді. Технологияны мыс балқыту және қорғасын өндірісінің көп компонентті шикізатының түрі мен құрамы бойынша бөлек өңдеу үшін пайдалануға болады.

Түйін сөздер: мыс-қорғасын штейні, мыс, қорғасын, мырыш, мышьяк, тотықтырып үрлеу, балқыту, бөлініп таралу.

	Авторлар туралы ақпарат:
Досмухамедов Нурлан Калиевич	*Т.ғ.к., профессор, Сәтбаев университеті, 050013, Сатпаев көш. 22, Алматы, Казақстан. E-mail: n.dosmukhamedov@satbayev.university; ORCID ID: https://orcid.org/0000-0002-1210-4363*
Жолдасбай Ержан Есенбайұлы	*PhD, Ө.А. Байқоңыров атындағы Жезқазған университеті, 100600, Алашахан көш. 1б, Жезқазған, Казақстан. E-mail: zhte@mail.ru; ORCID ID: https://orcid.org/0000-0002-9925-4435*
Арғын Айдар Әбділмәлікұлы	*PhD, Ө.А. Байқоңыров атындағы Жезқазған университеті, 100600, Алашахан көш. 1б, Жезқазған, Казақстан. E-mail: aidarargyn@gmail.com; ORCID ID: https://orcid.org/0000-0001-5001-4687*
Ичева Юлианна Борисовна	*Т.ғ.к., Ө.А. Байқоңыров атындағы Жезқазған университеті, 100600, Алашахан көш. 1б, Жезқазған, Казақстан. E-mail: isheva1967@mail.ru; ORCID ID: https://orcid.org/0000-0001-5914-9772*
Құрмансейтов Мұрат Бауыржанұлы	*Магистр, Сәтбаев университеті, 050013, Алматы, Сәтбаев көш. 22, Алматы, Казақстан. E-mail: murat.kmb@mail.ru; ORCID ID: https://orcid.org/0000-0001-5008-2866*

Укрупненные испытания по переработке медно-свинцовых штейнов, полученных после восстановительной плавки сбалансированной шихты

[1]Досмухамедов Н.К., [2]Жолдасбай Е.Е., [2]Аргын А.А., [2]Ичева Ю.Б., [1]Курмансейтов М.Б.

[1] Satbayev University, Алматы, Казахстан
[2]Жезказганский университет имени О.А. Байконурова, Жезказган, Казақстан

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АННОТАЦИЯ

В работе исследовано поведение меди, свинца, цинка и мышьяка при окислительной продувке промежуточного медно-свинцового штейна, представляющей вторую ступень общей технологии для переработки сбалансированного сырья медного и свинцового производства. Установлены оптимальные параметры окислительной продувки промежуточного штейна: время продувки расплава кислородом – 20 мин; расход кислорода – 1,4 раза превышающий его расход от СНК для окисления сульфида цинка и железа; температура – 1250 °С. Достигнуты высокие показатели по комплексному селективному извлечению металлов в целевые продукты: свинца в черновой свинец – 97,6%; меди в штейн – 98,6%; цинка в шлак – 56,8%, в штейн – 1,7, в пыль, газы – 41,5; мышьяка и сурьмы в пыль – до 97,4% и 90%, соответственно. Разработана общая технологическая схема для раздельной переработки сбалансированной шихты, составленной из полупродуктов медного и свинцового производства. Технология может быть использована для раздельной переработки различного по типу и составу многокомпонентного сырья медеплавильного и свинцового производства.

Ключевые слова: медно-свинцовый штейн, медь, свинец, цинк, мышьяк, окислительная продувка, плавка, распределение.

	Информация об авторах:
Досмухамедов Нурлан Калиевич	*К.т.н., профессор, Satbayev University, 050013, ул. Сатпаева 22, Алматы, Казахстан. E-mail: n.dosmukhamedov@satbayev.university; ORCID ID: https://orcid.org/0000-0002-1210-4363*
Жолдасбай Ержан Есенбайулы	*PhD, Жезказганский университет имени О.А. Байконурова, 100600, ул. Алашахана 1б, Жезказган, Казахстан. E-mail: zhte@mail.ru; ORCID ID: https://orcid.org/0000-0002-9925-4435*
Аргын Айдар Абдилмаликулы	*PhD, Жезказганский университет имени О.А. Байконурова, 100600, ул. Алашахана 1б, Жезказган, Казахстан. E-mail: aidarargyn@gmail.com; ORCID ID: https://orcid.org/0000-0001-5001-4687*
Ичева Юлианна Борисовна	*К.т.н., Жезказганский университет имени О.А. Байконурова, 100600, ул. Алашахана 1б, Жезказган, Казахстан. E-mail: isheva1967@mail.ru; ORCID ID: https://orcid.org/0000-0001-5914-9772*
Курмансейтов Мурат Бауыржанулы	*PhD, Satbayev University, 050013, Алматы, ул. Сатпаева 22, Казахстан. E-mail: murat.kmb@mail.ru; ORCID ID: https://orcid.org/0000-0001-5008-2866*

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Metallurgy

Study of the dispersing properties of microemulsion mercaptan-containing collectors based on oil products

[1]Kenzhaliyev B.K., [1*]Mukhanova A.A., [1]Samenova N.O., [2]Toktagulova K.N.,
[1]Kaldybaeva J.A., [1]Turysbekov D.K., [1]Yussupova S.Zh.

[1] Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Almaty, Kazakhstan
[2] JSC CNPC Aktobemunaigas, Aktobe, Kazakhstan

** Corresponding author email: a.mukhanova@satbayev.university*

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ABSTRACT
The article discusses the production of flotation reagents based on domestic sulfur-containing oil products using the ultrasonic cavitation method. Particular attention is paid to the selection of oil products based on the analysis of their qualitative and quantitative composition. Mercaptans (thioalcohols) are known to be the most effective sulfhydryl collectors. This study utilizes sulfur-containing oil products with the same sulfur content but differing in the form of sulfur presence in the starting materials: refined oil, mercaptan-containing product, and an intermediate product obtained during oil demercaptanization. Compositions were developed using these oil products and butyl xanthate. During ultrasonic cavitation, the dependence of emulsion stability over time on ultrasonic power and component ratios was determined. Optimal conditions for ultrasonic cavitation and the appropriate proportions of components in the compositions were established. Despite having the same sulfur content, it was found that only sulfur-containing products with specific structural characteristics are suitable for flotation reagent production. Additionally, ultrasonic cavitation's role in modifying the compositions' properties was identified, significantly influencing their efficiency as collectors.

Keywords: refined oil, mercaptan-containing product, butyl xanthate.

Information about authors:

Kenzhaliyev Bagdaulet Kenzhaliyevich	*Doctor of Technical Sciences, Professor, General Director-Chairman of the Management Board of the Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: bagdaulet_k@satbayev.university; ORCID ID: https://orcid.org/0000-0003-1474-8354*
Mukhanova Aynur Aitkazinovna	*Candidate of Technical Sciences, Leading Researcher of the Laboratory of Flotation Reagents and Enrichment, Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: a.mukhanova@satbayev.university; ORCID ID: https://orcid.org/0000-0002-2130-683X*
Samenova Nazira Orakkyzy	*Researcher, Laboratory of Flotation Reagents and Beneficiation, Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: nazira.orakkyzy@gmail.com; ORCID ID: http://orkid.org/0000-0002-3027-5246*
Toktagulova Kamshat Nadirovna	*Engineer chemist of 2 categories of the CNPC Aktobemunaigas JSC, Aktobe, Kazakhstan. Email: nadirovna_k@mail.ru*
Kaldybayeva Zhamikhan Abdildabekovna	*Junior Researcher, Laboratory of Flotation Reagents and Beneficiation, Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: zh.kaldybaeva@satbayev.university; ORCID ID: https://orcid.org/0000-0001-5763-9655*
Turysbekov Dulatbek Kadyrbekuly	*Candidate of Technical Sciences, Leading research of the laboratory of flotation reagents and Ore Benefication, Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: d.turysbekov@satbayev.university; ORCID ID: https://orcid.org/0000-0003-0904-1565*
Yussupova Saniya Zhabborovna	*Engineer of the Laboratory of Flotation Reagents and Beneficiation, Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: Saniya.yusupova02@mail.ru; ORCID ID: https://orcid.org/0009-0009-1728-3418*

Introduction

Flotation reagents play a key role in the flotation process, determining its efficiency and future development prospects. The success of flotation largely depends on their proper selection and economically justified application. One of the key characteristics of flotation reagents is their selective influence on phase boundaries, particularly on the surface of various minerals, due to the conditions created in the aqueous pulp medium. Of particular interest is the combined use of ionic and apolar collectors, which demonstrate high flotation efficiency [[1], [2], [3]].

In recent years, research by domestic scientists has intensified interest in the use of heteroorganic oil compounds for the beneficiation of polymetallic ores. Technologies are actively being developed to extract sulfur-containing compounds from oil products, including mercaptans, sulfides, and thiophenes, with the latter accounting for 80–95% of the total. It has been established that oil sulfides are predominantly represented by cyclic compounds, homologs of alkyl-substituted thiophenes, and thiocycloalkanes. As the boiling point of oil fractions increases, the proportion of condensed monocyclic sulfides decreases, while that of polycyclic sulfides rises. These compounds exhibit high surface activity, making them promising apolar reagents that do not require additional surfactants.

Studies by V. A. Glembotsky, L. Ya. Shubov, V. A. Esepkin, S. I. Chernykh, and others have shown that the most effective apolar reagents contain aromatic hydrocarbons and their homologs. These findings highlight the need for a more in-depth investigation of such compounds as flotation reagents.

This study aims to evaluate the potential use of sulfur-rich oil concentrates containing aromatic and heteroatomic compounds as selective apolar collectors. These substances remain insufficiently studied in the context of ore flotation [4].

Many collectors possess a complex heteropolar molecular structure, incorporating both polar and apolar fragments. The polar part of the molecule interacts with water, while the apolar part, represented by a hydrocarbon radical, exhibits hydrophobic properties. During adsorption on the mineral surface, the polar part of the molecule attaches to the mineral, while the apolar part extends into the aqueous phase, facilitating hydrophobization and subsequent flotation of the mineral [5].

The selective properties of collectors depend on their chemical composition and molecular structure. They are classified based on their interaction mechanism with minerals, their degree of dissociation into ions, and the type of ion responsible for the hydrophobizing effect (anion or cation). Collectors are generally divided into three main groups (Figure 1):

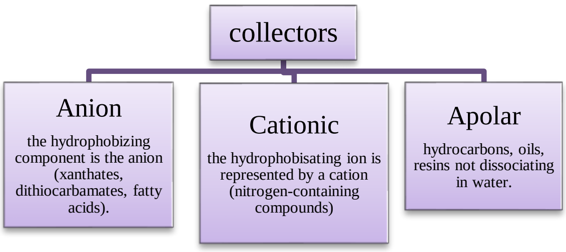

Figure 1 - A main group of collectors

One of the key directions for improving flotation efficiency is the development of new sulfur- and phosphorus-containing reagents that ensure high performance while reducing consumption.

In Kazakhstan, the production of flotation reagents, particularly collectors and foaming agents, is limited. Imported reagents are often expensive, of inconsistent quality, and have restricted applicability to different ore types. This situation necessitates the development of domestically produced flotation reagents from local raw materials, as well as the optimization of reagent regimes for their effective use in beneficiation processes [6].

According to the literature, xanthates, dithiophosphates, dixanthates, and their mixtures are widely used as collectors. For instance, butyl xanthate is the primary reagent in Russia, while amyl xanthate is more commonly used abroad [[7], [8], [9]]. It has been established that an increase in the hydrocarbon radical enhances the reactivity and oxidizability of xanthates [10].

For selective flotation, weak collectors such as aeroflots, or their combinations with xanthates, are recommended. The research of A. Abramov focuses on the development of new selective collecting reagents to improve the flotation efficiency of non-ferrous metals [11].

Flotation reagents used in mineral processing can be derived from crude oil. Crude oil contains not only water and salts but also a wide range of organic compounds, including sulfur-containing components, which are of particular interest for flotation. Compounds such as mercaptans, sulfides, and thiophenes have the potential to serve as effective collector reagents with selective effects on minerals.

The extraction of such compounds can be integrated into the early stages of oil processing, particularly during the breakdown of stable water-in-oil emulsions. This process offers a dual advantage: on one hand, the removal of these compounds enhances oil quality and reduces corrosiveness caused by sulfur-containing components; on the other, the recovered compounds can be utilized in the synthesis of innovative flotation reagents, which is especially valuable for polymetallic ore beneficiation [[12], [13], [14]].

Thus, the development of technologies for extracting sulfur-containing components from oil presents both environmental and industrial benefits. Environmentally, it improves oil properties and reduces pollution; industrially, it enables the creation of cost-effective and highly efficient flotation reagents from local raw materials.

The presented data confirm that advancing the composition of sulfur-containing reagents used as flotation collectors is a dynamic area of research. However, the challenge of selecting suitable initial components and optimizing their compositions for effective flotation of refractory ores remains unresolved. This is because reagent efficiency is directly influenced by the qualitative and quantitative composition of the raw material, as well as its structural characteristics, necessitating an individualized approach in each specific case [15].

Additionally, with the declining quality of raw materials and increasing flotation reagent consumption, reducing costs becomes essential. In this context, the synthesis of flotation reagents from inexpensive domestic raw materials, such as sulfur-containing oil products, is of particular importance, offering opportunities for the development of more economical and efficient flotation technologies [16].

Experimental part

For this study, three samples of oil and oil products from the Kenkiyak deposit, provided by Aktobeoilgas, were selected. The samples included dehydrated oil, purified oil, mercaptan-containing products, and a byproduct obtained during oil demercaptization.

At this stage of the research, the focus was on the raw oil from the Kenkiyak deposit and oil that had undergone the first stage of treatment. The physical and chemical characteristics, as well as the composition of oil with a water content of up to 30%, are presented in Table 1.

Table 1 - Physical and chemical characteristics of oil in the Kenkiyak deposits

Description of indicators	Averaged values
Viscosity, MPa × ^{0}C, at 200 ^{0}C - 500 ^{0}C	4.13 – 2.18
Density, g/cm^3	0.8259
Asphaltene, %	0.4
Paraffin, %	3.3
Pour point, 0S	8.70 ~ 14.0
Mass fraction of sulfur, %	0.70 ~ 1.21

The data presented in Table 1 indicate that the composition of the source oil is characterized by a sulfur content exceeding 1%. Sulfur is present in the form of both hydrogen sulfide and mercaptans. To effectively remove stable sulfur-containing compounds, a second purification stage is employed, utilizing specialized reagents—absorbers. This method is currently the most effective for reducing sulfur-containing components such as hydrogen sulfide and mercaptans in oil.

This study examined oil after the first and second refining stages. The first stage of oil treatment, based on existing technology, includes heating followed by electrostatic dehydration. This process relies on the destruction of petroleum emulsions through a thermochemical method combined with exposure to a high-voltage electric field.

The thermochemical method involves preheating the oil to a temperature of 48–50 °C with the addition of a demulsifier. As the temperature rises, the oil's viscosity decreases, facilitating better mixing of the emulsion with the demulsifier and enhancing the separation of oil and water. An essential condition for effective separation is the residence time of the emulsion in the apparatus. The demulsifier penetrates the surface layer of water droplets in the oil, breaking the protective coating composed of asphaltenes, resins, and paraffins. This process promotes the coalescence (fusion) of water droplets. The degree of dehydration achieved through thermochemical treatment ranges from 80% to 90% [17].

More stable water-in-oil emulsions are disrupted using a high-voltage electric field of 20–25 kV at an industrial frequency of 50 Hz in an electric dehydrator. Under the influence of an alternating electric field, dispersed water droplets in the oil coalesce, merging with other droplets and increasing in size. The enlarged water droplets settle in the intermediate layer below the lower electrode before passing into the drainage water layer.

When subjected to an electric field between two horizontally positioned lattice electrodes, the protective shell of the water emulsion droplets breaks down. As a result, water and the dissolved salts it contains separate from the oil due to differences in density and accumulate at the bottom of the electric dehydrator. Dissolved mineral salts are also removed from the oil along with the separated water [18].

Mercaptans can be considered hydrogen sulfide derivatives in which one hydrogen atom is replaced by a hydrocarbon radical. Their general formula is RSH.

With alkalis, they form mercaptides:

$$RSH + NaOH \text{ -> } RSNa + H_2O \quad (1)$$

At the first stage of the second stage of purification, a 2-5% solution of alkali NaOH of hydrogen sulfide, naphthenic acids and part of methyl, -ethylmercaptans is selectively extracted from oil by the following reactions:

$$H_2S + 2NaOH \rightarrow Na_2S + 2H_2O \tag{2}$$

$$RSH + NaOH \rightarrow RSNa + H_2O \tag{3}$$

$$RCOOH + NaOH \rightarrow RCOONa + H_2O \tag{4}$$

At the second stage of the second purification stage, there is additional extraction of methyl, - ethyl mercaptans, partial extraction of propyls, -isopropyl mercaptans with alkali solution 6.5-14.3 wt%. If necessary, mercaptides are oxidised into disulphide oil with air oxygen in the presence of a catalyst - cobalt phthalocyanine. The extraction and oxidation of mercaptans occurs according to the reactions:

$$RSH + NaOH \rightarrow RSNa + H_2O \tag{5}$$

$$2RSNa + 1/2O_2 + H_2O \rightarrow RSSR + 2NaOH \tag{6}$$

Reaction (4) shows that the oxidation of RSNa completely regenerates the consumed alkali.

To maintain effective purification, the alkaline solution should never contain a significant amount of unoxidized RSNa. For this reason, the amount of air introduced into the oil must always exceed the stoichiometric air flow rate for complete oxidation RSNa.

Table 2 presents the physical and chemical parameters that oil should have after the first and second stages of the second purification stage.

Table 2 - Physical and chemical parameters of oil after the second stage of treatment

Name products	Value of indicators	
	I oil treatment	II oil treatment
Mass fraction of sulfur, %	no more 0.8	no more 0.6
Density (at 20 ^{0}C), kg/m^3	no more 850	no more 850
Fraction yield to temperature, % 200^{0}C 300^{0}C 350^{0}C	no more 27 no more 47 no more 57	no more 27 no more 47 no more 57
Mass fraction of paraffin, %	no more 6.0	no more 5.0
Mass fraction of hydrogen sulfide, ppm	no more 80	no more 1
Mass fraction of methyl-ethyl mercaptans in total, ppm	no more 100	no more 40
Water mass fraction, %	no more 0.5	no more 0.5
Chloride salt concentration, mg/dm^3	no more 100	no more 90
Mass fraction of mechanical impurities,	no more 0.05	no more 0.05
Saturated vapour pressure, kPa	no more 66.7	no more 66.7 (500)

According to Table 2, the content of hydrogen in oil should not exceed 1 %, and the content of mercaptans - 40 ppm.

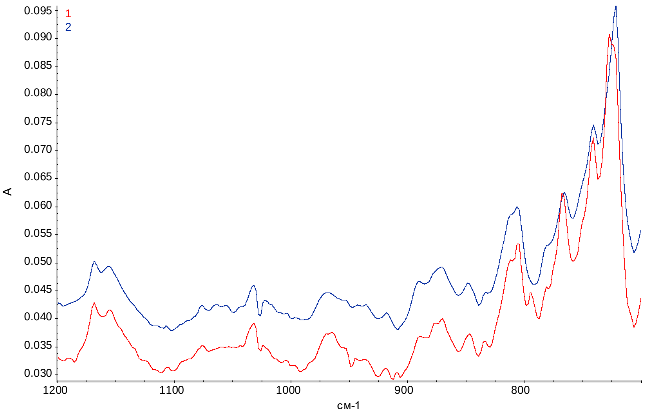

Figure 2 - Comparative figures of IR spectra of oil (Kenkyiak deposits) after stages 1 and 2 of treatment. (1 - the first stage of oil purification, 2 - the second stage of oil purification)

Figure 2 compares the spectra of oil samples after the first and second cleaning stages. A significant difference in spectral profiles is noted, which indicates changes in the ratio of compounds present. The second fraction contains a higher concentration of compounds including S = O groups, as well as sulfur compounds of aromatic and naphthenic structures.

One promising fine dispersion technique is ultrasonic cavitation. This process makes it possible to obtain stable mixtures of substances and significantly accelerate various chemical and mass exchange processes. Ultrasonic dispersion helps to obtain highly dispersed, homogeneous and pure chemical nanosuspensions and nanoemulsions.

In our study, ultrasonic cavitation is considered an advanced technology for dispersing solid materials. This method is highly effective in breaking down solid particles through mechanisms such as shock waves and frictional flows generated by collapsing cavitation bubbles. The dispersion of insoluble components simultaneously facilitates the formation of stable mixtures and enhances chemical processes, enabling the production of nanosuspensions and nanoemulsions with high dispersion and purity.

Additionally, ultrasonic treatment is applied in the pretreatment of crude oil, demonstrating high efficiency in demulsification and dewatering. This method not only improves oil refining quality but also enhances environmental safety by reducing waste volumes. The use of ultrasound in such processes underscores its role as a sustainable and environmentally friendly approach in modern industrial practices.

To obtain emulsions, oil-based compositions after the first stage of purification were used along with aeroflot and dispersed using the JY99-Ultrasonic Homogenizer. The model used was JY99-IIDN, with an ultrasound power of 1800 W, an operating frequency of 20–25 kHz, and a power supply of 220/110 V, 50/60 Hz.

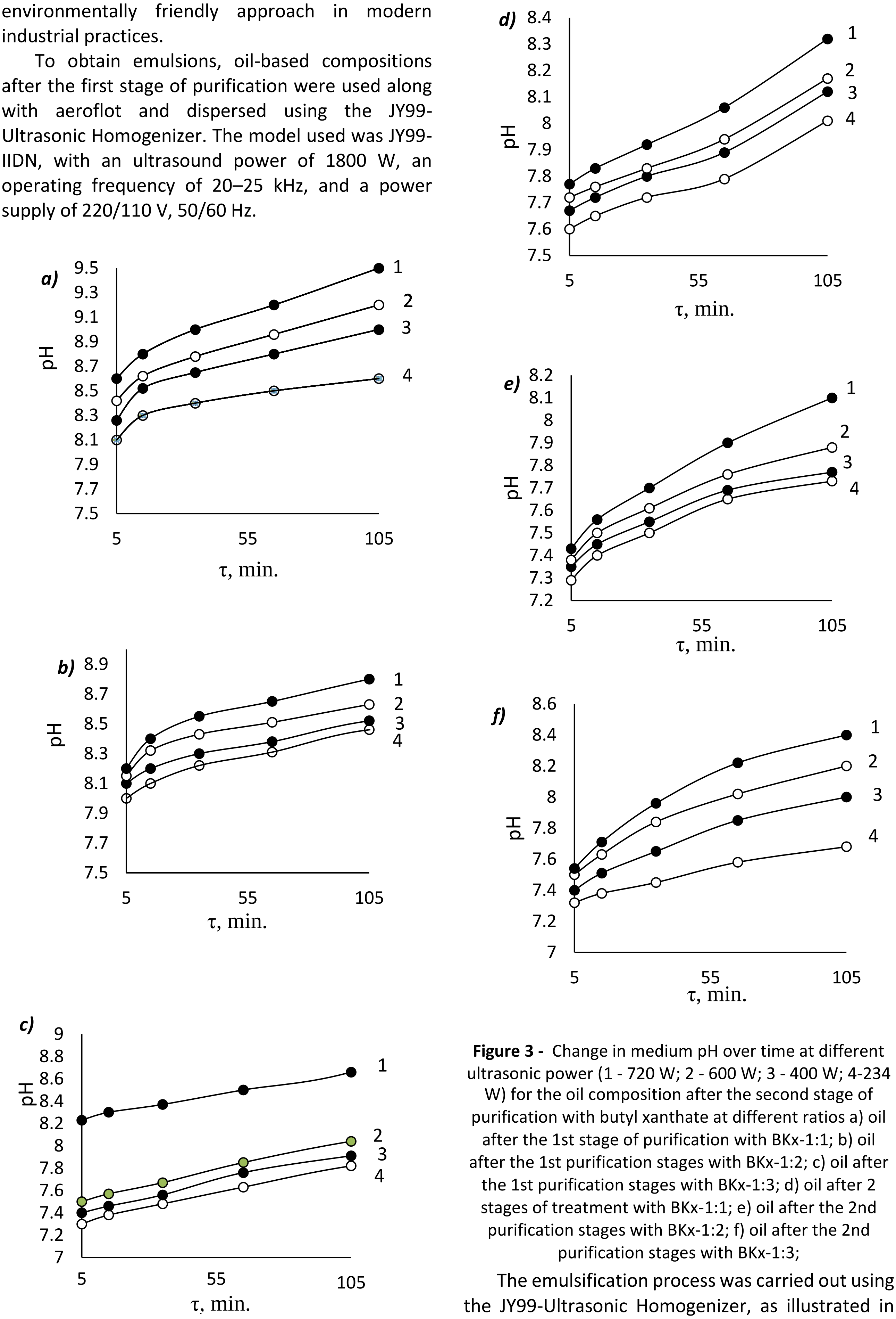

Figure 3 - Change in medium pH over time at different ultrasonic power (1 - 720 W; 2 - 600 W; 3 - 400 W; 4-234 W) for the oil composition after the second stage of purification with butyl xanthate at different ratios a) oil after the 1st stage of purification with BKx-1:1; b) oil after the 1st purification stages with BKx-1:2; c) oil after the 1st purification stages with BKx-1:3; d) oil after 2 stages of treatment with BKx-1:1; e) oil after the 2nd purification stages with BKx-1:2; f) oil after the 2nd purification stages with BKx-1:3;

The emulsification process was carried out using the JY99-Ultrasonic Homogenizer, as illustrated in

Figure 3, following this procedure: 50 ml of distilled water and 0.5 g of the test mixture were placed in a beaker. To evaluate the stability of the resulting microemulsions, three different ratios of oil (after the first purification stage) to butyl xanthate were selected: 1:1, 1:2, and 1:3. A titanium ultrasonic probe in the form of a cone concentrator was immersed in the sleeve, ensuring its tip was positioned in the central part of the liquid volume. The treatment was conducted at an operating frequency of 25 kHz, with adjustable net power in the liquid medium ranging from 234 W to 720 W. The sonication time varied from 5 to 105 minutes, and the temperature of the resulting microemulsion after sonication reached 45–50 °C.

Studies were conducted to determine the optimal sonication conditions for each mixture. The dependence of the pH variation of the emulsion medium overtime on ultrasonic power and the ratio of components (oil after the first purification stage with butyl xanthate) was established (Figure 2). The pH change plays a crucial role in flotation processes, as it influences the chemical state and behavior of reactants in the emulsion. The pH of the medium directly affects the surface charge of particles, altering their ability to adsorb flotation reagents, including butyl xanthate, thereby impacting flotation efficiency.

Ultrasonic treatment can modify the pH by influencing the dissociation of chemical bonds and the distribution of ions in solution, which affects the acid-base properties of the medium and, consequently, the adsorption of reagents and their interaction with minerals. These modifications can significantly impact selectivity and the recovery of valuable components during flotation.

Figure 4 presents the results of pH variation over time during the ultrasonic dispersion of microemulsion collectors, obtained from oil after the first and second purification stages in combination with sulfur-containing components and butyl sodium xanthate. The graphs illustrate the dependence of pH on processing time at different ultrasound power levels (720 W, 600 W, 400 W, and 234 W). The pH tends to increase as dispersion time progresses, with higher ultrasound power contributing to a more significant rise in pH.

The influence of collector composition on pH dynamics is also evident: an increase in the butyl sodium xanthate content (from 1 to 3) leads to a notable shift in the system's acid-base balance.

In further studies, the composition of the compositions was complicated. The collector used was a mercaptan-containing mixture consisting of oil, a sulphur-containing product and sodium butyl xanthate in ratios of 0.5: 0.5: 0.33; 0.5: 0.5: 0.5 and 0.5: 0.5: 1.0. The results are presented in Figure 3.

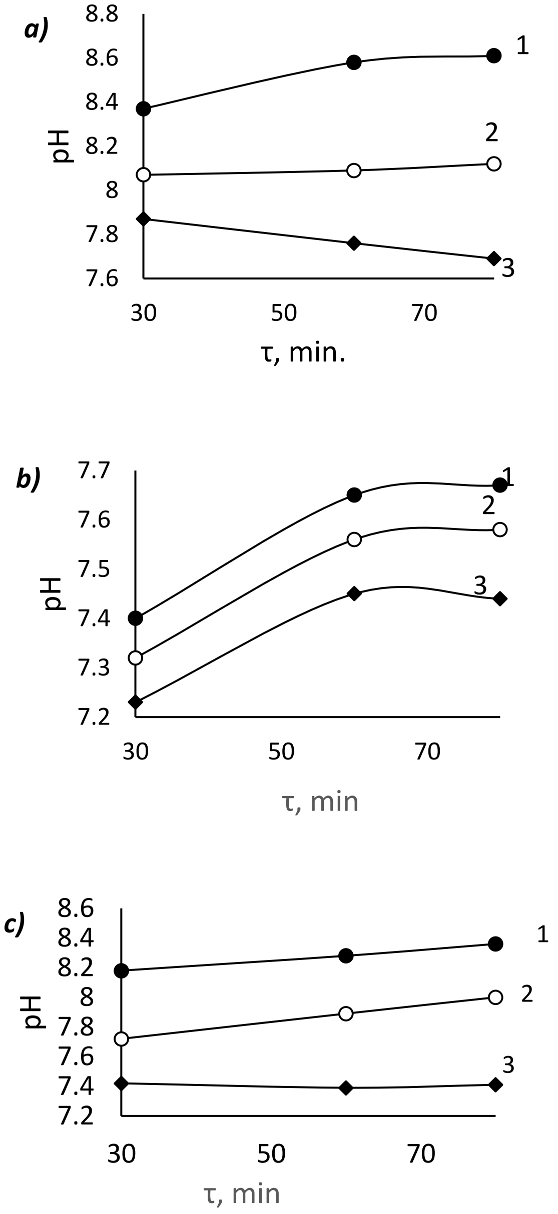

Figure 4 - Change in medium pH over time at different ultrasonic power (1-600 W; 2 - 400 W; 3-234 W) for a mercaptan-containing collector composition consisting of oil, sulfur-containing product and butyl xanthate, with the following ratios: a) 0.5: 0.5: 0.25; b) 0.5: 0.5: 0.5; c) 0.5: 0.5: 1.0.

The particle size measurement of the emulsions was performed using a Photocor Compact analyzer as shown in Table 3. The analyzer operates on the principles of static and dynamic light scattering known as photon correlation spectroscopy. The size of the particles dispersed in the liquid, as well as the molecular weight of the polymers, were determined by analyzing the correlation function of fluctuations in the intensity of scattered light and its total scattering intensity. Measured sizes range from 1 nm to 100 µm.

In previous studies, it was found that the use of a composition of oil and xanthate in equal proportions of 1:1 allows to achieve high flotation efficiency [19]. Paper [20] shows the results of experiments to determine the size of the dispersed phase in emulsions with different ratios of oil and xanthate (1:1; 1:2; 1:3) during ultrasonic treatment with different power (234, 400, 600 W) and at different time intervals (35, 65, 105 minutes). Analysis of the data shows that the minimum particle size, 10.73 nm, was observed at a power of 600 W and a treatment time of 65 minutes or more, especially at the ratio of oil to 1:3 xanthate. These results confirm the effectiveness of increasing ultrasound power and processing time to achieve fine particle sizes in predetermined proportions. With an increase in the concentration of xanthate, the size of the microemulsion decreases and its stability increases accordingly. Also, the presence of sulfur compounds in the oil emulsion, which can be natural emulsifiers, most likely has a significant effect on the stability of the microemulsion.

Additional experiments were performed using refined oil, mercaptan-containing product and butyl xanthate in a ratio of 1:1:0.5; 1:1:1 and 1:1:2. The results of these studies are presented in Table 3.

Table 3 shows the results of experiments to determine the size of the dispersed phase in emulsions using refined oil and butyl xanthate in three different proportions (1:1:0.5, 1:1:1, 1:1:2). The emulsions were sonicated at different powers (234, 400 and 600 W) and measured 30, 60 and 80 minutes after the start of the experiments.

It can be seen from Table 3 that with increasing processing time and ultrasonic power, the particle size decreases in most cases. This is especially noticeable in experiments with the proportion of 1:1:2, where the particle size decreased to 6.61 nm with a power of 600 W and a processing time of 60 minutes. This data may indicate that the optimum conditions for achieving the minimum particle size can be achieved with a higher power and processing time. Increasing the power of the exposure process increases cavitation and reduces the size of the dispersed phase of the emulsion.

This leads to the conclusion that it is important to select appropriate processing conditions to obtain emulsions with the desired particle size characteristics, which is important for the practical application of such systems in various processes.

Table 3 - Influence of ultrasonic treatment power and exposure time on disperse phase size in emulsions with different ratios of oil and xanthate

No.	Time to reach, min.	Dispersed phase value, nm			Experimental conditions
		Power, W			
		234 W	400 W	600 W	
[Refined oil + mercaptan]: butyl xanthate – 1:1:0,5					
1	30 60 80	59.71 59.43 61.45	43.65 18.95 21.97	31.09 24.81 30.79	0.5g/0.5g/0.25g
[Refined oil + mercaptan]: butyl xanthate – 1:1:1					
2	30 60 80	47.92 41.93 38.6	21.73 19.8 12.36	15.63 23.58 25.58	0.5g/0.5g/0.5g
[Refined oil + mercaptan]: butyl xanthate – 1:1:2					
3	30 60 80	28.39 18.95 13.51	15.79 7.16 7.73	17.58 6.61 9.63	0.5g/0.5g/1.0g

Conclusions

Based on the presented data and analysis of the experimental results, it was established that ultrasonic cavitation significantly enhances the efficiency of dispersing and modifying the properties of petroleum products for the production of flotation agents. The application of ultrasound enables control over emulsion stability, improving their characteristics by adjusting exposure power and processing time.

Additionally, the content and chemical form of sulfur in sulfur-containing oil products, particularly mercaptans, have a crucial influence on the properties of collectors. Experiments using refined oil, a mercaptan-containing product, and butyl xanthate in ratios of 1:1:0.5; 1:1:1; and 1:1:2 demonstrated that the 1:1:2 ratio yielded the best results, achieving a particle size reduction to 6.61 nm at 600 W and a treatment time of 60 minutes.

It was also found that ultrasonic cavitation influences not only particle size but also the colloidal properties of the compositions, significantly enhancing their efficiency as collectors. This finding opens up new prospects for the development of advanced mineral processing technologies. These conclusions underscore the importance of innovative approaches in optimizing the production and application of flotation reagents.

Conflicts of interest.

The correspondent author declares that there is no conflict of interest on behalf of all authors.

CRediT author statement: **B. Kenzhaliyev**: Conceptualization; **A. Mukhanova**: Conceptualization, Methodology, Software; **D. Turysbekov**: Methodology; **N. Samenova**: Data curation, Writing-Original draft preparation, Software, Validation; **Zh. Kaldybaeva, K. Toktagulova, S. Yussupova**: Visualization, Investigation.

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Мұнай өнімдері негізінде алынған құрамында меркаптан бар микроэмульсиялық жинағыштардың диспергирлеу қасиеттерін зерттеу

[1] Кенжалиев Б.К., [1] Муханова А.А.,[1] Саменова Н.О., [2]Тоқтагулова К.Н., [1]Қалдыбаева Ж.А., [1]Тұрысбеков Д.К., [1]Юсупова С.Ж.

[1] Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Алматы, Қазақстан
[2] СНПЗ АқтөбеМұнайгаз АҚ, Ақтөбе, Қазақстан

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ТҮЙІНДЕМЕ

Мақалада ультрадыбыстық кавитация әдісін қолдана отырып, құрамында күкірті бар отандық мұнай өнімдері негізінде флотореагенттер алу мәселесі қарастырылады. Зерттеу барысында мұнай өнімдерінің сапалық және сандық құрамы талданып, олардың негізінде реагенттерді іріктеуге ерекше мән берілді. Сондай-ақ, неғұрлым тиімді сульфгидрильді жинағыштар меркаптандар (тиоспирттер) екені белгілі. Жұмыста құрамындағы күкірт мөлшері бірдей, бірақ бастапқы материалдардағы күкірттің химиялық формасы әртүрлі болатын мұнай өнімдері қолданылды. Оларға тазартылған мұнай, құрамында меркаптан бар өнім, сондай-ақ мұнайды демеркаптанизациялау арқылы алынған аралық өнім жатады. Аталған мұнай өнімдері мен бутил ксантогенаты негізінде композициялар жасалды. Ультрадыбыстық кавитация процесінің әсерінен уақыт өте келе эмульсиялардың тұрақтылығының өзгеруі, ультрадыбыстық әсер ету қуаты және компоненттердің әртүрлі қатынастағы тәуелділігі зерттелді. Ультрадыбыстық кавитация мен композициядағы компоненттердің пропорциясын оңтайландыру шарттары анықталды. Күкірт мөлшерінің бірдей болуына қарамастан, тек белгілі бір құрылымдағы күкіртқұрамды өнімдер ғана флотореагенттер алу үшін жарамды екені дәлелденді. Сондай-ақ, композициялардың қасиеттерін модификациялауда ультрадыбыстық кавитацияның маңызды рөл атқаратыны анықталды. Бұл фактор олардың жинағыш реагент ретіндегі тиімділігіне айтарлықтай әсер етеді.

Түйін сөздер: тазартылған мұнай, құрамында меркаптан бар жинағыш, бутилді ксантогенат.

	Авторлар туралы ақпарат:
Кенжәлиев Бағдәулет Кенжәліұлы	*Техника ғылымдарының докторы, профессор, Бас-директор - Басқарма төрағасы, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: bagdaulet_k@satbayev.university; ORCID ID: https://orcid.org/0000-0003-1474-8354*
Муханова Айнұр Айтқазықызы	*Техника ғылымдарының кандидаты, Флотациялық реагенттер және кен байыту зертханасының жетекші ғылыми қызметкері, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: a.mukhanova@satbayev.university; ORCID ID: https://orcid.org/0000-0002-2130-683X*
Саменова Нәзира Ораққызы	*Флотациялық реагенттер және кен байыту зертханасының ғылыми қызметкері, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: nazira.orakkyzy@gmail.com; ORCID ID: http://orkid.org/0000-0002-3027-5246*
Тоқтағұлова Кәмшат Надірқызы	*СНПЗ АқтөбеМұнайгаз АҚ 2-дәрежелі инженер-химикі, Ақтөбе, Қазақстан. Email: nadirovna_k@mail.ru*
Қалдыбаева Жәмихан Әбділдәбекқызы	*Флотациялық реагенттер және кен байыту зертханасының кіші ғылыми қызметкері, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: zh.kaldybaeva@satbayev.university; ORCID ID: https://orcid.org/0000-0001-5763-9655*

Тұрысбеков Дулатбек Қадырбекұлы	*Техника ғылымдарының кандидаты, Флотациялық реагенттер және кен байыту Зертханасының жетекші ғылыми қызметкері, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: d.turysbekov@satbayev.university; ORCID ID: https://orcid.org/0000-0003-0904-1565*
Юсупова Сания Жабборқызы	*Флотациялық реагенттер және кен байыту зертханасының инженері, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: Saniya.yusupova02@mail.ru; ORCID ID: https://orcid.org/0009-0009-1728-3418*

Изучение диспергирующих свойств микроэмульсионных меркаптансодержащих собирателей, полученных на основе нефтепродуктов

[1]Кенжалиев Б.К., [1]Муханова А.А.,[1]Саменова Н.О., [2]Токтагулова К.Н., [1]Калдыбаева Ж.А., [1]Турысбеков Д.К., [1]Юсупова С.Ж.

[1] АО Институт металлургии и обогащения, Satbayev University, Алматы, Казахстан
[2] АО СНПЗ АктобеМунайгаз, Актобе, Қазақстан

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АННОТАЦИЯ

В статье рассматривается получение флотореагентов на основе отечественных серосодержащих нефтепродуктов с применением метода ультразвуковой кавитации. Особое внимание уделено подбору нефтепродуктов, основанному на анализе их качественного и количественного состава. Известно, что наиболее эффективными сульфгидрильными собирателями являются меркаптаны (тиоспирты). В работе использованы серосодержащие нефтепродукты с одинаковым содержанием серы, но различной формой ее присутствия в исходных материалах: очищенная нефть, меркаптансодержащий продукт, а также промежуточный продукт, полученный при демеркаптанизации нефти. На основе указанных нефтепродуктов и бутилового ксантогената были созданы композиции, для которых в процессе ультразвуковой кавитации определялись зависимости изменения устойчивости эмульсий во времени от мощности ультразвукового воздействия и соотношения компонентов. Были разработаны оптимальные условия ультразвуковой кавитации и пропорции компонентов в композициях. Установлено, что, несмотря на одинаковое содержание серы, для получения флотореагентов пригодны только серосодержащие продукты определенной структуры. Также выявлена роль ультразвуковой кавитации в модификации свойств композиций, что существенно влияет на их эффективность в качестве собирателей.

Ключевые слова: очищенный нефть, меркаптан-содержащий продукт, бутиловый ксантогенат.

	Информация об авторах:
Кенжалиев Багдаулет Кенжалиевич	*Доктор технических наук, профессор, Генеральный директор-Председатель правления АО Институт металлургии и обогащения, Satbayev University, ул.Шевченко 29/133, 050010, Алматы, Казахстан. Email: bagdaulet_k@satbayev.university; ORCID ID: https://orcid.org/0000-0003-1474-8354*
Муханова Айнур Айтказыновна	*Кандидат технических наук, ведущий научный сотрудник лаборатории флотореагентов и обогащения, АО Институт металлургии и обогащения, Satbayev University, ул.Шевченко 29/133, 050010, Алматы, Казахстан. Email: a.mukhanova@satbayev.university; ORCID ID: https://orcid.org/0000-0002-2130-683X*
Саменова Назира Ораққызы	*Научный сотрудник лаборатории флотореагентов и обогащения, АО Институт металлургии и обогащения, Satbayev University, ул.Шевченко 29/133, 050010, Алматы, Казахстан. Email: nazira.orakkyzy@gmail.com; ORCID ID: http://orkid.org/0000-0002-3027-5246*
Токтагулова Камшат Надировна	*АО СНПЗ АқтобеМунайгаз, инженер-химик 2-категорий, Ақтөбе, Қазақстан. Email:nadirovna_k@mail.ru*
Калдыбаева Жамихан Абдилдабековна	*Младший научный сотрудник лаборатории флотореагентов и обогащения, АО Институт металлургии и обогащения, Satbayev University, ул.Шевченко 29/133, 050010, Алматы, Казахстан. Email: zh.kaldybaeva@satbayev.university; ORCID ID: https://orcid.org/0000-0001-5763-9655*
Турысбеков Дулатбек Кадырбекулы	*Кандидат технических наук, ведущий научный сотрудник лаборатории флотореагентов и обогащения, АО Институт металлургии и обогащения, Satbayev University, ул.Шевченко 29/133, 050010, Алматы, Казахстан. Email: d.turysbekov@satbayev.university; ORCID ID: https://orcid.org/0000-0003-0904-1565*
Юсупова Сания Жабборовна	*Инженер лаборатории флотореагентов и обогащения, АО Институт металлургии и обогащения, Satbayev University, ул.Шевченко 29/133, 050010, Алматы, Казахстан. Email: Saniya.yusupova02@mail.ru; ORCID ID: https://orcid.org/0009-0009-1728-3418*

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Metallurgy

Lithium extraction methods and its application prospects: a review

[1*]Yersaiynova A.A., [1]Karshyga Z.B., [2]Muhammad N.A.A., [1]Yessengaziyev A.M., [1]Orynbayev B.M.

[1] Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Almaty, Kazakhstan
[2]University Pendidikan Sultan Idris, 35900 Tanjong Malim, Perak, Malaysia

** Corresponding author email: a.yersaiynova@satbayev.university*

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ABSTRACT
Lithium is the most important raw material for the production of modern electronics and electric vehicles. Today, it is impossible to imagine any mobile device without lithium batteries. The role of lithium in the global economy is only growing. The production of electric vehicles and batteries contributes to the reduction of carbon dioxide emissions. Nevertheless, end-of-life lithium-ion batteries pose a danger to the ecosystem. The article presents technological developments in the field of lithium extraction. The main sources of lithium are pegmatites, continental and geothermal brines, as well as clays, seawater and industrial brines. The main commercial lithium product is lithium carbonate (Li_2CO_3), which is obtained mainly from the mining, extraction and processing of spodumene ores and saltlake, oilfield brines. The effective role of lithium in addressing important issues such as pollution, climate change and the increasing depletion of natural resources used to produce lithium-ion batteries for these electric vehicles is also discussed.

Keywords: lithium outlook, lithium minerals, demand and use, lithium resources, key technologies.

Information about authors:

Yersaiynova Albina Abatkyzy	*Doctoral student, laboratory of titanium and rare refractory metals, Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: a.yersaiynova@satbayev.university; ORCID ID: https://orcid.org/0000-0003-0638-380X*
Karshyga Zaure Baitaskyzy	*Ph.D., Leading Researcher, Associate Professor, laboratory of titanium and rare refractory metals, Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: z.karshyga@satbayev.university; ORCID ID: https://orcid.org/0000-0002-3025-7363*
Muhammad Noorazlan Abd Azis	*Ph.D., Associate Professor, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, Tanjung Malim, Perak, 35900, Malaysia. Email:azlanmn@fsmt.upsi.edu.my; ORCID ID: https://orcid.org/0000-0002-2792-4145*
Yessengaziyev Azamat Muratovich	*Ph.D., Head of the Laboratory of Titanium and Rare Refractory Metals of the Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: a.yessengaziyev@satbayev.university; ORCiD ID: https://orcid.org/0000-0002-4989-4119*
Orynbayev Bauyrzhan Munarbaiuly	*Doctoral student, laboratory of titanium and rare refractory metals, Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Shevchenko str., 29/133, 050010, Almaty, Kazakhstan. Email: Bauyrzhan.Orynbayev@stud.satbayev.university; ORCID ID: https://orcid.org/0000-0002-7730-9060*

Introduction

Lithium and lithium-containing compounds are vital strategic resources for the economy, and their consumption is growing rapidly every year. Lithium is also one of the most important metals that set the direction of scientific and technological progress in the modern world. Lithium and its alloys are used in a wide variety of industrial applications, such as: batteries, ceramics and glass, lubricating greases, polymer production, medical, continuous casting mold flux powders, air treatment, and other uses [1].

In glass and ceramics manufacturing, lithium is used as a flux where it helps lower the melting point and increase durability [2]. Lithium can be used as an absorbent medium in industrial refrigeration systems as well as in humidity control and dehumidification systems. In metallurgy, lithium is used in the production of lightweight alloys used in aviation and aerospace, as well as in the production of high-strength, corrosion-resistant military components. In medicine, it is used to treat bipolar disorder, depression and other mental disorders [3]. Lithium is also used as a thickener in the production of lubricants. These lubricants have excellent heat resistance and water repellency properties, making them ideal for use in heavy equipment and automotive applications. Lithium is widely used today in the manufacture of batteries, especially lithium-ion batteries, which are used in electric transport, stationary energy storage systems, as well

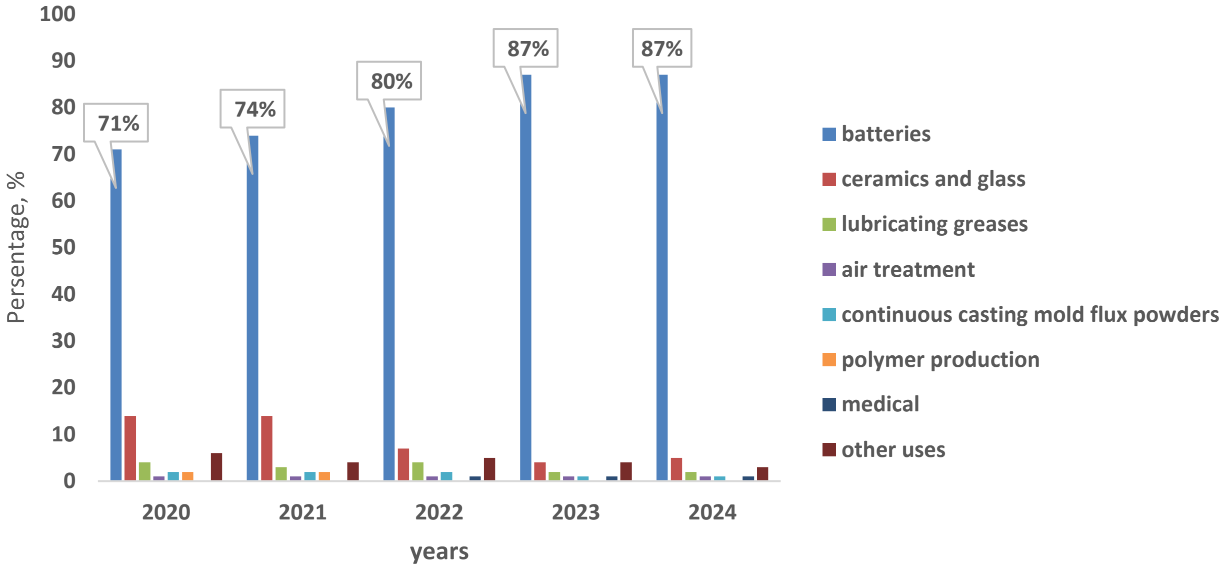

Figure 1 - Use of lithium and its compounds in the world from 2019 to 2023

as in compact power supplies for computer and telecommunications equipment. According to U.S. Geological Survey, Mineral commodity summaries over the last 5 years, the application area of lithium is changing [[4], [5], [6], [7], [8]]. Figure 1 shows a steady annual increase in the use of lithium in batteries, while other areas show a decrease.

Since Sony and Asahi Kasei introduced lithium-ion batteries in 1991, lithium-ion batteries have significantly changed many aspects of our daily lives [[9], [10]]. In recent years, the demand for lithium has been steadily increasing as global energy storage markets are growing rapidly [11]. The electric vehicle industry is experiencing particularly rapid growth, significantly accelerating this process [12].

Between 2021 and 2022, global fossil fuel-derived carbon dioxide (CO_2) emissions increased and reached the highest level in history [[13], [14]]. The main sources of atmospheric pollution are industrial enterprises and automobiles [15]. Industrial facilities emit gases and dust into the atmosphere, and vehicles emit significant amounts of exhaust gases [16]. Atmospheric pollutants include not only gaseous substances but also fine dust containing heavy metals such as lead (Pb), cadmium (Cd), and mercury (Hg), which pose a serious threat to human health [[17], [18], [19]]. Also, due to the emission of CO_x, NO_x and other gases into the atmosphere in large quantities, we intensively observe climate change every year: floods, forest fires, drought and other major disasters [[20], [21]]. In this regard, international organizations intend to switch to renewable energy sources to reduce the spread of greenhouse gases and to reduce the load of fossil fuels [22].

Increased demand for lithium has an impact on its price. The formation of lithium prices depends on its supply, which, in turn, may be constrained by its natural reserves, as they are concentrated in a limited number of countries.

Main lithium minerals and world reserves

The main sources of lithium are pegmatites, continental and geothermal brines, also clays, seawater and industrial groundwater [23]. The main commercial lithium product is lithium carbonate (Li_2CO_3), which is obtained mainly by mining, extraction and processing of spodumene ores and saltlake, oilfields brines.

More than 100 natural lithium minerals have been identified [[24], [25]]. Lithium is predominantly found in the form of silicates, less frequently in the form of phosphates, and lithium minerals from other classes are extremely rare [26]. Commercial lithium minerals are: spodumene, lepidolite, petalite, eucryptite and cinnwaldite, also other minerals are presented in Table 1 [27].

According to U.S. Geological Survey, Mineral commodity summaries U.S. Geological Survey lithium reserves worldwide in January 2024 are about 105 million tons [7]. Figure 2 shows that Bolivia, Argentina, USA and Chile have the largest lithium reserves. In Kazakhstan, according to the USGS, lithium reserves amount to 50 thousand tons.

Table 1 - The main lithium minerals

Mineral name	Chemical formula	Content of Li_2O, %
Spodumene	$LiAl[Si_2O_6]$	5.9-7.6
Bikitaite	$LiAlSi_2O_6*H_2O$	6.51
Holmquistite	$Li_2(Mg_3Al_2)Si_8O_{22}(OH)_2$	1.1
Lepidolite	$KLi_2Al(Al,Si)_4O_{10}(F,OH)_2$	4.1-5.5
Cinnwaldite	$K(Li,Fe,Al)_3(OH,F)_2[AlSi_3O_{10}]$	2.9-4.5
Tyniolite	$KLiMg_2[Si_4O_{10}](OH, F)_2$	3.70
Polylithionite	$KLi_2Al[Si_4O_{10}]F_2$	3.70-7.70
Bitite	$Ca_4(Li,Be,Al)_{12}[(Si,Al)_4O_{10}][OH]_2$	2.73
Kukeit	$LiAl_4[Si_3AlO_{10}](OH)_8$	0.80-4.33
Eucryptitis	$LiAl[SiO_4]$	6.1
Petalite	$LiAl(Si_4O_{10})$	3.4-4.1

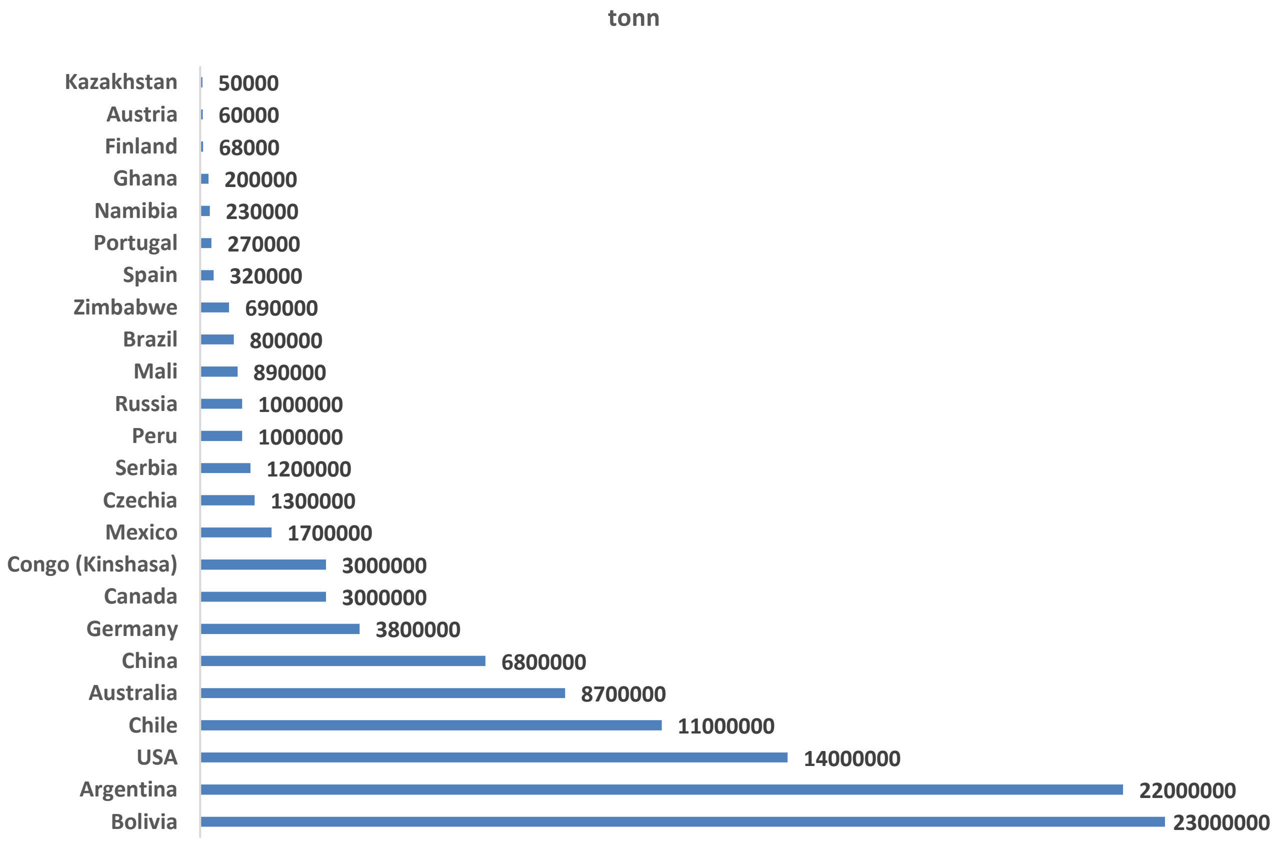

Figure 2 - Lithium reserves by country

President of Kazakhstan Kasym-Jomart Tokayev in 2022 during a meeting with the public of Zhetysu region stated about the large reserves of lithium in the bowels of Kazakhstan and the need for serious investment in the exploration and development of lithium, also instructed the Geological Service to intensify work in this direction [28].

Lithium deposits are mainly located: in East Kazakhstan region Akhmetkino, Akhmirovskoye, Bakennoye, Verkhne-Baimurzinskoye, Yubileynoye, Medvedka, Karasu, Kokkol, Targynskoye, in Aktobe region Verkhne-Irgizskoye. in Kostanay region Smirnovskoe and Drozhzhzhilovskoe, in Karaganda region lithium (Li) is established at the Zhanet deposit, in Almaty region at the Karagailyaktas deposit, in Zhambyl region lithium mineralization is established at the quartz vein deposit Maikol [29]. Currently, the above-mentioned sites are being explored, or the sites are mothballed or in reserve.

Mining activities are gaining momentum every year. Reserves of some rare elements have significantly decreased, while their demand has increased dramatically due to their increased use in new areas of engineering and technology. In this connection it is becoming more and more important to search for and involve in industrial development new types of mineral raw materials. One of such sources for many valuable elements necessary for various branches of economy can be underground industrial brines, which are a kind of “liquid ore” [30]. Its advantage over traditional ores is obvious: hydro-mineral raw materials have a polycomponent and can simultaneously serve as a source for the extraction of various valuable metals, such as lithium [31].

The comparison shows that the average brine deposit (1.45 million tons of lithium) is significantly larger than the average pegmatite deposit (0.11 million tons of lithium). Especially large brine deposits, such as Salar de Atacama in Chile and Uyuni in Bolivia, have a much larger total lithium resource (21.6 million tons of lithium) [[32], [33]]. Also lithium-bearing brine resources around the world are: Clayton Valley, USA, Salton Sea, USA, Salar de Atacama, Chile, HombreMuerto, Argentina, Salar de Uyuni, Bolivia, Searles Lake, USA, Great Salt Lake, USA, Dead Sea, Israel, Sua Pan, India, Bonneville, USA, Zabuye, Taijinaier, China. Thus, brine deposits have a much greater capacity for large-scale and long-term mining compared to pegmatite deposits. Lithium mining from brines is considered more favorable than mining from ores because it is more environmentally friendly and cost-effective [33].

On the territory of Kazakhstan 4 perspective provinces of industrial waters are identified: Pre-Caspian, Mangistau-Ustirt, Shu-Sarysu and South-Torgai. All provinces of industrial waters are associated with oil and gas bearing fields [34]. Locating lithium feedstock in or near industrial areas has significant advantages.

All natural sources of lithium raw materials have their own characteristics: the type of raw materials, the quantitative content of the main component, the accompanying impurity components and their quantity, and others.

Therefore, when processing existing raw materials and creating own production facilities, an individual approach is required with the study of modern technological developments and the involvement of new advanced solutions.

Lithium extraction methods

For spodumene concentrate, which is currently the main one among lithium concentrates, four treatment methods are known: sulfuric acid, sulfate, lime and chloride roasting. All these methods are used in industry, and the choice of a particular one depends on the economic efficiency [[35], [36]].

Sulfuric acid method. Lithium extraction by acid leaching is much more efficient when using β-spodumene as a starting material than α-spodumene [[37], [38], [39]]. Therefore, before sulfatization, natural spodumene is heat treated to convert the minerals into a more reactive form [[40], [41]]. After dicrypitation is followed by sulfatization with sulfuric acid, Decomposition reaction of spodumene with sulfuric acid to form lithium sulfate [[42], [43]]:

$$Li_2O \cdot Al_2O_3 \cdot 4SiO_2 + H_2SO_4 \rightarrow Li_2SO_4 + 4SiO_2 \cdot Al_2O_3 \cdot \cdot H_2O \quad (1)$$

$$Li_2SO_{4(s)} \rightarrow Li_2SO_{4(aq)} \quad (2)$$

$$Li_2SO_{4(aq)} + Na_2CO_{3(aq)} \rightarrow Li_2CO_{3(s)} + Na_2SO_{4(aq)} \quad (3)$$

In this process, H^+ ions from the acid chemoselectively replace Li^+ ions in spodumene as described in equation (1), forming water-soluble $Li_2SO_{4(s)}$ [42]. The resulting soluble compound is leached with water according to equation (2), after which the solution is treated with lime at 90°C to adjust pH and remove impurities. Further, lithium carbonate is extracted by adding sodium carbonate solution to the extract, which is reflected in equation (3) [[42], [43]]. The technological scheme of the sulfuric acid method of spodumene concentrate processing is presented in Figure 3.

The advantage of the sulfuric acid method is: no need to grind the ore due to the fact that preliminary decryption leads to its loosening, so that sulfatization with sulfuric acid is successfully carried out even in the treatment of large fractions, and there are no long high-temperature processes.

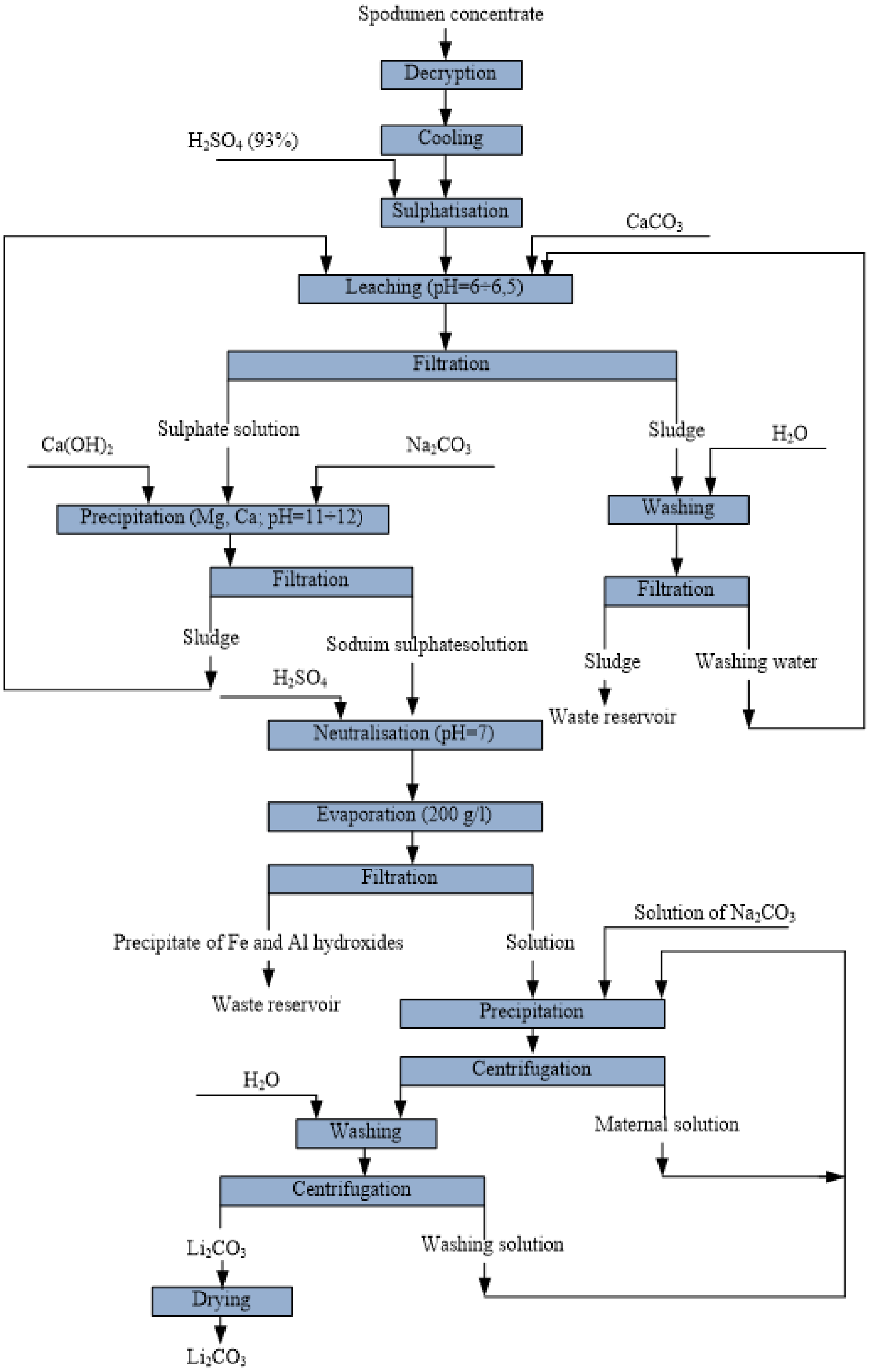

Figure 3 - Technological scheme of sulfuric acid method of spodumene concentrate processing

Sulfate method. This method is based on sintering of lithium ores and concentrates with potassium sulfate [[26], [44]]. The process is based on direct substitution of lithium with potassium [45]. Sintering of natural spodumene with potassium sulfate takes place at temperatures of 920 -1050°C, where α-spodumene under heating changes into β-spodumene, which reacts with potassium sulfate:

$$Li_2O\cdot Al_2O_3\cdot 4SiO_2 + K_2SO_4 = Li_2SO_4 + K_2O\cdot Al_2O_3\cdot 4SiO_2 \quad (4)$$

The obtained sinter is quenched with water and leached, separating the insoluble residue by decantation or filtration. The solution containing lithium sulphate and impurities is purified with potassium or sodium hydroxides, after which lithium carbonate (Li_2CO_3) is precipitated with sodium carbonate according to equation (5). The precipitated lithium carbonate is squeezed, washed and dried:

$$Li_2SO_{4(aq)} + Na_2CO_{3(aq)} \rightarrow Li_2CO_{3(s)} + Na_2SO_{4(aq)} \quad (5)$$

The technological scheme of spodumene concentrate processing by sulphate scheme is presented in Figure 4.

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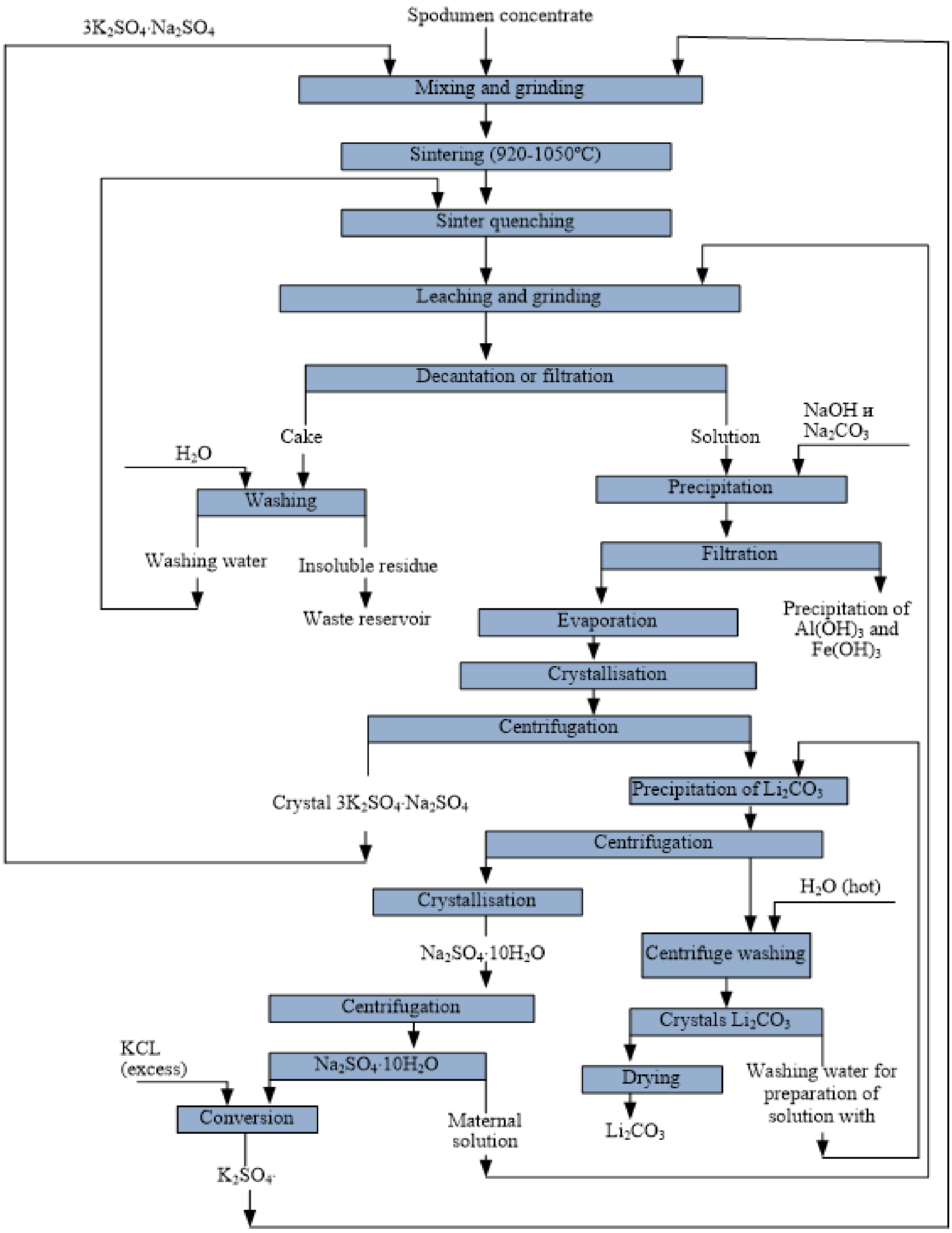

Figure 4 - Technological scheme of spodumene concentrate processing by sulfate scheme

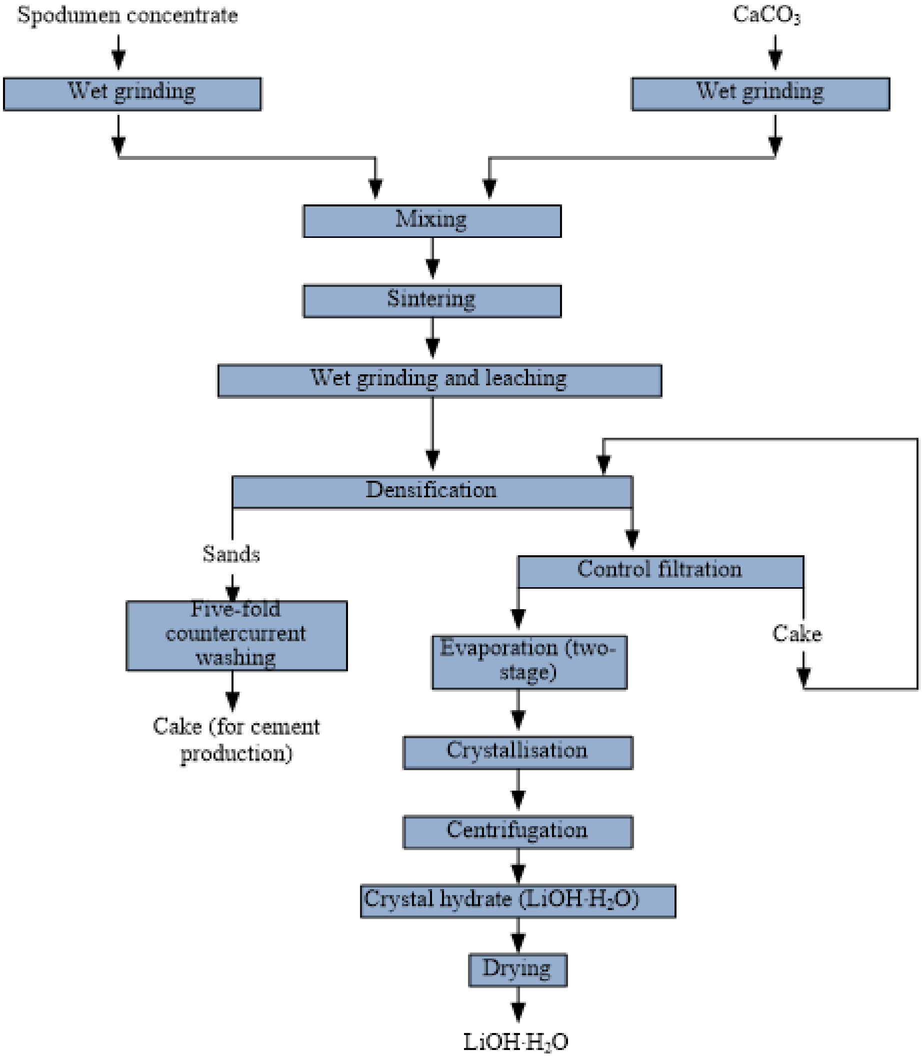

Figure 5 - Technological scheme of spodumene concentrate processing by lime method

Under optimal sintering conditions, the degree of mineral penetration reaches 90-95%, which indicates the high efficiency of the method. Also this method allows to obtain lithium carbonate with high purity, which is important for further use in industry. The disadvantage of the method is a large consumption of potassium sulphate.

Lime method. The lime method is based on the sintering of lithium concentrates with the adding of lime or limestone, and then decomposition of the resulting sinter with water [44].

The technological scheme of spodumene concentrate processing by the lime method is presented in Figure 5.

Sintering of spodumene with limestone is carried out at a temperature of 1150-1200°C. The following reaction takes place:

$$Li_2O \cdot Al_2O_3 \cdot 4SiO_2 + 8CaCO_3 \rightarrow Li_2O \cdot Al_2O_3 + 4(2CaO \cdot SiO_2) + 8CO_2 \quad (6)$$

The reaction of spodumene with lime can be described as follows:

$$Li_2O \cdot Al_2O_3 \cdot 4SiO_2 + 4CaO + 4H_2O \rightarrow 2LiOH + 2Al(OH)_3 + 4CaSiO_3 \quad (7)$$

The lime method has the following advantages: it is universal for opening of all lithium minerals, it does not require scarce reagents (only natural limestone is needed). However, the lime scheme has serious disadvantages: the initial concentrate should be rich in lithium content, and limestone should be of high quality, with low content of silicon, aluminum, iron [46]. Due to the low concentration of lithium in solutions after leaching, large volumes of process equipment and high energy consumption for evaporation are required.

Chlorinating method. One of the methods of processing ores and concentrates containing lithium is chlorinating roasting [[45], [47]]. This method was used to treat concentrates containing lepidolite and spodumene. The material consisting of lepidolite, NH_4Cl and $CaCO_3$ was roasted at 750 - 800°C and then leached with water [48]. The mineral decomposes by the reaction:

$$Li_2O\cdot Al_2O_3\cdot 4SiO_{2(тв)} + 4CaCO_{3(тв)} + 2NH_4Cl \rightarrow 2LiCl_{(тв)} + 4CaSiO_3 + Al_2O_3 + 4CO_{2(г)} + 2NH_3 + H_2O \quad (8)$$

Evaporation of the solution crystallizes a mixture of lithium and calcium chlorides, which can be used for electrolysis or lithium hydroxide production by causticization. This method has been commercialized in the USA for the extraction of lithium from spodumene concentrate.

Chlorination roasting allows to obtain sufficiently pure lithium chloride directly from ores without using expensive reagents. The disadvantages of this method include the difficulty of capturing lithium chloride and high aggressiveness of furnace gases.

In recent years, new methods like hydrofluoric acid method, autoclave method, and microwave firing have been proposed [[49], [50]]. However, there are still some disadvantages associated with these methods, such as the use of highly toxic reagents and high energy consumption.

Extraction of lithium from hydromineral raw materials become popular among lithium producers due to its low production costs.

According to literature data, lithium ions from hydromineral raw materials can be extracted using conventional methods such as: evaporation, precipitation, as well as direct lithium extraction methods: solvent extraction, electrochemical, adsorption and membrane methods [[51], [52], [53]].

The conventional method of extracting lithium from underground brine is to pump it to the surface and further place it in huge ponds. Over a long period (up to a year or more), the water gradually evaporates, allowing the concentration of lithium to rise to a level sufficient to allow it to be precipitated by chemicals. However, this method is only applicable in sunny, dry regions [54].

As a result of the limitations of conventional technologies, direct lithium extraction (DLE) techniques have been developed to address these shortcomings and provide more efficient, environmentally friendly and cost-effective recovery of lithium from resources with relatively low grades of this metal.

Membranes. The membrane method is classified based on their driving mechanisms: electrodialysis, which relies on electrical potential, and nanofiltration, which operates under pressure. The main membrane technologies are nanofiltration, microfiltration and ultrafiltration. Nanofiltration membranes (NF) are capable of passing monovalent ions while simultaneously trapping multivalent ones. This property allows them to be used to separate lithium from divalent ions such as Mg^{2+} and Ca^{2+} [55]. In recent years, they have become a key method for lithium extraction, showing a rapid growth of interest from researchers and progressing steadily towards full-scale industrial utilization [56]. This interest is due to the low energy consumption, excellent cyclic stability, high separation efficiency, large porosity, permeability, surface area, and good mechanical stability of membranes [57]. In addition, they are easily adaptable to different operating conditions However, this process has its disadvantages, especially that membranes clog quickly when working with saturated solutions.

Solvent extraction. This method, known as liquid-liquid extraction, uses the difference in solubility properties of different salts to isolate lithium. The extraction process usually involves separating the solution into two separate phases: aqueous and organic. During this process, the impurities remain in the aqueous phase, while the lithium ions pass into the organic phase. Extractants include neutral organic phosphorous compounds, β-diketones, crown esters, and ionic liquids. One of the widely used extractants is $TBP/FeCl_3$ [58]. This is a promising method for lithium production, but it has drawbacks, including the need for an additional extractant purification step, possible loss of target material, and risks of environmental contamination when using organic reagents.

Electrochemical method. Electrochemical processes are one of the most studied and effective methods of lithium extraction, characterized by a high degree of extraction and ease of operation, as well as environmental safety [59]. The process is based on a principle similar to battery reverse charging technology, where an external electrostatic field is used to stimulate the movement of ions between a pair of specially designed electrodes designed to extract lithium ions [60]. The process is usually carried out cyclically: firstly, the target ions are introduced into the working electrode, and then released back into the solution during the regeneration stage to repair the electrode. In electrochemical methods, the performance of electrode materials plays a key role [61]. Therefore, it is necessary to optimize electrode materials that have a high ability to capture and release lithium, as well as demonstrate stability and corrosion resistance.

Adsorption. Methods of lithium sorption from brines can be carried out using both organic and inorganic sorbents. But organic ionites are selective not only to lithium, but also to other ions, which can complicate the separation and purification of extracted lithium, as well as lead to the formation of organic waste, which may require special methods of disposal or treatment, which increases the environmental burden of the process [62].

For the treatment of low lithium concentrations, the ion exchange adsorption method can be applied, where ionic sieves are used as adsorbents [63]. Lithium ion sieves are considered one of the most promising materials for lithium extraction from low lithium brines due to their high adsorption capacity and excellent lithium selectivity. Studies show that manganese oxide (LMO) and titanium oxide (LTO) based sorbents as an effective method for lithium extraction from brines [[64], [65]].

The greatest success has been achieved in developing ion sieves based on lithium manganese oxide (LMO). In particular, the lithium ion sieve material ($H_2Mn_2O_4$), derived from the spinel structure $LiMn_2O_4$ after acid treatment, exhibits high selectivity for Li^+ ion adsorption in aqueous solutions. This makes it a promising candidate for lithium extraction from natural brines. Such materials possess a high adsorption capacity.

Thus, according to the review, innovative approaches such as direct lithium extraction technologies present opportunities to develop a more sustainable and efficient lithium supply chain. This highlights the dynamic and ever-evolving nature of the lithium mining industry as it adapts to technological advancements and environmental challenges.

Conclusions

As the literature review has shown, lithium is currently one of the highly demanded metals of strategic importance, as well as contributing to solving environmental problems and improving the quality and level of our daily lives. Significant lithium reserves available in Kazakhstan allow to create production facilities for extraction and processing of domestic lithium-containing raw materials to produce valuable lithium products. Existing processing methods are designed for raw materials of appropriate composition, so research and development of technology acceptable for Kazakhstani lithium raw materials is an urgent task. The development of efficient and environmentally friendly lithium extraction technologies will enhance Kazakhstan's position in the global lithium supply chain, contributing to economic growth and technological advancement. Future studies will focus on optimizing extraction processes, minimizing environmental impact, and exploring innovative approaches to utilize Kazakhstan’s lithium resources effectively.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Литийді алу әдістері және оны қолдану перспективалары: шолу

[1]Ерсайынова А.А., [1]Қаршыға З.Б., [2]Muhammad N.A.A.,
[1]Есенгазиев А.М., [1]Орынбаев Б.М.

[1] Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Алматы, Қазақстан
[2]Пендикан Сұлтан Идрис университеті, Перак, Малайзия.

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ТҮЙІНДЕМЕ

Литий қазіргі заманда электроника мен электр көліктері үшін маңызды шикізат болып табылады. Бүгінгі күні литий батареялары жоқ кез келген мобильді құрылғыны елестету мүмкін емес. Литийдің әлемдік экономикадағы рөлі тек қана артып келеді. Электрлік көліктер мен батареяларды өндіру көмір қышқыл газының шығарындыларын азайтуға көмектеседі. Дегенмен, қызмет ету мерзімі біткен литий-иондық батареялар экожүйеге қауіп төндіреді. Мақалада литийді өндіру саласындағы технологиялық әзірлемелер берілген. Литийдің негізгі көздері пегматиттер, континенттік және геотермалдық тұзды ерітінділер, сондай-ақ саз, теңіз суы және өнеркәсіптік тұзды ерітінділер болып табылады. Литийдің негізгі коммерциялық өнімі литий карбонаты (Li_2CO_3) болып табылады, ол негізінен сподумен кендері мен тұзды ерітінділерді өндіруден және өңдеуден алынады. Сондай-ақ, қоршаған ортаның ластануы, климаттың өзгеруі және электр көліктеріне арналған литий-иондық аккумуляторларды өндіру үшін пайдаланылатын табиғи ресурстардың сарқылуы сияқты маңызды мәселелерді шешуде литийдің тиімді рөлі талқыланады.

Түйін сөздер: литий перспективалары, литий минералдары, сұраныс және пайдалану, литий ресурстары, негізгі технологиялар.

Авторлар туралы ақпарат:

Ерсайынова Альбина Абатқызы
Докторант, титан және сирек қиын балқитын металдар зертханасы, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: a.yersaiynova@satbayev.university; ORCID ID: https://orcid.org/0000-0003-0638-380X

Қаршыға Зәуре Байтасқызы
Ph.D., жетекші ғылыми қызметкер, қауымдастырылған профессор, титан және сирек қиын балқитын металдар зертханасы, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: z.karshyga@satbayev.university; ORCID ID: https://orcid.org/0000-0002-3025-7363

Мухаммед Нуразлан Абд Азис
Ph.D., қауымдастырылған профессор, ғылым және математика факультеті, Пендикан Сұлтан Идрис университеті, 35900 Танджонг Малим, Перак, Малайзия. Email: azlanmn@fsmt.upsi.edu.my; ORCID ID: https://orcid.org/0000-0002-2792-4145

Есенгазиев Азамат Муратович
Ph.D., титан және сирек қиын балқитын металдар зертханасының меңгерушісі, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: a.yessengaziyev@satbayev.university; ORCiD ID: https://orcid.org/0000-0002-4989-4119

Орынбаев Бауыржан Мұнарбайұлы
Докторант, титан және сирек қиын балқитын металдар зертханасы, Металлургия және кен байыту институты АҚ, Сәтбаев университеті, Шевченко көш., 29/133, 050010, Алматы, Қазақстан. Email: Bauyrzhan.Orynbayev@stud.satbayev.university; ORCID ID: https://orcid.org/0000-0002-7730-9060

Методы получения лития и перспективы его применения: обзор

[1]Ерсайынова А.А., [1]Қаршыға З.Б., [2]Muhammad N.A.A.,
[1]Есенгазиев А.М., [1]Орынбаев Б.М.

[1] АО Институт металлургии и обогащения, Satbayev University, Алматы, Казахстан
[2]Университет Пендидикан Султан Идрис, Перак, Малайзия

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АННОТАЦИЯ

Литий - важнейшее сырье для производства современной электроники и электромобилей. Сегодня невозможно представить ни одно мобильное устройство без литиевых батарей. Роль лития в мировой экономике только возрастает. Производство электромобилей и аккумуляторов способствует сокращению выбросов углекислого газа. Тем не менее отслужившие свой срок литий-ионные батареи представляют опасность для экосистемы. В статье представлены технологические разработки в области добычи лития. Основными источниками лития являются пегматиты, континентальные и геотермальные рассолы, а также глины, морская вода и промышленные рассолы. Основным коммерческим продуктом лития является карбонат лития (Li_2CO_3), который получают в основном при добыче, извлечении и переработке сподуменовых руд и рассолов. Также обсуждается эффективная роль лития в решении таких важных проблем, как загрязнение окружающей среды, изменение климата и растущее истощение природных ресурсов, используемых для производства литий-ионных батарей для электромобилей.

Ключевые слова: перспективы лития, минералы лития, спрос и использование, ресурсы лития, ключевые технологии.

Информация об авторах:

Ерсайынова Альбина Абатқызы
Докторант, лаборатория титана и редких тугоплавких металлов, Институт металлургии и обогащения АО, Satbayev University, ул.Шевченко, 29/133, 050010, Алматы, Казахстан. Email: a.yersaiynova@stud.satbayev.university; ORCID ID: https://orcid.org/0000-0003-0638-380X

Қаршыға Зәуре Байтасқызы	*Ph.D., ведущий научный сотрудник, ассоциированный профессор, лаборатория титана и редких тугоплавких металлов, Институт металлургии и обогащения АО, Satbayev University, ул. Шевченко, 29/133, 050010, Алматы, Казахстан. Email: z.karshyga@satbayev.university; ORCID ID: https://orcid.org/0000-0002-3025-7363*
Мухаммед Нуразлан Абд Азис	*Ph.D., ассоциированный профессор, факультет естественных наук и математики, Университет Пендидикан Султан Идрис, 35900 Танджонг Малим, Перак, Малайзия. Email:azlanmn@fsmt.upsi.edu.my; ORCID ID: https://orcid.org/0000-0002-2792-4145*
Есенгазиев Азамат Муратович	*Ph.D., заведующий лабораторией титана и редких тугоплавких металлов, Институт металлургии и обогащения АО, Satbayev University, ул.Шевченко, 29/133, 050010, Алматы, Казахстан. Email: a.yessengaziyev@satbayev.university; ORCiD ID: https://orcid.org/0000-0002-4989-4119*
Орынбаев Бауыржан Мұнарбайұлы	*Докторант, лаборатория титана и редких тугоплавких металлов, Институт металлургии и обогащения АО, Satbayev University, ул.Шевченко, 29/133, 050010, Алматы, Казахстан. Email: Bauyrzhan.Orynbayev@stud.satbayev.university; ORCID ID: https://orcid.org/0000-0002-7730-9060*

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Metallurgy

Development of a technology for the production of aluminum castings using 3d printing of models and lost-was casting

[1*]Merkibayev Y.S., [1]Chepushtanova T.A., [1]Berlibek A.M., [1]Tolegenova A.K. , [2]Nugumarov Sh.T.

[1]Satbayev University, Almaty, Kazakhstan
[2]Holding Inc, Almaty, Kazakhstan

** Corresponding author email: y.merkibayev@satbayev.university*

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ABSTRACT

Industrial development of the Republic of Kazakhstan requires accelerated formation of industries with high added value, capable of meeting domestic needs and increasing export potential. Despite the potential for innovative development, additive technologies for the production of metal products have limited application in traditional mechanical engineering industries due to the high cost of equipment (for example, 3D printers for metals) and, as a consequence, the high cost of production. This factor limits their use in serial production. Thus, the development and implementation of new casting technologies based on the integration of modern scientific achievements and advanced technical solutions is an important task for ensuring sustainable growth of high-tech and competitive industries in Kazakhstan. Development and implementation of technology for the production of aluminum castings of complex shapes using 3D printing of models and investment casting, which will reduce production costs, shorten manufacturing time and improve product quality for strategically important industries. The study used comparative analysis methods and experimental studies aimed at studying the mechanical properties of products manufactured using various technologies, as well as optimizing 3D printing parameters to achieve better product characteristics. The results showed that additive technologies provide high accuracy, allow you to create complex geometric shapes and reduce waste. However, to improve the mechanical properties of products, such as strength and wear resistance, it is necessary to optimize the extrusion parameters during 3D printing. The findings of the study confirm that the choice of technology depends on the specific conditions and requirements for the product. Additive technologies, despite the existing advantages, require further research to improve the properties of final products. The practical significance of the work is that the results of the study can help manufacturers choose the most efficient and cost-effective production methods, which in turn will lead to reduced costs and improved product quality, as well as improved environmental performance.

Keywords: 3D printing, investment casting, aluminum alloys, additive manufacturing, industrial manufacturing, innovative manufacturing processes.

	Information about authors:
Merkibayev Yerik Serikovich	*Ph.D., senior Lecturer of the Mining and Metallurgical Institute, Satbayev University, Almaty, Kazakhstan. Email: y.merkibayev@satbayev.university; ORCID ID:https://orcid.org/0000-0003-3869-6835*
Chepushtanova Tatyana Alexandrovna	*Candidate of Technical Sciences, PhD, Associate Professor Department Metallurgy and Mineral Processing, Associate Professor, Mining and Metallurgical Institute, Satbayev University, Almaty, Kazakhstan Email: T.Chepushtanova@satbayev.university; ORCID ID: https://orcid.org/ 0000-0002-6526-0044*
Berlibek Arailym Muratqyzy	*Doctoral student, Master of the Department of Metallurgy and Mineral Processing, Mining and Metallurgical Institute named after O.A. Baikonurov, Satbayev University, Almaty, Kazakhstan. Email: berlibek99mail.ru; ORCID ID: https://orcid.org/0009-0001-0163-6911*
Tolegenova Aigerim Kairatovna	*Doctor PhD, lecturer of the Department of Construction and Building Materials, Institute of Architecture and Construction, Satbayev University, Almaty, Kazakhstan. Email: a.tolegenova@satbayev.university; ORCID ID: https://orcid.org/0000-0003-1312-4101*
Nugumarov Shyngys Tursuntayuly	*Director of Atlanta Holding Inc, Almaty, Kazakhstan.*

Introduction

The industrial development of the Republic of Kazakhstan requires accelerated formation of high-value-added production facilities capable of meeting domestic needs and increasing export potential [1]. Cast aluminum products are widely used in such strategically important industries as mechanical engineering, oil and gas industry, electrical engineering, automotive industry and agricultural machinery [2]. Despite this, the country's domestic market remains significantly dependent on imports of finished products, which indicates the need to develop local production [3].

Traditional aluminium casting methods such as sand casting, chill casting and die casting are characterized by high process complexity and significant tooling costs [4]. This leads to increased cost of finished products and longer production cycles. In addition, these methods demonstrate low environmental sustainability and efficiency, which is important in the context of modern environmental compliance requirements [5].

On the other hand, additive manufacturing technologies for metal products, despite their great innovation potential, have limited application in traditional mechanical engineering industries. The main obstacles are the high cost of equipment, such as 3D printers for metals, and, as a result, the high cost of production [6]. These factors significantly limit their use in serial production, preventing the full potential of additive technologies from being realized. Thus, the development and implementation of new casting technologies based on the integration of modern scientific achievements and advanced technical solutions is an important task for ensuring sustainable growth of high-tech and competitive industries in Kazakhstan.

Mechanical response measurements of unidirectional 3D printed PLA

In this study, all samples were manufactured using a Flashforge Replicator FDM 3D printer with 1.75 mm diameter PLA filament purchased from IMAKR. The printer hot extrudes PLA filament through a 0.4 mm diameter nozzle, depositing the extruded filament onto a heated metal platform using a moving print head in a pre-set pattern. After each layer is completed, the print head rises to move to the next layer [[7], [8], [9]]. The user can control the layer thickness, deposition rate, extrusion temperature, and PLA filament feed rate, while the printer automatically adjusts the distance between adjacent layers based on these parameters, maintaining volume. In the subsequent study, the deposition directions were defined as axial (x), transverse (y), and out-of-plane (z) [9]. Although the printer is capable of producing parts with multi-directional texture, this study focuses on uniaxial 3D printed materials, where the deposition direction is the same in all layers. Before block fabrication, the sensitivity of the material microstructure to the printing parameters was assessed. Uniaxial blocks were fabricated with different printing parameters as listed in Table 1. Sensitivity of material porosity to user-defined 3D printing parameters. The shaded row indicates the fabrication conditions (T10) for the samples tested in this study.

Table 1 - Different printing parameters of uniaxial blocks

Pr. ex-t	Layer heigt (mm)	Extruder temp. (°C)	Extruder speed (mm/s)	Porosity (%)
T1	0.2	200	45	5.66
T2	0.2	200	45	7.53
T3	0.2	210	45	4.71
T4	0.2	230	45	5.83
T5	0.2	240	45	7.28
T6	0.1	220	45	3.43
T7	0.3	220	45	13.54
T8	0.4	220	45	7.01
T9	0.2	220	30	4.49
T10	0.2	220	60	1.46
T11	0.2	230	75	5.45
T12	0.2	230	100	6.67
T13	0.2	240	100	5.79
T14	0.2	240	125	6.32
T15	0.2	240	150	7.70

The blocks were polished and examined using optical and SEM microscopy to measure porosity and analyze the microstructure. Porosity was determined by converting optical micrographs into binary images to estimate the void-to-total area ratio, with a minimum of three photographs analyzed and average porosity values recorded. No additional surface features were detected during the study, except for rare rounded pores. Extrusion parameters ranged from 200-240°C, layer heights ranged from 0.1 to 0.4 mm, and deposition speeds ranged from 45 to 150 mm/s. The optimal parameters for printing were the extrusion temperature of 220°C, 0.2 mm layer height and 60 mm/s deposition speed resulted in an average porosity of 1.5% with random spherical pores of about 100 µm in diameter. These data demonstrated the high sensitivity of the 3D printer material porosity to the printing parameters [[10], [11]]. PLA was chosen for the study for practical reasons, as it was the most suitable material for this printer, allowing the production of low-porosity blocks. In contrast to carbon fibre-reinforced PLA, ABS and nylon, which yielded porosity in the order of 5-10%, PLA showed significantly lower porosity. Preliminary tests showed that the strength of PLA was twice that of ABS and 20% higher than carbon fibre-reinforced PLA. The mechanical response of the 3D printed PLA produced by the fused deposition method was measured by testing the material along several directions. The study revealed anisotropic and asymmetric fracture behavior depending on the deposition direction. The presented dataset demonstrates that 3D printing enhances the mechanical properties of PLA compared to injection molded PLA [11]. The porosity of the 3D printed material can be minimized, which improves the mechanical response, by optimizing the extrusion temperature, extrusion speed, and print head speed. In this study, samples with a porosity of about 1% were tested. Compared to homogeneous polymers, the 3D printing process increases the crystallinity of the material, reduces its plasticity, and increases its fracture toughness and strain rate sensitivity. The elastic response of the 3D printed material is transversely isotropic, although the anisotropy is mild. The stiffness in both the axial and transverse directions is similar to that of injection-molded PLA, indicating that 3D printing does not significantly affect the elasticity of the material. The inelastic response of the material is plastic and orthotropic. When stretched, the material is more brittle in the out-of-plane direction and more plastic in the plane.

Investment Casting: From 3D Printing to Functional Parts

The process begins with the development of a 3D model, which is created in Fusion360 CAD software. At this stage, elements such as the core and sprue are taken into account, which will ensure the correct flow of aluminum into the target mold [12]. It is also important to determine the level of detail of the future model. After this, the model is exported and sliced using Flashprint, a program compatible with the 3D printer used. The sliced model is then printed using the Flash Forge Creator 3 Pro 3D printer [13].

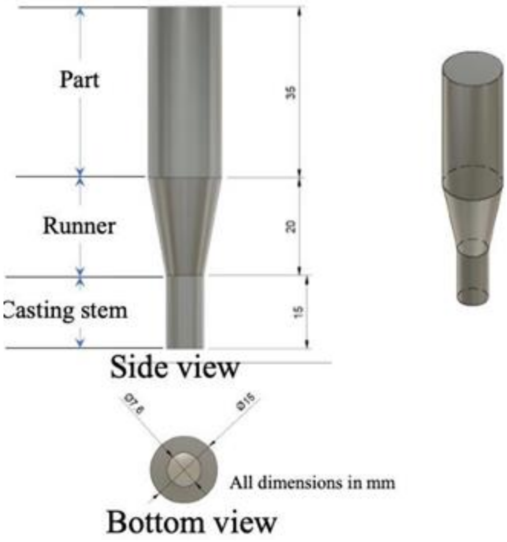

Figure 1 - Proposed 3D model

Next comes the preparation of the plaster mold. To do this, 1 kg of Prestige ORO Plaster is mixed with 380 ml of water, after which the mixture is vacuumed using a Kaya Vacuum Casting machine for 5 minutes. The 3D printed model is fixed in a vacuum casting flask, after which the mold is filled with plaster and vacuumed again. The plaster mold is then fired at 600 °C in a Thermolyne Premium muffle furnace for two hours. This step is necessary to improve the mechanical properties of the plaster and melt the PLA, leaving a hollow mold with precise details. After preparing the mold, the aluminum is melted in a MIFCO induction furnace, where it is heated to 930 °C for about one hour [[14], [15]]. Once the aluminum is melted, it is poured into the prepared plaster mold, which is then cooled, ensuring that the metal hardens in the specified contours. After cooling, the plaster mold is dissolved in water and the cast aluminum element is extracted using a high-pressure hose. Then, the post-processing steps are performed: excess aluminum that has solidified in the sprue area is removed and the surface is ground to achieve the required smoothness. This method allows for the acceleration of the product creation process, ensuring accuracy, repeatability and high quality. The process includes several key steps, starting from the development of the 3D model and finishing with post-processing, which makes it possible to obtain high-quality functional parts with high detail, as shown in Figure 1 [16].

Influence of process parameters on surface looseness and quality in rapid investment casting.

The rapid investment casting (RIC, Fig. 2) process differs from traditional investment casting (IC) by using additive manufacturing technologies to produce sacrificial patterns [[18], [19]]. The time and cost savings offered by RIC make it attractive for low-volume production and complex patterns that are difficult to manufacture using injection molding, such as molds created by topology optimization [19]. The method provides the flexibility to create complex patterns directly from a CAD file and avoids the required tooling step used in the traditional process [20]. Wax-based patterns in traditional IC are prone to shrinkage in thicker sections and distortion of thin features when the pattern is removed from the mold [21]. However, RIC offers the ability to produce thin-walled geometries with precise dimensional tolerance control due to the rigidity of 3D-printed patterns. Most importantly, RIC offers a much lower capital cost compared to direct metal 3D printing technologies such as selective laser sintering (SLS) [22].

The study evaluated the effect of different materials and technologies on the properties of investment casting shells. Specimens were produced using two coating types: with and without 50/100-grain plaster. Thermoplastics ABS, PLA, and PVB, as well as IC wax, were used for the analysis.

The thermoplastic templates were 3D printed, while the wax templates were cast in a mold. The effect of the materials on the brittleness of the shells was related to their melting point and burnout temperature, which affected the rate of melting and absorption of the material by the shell. Micrographs and SEM images showed that the most pronounced surface damage was observed in ABS samples, which corresponds to high brittleness values. Mass loss occurs due to the separation of loosely bonded particles from the shell during the burnout process. Specimens with plaster had increased friability caused by cracks and voids forming around large plaster particles. This weakened the shell matrix, leading to greater failure during the burnout process. Specimens without plaster showed less erosion due to the thicker layer of quartz flour providing better protection [23].

It was also found that PVB, unlike ABS and PLA, does not cause cracking of the shell due to thermal expansion, making it a more suitable material for patterns. The thermomechanical properties of PVB, in particular its low maximum tan(δ) temperature, make it more fluid at lower temperatures, which helps to better match the texture of the particles on the inner surface of the mold and reduces the likelihood of fragmentation. This is also confirmed by the low friability and reduced fragmentation of shells using PVB. Shell fragmentation is important for the quality of cast surfaces, as broken fragments can cause defects in castings. The example in Fig. 2 shows that ABS and PLA castings have higher surface accuracy compared to PVB castings, where 3D printer lines are visible [24].

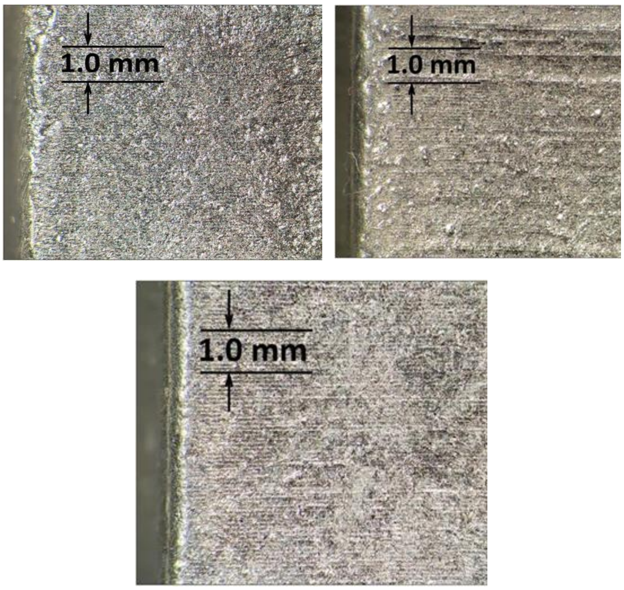

Figure 2 - ABS and PLA castings

However, the surfaces of PVB castings show a lower degree of damage, confirming its advantages for use in investment casting. The present study showed that using PVB patterns instead of ABS and PLA significantly reduces the embrittlement of ceramic shells and the associated casting defects in the FFF-based RIC process. The effect of the distance between the coarse plaster particles and the pattern surface on the embrittlement of ceramic molds was found to be significant. The embrittlement of shells was reduced by 25%, 35%, 55% and 80% for ABS, PLA, PVB and wax, respectively, when the face coat did not contain plaster. The greatest reduction in embrittlement was observed for wax patterns, confirming the relevance of the results for the traditional IC industry. The use of PVB patterns in combination with a modified face coat (without plaster) leads to the minimization of surface defects such as roughness and pitting. Since shell inclusion defects are associated with embrittlement, they can also be reduced. ABS and PLA models with plaster in the face layer show the worst results in terms of surface quality and inclusion defects.

The methodology proposed in the study can be adapted for use in other 3D printing technologies such as SLA and SLS. Alternative materials for the face layer plaster can be considered for further research, such as mullite fireclays with higher surface roughness and different particle size distributions. The cell size and particle shape of the plaster can affect the formation of cracks due to drying shrinkage. Spherical plaster particles have a low surface-to-volume ratio, which can reduce the surface area of cracks. The plaster application rate and drying conditions, which can affect the shrinkage and friability of the shell, should also be investigated.

Thermal Properties Of 3d Printed Light Metal Casting Molds

In foundries, various types of sand and aggregates are used. However, most foundries process silicon ($SiO2$) due to its low cost, availability and chemical resistance [25].

Alternative mold materials such as chromite ($FeCr2O4$ $Al2O3$ MgO) or ceratites ($Al2O3$ $SiO2$) behave better in terms of thermal stability but are also associated with higher costs. Especially for 3D printing, sand properties such as flowability play a major role in creating a smooth and homogeneous surface on the powder bed [26].

Established binders such as furan or phenolic resins are categorized as organic systems. Mold materials associated with organic systems release hazardous compounds during combustion such as BTX (benzene, toluene and xylene), CO and CO2 [25].

Inorganic systems are used due to lower emissions during casting compared to organic systems. When using cores made using inorganic systems, water vapour is mainly emitted [24].

Furan-based technologies account for 80% of the current sand 3D printing market share, while phenolic and inorganic systems share the remaining market shares [17].

In this study, an in-house developed measuring setup was used to determine the thermal properties of the materials. Table 2 presents the sands and binders studied along with their AFS fineness number information.

The density of each sample was calculated and the binder composition was also reported. Sand and binder configurations used layer thicknesses ranging from 0.28 to 0.30 mm. The furan printing process used sand pre-mixed with sulfuric acid followed by selective injection of furfuryl alcohol. The cold cure system uses these components, while the phenolic system uses untreated sand and infrared lamps for curing. The inorganic system uses liquid glass, where resin bridges are formed and cured by dehydration. The binder type does not significantly affect the heat capacity, since its content in the sand varies from 1.2 to 3.4 wt%. However, the heat capacity related to volume can change depending on the thermal behaviour of different sand types. The graphs show the heat capacity measurement for two types of materials, silicon and cerabeads, printed with a phenolic binder. When silica is heated, quartz undergoes an inversion at about 573°C, resulting in a volume expansion of 0.8%. This effect significantly affects the heat capacity of the material. Specific heat capacity was measured at 100°C, 300°C, 500°C and 700°C. The average heat capacity values at these temperatures are plotted for different sand and binder combinations. Of all silica types, the furan (SiFu) variation showed the highest heat capacity values. Chromite showed low heat capacity values by weight, but when density was taken into account, its ability to absorb thermal energy was significantly higher than that of silica-based products or cerabeads. All plots showed a gradual increase in heat capacity with increasing temperature, followed by a decline after reaching a peak. Inorganically bound sand (SiO) showed much lower heat absorption capacity, which is attributed to chemical reactions such as pyrolysis or combustion occurring in the organically bound sand when exposed to heat. Molding sand is a complex

Table 2 - Samples printed on a 3D sand printer, overview

Aggregate	Binder	Short-term	Density ½g/cm^3]	Binder content ½wt%]
Silica (AFS97)	Furan	SiFu	1.28	1.2
Silica (AFS97)	Phenol	SiPh	1.32	2.2-2.4
Silica (AFS97)	Inorganic	SiIO	1.25	2.8-3.4
Cerabeads (AFS65)	Furan	CBFu	1.48	1.1
Cerabeads (AFS65)	Phenol	CBPh	1.43	1.9
Chromite (AFS71)	Phenol	ChroPh	2.31	3.2

composite of grains and bonding bridges that wet the sand to varying degrees. The surface roughness and texture of the sand affect the heat transfer in the STA/DSC measuring crucible, which may explain the variance in the measurements. Each sample was placed in the crucible as a whole to simulate mold conditions. Conduction and convection are responsible for heat transfer rather than radiation, despite the air content of the material being around 50%. These differences are not visually noticeable, so the term thermal conductivity is applicable in a general context. STA/DSC measurements were performed under argon to prevent combustion and to induce pyrolysis of the binder. The influence of chemical reactions on the parameters requires further investigation.

The Role and Impact of 3D Printing Technologies in Casting

The casting process is complex and involves several steps such as making patterns by hand or using machine tools, and creating cores and molds, which opens up the possibility of applying 3D printing technologies in the foundry. The core is formed in a core box, and the pattern is used to create the mold cavity and the core. Thus, making the pattern, core and mold is the first step in casting, but the processes for creating them can vary significantly. Some of them are done by hand, using machine tools, forging, firing clay or casting with low melting point alloys. All of these processes can be difficult and confusing, sometimes even similar to the dilemma of "what comes first: the chicken or the egg". There are many methods of casting, such as sand casting, investment casting and shell molding. Investment casting uses foam or wax templates that are then melted out during the molding process. Clay or foam is used to produce the templates and the mold cavity dies can be made by hand or by machine [27].

Conclusion

This study has demonstrated important aspects regarding the mechanical properties and manufacturing technology of 3D printed parts and their interaction with aluminum casting. The revealed asymmetry in tension and compression highlights the need to consider the material properties when using it in various applications. 3D printed PLA, being stiffer than cast PLA, opens up new possibilities for designing and creating parts, but also requires a careful strategic approach to heat treatment, as this can significantly affect the strength of the product.

Using the investment casting process for 3D printed parts has shown significant advantages over traditional methods, especially in terms of cost and waste reduction. The results confirmed that investment casting can be an optimal solution for obtaining metal parts with high accuracy and quality, which is relevant for serial production, including using recycled materials.

However, further research into the interactions at the shell-template interface and the mechanisms affecting strength is necessary to better understand the processes occurring under these conditions. This will allow for a more accurate prediction of the characteristics of the final products. The measurement methodology used, taking into account all its nuances and potential sources of errors, leaves room for improvement and increased accuracy, which will contribute to the development of this technology. In the future, to expand and improve casting methods using 3D printed templates, it is advisable to conduct more detailed thermomechanical and thermal analyses, which will help to obtain a more complete understanding of the properties of materials and their behaviour in different temperature ranges. Taking into account the above, the results of the work can have a positive impact on industrial development and innovative approaches in production, including possible implementation in wider practice.

Conflicts of Interest. On behalf of all authors, the correspondent author declares that there is no conflict of interest.

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Үлгілерді 3D басып шығару және балқытылатын үлгілерді құюды қолдану арқылы алюминий құймаларын өндіру технологиясын әзірлеу

[1]Меркибаев Е.С., [1]Чепуштанова Т.А. , [1]Берлібек А.М., [1]Толегенова А.К., [2]Нугумаров Ш.Т.

[1]Сәтбаев университеті, Алматы, Қазақстан
[2]Holding Inc, Алматы, Қазақстан

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ТҮЙІНДЕМЕ

Жұмыстың өзектілігі. Қазақстан Республикасының өнеркәсіптік дамуы ішкі қажеттіліктерді қанағаттандыруға және экспорттық әлеуетті арттыруға қабілетті, қосылған құны жоғары өндірістерді жеделдетіп қалыптастыруды талап етеді. Металл бұйымдарын өндірудің аддитивті технологиялары, инновациялық даму әлеуетіне қарамастан, жабдықтың жоғары құнына (мысалы, металдарға арналған 3D принтерлер) және соның салдары ретінде өндірістің жоғары құнына байланысты дәстүрлі машина жасау салаларында қолданудың мүмкіндігі шектеулі. Бұл фактор оларды жаппай өндірісте пайдалануды шектейді. Осылайша, қазіргі заманғы ғылыми жетістіктерді және озық техникалық шешімдерді біріктіру негізінде құюдың жаңа технологияларын әзірлеу және енгізу Қазақстандағы жоғары технологиялық және бәсекеге қабілетті өндірістердің тұрақты өсуін қамтамасыз етудің маңызды міндеті болып табылады. Жұмыстың мақсаты. Үлгілерді 3D басып шығаруды және балқытылатын үлгілерді құюды қолдана отырып, күрделі пішіндегі алюминий құймаларын өндіру технологиясын әзірлеу және енгізу, бұл стратегиялық маңызды салалар үшін өнімнің өзіндік құнын төмендетуге, өндіру уақытын қысқартуға және өнімнің сапасын жақсартуға мүмкіндік береді. Алынған нәтижелер аддитивті технологиялардың жоғары дәлдікті қамтамасыз ететінін, күрделі геометриялық фигураларды жасауға және қалдықтарды азайтуға мүмкіндік беретінін көрсетті. Дегенмен, өнімнің беріктігі мен тозуға төзімділігі сияқты механикалық қасиеттерін жақсарту үшін 3D басып шығарудағы экструзия параметрлерін оңтайландыру қажет. Аддитивті технологиялар, олардың артықшылықтарына қарамастан, соңғы өнімнің қасиеттерін жақсарту үшін қосымша зерттеулерді қажет етеді. Жұмыстың практикалық маңыздылығы мынада: зерттеу нәтижелері өндірушілерге ең тиімді және үнемді өндіріс әдістерін таңдауға көмектеседі, бұл өз кезегінде шығындарды азайтуға және өнім сапасын жақсартуға, сонымен қатар қоршаған ортаны қорғау көрсеткіштерін жақсартуға әкеледі.

Түйін сөздер: 3D басып шығару, балқытылатын үлгілер бойынша құю, алюминий қорытпалары, аддитивті технологиялар, өнеркәсіптік өндіріс, инновациялық өндіріс процестері.

	Авторлар туралы ақпарат:
Меркибаев Ерик Серикович	*PhD докторы, Металлургия және пайдалы қазбаларды байыту кафедрасының аға оқытушысы, Ө.А. Байқоңыров атындағы тау-кен-металлургия институты, Сәтбаев университеті, Алматы, Қазақстан. Email: y.merkibayev@satbayev.university; ORCID ID: https://orcid.org/0000-0003-3869-6835*
Чепуштанова Татьяна Александровна	*PhD докторы, техника ғылымдарының кандидаты, Металлургия және пайдалы қазбаларды байыту кафедрасының қауымдастырылған профессоры, Ө.А. Байқоңыров атындағы тау-кен-металлургия институты, Сәтбаев университеті, Алматы, Қазақстан. Email: T.Chepushtanova@satbayev.university; ORCID ID: https://orcid.org/ 0000-0002-6526-0044*

Берлібек Арайлым Мұратқызы	*Докторант, Металлургия және пайдалы қазбаларды байыту кафедрасының магистры, Ө.А. Байқоңыров атындағы тау-кен-металлургия институты, Сәтбаев университеті, Алматы, Қазақстан. Email: berlibek99mail.ru; ORCID ID: https://orcid.org/0009-0001-0163-6911*
Толегенова Айгерим Кайратовна	*PhD докторы, Құрылыс және құрылыс материалдары кафедрасының оқытушысы, Сәулет және құрылыс институты, Сәтбаев университеті, Алматы, Қазақстан. Email: a.tolegenova@satbayev.university; ORCID ID: https://orcid.org/0000-0003-1312-4101*
Нугумаров Шынгыс Тұрсұнтайұлы	*Директор Atlanta Holding Inc, Алматы, Қазақстан.*

Разработка технологии производства алюминиевых отливок с использованием 3d-печати моделей и литья по выплавляемым моделям

[1]Меркибаев Е.С., [1]ЧепуштановаТ.А., [1]БерлибекА.М., [1]Толегенова А.К., [2]Нугумаров Ш.Т.

[1]Satbayev University, Алматы, Казахстан
[2]Holding Inc, Алматы, Казахстан

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АННОТАЦИЯ

Актуальность работы. Индустриальное развитие Республики Казахстан требует ускоренного формирования производств с высокой добавленной стоимостью, способных обеспечить внутренние потребности и повысить экспортный потенциал. Аддитивные технологии производства металлических изделий, несмотря на потенциал для инновационного развития, имеют ограниченное применение в традиционных отраслях машиностроения из-за высокой стоимости оборудования (например, 3D-принтеров для металлов) и, как следствие, высокой себестоимости продукции. Этот фактор ограничивает их использование в серийном производстве. Таким образом, разработка и внедрение новых технологий литья, основанных на интеграции современных научных достижений и передовых технических решений, является важной задачей для обеспечения устойчивого роста высокотехнологичных и конкурентоспособных производств в Казахстане. Цель работы. Разработка и внедрение технологии производства алюминиевых отливок сложной формы с использованием 3D-печати моделей и литья по выплавляемым моделям, что позволит снизить производственные затраты, сократить время изготовления и повысить качество продукции для стратегически важных отраслей промышленности. В процессе исследования применены методы сравнительного анализа и экспериментальные исследования, направленные на изучение механических свойств изделий, изготовленных с использованием различных технологий, а также оптимизацию параметров 3D-печати для достижения лучших характеристик продукции. Полученные результаты показали, что аддитивные технологии обеспечивают высокую точность, позволяют создавать сложные геометрические формы и снижают отходы. Однако для улучшения механических свойств изделий, таких как прочность и износостойкость, требуется оптимизация параметров экструзии при 3D-печати. Выводы исследования подтверждают, что выбор технологии зависит от конкретных условий и требований к изделию. Аддитивные технологии, несмотря на имеющиеся преимущества, требуют дальнейших исследований для улучшения свойств конечных продуктов. Практическая значимость работы заключается в том,что результаты исследования могут содействовать производителям выбрать наиболее эффективные и экономичные методы производства, что в свою очередь приведет к снижению затрат и повышению качества продукции, а также улучшению экологических показателей.

Keywords: 3D-печать, литье по выплавляемым моделям, алюминиевые сплавы, аддитивные технологии, промышленное производство, инновационные производственные процессы.

	Информация об авторах:
Меркибаев Ерик Серикович	*Доктор PhD, старший преподаватель кафедры металлургии и обогащения полезных ископаемых, Горно-металлургический институт имени О.А. Байконурова, Satbayev university, Алматы, Казахстан. Email: y.merkibayev@satbayev.university; ORCID ID: https://orcid.org/0000-0003-3869-6835*
Чепуштанова Татьяна Александровна	*Доктор PhD, кандидат технических наук, ассоциированный профессор кафедры металлургии и обогащения полезных ископаемых, Горно-металлургический институт имени О.А. Байконурова, Satbayev University, Алматы, Казахстан. Email: T.Chepushtanova@satbayev.university; ORCID ID: https://orcid.org/0000-0002-6526-0044*
Берлібек Арайлым Мұратқызы	*Докторант, магистр кафедры металлургии и обогащения полезных ископаемых, Горно-металлургический институт имени О.А. Байконурова, Satbayev University, Алматы, Казахстан. Email: berlibek99mail.ru; ORCID ID: https://orcid.org/0009-0001-0163-6911*
Толегенова Айгерим Кайратовна	*Доктор PhD, преподаватель кафедры строительства и строительных материалов, Институт архитектуры и строительства, Satbayev University, Алматы, Казахстан. Email: a.tolegenova@satbayev.university; ORCID ID: https://orcid.org/0000-0003-1312-4101*
Нугумаров Шынгыс Тұрсұнтайұлы	*Директор Atlanta Holding Inc, Алматы, Казахстан.*

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Техникалық редакторлар:
Г.К. Қасымова, Н.М.Айтжанова, Т.И. Қожахметов

Компьютердегі макет:
Г.К. Қасымова

Дизайнер:
Г.К. Қасымова, Н.М.Айтжанова

“Металлургия және кен байыту институты” АҚ
050010, Қазақстан Республикасы, Алматы қаласы, Шевченко к-сі, 29/133

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Технические редакторы:
Г.К. Касымова, Н.М. Айтжанова, Т.И. Кожахметов

Верстка на компьютере:
Г.К. Касымова

Дизайнер:
Г.К. Касымова, Н.М.Айтжанова

АО “Институт металлургии и обогащения”
050010, г. Алматы, Республика Казахстан. ул. Шевченко, 29/133

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G.K. Kassymova, N.M. Aitzhanova, T.I. Kozhakhmetov

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“Institute of Metallurgy and Ore Benficiation” JSC
050010, Almaty city, the Republic of Kazakhstan. Shevchenko str., 29/133

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