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THE USE OF METAKAOLIN-BASED GEOPOLYMERS FOR WASTEWATER TREATMENT

Abstract. The article presents methods for the purification and removal of cadmium and lead ions using materials based on metakaolin, cement, Kokshetau clay, and geopolymers. The adsorption properties were studied during the preparation of a model solution using cadmium and lead salts. The efficiency of the purification process was evaluated using infrared (IR) spectroscopy and atomic emission spectroscopy methods. The results demonstrate that geopolymers serve as an effective and eco-friendly alternative for treating wastewater contaminated with heavy metals. The findings highlight the potential of geopolymers as an innovative and sustainable approach to wastewater management, reducing environmental pollution and associated health risks. The results of the work showed the possibility of using Geopolymers as an environmentally effective and alternative method for treating wastewater contaminated with heavy metals.

Keywords: wastewater, water treatment, metacaolin, Geopolymers, heavy metals, adsorption, IR spectroscopy.

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Introduction. The increasing contamination of wastewater with heavy metals worldwide leads to environmental degradation, health problems, and ecosystem disruption. Approximately 25% of the global population consumes drinking water contaminated with heavy metals and other hazardous substances, posing a significant threat to human health. Addressing this issue involves a well-established process known as wastewater management, which includes wastewater collection, purification using geopolymers, and reuse [1,2]. Efforts to combat water pollution in many countries have undoubtedly been crowned with some success. However, the problems continue to get more complicated and become more serious. In developing Asia, about 785 million people do not have access to safe and sustainable water sources. This pollution is mainly caused by the discharge of industrial wastewater. As in many other countries, wastewater pollution and treatment remain urgent problems in Kazakhstan. According to various sources, only 58 out of 87 cities in Kazakhstan have sewage treatment plants, and many of these facilities do not meet regulatory standards or function improperly [3].

Thus, the removal of heavy metals harmful to human health (such as cadmium and lead) using geopolymers is an important and environmentally safe alternative. Currently, wastewater filtration sites in the Zhambyl region are in unsatisfactory condition. This negatively affects the drinking water supply of settlements throughout the region, harms human health, and damages agricultural lands [4]. The main source of pollution in the southwestern part of the region is related to phosphate mining and processing. According to data, the annual volume of untreated wastewater reaches approximately 23,028,000 cubic meters per year [5]. If wastewater is not pretreated, it may lead to groundwater contamination and pose a threat of hazardous flooding in settlements near Zhambyl district [6]. Therefore, the removal of heavy metals from water using geopolymers remains one of the most urgent issues today.

Biological treatment of wastewater represents significant progress in environmental protection. However, wastewater treatment plants generate sludge, which is one of the most common types of waste. Incineration is one method of sludge disposal, but it does not fully meet all requirements. Firstly, due to the high water content (80-90%), it requires energy-intensive and inefficient drying. Secondly, the incineration process leads to the release of toxic gases, such as carbon dioxide [7].

In recent years, the need for effective water resource management and solutions to environmental problems has increased. In this context, the use of metakaolin-based geopolymers in wastewater treatment has gained significant attention [8]. Geopolymers are recognized as environmentally friendly materials due to their high mechanical and chemical stability.

Geopolymers are a new class of synthetic aluminosilicate materials obtained through the alkaline activation of various aluminosilicate raw materials, such as fly ash, natural clays, zeolites, and feldspar [9]. These eco-friendly materials compete significantly with Portland cement-based construction materials since their production does not emit CO_2, involves minimal costs, and exhibits superior mechanical properties compared to other materials [10].

The structure of geopolymers consists of alternating chains of silicon and aluminum atoms, classified by J. Davidovits as polysilicates (–Si–O–Al–O–), poly (silicate-sialate) (–Si–O–Al–O–Si–O–), and poly (silicate-disialate) (–Si–O–Al–O–Si–O–Si–O–) [11]. Similar to zeolites, silicon $[SiO_4]^{4-}$ and aluminum $[AlO_4]$ tetrahedra in geopolymers form two- and three-dimensional networks, with excess negative charge compensated by exchangeable cations such as Na+, K+ and Ca2+ [12]. However, unlike zeolites, geopolymers mostly have an amorphous structure, and the presence of zeolite phases strongly depends on the synthesis conditions. The addition of an alkaline activator (NaOH, Na_2SiO_3, KOH, K_2SiO_3) to a source of silicon and aluminum leads to their dissolution, followed by a polymerization reaction to form a Si–O–Al–O geopolymer grid. The result is a material with excellent bonding properties, mechanical strength and heat resistance [13]. Due to their low calcium content, copolymers have a higher resistance to acids and salt solutions compared to Portland cement. These properties make geopolymers suitable not only for the construction industry, but also for the disposal and localization of industrial waste [14].

To enhance the mechanical strength and adsorption capacity of geopolymers for pollutants, zeolites are incorporated as fillers during the synthesis process. This addition increases compressive strength and improves the adsorption of methylene blue and copper ions (7.8 mg Cu^{2+}/g) from aqueous solutions. Additionally, the dispersion of Cu_2O particles within the geopolymer matrix results in a novel photoactive composite capable of removing methylene blue from water or air [15,16].

Materials and methods. Preparation of the suspension. Metakaolin powder is added to the solution of the compound Na_2O/Al_2O_3 in molar proportions of 1.0, 1.2, 1.4, 1.6 and 1.8. Then hydrogen peroxide, H_2O_2 and polysorbate 80 are added to the aqueous solution as surfactants.To obtain a foamed suspension, the mixture must be mixed.

Preparation of geopolymer balls. The resulting homogeneous suspension is continuously injected into a polyethylene glycol solution in a water bath at a temperature of 70℃ using syringes for making balls of porous geopolymers. The granules have the shape of a ball and are scattered in solution using a syringe. Inside the solution, the suspension freezes instantly, and when the excess sodium hydroxide is removed, the suspension, which has turned into a ball, can be easily collected.Thus, porous polymer beads were obtained within 24 hours after drying at room temperature. To avoid damage during adsorption, the obtained geopolymer samples were washed with deionized water at 60℃ for 1 hour, and then steamed for 24 hours. Before filtration and drying, the samples were repeatedly washed with deionized water for at least 24 hours until the PH reached 7 ± 0.5. The adsorption process was carried out using a washed geopolymer as a heavy metal absorber.However, the adsorption of Pb(II), Cd(II), Ni(II), and Cu(II) was significantly reduced. Thanks to clay minerals (Kokshetau clay, metakaolin), water was successfully adsorbed.

The following materials were used as the object of research in this article: cement, metakaolin, natural clayey clay. To prepare samples, Kokshetau clay was fired at 650℃, 800℃ for 6 and 12 hours in a muffle furnace. Wastewater contaminated with heavy metals (Cd, Pb) was used as a model solution. The total concentration of the model solution is 50 mg/l.

To obtain the geopolymer material, natural clay was first modified. That is, the materials obtained by firing were prepared (natural clay 6 hours – 650℃, 12 hours – 650℃, 6 hours – 800℃ 12 hours – 800℃. In order to increase the activity of the sample taken as an adsorbent, it was modified according to the method given below. Each piece of Kokshetau clay was weighed on a porcelain plate weighing 100 g. The first 100 grams of clay were fired in a muffle furnace at a temperature of 800℃ for 6 hours. The second clay pot weighing 100 grams was fired in a muffle furnace at a temperature of 800℃ for 12 hours. The third 100-gram clay pot was fired in a muffle furnace at 650℃ for 6 hours. The fourth 100-gram clay pot was fired in a muffle furnace at 650℃ for 6 hours. As a result, 6 samples were prepared, 4 of which were made of clay and metacaolin, cement. Cadmium and lead ions were used to remove metal ions using the obtained adsorbents.

The following tools were used to determine the physico-chemical properties of the obtained adsorbents and geopolymers:

- Elemental analysis;
- Infrared spectroscopy;
- Atomic emission spectroscopy method.

Research results and discussion. According to the results of the analysis, it was found that the metacaolin-based geopolymer contains more of the element O (47.51%). At the same time, Al (22.78%) and Si (27.75) are found in greater quantities than other metals (Table 1).

Table 1

The elemental composition of metacaolin

Metacaolin	Percentage of elemental composition (%)						
	O	Al	Si	K	Ca	Ti	Fe
Average calculation	47.51	22.78	27.75	0.18	0.16	0.66	0.95

According to the results of this analysis, it was seen that in 12 hours the element O (42.03%) in the composition of the material was more. In addition,in terms of metals, Si (23.24%), Al (16.50%) and Fe (15.52%) are found. It has been shown that in 6 hours the element O (40.43%) in the composition of the material is more. In addition , the result shows that there are much more metals in Si (23.47%) and Fe (17.67%), Al (15.57%) than in 12 hours in comparison (Table 2).

Table 2

Elemental composition of Kokshetau clay at 800℃ for 12.6 hours

Kokshetau clay (12.6 hours 800℃)	Percentage of elemental composition (%)							
	O	Mg	Al	Si	K	Ca	Ti	Fe
Average values 12 hours	42.03	0.25	16.50	23.24	0.50	0.24	1.72	15.52
Average values 6 hours	40.43	0.12	15.57	23.47	0.51	0.34	1.89	17.67

According to the results of the analysis, it turned out that in 12 hours the material contains more oxides SiO_2 (47.10%) and Al_2O_3 (29.85%). Also found in the amount of FeO (19.03%). It has been shown that in 6 hours the element O (40.43%) in the composition of the material is more. In addition , the result shows that there are much more metals in Si (23.47%) and Fe (17.67%), Al (15.57%) than in 12 hours in comparison (Table 3).

Table 3

Kokshetau clay oxygen analysis by stoichiometry at 800℃ for 12.6 hours

Kokshetau clay (12.6 hours 800℃)	Percentage of elemental composition (%)						
	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	FeO
Average values 12 hours	0,19	27,76	46,74	0,57	0,44	2,95	21,33
Average values 6 hours	0,40	29,85	47,10	0,57	0,31	2,73	19,03

IR spectroscopy results. The IR spectra of 6 different adsorbents obtained were studied and discussed. The IR spectra of the studied samples were determined based on the results of mineral and inorganic groups present in their composition. In the composition of adsorbents, antigorite, analzyme, akmite,kaolinite, anthrophylite, actionolite, Andalusite, arfvedsonite, astrophyllite, albite and oxides of magnesium and calcium are found. The absorption lines 540 and 563 cm^{-1} relate to the absorption lines for antigorite, actinolite 673 and 683 cm^{-1}, Andalusite 732 and 746 cm-1 and other minerals (Table 4).

Table 4

Results of 6H IR spectroscopy of 1 sample of metacaolin

Natural clay	νAntigorite (cm^{-1})	νActinolite (cm^{-1})	νAndalusian (cm^{-1})	νArfvedsonite (cm^{-1})	νCalcium oxide (cm^{-1})	νMagnium oxide (cm^{-1})
Metacaolin sample 1 6H	554 560	673 683	732 746	784 795	1014 1027	1133 1151

The infrared spectroscopy results of the sample of natural Kokshetau clay, calcined at 650°C for 12 hours, revealed various mineral and inorganic absorption spectra. Specifically, the absorption bands were identified as follows: 555-563 cm^{-1} for antigorite, 609-618 cm^{-1} for analcime, and 728-736 cm^{-1} for acmite. In addition, the absorption bands at 995-1020 cm^{-1} corresponded to calcium oxide, while those at 1125-1140 cm^{-1} indicated the presence of magnesium oxide (Table 5).

Table 5

Results of 12H IR spectroscopy of 1 sample of metakaolin

Natural clay	νAntigorite (cm^{-1})	νAnalcim (cm^{-1})	νAcmite (cm^{-1})	νCalcium oxide (cm^{-1})	νMagnium oxide (cm^{-1})	νAntigorite (cm^{-1})
Metacaolin sample 1 12H	555 563	609 618	728 736	995 1020	1125 1140	555 563

The infrared spectroscopy results of the cement sample showed the absorption spectra of various minerals and inorganic compounds (Table 6). Specifically, the following were observed: 613-621 cm^{-1} for Analcime, 671-677 cm^{-1} for Anhydrite, 1076-1084 cm^{-1} for Antigorite, 725-750 cm^{-1} for Aegirine, and 824-921 cm^{-1} for Anthophyllite. Additionally, Calcium oxide was detected at 964-972 cm^{-1}.

Table 6

IR spectroscopy results of cement sample

Studied samples	νAntigorite (cm^{-1})	νAnalcim (cm^{-1})	νAnghydrite (cm^{-1})	νAcmite (cm^{-1})	νAntrophylitis (cm^{-1})	νCalcium oxide (cm^{-1})
Cement	1076 1084	611 623	671 678	725 750	824 921	1008 1028

As a result of infrared spectroscopy, the metacaolin sample showed spectra of various minerals and inorganic absorption, namely 613-621 cm^{-1} Analzyme, 671-677 cm^{-1} anhydrite, 785-792 cm^{-1} Arfedsonite, 549-557 cm^{-1} Astrophyllite,1134-1138cm^{-1} albite. In addition, 964-972 cm^{-1} showed calcium oxide (Table 7).

Table 7

IR spectroscopy results of the metacaolin sample

Studied samples	νAnalcim (cm^{-1})	νAnghydrite (cm^{-1})	νArfvedsonite (cm^{-1})	νAstrophyllite (cm^{-1})	νCalcium oxide (cm^{-1})	νAlbite (cm^{-1})
Metacaolin	613 621	671 677	785 792	549 557	964 972	1134 1138

Study of the adsorption effect. To determine the effect of the weight of the adsorbent on the adsorption process, 4 different weights ($m_{(adsorbent)}$ = 0.2;0.4;0.6;0.8 g) of each sample were prepared and studied. As a sample, we studied 6 different adsorbents, namely 4 samples of fired Natural Clay, i.e. 6 and 12 hours at 650,800℃ (MC1 6H, MC1 12H, MC2 6H, MC2 12h), metacaolin and cement.According to the results of the studies obtained, the adsorption of the heavy metal ion Cd (II) and Pb (II) increased due to the increase in the weight of MC1 6 H (650℃ 6H fired clayfish). The yield of MS1 6H adsorbent to Cd (II) heavy metal ion was reduced to 0.2 g/l – 46%; 0.4 g/l – 45%; 0.6 g/l – 80%; 0.8 g/l – 94%, respectively. We found that the yield of PB (II) heavy metal ions decreased by 0.2 g/l – 91%; 0.4 g/l – 86%; 0.6 g/l – 94%; 0.8 g/l – 95%, respectively. These results have already reached good indicators due to the increase in the surface area of MS1 6H, can be applied to Cd (II) and Pb (II) adsorption. Based on the results obtained, we can see in the figure 1 below that it has achieved good results in the adsorption of two metal ions.

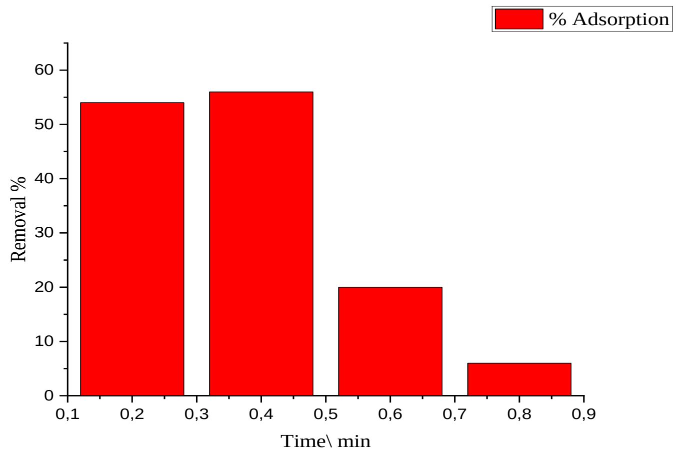

Fig. 1. Study of the adsorption effect

The results of the analysis obtained using the atomic emission spectroscopy instrument for the adsorbent are presented in Tables 8 and 9 below. According to the obtained results, for the Cd (II) ion, at a concentration of 0.8 g/L with a contact time of 12 hours, the yield was 21%, while for the Pb (II) ion, at the same concentration of 0.8 g/L, the yield reached 91%. In these four different samples, as the adsorbent mass increased, the adsorption capacity of the heavy metal also increased. However, for the cadmium ion, adsorption was weak, leading to the conclusion that it is ineffective as an adsorbent.

Table 8

Results of NPP analysis of the effect of different weights on the adsorption process on a heavy metal ion Cd(II)

No.	Name of the adsorbent	Weight, g/l	Tool indication	$C_{Cd(II)}$	Not adsorbed Cd(II), %	Total adsorption yield, %
1	Metacaolin sample1 (650°C, 6h)	*Primary*	*50*	*51.56*	*100%*	
		0.2	1.12	28	54%	46%
		0.4	1.13	28.25	56%	45%
		0.6	0.41	10.25	20%	80%
		0.8	0.12	3	6%	94%
2	Metacaolin sample1 (800°C, 12h)	*Primary*	*1.83*	*52.31*	*100%*	
		0.2	1.67	41.75	80%	20%
		0.4	1.62	40.5	77%	23%
		0.6	1.46	36.5	70%	30%
		0.8	0.98	24.5	47%	53%
3	Metacaolin	*Primary*	*50*	*49.20*	*100%*	
		0.2	1.61	40.25	82%	18%
		0.4	1.45	36.25	74%	26%
		0.6	1.05	26.25	53%	47%
		0.8	1.11	27.75	56%	44%
4	Cement	*Primary*	*50*	*51.28*	*100%*	
		0.2	2.16	54	105%	5%
		0.4	2.07	51.75	101%	1%
		0.6	2.05	51.25	99.94%	0.06%
		0.8	2.08	52	101%	1%
5	Metacaolin sample2 (650°C, 6h)	*Primary*	*50*	*51.15*	*100%*	
		0.2	0.15	3.75	19%	81%
		0.4	0.07	1.75	11%	89%
		0.6	0.10	2.5	14%	86%
		0.8	0.10	2.5	14%	86%
6	Metacaolin sample2 (800°C, 12h)	*Primary*	*50*	*50.35*	*100%*	
		0.2	1.96	49	97%	3%
		0.4	1.85	46.25	92%	8%
		0.6	1.85	46.25	92%	8%
		0.8	1.60	40	79%	21%

Table 9

Results of AES Analysis on the Effect of Different Weights on the Adsorption Process of Pb(II) Heavy Metal Ion

№	Name of the adsorbent	Weight, g/l	Tool indication	$C_{Pb(II)}$	Not adsorbed Pb(II), %	Total adsorption yield, %
1	2	3	4	5	6	7
1	Metacaolin sample1 6h (650°C, 6h)	*Primary*	*50*	*47.06*	*100%*	
		0.2	0.16	4	9%	91%
		0.4	0.26	6.5	14%	86%
		0.6	0.12	3	6%	94%
		0.8	0.10	2.5	5%	95%
2	Metacaolin sample1 12h(800°C, 12h)	*Primary*	*50*	*48.07*	*100%*	
		0.2	0.28	7	15%	85%
		0.4	0.27	6.75	14%	86%
		0.6	0.11	2.75	6%	94%
		0.8	0.08	2	4%	96%

Table 9 (continued)

1	2	3	4	5	6	7
3	Metacaolin	*Primary*	*50*	*43.79*	*100%*	
		0.2	0.44	11	25%	75%
		0.4	0.22	5.5	13%	87%
		0.6	0.16	4	9%	91%
		0.8	0.16	4	9%	91%
4	Cement	*Primary*	*50*	*43.21*	*100%*	
		0.2	0.34	8.5	20%	80%
		0.4	0.26	6.5	15%	85%
		0.6	0.28	7	16%	84%
		0.8	0.38	9.5	22%	78%
5	Metacaolin sample2 6h (650°C, 6h)	*Primary*	*50*	*44.75*	*100%*	
		0.2	0.34	8.5	8%	92%
		0.4	0.20	5	4%	96%
		0.6	0.25	6.25	5%	95%
		0.8	0.25	6.25	5%	95%
6	Metacaolin sample2 12h (800°C, 12h)	*Primary*	*50*	*44.89*	*100%*	
		0.2	0.40	10	22%	78%
		0.4	0.26	6.5	14%	86%
		0.6	0.22	5.5	12%	88%
		0.8	0.16	4	9%	91%

After studying all the samples, our conclusion proved that MS2 as an adsorbent is effective if it takes a weight of 6h at 0.4 g/l. For this work, we examined the purification of Cd(II) metal by 89%, and for Pb(II) metal by 96%. In fact, if we analyze all the results Cd(II) heavy metal ion it has been observed that it is more difficult to purify in water than PB(II) ion. There are currently 3 known ways to purify this cadmium, but only one way to eliminate cadmium in this drinking water is the ion exchange method. Ionites extract 98% of cadmium from water. The disadvantages of water purification from cadmium by Ion Exchange are the limited resource of cartridge operation, the need for frequent resins. Pb (II) uses reverse osmosis and electrodialysis when it comes to metal ion purification. These methods provide only 70-90% lead purification of water, and our developed treatment method provided 96% lead. That is why we used geopolymer adsorbent in this cleaning work. The geopolymer adsorbent, consisting of a layer of metacaolin, has been found not only cheap, simple and environmentally safe, but also reliable. During wastewater treatment, the predominant amount of metal ions was destroyed and reduced.

Conclusion. Geopolymers i.e. metacaolin, cement, clayfoil as adsorbents in recent years for highly environmentally friendly materials, they have aroused great interest. At the moment, they are inexpensive and affordable materials. The basis for wastewater treatment is now cadmium and lead heavy metal ions, both economically and ecologically causes trouble.

According to the results of the study, we took 6 samples, that is, Kokshetau Clay was fired in a muffle furnace for 6,12 hours at 650°C and 6.12 hours at 800°C, cement and metacaolin. Examination of the elemental composition and infrared spectroscopic analyzes were carried out to determine the physico – chemical properties of adsorbents.

After examining all the samples, our conclusion proved that metacaolin as an adsorbent is effective if the sample2 6H weighs 0.4 g/l. For this work, we examined the purification of Cd(II) metal by 89%, and for Pb(II) metal by 96%. In fact, if we analyze all the results Cd(II) heavy metal ion it has been observed that it is more difficult to purify in water than PB(II) ion.

In general, good, that is, positive results were obtained on the studied samples. The geopolymer adsorbent, consisting of a layer of metacaolin, has been found not only cheap, simple and environmentally safe, but also reliable. During wastewater treatment, the predominant amount of metal ions was destroyed and reduced.

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АҒЫНДЫ СУЛАРДЫ ТАЗАРТУ МАҚСАТЫНДА МЕТАКАОЛИН НЕГІЗІНДЕГІ ГЕОПОЛИМЕРЛЕРДІ ҚОЛДАНУ

Аңдатпа. Мақалада метакаолин, цемент, Көкшетау сазы және геополимерлер негізіндегі материалдарды қолданып, кадмий мен қорғасын иондарын тазарту және жою әдістері қарастырылған. Адсорбциялық қасиеттер модельдік ерітіндіні кадмий және қорғасын тұздарын пайдалана отырып дайындау барысында зерттелді. Тазарту үдерісінің тиімділігі инфрақызыл (IR) спектроскопиясы және атомдық эмиссиялық спектроскопия әдістері арқылы бағаланды. Зерттеу нәтижелері геополимерлердің ауыр металдармен ластанған ағын суларды тазартуда тиімді және экологиялық таза балама екенін көрсетті. Бұл нәтижелер геополимерлердің қоршаған ортаны ластауды азайтып, денсаулыққа тигізетін қауіпті төмендету арқылы, инновациялық және тұрақты шешім ретінде әлеуетін айқындайды. Жұмыс нәтижелері геополимерлерді ауыр металдармен ластанған ағын суларды тазартудың экологиялық тиімді және балама әдісі ретінде қолдануға болатындығын көрсетті.

Тірек сөздер: ағынды сулар, суды тазарту, метакаолин, геополимерлер, ауыр металдар, адсорбция, ИҚ спектроскопиясы.

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ИСПОЛЬЗОВАНИЕ ГЕОПОЛИМЕРОВ НА ОСНОВЕ МЕТАКАОЛИНА ДЛЯ ОЧИСТКИ СТОЧНЫХ ВОД

Аннотация. В статье представлены методы очистки и удаления ионов кадмия и свинца с использованием материалов на основе метакаолина, цемента, кокшетауской глины и геополимеров. Адсорбционные свойства были изучены при приготовлении модельного раствора с использованием солей кадмия и свинца. Эффективность процесса очистки оценивалась с использованием методов инфракрасной (ИК) спектроскопии и атомно-эмиссионной спектроскопии. Результаты показывают, что геополимеры служат эффективной и экологичной альтернативой для очистки сточных вод, загрязненных тяжелыми металлами. Полученные результаты подчеркивают потенциал геополимеров как инновационного и устойчивого подхода к управлению сточными водами, снижающего загрязнение окружающей среды и связанные с этим риски для здоровья. Результаты работы показали возможность использования геополимеров в качестве экологически эффективного и альтернативного метода очистки сточных вод, загрязненных тяжелыми металлами.

Ключевые слова: сточные воды, водоподготовка, метакаолин, геополимеры, тяжелые металлы, адсорбция, ИК-спектроскопия.