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«Жанармай, катализ және электрохимия институты» АҚ

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**OBTAINING COMPOSITE MATERIAL BASED ON HUMIC
ACID AND MICROSPHERE AND APPLICATION FOR WATER
TREATMENT FROM HEAVY METALS**

Abstract. A chemical analysis of humic acids based on brown coal from the Maikuben basin (Kazakhstan) and their modified form was carried out. Humic acids (HA) were obtained on the basis of potassium humate. To obtain a composite, the initial HA was modified with a microsphere in the ratio 1/1 and then heat-treated in a tube furnace at 600 °C in an inert atmosphere. The elemental composition of the initial sample based on HA/microsphere=1:1 was (wt.%): C 29.15, O 38.43, Na 0.27, Mg 0.29, Al 14.14, Si 13.72, S 0.22, Cl 0.16, K 1.86, Ca 0.42, Ti 0.36, Fe 0.97; activated form of the composite sample (wt %): C 52.50, O 23.53, Na 0.51, Mg 0.25, Al 6.11, Si 10.81, K 3.78, Ca 0.76, Ti 0.43, Fe 1.33, S 0.36. The physicochemical characteristics and surface morphology of the samples under study have been studied. It has been established by the BET method that thermal and vapor-gas activation of a composite material based on humic acid and microspheres leads to a significant increase in the specific surface area from 1.14 to 46.74 m²/g, respectively. The results of the analysis of micrographs showed that after thermal treatment of the composite based on humic acid and microspheres, the surface structure of the spherical shape changes and becomes more developed, where pores with particle sizes from ~343.4 nm to ~16.41 μm can be observed, and small particles are also observed. with a size from 49.5 nm to 158.1 nm. The obtained samples were tested as an adsorbent for

water purification from heavy metals and showed a high degree of purification (%): Zn 100.00, Cd 97.10, Pb 90.00 and Cu 93.5.

Key words: humic acid, microsphere, composite, carbonization, activation, adsorbent, heavy metals.

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ГУМИН ҚЫШҚЫЛЫ МЕН МИКРОСФЕРА НЕГІЗІНДЕГІ КОМПОЗИТТІК МАТЕРИАЛДЫ АЛУ ЖӘНЕ СУДЫ АУЫР МЕТАЛДАРДАН ТАЗАРТУДА ҚОЛДАНУ

Аннотация. Майкөбен бассейнінің (Қазақстан) қоңыр көмірі негізіндегі гумин қышқылдарына және олардың модификацияланған түріне химиялық талдау жүргізілді. Калий гуматы негізінде гумин қышқылдары (ГҚ) алынды. Композитті алу үшін ГҚ-ы микросфера бөлшектермен 1/1 қатынасында модификацияланып 600⁰С-та инертті атмосферада құбырлы пеште термиялық өңдеуден өтті. ГҚ/микросфера=1:1 негізіндегі бастапқы үлгінің элементтік құрамы (масс.%): С 29.15, О 38.43, Na 0.27, Mg 0.29, Al 14.14, Si 13.72, S 0.22, Cl 0.16, K 1.84, Ca 0.42, Ti 0,36, Fe 0.97, композициялық үлгінің активтендірілген формасын үшін (масс.%): С 52.50, О 23.53, Na 0.51, Mg 0.25, Al 6.11, Si 10.81, K 3.78, Ca 0,76, Ti 0.43, Fe 1.33, S 0.36 құрады. Аталған үлгілердің физика-химиялық сипаттамалары мен беткі морфологиясы зерттелді. БЕТ әдісімен гумин қышқылды мен микросфера негізіндегі композициялық материалдың термиялық және бу-газды активтенуі меншікті бетінің сәйкесінше 1,14-тен 46,74 м²/г-ға дейін айтарлықтай өсуіне әкелетіні анықталды. Микросуреттерді талдау нәтижелері гумин қышқылды мен микросфералар негізіндегі композитті термиялық өңдеуден кейін сфералық пішіннің беткі құрылымы өзгеріп, өлшемдері ~343,4 нм-ден ~16,41 мкм-ге дейінгі кеуектердің пайда болғанын және 49,5 нм-ден 158,1 нм-ге дейінгі ұсақ бөлшектердің түзілгенін байқауға болады. Алынған үлгілер ауыр металдардан суды тазарту үшін адсорбент ретінде сыналды және жоғары тазарту дәрежесін (%): Zn 100,00, Cd 97,10, Pb 90,00 және Cu 93,5 көрсетті.

Түйін сөздер: гумин қышқылды, микросфера, карбонизация, активация, адсорбент, ауыр металдар.

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ПОЛУЧЕНИЕ КОМПОЗИТНОГО МАТЕРИАЛА НА ОСНОВЕ ГУМИНОВОЙ КИСЛОТЫ И МИКРОСФЕРЫ И ПРИМЕНЕНИЕ ДЛЯ ОЧИСТКИ ВОДЫ ОТ ТЯЖЕЛЫХ МЕТАЛЛОВ

Аннотация. Проведен химический анализ гуминовых кислот на основе бурого угля Майкубенского бассейна (Казахстан) и их модифицированной формы. Гуминовые кислоты (ГК) были получены на основе гумата калия. Для получения композита, исходный ГК модифицирован с микросферой в соотношениях 1/1 и далее термически обработан в трубчатой печи при 600°C в инертной среде. Элементный состав исходного образца на основе ГК/микросферы=1:1 составил (масс.%): С 29.15, О 38.43, Na 0.27, Mg 0.29, Al 14.14, Si 13.72, S 0.22, Cl 0.16, K 1.86, Ca 0.42, Ti 0.36, Fe 0.97, активированной формы композитного образца (масс.%): С 52.50, О 23.53, Na 0.51, Mg 0.25, Al 6.11, Si 10.81, K 3.78, Ca 0.76, Ti 0.43, Fe 1.33, S 0.36. Изучены физико-химические характеристики и морфология поверхности исследуемых образцов. Методом БЭТ установлено, что термическая и парогазовая активация композитного материала на основе гуминовой кислоты и микросферы приводит к существенному увеличению удельной поверхности от 1.14 до 46.74 м²/г соответственно. Результаты анализа микроснимков показали, что после термической обработки композита на основе гуминовой кислоты и микросферы поверхностная структура сферической формы изменяется и становится более развитой, где можно наблюдать поры с размерами частиц от ~343,4 нм до ~16,41 мкм, также наблюдаются мелкие частицы размером от 49,5 нм до 158,1 нм. Полученные образцы апробированы в качестве адсорбента для очистки воды от тяжелых металлов и показали высокую степень очистки (%): Zn 100.00, Cd 97.10, Pb 90.00 и Cu 93.5.

Ключевые слова: гуминовая кислота, микросфера, композит, карбонизация, активация, адсорбент, тяжелые металлы.

Introduction. Humic substances (HS) obtained from oxidized coals and peat are complex polymer complexes that effectively bind pollutants of organic and inorganic nature and remove them from the biological cycle, prevent the

degradation of natural biocenoses. Humic acids (HA) have a significant ability to form complex and intra-complex (chelate) compounds with iron and aluminum, as well as copper and other polyvalent cations, which, depending on the pH of the medium, acquire greater mobility or form strong water-insoluble compounds. At the same time, HAs form complex compounds with metals mainly in the region of low pH values. Due to the presence of oxygen-containing functional groups, such as carboxyl $-\text{COOH}$, phenolic and alcohol $-\text{OH}$, as well as carbonyl $>\text{C}=\text{O}$, humic substances can form stable complexes with metal ions. Amine ($-\text{NH}_2$)-, amide ($\text{CO}-\text{NH}_2$)- and imine ($>\text{C}=\text{NH}$)-groups also participate in the formation of organometallic complexes (Popov et al., 2004). Humic substances as a natural polymeric material can be used as a matrix in the synthesis of composite nanomaterials due to their structural features and the presence of opposite charges.

Humic acids are considered as a promising and inexpensive source of valuable products and new materials due to the “green” chemistry approach (Xu et al., 2019; Brezoiu et al., 2019). The chemical heterogeneity and metastable conformation of humic substances, as well as their various reactive functional groups, determine a wide range of useful properties, including adsorption capacity with respect to metals and organic pollutants (Pota et al., 2020; Afzal et al., 2019; Pukalchik et al., 2019). Moreover, they show a strong effect in reducing reactive oxygen species due to their inherent paramagnetic properties, which leads them to be effective antioxidant agents.

The progressive explosive growth of the population, industrialization and urbanization lead to the release of various harmful pollutants into the environment (Xu et al., 2012). Water pollution is a growing environmental problem faced worldwide due to the displacement of environmental pollution and the deterioration of water resources (Sahbaz et al., 2018).

To remove and purify harmful impurities contained in wastewater, various advanced methods are used in industrial wastewater treatment, such as ultrafiltration, biological treatment, chemical oxidation, chemical precipitation, reverse osmosis, electrochemical treatment, photocatalytic oxidation, bioremediation and adsorption (Mahdavinia et al., 2016).

Recently, nanocomposite materials have been used in various wastewater treatment processes, such as adsorption, catalytic oxidation, membrane process, probing, and disinfection (Ganesan et al., 2019).

Most of the nanomaterials used in the wastewater treatment process are in the form of nanoparticles (NPs). These nanoparticles, when in contact with wastewater, experience some practical difficulties such as difficult separation, settling, cross-reaction, unhealthy disturbances, etc. To overcome the above problems, one of the successful strategies applied in wastewater treatment is the

introduction of nanocomposites. Nanocomposites come in various forms such as gels, colloids, porous structures, and copolymers (Yanyang et al., 2016).

It is known (Joseph et al., 2014, Bashar et al., 2013) that nanodispersed systems, including oxides and hydroxides of metals stabilized by various polymer matrices, expand the range of new promising functional materials, in particular, selective sorbents, magnetic and photosensitive coatings, and broad-spectrum composites. It should be noted that new composite materials obtained as a result of the stabilization of nanoparticles and the introduction of the latter into the structure of the natural polymer matrix are characterized by a qualitative expansion of the physicochemical properties of the latter (Yang et al., 2015).

In this regard, the issue of searching for natural polymeric stabilizers of nanoparticles is of particular relevance, since in their synthesis it is very difficult to stabilize nanoparticles, an increase in the surface energy of which leads to their rapid aggregation, which, in turn, may be accompanied by an uneven distribution of the latter in the structure of the polymer carrier (Mambetzhanova et al., 2019).

The use of humic acids as a natural polymeric stabilizer of nanoparticles seems to be very relevant, since the macromolecular nature and multifunctional nature of this carrier, as well as varying the conditions of in situ synthesis, make it possible to obtain commercial materials characterized by good selective, sorption, and magnetic properties (Pourmortazavi et al., 2017).

Aluminosilicate hollow microspheres - glass-ceramic balls formed during high-temperature flaring of coal and are hollow, almost ideally shaped silicate balls with a smooth surface, with a diameter of 10 to several hundred micrometers, on average, about 100 microns. The walls are solid non-porous with a thickness of 2 to 10 microns, melting point 1400-1500 °C, density 580-690 kg/m³. The internal cavity of the particles is filled mainly with nitrogen and carbon dioxide. They are the most valuable components of waste ash from thermal power plants.

The purpose of this work is to study the physicochemical properties of a modified composite material based on humic acids from the oxidized coal of the Maikuben pool and microspheres obtained on the basis of ash and slag waste, as well as its application for water purification from heavy metals.

Research material and methods. The raw material used was oxidized brown coal from the Maikuben deposit, preliminarily crushed to a particle size of less than 0.5 mm and having the following characteristics (wt %): Ad 66.09; Wr 5.73; Vd 17.78; Std 0.71; Std 21.01; Htd 1.68; Ntd 2.09; Na 0.61; Al 0.89; K 0.58; Ca 0.31; Ti 0.22; Fe 1.11; Zr 0.08. The yield of free humic acids was (per analytical state) 56%. The particle size of the coal is: 2.95 microns - 10%, 63.8 microns - 50%, 452 microns - 90%. The X-ray phase composition of the sample contains: halloysite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), silicon oxide (SiO_2), albite $\text{Na}(\text{AlSi}_3\text{O}_8)$ (Yermagambet et al., 2021).

Humic substances are obtained from oxidized brown coal, purified and determined in accordance with the recommendations of the International Society for Humic Substances (INSS). Humic acids were obtained on the basis of potassium humate by acidifying them with a 20% acid solution; as a result of the reaction, humic acids precipitated in the form of amorphous brown precipitates (pH=4.59). The resulting gel is dried at 80°C in a muffle furnace. Aluminosilicate microspheres were obtained on the basis of ash and slag waste from the combustion of coal from the Bogatyr open-pit mine of the Ekibastuz basin (Kazakhstan).

To obtain the HA/microsphere modification and its activated form, the dried HA sample is crushed to a powder state and mixed with the microsphere in an ultrasonic bath at a ratio of 1/1 (HA/microsphere), then dried in a muffle furnace. Further, the resulting composite material is briquetted with starch on a laboratory press «TP-1000». The maximum pressing force was 0.7 kN (700 kg/cm²), the briquette diameter was 0.8 cm.

Samples of the activated composite based on humic acid/microspheres (1/1) were obtained by carbonization at 600°C in argon and steam activation for 1 hour at this temperature.

Moisture, ash content and volatility of the samples were determined on a Thermoster Eltra thermogravimetric analyzer (according to ASTM D7582-12). The total pore volume, bulk density, pH of the aqueous extract, and methyl orange adsorption activity were determined in accordance with the procedures (Yermagambet et al., 2021). The adsorption characteristics of sorbents (specific surface area) were studied by the Brunauer-Emmett-Teller (BET) method, the measurements were performed on a KATAKON Sorbtometer M instrument. Chemical analysis and surface morphology were studied by energy-dispersive X-ray spectroscopy on an SEM instrument (Quanta 3D 200i) with an attachment for energy-dispersive analysis from EDAX.

To conduct research on the removal of heavy metals using humic acids and their modified form, a model solution of heavy metals was prepared from state standard samples (SSS). SSS 7256 of the composition of a solution of zinc ions is a solution of zinc sulfate 7-water, SSS 7012 of lead ions is an aqueous solution of lead (II) nitrate, SSS 7998 of copper ions is an aqueous solution of copper (II) sulfate, SSS 6690 cadmium ions - an aqueous solution of cadmium nitrate. A weighed portion of the sorbent (0.5 g) was mixed in a conical flask (250 ml) with a model solution (50 ml, pH=3.26) and stirred in a shaker at room temperature (25°C) at a stirring speed of 200 rpm for 3 h. process, the adsorbent with heavy metals was removed from the solution by filtration. The solution filtered from the adsorbent was analyzed for the content of heavy metals on a Ta-lab voltammetric analyzer.

Results and discussions. The results of the elemental analysis of the samples presented in Table 1 show that after the thermal treatment of the composite material based on humic acid and microspheres, most of the volatile components are removed in the form of gaseous products, respectively, the concentration of mineral components increases. The carbon content increases almost 2 times.

Table 1 - Chemical composition of samples

Nomination	The content of elements, wt. %											
	C	O	Na	Al	Si	K	Fe	Mg	Ti	S	Ca	Cl
Composite based on humic acid/microspheres (1/1)	29.15	38.43	0.27	14.14	13.72	1.86	0.97	0.29	0.36	0.22	0.42	0.16
Activated composite based on humic acid/microspheres (1/1)	52.50	23.53	0.51	6.11	10.81	3.78	1.33	0.25	0.43	0.36	0.76	-

The physicochemical characteristics of the samples are presented in Table 2.

Table 2 - Physical and chemical characteristics of the samples

Nomination	W ^r , %	A ^r , %	V ^d , %	V _Σ on water, cm ³ /g	ρ	pH _{water extract}	A _{methyl orange} , mg/g	A _{methylene blue} , mg/g	A _{iodine} , %	S _{BET} , m ² /g
Composite based on humic acid/microspheres (1/1)	3.29	67.59	36.58	-	0.45	5.62	14.5	-	-	1.14
Activated composite based on humic acid/microspheres (1/1)	0.75	91.02	8.90	0.77	0.33	10.25	30.5	102.5	17.78	46.74

Micrographs of the original samples and their activated form are shown in Figs. 1 and 2. When analyzing the surface morphology of the initial composite material, it was found that the cleavage surface is represented by a heterogeneity of the structure; in Figure 1.1, spherical particles with a diameter of 50-200 microns are observed.

The wall thickness of the spherical shape of the aluminosilicate microsphere is from 2 to 10 microns. Figures 1.1 and 1.2 show pores ranging in size from 300 nm to 500 μm on the particle surface. As shown in Figure 1.3, the remains of

microorganisms are visible, on the surface of which cavities with a diameter of 111 to 272 nm are evenly distributed.

The results of the analysis of micrographs after thermal treatment of the composite based on humic acids and microspheres show that the surface structure of the spherical shape changes and becomes more developed, where pores with particle sizes from ~ 343.4 nm to ~ 16.41 μm can be observed, and small particles with a size of 49.5 nm to 158.1 nm. The elemental composition of these nanoparticles is (w_i %): C – 6.92; O – 43.23; Na – 0.86; Mg – 0.93; Al – 9.69; Si – 20.39; K – 4.20; Ca – 2.34; Fe–11.43, and the values of the specific surface area of the activated composite based on humic acid/microspheres (1/1) increases in comparison with the initial sample - from 1.14 to 46.74 m^2/g . An increase in the adsorption activity for methyl orange by a factor of 2 from 14.5 mg/g to 30.5 mg/g is also observed.

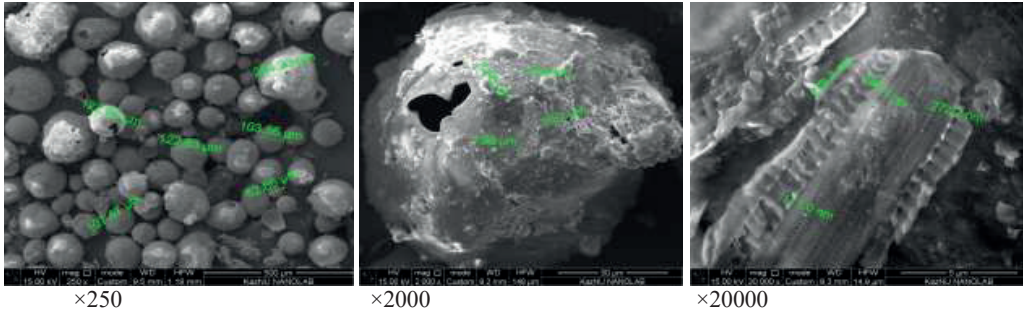


Figure 1.1

Figure 1.2

Figure 1.3

Figure 1. Electron microscopic images of the composite based on humic acid/microspheres: (1.1) $\times 250$ (1.2) $\times 2000$ and (1.3) $\times 20000$

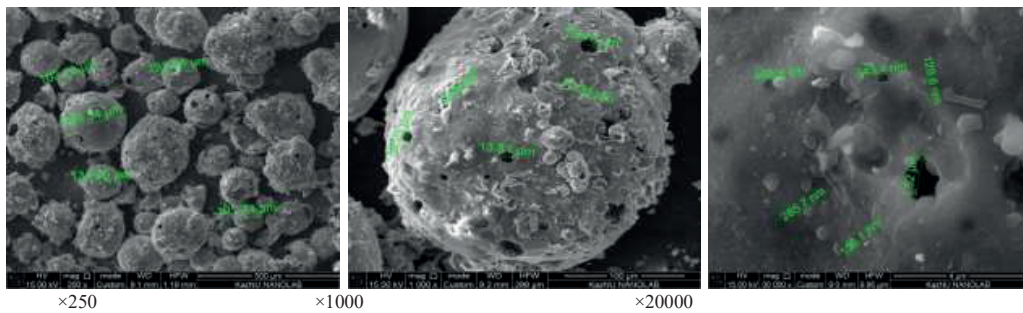


Figure 2.1

Figure 2.2

Figure 2.3

Figure 2. Electron microscopic images of the activated composite based on humic acid/microspheres: $\times 250$ (1.1), $\times 1000$ (1.2) and $\times 20000$ (1.3)

The samples obtained were tested for the purification of heavy metals under dynamic conditions. Analysis of the data obtained showed that after water purification, the values of all heavy metals decrease (Table 3).

Table 3 - Results of water purification from heavy metals

Heavy metals, mg/l	$C_0(\text{Me}), \text{mg/l}$	Humic acids		Activated composite based on humic acid/microspheres (1/1)	
		$C_{\text{residual}}(\text{Me}), \text{mg/l}$	degree of purification, %	$C_{\text{residual}}(\text{Me}), \text{mg/l}$	degree of purification, %
Zinc	0.10±0.01	0.0010±0.0004	99.00	Not detected	100.00
Cadmium	0.10±0.01	0.0033±0.0003	96.70	0.0029±0.0005	97.10
Lead	0.10±0.01	0.0216±0.0005	78.40	0.010±0.002	90.00
Copper	0.10±0.01	0.0171±0.0003	82.90	0.0065±0.0029	93.50

As a result of sorption, the resulting carbon materials adsorbed most of the heavy metals from the solution and showed a high degree of purification: 99.00-100.00% zinc, 96.70-97.1% cadmium, 78.40-90.00% lead and 82.90-93.50% copper. This is explained by the fact that when interacting with heavy metals, humic acids and their activated modified form form a chelate complex due to carboxyl, phenolic and amine groups. When interacting with heavy metals at pH = 4-5, the initial HAs form coordination bonds with carboxyl groups, since at pH below 6 strongly acidic carboxyl functional groups, as well as medium-strength carboxyl groups, will be completely dissociated. The activated form of humic acids with the microsphere binds copper cations to a large extent (the excess is ~ 10%) compared to the initial humic acids. This is due to additional physical adsorption of the porous material and complexation with phenolic groups at pH=7-8, at which the activated form of the composite material was adsorbed with heavy metals. As is known, under alkaline conditions, metals are bound by phenolic and alcohol groups. As a result of sorption, the resulting composite showed the highest degree of purification with respect to all heavy metals in comparison with analogues, which may be due to additional adsorption with microsphere nanoparticles.

Conclusion. Thus, as a result of the high-temperature process of carbonization and activation (in an inert medium) and modification (microsphere) of HA based on the coal of the Maikuben basin (Kazakhstan), an activated composite form of humic acids was obtained, which has a more developed and ordered surface structure and high adsorption properties. In this regard, the obtained samples based on HA from the coal of the Maikuben basin can be considered as an adsorbent for water purification from heavy metal ions, such as zinc, cadmium, lead, and copper. Further research on the resulting composite material may lead to improved performance and applications of this material in various other areas such as medicine, engineering, agriculture, and materials science.

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