IRSTI 61.31.57

A.M. Serikbayeva[1] – *main author*,
K.R. Toktybayeva[2], **E.N. Abdulova**[3], **R.U. Baigulova**[4],
T.K. Akilov[5], **T.G. Helder**[6], **M.S. Kalmakhanova**[7]

©

[1,2,3,4]Senior Lecturer, [5.6]Associate Professor, [7]PhD, Associate Professor

ORCİD
[1]https://orcid.org/0000-0002-8204-7851 [2]https://orcid.org/0000-0001-6193-944X
[3]https://orcid.org/0009-0009-0660-7616 [4]https://orcid.org/0009-0002-5148-559X
[5]https://orcid.org/0009-0001-5314-3983 [6]https://orcid.org/0000-0001-6898-2408
[7]https://orcid.org/0000-0002-8635-463X

[1,2,3,4,5]M. Auezov South Kazakhstan Research University, Shymkent, Kazakhstan
[6]Instituto Politecnico de Braganca, Braganca, Portugal
[7]M. Kh. Dulaty Taraz University, Taraz, Kazakhstan

@ *[1]ali_2006.82@mail.ru*

https://doi.org/10.55956/OQOD1949

DEVELOPMENT AND INVESTIGATION OF THE PROPERTIES OF ORGANICALLY MODIFIED PAVLODAR CLAY MINERALS FOR EFFECTIVE REMOVAL OF METHYLENE BLUE FROM THE AQUATIC ENVIRONMENT

Abstract. Due to the increasing pollution of water resources by industrial emissions in recent years, special attention has been paid to the development of effective and affordable adsorbents. In this work, the ability of modified clay materials to remove methylene blue (MB) from aqueous solutions using it as a model contaminant was studied. Modification of the natural Pavlodar clay was carried out by interlayer grafting of dimethyl sulfoxide (DMSO) and triethanolamine (TEOA). The process included the pre-introduction of DMSO, the subsequent addition of TEOA at 180°C with an exposure of 2 hours and drying at 60°C for 24 hours. The obtained materials were characterized by XRD, FT-IR, elemental analysis, thermogravimetry (DTA and TGA) and specific surface area analysis (SAA) methods. Adsorption tests were carried out at a concentration of MB 50 mg/l, adsorbent doses of 0.25-2.5 g/l, and a temperature of 50°C. The modified clay showed high efficiency: up to 97% of MB was removed within the first 15 minutes, and at a dosage of 0.5 g/l, the removal rate was 85% versus 40% for natural clay. Adsorption remained high over a wide pH range, which highlights the potential of such materials for practical applications in wastewater treatment.

Keywords: adsorption, dimethyl sulfoxide, methylene blue, organoclays, triethanolamine.

Serikbayeva A.M., Toktybayeva K.R., Abdulova E.N., Baigulova R.U., Akilov T.K., Helder T.G., Kalmakhanova M.S. Development and investigation of the properties of organically modified pavlodar clay minerals for effective removal of methylene blue from the aquatic environment //Mechanics and Technology / Scientific journal. – 2025. – No.2(88). – P.206-214. https://doi.org/10.55956/OQOD1949

Introduction. Dyes are colored organic compounds based on functional groups such as the chromophore group (NR_2, NHR, NH_2, COOH, and OH) and auxochromes (N_2, NO, and NO_2). There are different classes of dyes used to color various substrates, acidic dyes are commonly used to color silk, wool, modified acrylic, and nylon. They are also used in cosmetics, paper, food, inkjet printing, and skin coloring. The main classes of acidic dyes are azine, xanthene, anthraquinone, triphenylmethane, nitroso, nitro- and azo dyes. Acid blue 2, acid red 57, methyl orange, orange (I, II) are common acid dyes. The main dyes are used for dyeing modified polyesters, modified nylons, polyacrylonitrile, as well as in the paper industry and medicine. They are also used for tanning of cotton, silk and wool [1]. This class of dyes is soluble in water and gives colored cations, they are also called cationic dyes. The main classes are cyanine, thiazine, acridine, oxazine, hemicyanin and diazagemicyanin, i.e. basic red 46, malachite green, basic yellow 28, crystalline purple, methylene blue, basic brown and basic red 9 are the main dyes. Dispersed dyes are used on cellulose acetate, nylon, acrylic fibers and cellulose fiber. These are non-ionic dyes, insoluble in water and from aqueous solutions, also used for acrylic fibers. The main classes are benzodifuranone, nitro, styryl, azo, and anthraquinone groups, and some common examples are dispersed yellow, dispersed blue, dispersed orange, and dispersed red. Direct dyes are used for dyeing leather, cotton, viscose and in the paper industry. These dyes have an affinity for cellulose fiber if the dyeing process takes place in an aqueous solution containing electrolytes. The main classes are oxazine, stilbenes, and polyazo compounds. Examples are straight orange 34, straight black, straight purple and straight blue, etc. Reactive dyes are used for nylon, wool, as well as cellulose and cotton fiber. The chromophores in these dyes are phthalocyanine, azo, and triarylmethane, and a covalent bond is formed between the dye and the fiber. Common examples are reactive yellow 2, reactive red, remazole and reactive black 5, etc. Vat dyes are used for dyeing wool, viscose fibers, linen wool and cotton (mainly on cellulose fibers), i.e. indigoids, anthraquinone, etc.; vat dyes are insoluble in water. Common examples of VAT dyes are VAT-green 6, VAT-blue and indigo. Application of adsorption using clay sorbent (bentonite – 0.05-0.2; red sludge – 0.025; Clinoptilolite – 0.14-0.29) Units/kg for wastewater treatment will be economically efficient due to cost savings that are used when importing commercial activated carbon (0.8-1.1 units/kg), natural zeolite (0.08 units/kg), chitin (15-20 units/kg), chitosan (16.5-10 units/kg), crosslinked chitosan (5-10 units/kg). At the same time, from a technical point of view, the adsorption process is also considered the best alternative for water and wastewater treatment due to its convenience, ease of operation and design, as well as the fact that it helps to solve the problem of high energy consumption (used in reverse osmosis and ultraviolet sterilization) in most developing countries. there is not enough electricity [2]. A wide variety of organic pollutants (residual dyes) enter various water bodies from various sources, such as the pharmaceutical industry, the pulp and paper industry, the leather industry, the bleaching industry, the textile industry, and other anthropogenic activities. Currently, there are about 100,000 dyes on sale (azo dyes account for about 70% by weight of these dyes), and more than 1 million tons of dyes are produced per year, of which 50% are textile dyes. According to one estimate, 2% of the dyes produced end up directly in water waste, 10% is lost during subsequent staining, and 20% of these colored compounds enter the environment through waste [3]. Data on the percentage of various dyes on various substrates and their release in wastewater.

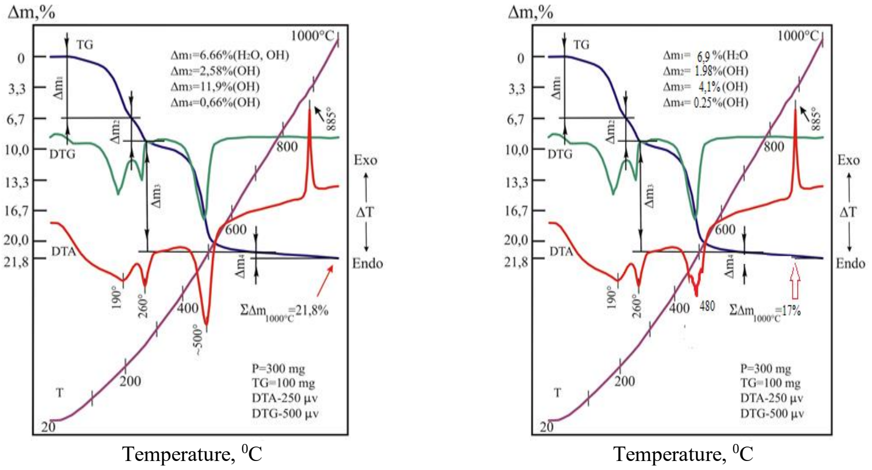
Materials and methods. *Materials and Reactants.* Two natural clays were collected at the clay deposit of Kazakhstan in the city of Pavlodar, namely Pavlodar clay and Pavlodar clay. Dimethylsufoxide (DMSO, C_2H_6OS ,99.5%) was supplied by Stanlab PURE, dioxane ($C_4H_4O_2$, 99.5%) was supplied by DiAKiT, isopropanol (C_3H_8O, 99.7%) was supplied by Chemistry and Technology, TEAO ($C_6H_{15}NO_3$,95%) was supplied by Tomas.kz. Methylene blue (98%) was supplied by Merck. All reactants were used without any modification.

Development of materials. For the manufacture of a material with adsorption properties, two natural clays from the Pavlodar deposits were used, named as Pavlodar and Pavlodar. The natural clay was first grind into powder in a mill and sieved through a sieve No. 0.063. To modify the natural clay, 6 g of the clay was initially suspended in 6 L of water and then added to a mixture containing 30 mL of DMSO and 2.5 mL of water. The suspension was kept under magnetic stirring at a temperature of 80°C for 5 days, and then the mixture was left at room temperature for 2.5 days. The resulting material was extracted after two series of centrifugation using first dioxane (2×25 mL), and then isopropanol (2×25 mL). The product, named K-DMSO (K standing for the chosen clay), was dried at 50°C, leading to two modified clay: Pavlodar-DMSO. Then, 1 g of K-DMSO was added to 6 g of TEOA and stirred at 180°C for 2 h, and then washed with isopropanol (3×50 mL). The resulting material (K-TEAO) was dried at 50°C for 24 h, leading to Pavlodar-TEAO.

Characterization of Materials. The textural properties of the materials were determined by analysis of N_2 adsorption-desorption isotherms at 77 K, obtained in a Quantachrome NOVATOUCH XL4 adsorption analyzer using procedures reported elsewhere [4-6]. All calculations were carried out using the software version NovaWin 11.02. Prior to analysis, the materials were degassed at 120°C for 16 hours. The surface areas (S_{BET} and $S_{Langmuir}$) were determined using the methods of BET and Langmuir at p/p0 in the range 0.05-0.35. The total pore volume (V_{Total}) was determined at p/p0 = 0.98. Thermogravimetric analysis were performed on a derivatograph of the company "MOM" – Budapest (Hungary). The method used is based on the recording by the device of changes in the thermochemical and physical parameters of a substance that can be caused when it is heated. The thermochemical state of the sample is described by the curves: T (temperature), DTA (differential thermoanalytical), TG (thermogravimetric) and DTG (differential thermogravimetric), the latter curve being a derivative of the TG function. The analysis of the materials was carried out carried out in air atmosphere, in the temperature range from 20 to 1000°C with a heating rate of 10°C min^{-1}.

Adsorption process and analytical methods. The methylene blue adsorption tests were carried out adding 0.125 g of adsorbent (natural and modified clays) to 50 mL of a methylene blue solution of 50 mg/L with a total contact time of 8 h. Sampling was undertaken at predetermined intervals (0, 15, 30, 60, 120, 240, 360 and 480 min). Tests with different pH values of the methylene blue solution were considered (pH 1, 3, 6, 9 and 12). The adsorption tests also considered variations in the concentration of the adsorbent (2.5, 0.5 and 0.25 g/L) and variations in the concentration of methylene blue solution (50, 100 and 500 ppm). The adsorption of methylene blue was monitored using a UV-VIS spectrophotometer.

Research results and discussion. *Thermal analysis (DTA and TGA).* As a result of the dynamic heating of these samples, the curves DTA, DTG and TG noted the manifestations caused by the occurrence of various types of reactions in the system, reported in Figure 1. Among them are the processes associated with the release of H_2O and hydroxyls into the atmosphere during the decomposition of clay minerals, reactions with CO_2 emissions as a result of the combustion of organic matter, as well as during the destruction of calcite. During the thermal decomposition of its mineral components, two pronounced effects were recorded, associated with the decomposition of Gibbs (in the range of 20-200°C) and with the dissociation of calcium carbonate (in the range of 400-725°C), as observed in Figure 1a.



a) b)

Fig. 1. Thermogravimetrical analysis of (a) Pavlodar natural clay and (b) Pavlodar-TEAO

The quantitative analysis are reported in Table 1. According to the thermogravimetric curve, in the first case, Gibbs is dehydrated (in two stages) with a weight loss equal to Δm1-0.3%=1.7%, which corresponds to the presence of 8.1% of Gibbs in the Pavlodar modified clay under dynamic heating from 20 to 1000°C, leaving a series of endothermic manifestations on the DTA curve at 190, 260 and ~500°C and one exothermic peak in the 900°C region, Fig.1. These endothermic reactions also took shape on the DTG curve in the form of clearly defined peaks in the same temperature regions. Such processes are caused by thermal dehydration of minerals that are part of the test sample. At these temperatures, each such discharge from the water system left a corresponding step of weight loss on the thermogravimetric (TG) curve, Table 1. According to the atlas of thermal curves of minerals and rocks, the endothermic peak at 260°C is caused by the decomposition of gibbsite (Gibbsit). Dehydration of this mineral, due to the defect of its structure, proceeds in two stages. The total weight loss in this case reaches the value of Δm1 + Δm2 = 8.58%, which corresponds to 13.1% of gibbsite in the sample. Along with this mineral, kaolinite is present in the sample, the diagnosis of which was carried out according to the above–mentioned signs - according to the endothermic effect at 500°C and the exothermic manifestation occurring at 900°C. The effect found in the 500°C region is caused by the removal of constitutional water from the kaolinite structure. When heated, kaolinite leaves on the DTA curve a weakly expressed endothermic manifestation in the range of 300-500°C and an equally weak exothermic peak in the region of 900°C. Thermal dissociation of this mineral occurs together with the combustion of organic matter(s). At the same time, kaolinite registers a weight loss of the modified

Table 1

Thermogravimetric readings of the natural and modified Pavlodar clay and of the modified Pavlodar clay, in the range of 20-1000°C

Material	Weight loss sequence	Weight loss (%)	Volatile components	Temperature range (°C)
Natural Pavlodar clay	Δm1	2.0	H_2O	20-200
	Δm2	0.875	OH	200-400
	Δm3	7.38	CO_2	400-725
	Δm4	0.125	OH	725-1000
	∑Δm1000°C	10.38	H_2O, OH, CO_2	20-1000
Modified Pavlodar clay	Δm1	6.66	H_2O	20-190
	Δm2	2.58	OH	190-260
	Δm3	11.9	OH	260-700
	Δm4	0.66	OH	700-1000
	∑Δm1000°C	21.8	H_2O, OH	20-1000

N_2 adsorption-desorption isotherms at –196°C. The N_2 adsorption-desorption isotherms of natural and prepared adsorbents are shown in Figure 2, and the textural properties are reported in Table 2. The isotherm pattern was identified as type IV [7] with loop hysteresis of type H3 [8], in accordance with the IUPAC classification. Type IV shows that the material has a mesoporous character, and loop hysteresis of type H3 shows the characteristics of a layered material with gaps similar to the pores formed by a plate layer with a space between arrays of plates [9]. This also implies that DMSO and TEOA can maintain the area between the clay layers to remain supported to create a wider gallery.

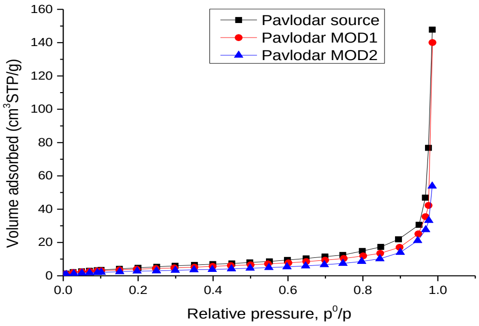


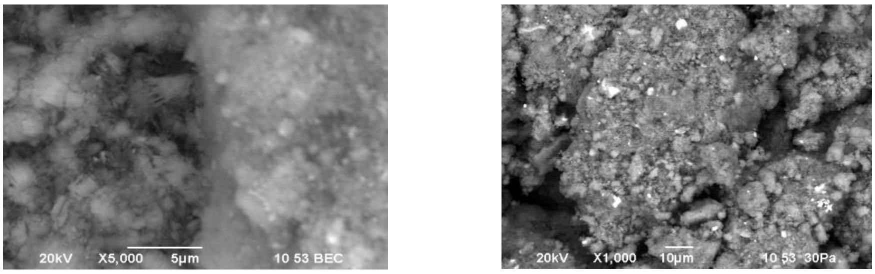
Fig. 2. N_2 adsorption-desorption isotherms at 77 K of the Pavlodar natural clays, and their derived materials

Table 2

Textural properties of the materials

Sample	S_{BET} (m^2g^{-1})	$S_{Langmuir}$ (m^2g^{-1})	S_{ext} (m^2g^{-1})	V_{mic} (cm^3g^{-1})	V_{Total} (cm^3g^{-1})	S_{mic} (m^2g^{-1})	W_{mic} (nm)
Pavlodar	20	20	20	0	0.1200	0	-
Pavlodar-DMSO	16	17	16	0	0.0660	0	-
Pavlodar-TEAO	11	12	11	0	0.0500	0	-

Scanning electron microscopy (SEM). Scanning electron microscopy (SEM) images of the original and modified clays are shown in Figure 3. There are obvious differences in the morphology between the original and the modified clays. The SEM images showed that kaolinite has a facial aspect, manifested in its typical hexagonal plates and stacks [10]. The Pavlodar clay modified with TEOA retains this aspect, but the size of kaolinite particles has been reduced due to intercalation of TEAO molecules. The modified clay has more spongy particles, this behavior occurs due to changes in the surface charge of the particle and the time spent on the preparation of this material.



a) Pavlodar b) Pavlodar-TEAO

Fig. 3. SEM images of the (a) Pavlodar natural clay, (b) Pavlodar-TEAO clay

Adsorption of methylene blue. Effect of modification. Figure 4. shows that the ability of adsorption of methylene blue by the natural clay and by Pavlodar-TEAO present similar results under the chosen operating conditions, in which the adsorbed methylene blue is stable at 96-97% for all adsorbents. This means that the adsorbents have the same adsorption rate of methylene blue under the same operating conditions. In addition, it can be said that the adsorption process is completed in the first minutes of contact between the adsorbent and the methylene blue solution, since additional time does not increase the amount of adsorbed methylene blue. The number of active centers on an empty adsorbent leads to the fact that at first the adsorption process proceeds quickly, and then becomes constant. Due to this observation, it is anticipated that the adsorbent dosage is superior to what maybe needed for an efficient adsorption process. Accordingly, the study of adsorbent dosage was performed to confirm.

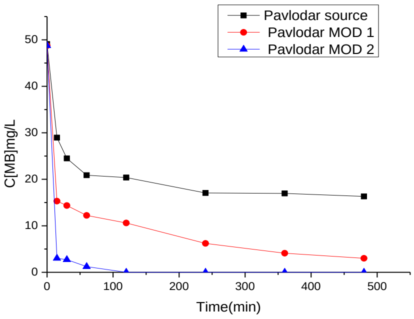


Fig. 4. Effect of clay modification on adsorption of methylene blue. Conditions: $[MB]_0$=50 mg/L, $C_{adsorbent}$=2.5 g/L, T=50ºC, pH=3.0

Effect of adsorbent dosage. The adsorbent dosage ranged from 2.5 to 0.25 g/l for both natural Pavlodar clay and clay modified with TEAO (Figure 9). At 2.5 g/l, natural clay showed rapid adsorption: in the first 15 minutes, more than 98% of methylene blue was removed (according to Figure 5). However, when the dosage was reduced to 0.5 g/l, the effectiveness dropped sharply to 35%, and at 0.25 g/l – to 21%. In contrast, modified clay (Pavlodar-TEAO) at the same doses (0.5 and 0.25 g/l) demonstrated significantly better efficiency: more than 90% of MB was removed in both cases. Therefore, 0.25 g/l was chosen as the optimal dose for further research.

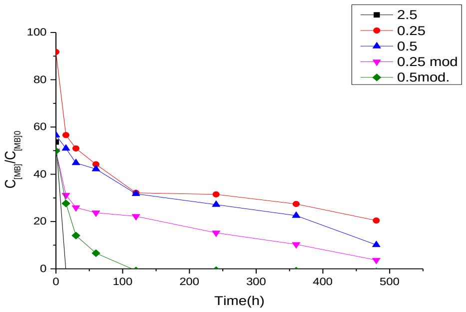


Fig. 5. Effect of adsorbent concentration on methylene blue adsorption by Pavlodar and Pavlodar-TEAO. Conditions:
$[MB]_0$=50 mg/L, $C_{adsorbent}$=0.25 – 2.5 g/L, T=50 ºC, pH_0=3.0

Effect of concentration of methylene blue. Figure 6 shows the dependence of the efficiency of adsorption of methylene blue on natural Pavlodar clay and its modification with TEAO on the concentration of the dye. As the concentration of methylene blue increases, the percentage of adsorption increases until the maximum capacity of the adsorbent is reached. The optimal concentration is 50 mg/l. At high concentrations, natural clay exhibits greater adsorption capacity than modified clay, due to its ability to expand and absorb dye molecules. The modified clay has a rigid porous structure that limits diffusion. The diameter of the methylene blue molecule (13-15 Å) and the pore size of clays (30.5 Å) ensure effective penetration of the dye into natural clay. The adsorption mechanism is based on the diffusion of molecules into the pores and galleries of the material.

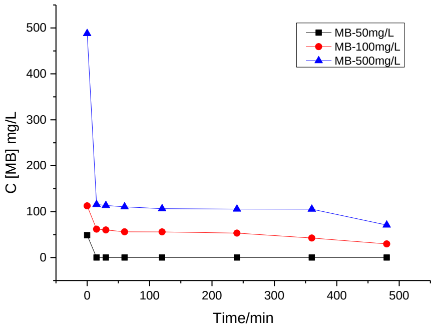


Fig. 6. Effect of the initial concentration of MB with the Pavlodar -TEAO clay. Conditions: $C_{adsorbent}$=0.25 gL^{-1}; $[MB]_0$=50, 100, and 500 mgL^{-1}; T=50°C ; pH_0=3.0

Adsorption is the result of complex interactions between adsorbate molecules and functional groups on the surface of the adsorbent. The main active centers are hydroxyl and silanol groups, the effectiveness of which depends on the structure and chemical composition of the surface. Modification of clay enhances the active centers. For methylene blue, the key is a nitrogen atom with an unshared pair of electrons. The mechanism of adsorption includes diffusion, electrostatic interactions, hydrogen bonds, and ion exchange.

Conclusion. Natural and modified clays are economically available and effective adsorbents for removing dyes from wastewater. Modification of clay using the tertiary amine TAO has significantly increased its adsorption capacity, despite the absence of significant changes in textural properties. The increased efficiency is explained by the introduction of nitrogen-containing functional groups that play the role of active Lewis centers, which enhance interaction with methylene blue molecules. This was especially evident in the modified clay "Pavlodar-TEAO", which showed high efficiency in cleaning aqueous solutions. These results indicate the potential for developing inexpensive and efficient water treatment technologies based on modified clays. At the same time, additional studies are needed for real use, including an assessment of environmental safety in the presence of various micro-pollutants in low concentrations.

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This research is funded by the Committee of Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. BR24992867).

Received: 22 May 2025
Accepted: 27 June 2025

А.М. Серикбаева, К.Р. Токтыбаева, Е.Н. Абдулова,
Р.У. Байгулова, Т.К. Акилов, Т.Г. Хелдер, М.С. Калмаханова

[1]М. Әуезов Оңтүстік Қазақстан университеті, Шымкент қ., Қазақстан
[2]М.Х. Дулати атындағы Тараз университеті, Тараз қ., Қазақстан
[3]Браганса политехникалық институты, Браганса қ., Португалия

СУ ОРТАСЫНАН МЕТИЛЕН КӨКІН ТИІМДІ ЖОЮ ҮШІН ОРГАНИКАЛЫҚ ТҮРЛЕНДІРІЛГЕН ПАВЛОДАРЛЫҚ САЗДЫ МИНЕРАЛДАРДЫҢ

ҚАСИЕТТЕРІН ӘЗІРЛЕУ ЖӘНЕ ЗЕРТТЕУ

Аңдатпа. Соңғы жылдары су ресурстарының өнеркәсіптік шығарындылармен ластануының артуына байланысты тиімді және қол жетімді адсорбенттерді дамытуға ерекше назар аударылады. Бұл жұмыс модификацияланған саз материалдарының метилен көкін (МБ) Сулы ерітінділерден модельдік ластаушы зат ретінде алып тастау қабілетін зерттеді. Табиғи Павлодар сазын модификациялау диметилсульфоксидті (ДМСО) және триэтаноламинді (TEOA) қабатты егу арқылы жүргізілді. Процесс DMSO-ны алдын-ала енгізуді, содан кейін TEOA-ны 180℃ температурада 2 сағаттық экспозициямен қосуды және 60℃ температурада 24 сағат бойы кептіруді қамтыды. Алынған материалдар рентгенография, инфрақызыл спектроскопия, элементтік талдау, термогравиметрия (DTA және TGA) және меншікті бетті талдау әдістерімен сипатталды. Адсорбция сынақтары mb концентрациясы 50 мг/л, адсорбент дозалары 0,25-2,5 г/л және 50℃ температурада жүргізілді. Модификацияланған саз жоғары тиімділікті көрсетті, алғашқы 15 минут ішінде 97% МК жойылды, ал 0,5 г/л дозада жою жылдамдығы табиғи саз үшін 40%-ға қарсы 85% құрады. Адсорбция PH-ның кең ауқымында жоғары болып қалады, бұл ағынды суларды тазартуда практикалық қолдану үшін осындай материалдардың әлеуетін көрсетеді.

Тірек сөздер: адсорбция, диметилсульфоксид, метилен көк, органоглин, триэтаноламин.

А.М. Серикбаева, К.Р. Токтыбаева, Е.Н. Абдулова,
Р.У. Байгулова, Т.К. Акилов, Т.Г. Хелдер, М.С. Калмаханова

[1]Южный Казахстанский университет им. М. Ауэзова, г. Шымкент, Казахстан
[2]Таразский университет им. М.Х. Дулати, г. Тараз, Казахстан
[3]Политехнический институт Браганса, г. Браганса, Португалия

РАЗРАБОТКА И ИССЛЕДОВАНИЕ СВОЙСТВ ОРГАНИЧЕСКИ МОДИФИЦИРОВАННЫХ ПАВЛОДАРСКИХ ГЛИНИСТЫХ МИНЕРАЛОВ ДЛЯ ЭФФЕКТИВНОГО УДАЛЕНИЯ МЕТИЛЕНОВОГО СИНЕГО ИЗ ВОДНОЙ СРЕДЫ

Аннотация. В связи с растущим в последние годы загрязнением водных ресурсов промышленными выбросами особое внимание уделяется разработке эффективных и доступных по цене адсорбентов. В этой работе была изучена способность модифицированных глинистых материалов удалять метиленовый синий (МБ) из водных растворов, используя его в качестве модельного загрязняющего вещества. Модификация природной павлодарской глины проводилась путем послойной прививки диметилсульфоксида (ДМСО) и триэтаноламина (ТЕОА). Процесс включал предварительное введение ДМСО, последующее добавление ТЕОА при температуре 180℃ с выдержкой 2 часа и сушку при температуре 60℃ в течение 24 часов. Полученные материалы были охарактеризованы методами рентгенографии, инфракрасной спектроскопии, элементного анализа, термогравиметрии (DTA и TGA) и анализа удельной поверхности (SAA). Испытания на адсорбцию проводили при концентрации MB 50 мг/л, дозах адсорбента 0,25-2,5 г/л и температуре 50℃. Модифицированная глина показала высокую эффективность: до 97% MC удалялось в течение первых 15 минут, а при дозировке 0,5 г/л скорость удаления составляла 85% против 40% для натуральной глины. Адсорбция остается высокой в широком диапазоне pH, что подчеркивает потенциал таких материалов для практического применения в очистке сточных вод.

Ключевые слова: адсорбция, диметилсульфоксид, метиленовый синий, органоглина, триэтаноламин.