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Original Empirical Research

Interaction of Methylene Blue with a Sorption Material Obtained from Engine Oil Regeneration Waste

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Abstract

Introduction. In light of the increasing pollution of water resources by organic compounds, particularly synthetic dyes, it is a pressing issue to develop effective, affordable, and environmentally friendly sorption materials. Despite the widespread use of activated carbons, clays, and organomineral composites, there remains a need to find low-cost sorbents based on industrial waste. One promising approach is the use of clay sludge generated during the regeneration of machine oil, but their sorption properties have not been sufficiently studied. The aim of this research was to obtain and characterize the sorption characteristics of a material based on clay sludge waste during the removal of methylene blue from aqueous solutions.

Materials and Methods. The sorption material was obtained by thermal treatment of oily clay sludge at various temperatures, with the optimal mode selected. Pore structure was studied by low-temperature nitrogen adsorption using BET, t-Plot, and BJH models. Sorption properties were evaluated using model methylene blue solutions and photocolorimetry at a wavelength of 670 nm. Adsorption capacity and purification efficiency were calculated using standard methods, and sorption isotherms were approximated using the Langmuir, Freundlich, and Dubinin-Radushkevich models.

Results. It was found that the sample (CS400) heat-treated at 400°C had a developed mesoporous structure with a specific surface area of 69.148 m^2/g and a total pore volume of 0.159 cm^3/g. The average pore diameter was approximately 4–6 nm, with no micropores present. The material demonstrated high sorption activity for methylene blue, effectively decolorizing solutions. The maximum sorption capacity reached 0.139 mmol/g (44.8 mg/g). The sorption process was best described by the Langmuir model ($R^2 = 0.9645$), indicating monolayer nature of adsorption. The calculated sorption energy (9.608 kJ/mol) suggested a predominance of physical interaction.

Discussion. The results obtained demonstrated that the high sorption activity of the material was due to the formation of a mesoporous structure during heat treatment. Pores with a diameter of 4–6 nm were predominant, which ensured accessibility of the active surface to dye molecules. Hysteresis indicated the contribution of capillary condensation to the sorbate retention process. The compliance with the Langmuir model indicated relative homogeneity of active sites. The established physical nature of adsorption suggested the predominance of weak intermolecular interactions.

Conclusion. The feasibility of the effective use of thermally modified clay sludge waste as a sorbent for the purification of water from cationic dyes has been experimentally confirmed. The CS400 material has been shown to have a high sorption capacity, effectively removing methylene blue from aqueous solutions. The obtained results demonstrated the potential of the developed sorbent for use in water treatment technologies and highlighted the feasibility of recycling industrial waste to produce functional materials.

Keywords: sorbent, water purification, sorption capacity, waste disposal

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Оригинальное эмпирическое исследование

Особенности взаимодействия красителя «метиленовый голубой» с сорбционным материалом, полученным на основе отхода регенерации машинных масел

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Аннотация

Введение. В условиях нарастающего загрязнения водных ресурсов органическими соединениями, в частности синтетическими красителями, актуальной задачей является разработка эффективных, экономически доступных и экологически безопасных сорбционных материалов. Несмотря на широкое применение активированных углей, глин и органоминеральных композитов, сохраняется необходимость поиска недорогих сорбентов на основе промышленных отходов. Перспективным направлением является использование глинистых шламов, образующихся при регенерации машинных масел, хотя их сорбционные свойства остаются недостаточно изученными. Целью настоящей работы было получение и исследование сорбционных характеристик материала на основе глинистого шламового отхода при удалении метиленового голубого из водных растворов.

Материалы и методы. Сорбционный материал получали путём термической обработки замасленного глинистого шлама при различных температурах с выбором оптимального режима. Пористую структуру исследовали методом низкотемпературной адсорбции азота, применяя модели БЭТ, t-Plot и BJH. Сорбционные свойства оценивали на модельных растворах метиленового голубого фотоколориметрическим методом при длине волны 670 нм. Адсорбционную ёмкость и эффективность очистки рассчитывали стандартными методами, а изотермы сорбции аппроксимировали моделями Ленгмюра, Фрейндлиха и Дубинина-Радушкевича.

Результаты исследования. Установлено, что образец, термообработанный при 400 °C (ГШ400), обладает развитой мезопористой структурой с удельной поверхностью 69,148 $м^2/г$ и общим объёмом пор 0,159 $см^3/г$. Средний диаметр пор составляет примерно 4-6 нм, микропоры отсутствуют. Материал демонстрирует высокую активность в адсорбции метиленового голубого, обеспечивая эффективное обесцвечивание растворов с максимальной сорбционной ёмкостью 0,139 ммоль/г (44,8 мг/г). Процесс сорбции наиболее адекватно описывается моделью Ленгмюра (R^2 = 0,9645), указывая на монослойный характер адсорбции. Рассчитанная энергия адсорбции (9,608 кДж/моль) свидетельствует о преобладании физического механизма взаимодействия.

Обсуждение. Полученные результаты показывают, что высокая сорбционная активность обусловлена формированием мезопористой структуры в процессе термообработки. Преобладание пор диаметром 4-6 нм обеспечивает доступность активной поверхности для молекул красителя, а наличие гистерезиса указывает на вклад капиллярной конденсации в удержание сорбата. Соответствие модели Ленгмюра свидетельствует об относительной однородности активных центров. Установленный физический характер адсорбции указывает на доминирование слабых межмолекулярных взаимодействий.

Заключение. Экспериментально подтверждена возможность эффективного использования термически модифицированного глинистого шламового отхода в качестве сорбента для очистки вод от катионных красителей. Материал ГШ400 обладает высокой сорбционной ёмкостью и обеспечивает эффективное удаление метиленового голубого из водных растворов. Полученные данные свидетельствуют о перспективности применения разработанного сорбента в технологиях водоочистки и подчёркивают целесообразность утилизации промышленных отходов для получения функциональных материалов.

Ключевые слова: сорбент, очистка воды, сорбционная емкость, утилизация отхода

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Для цитирования. Мурзаханов Я.А., Сапронова Ж.А., Свергузова С.В. Святченко А.В. Особенности взаимодействия красителя «метиленовый голубой» с сорбционным материалом, полученным на основе отхода регенерации машинных масел. Безопасность техногенных и природных систем. *Безопасность техногенных и природных систем*. 2026;10(2):142–151. https://doi.org/10.23947/2541-9129-2026-10-2-142-151

Introduction. The concept of environmental safety involves protecting the population from environmental hazards caused by natural and anthropogenic processes, preventing the depletion of natural resources, and addressing the growing gap between environmental demands and supply. Pollution and scarcity of water resources are among the main threats to environmental safety. Water purity is crucial for ecosystems and human health, as exposure to pollutants can cause serious health problems and affect food security [1].

According to the World Health Organization, approximately 2 billion people around the world use drinking water sources that are contaminated with chemicals [2]. By 2030, the global demand for fresh water is expected to exceed supply by up to 40% [3]. In industrialized and developing countries, there is a steady upward trend in wastewater volumes, which increases the burden on aquatic ecosystems. Rapid development of technology, industry, and urbanization has significantly affected the state of water bodies worldwide. The discharge of wastewater containing dyes from various industries poses a serious environmental challenge. Approximately 280,000 tons of dyes are released into the environment annually, leading to pollution of water resources on a global scale [4]. The textile industry generates a significant amount of polluted wastewater, containing a complex mixture of dyes, surfactants, salts, and heavy metals. In addition to environmental degradation, dye-containing pollutants have a profound negative impact on the local population, especially in developing regions. Polluted water sources reduce agricultural productivity, damage fisheries, and increase health care costs [5].

In recent years, the issue of wastewater treatment from dyes has become increasingly important due to more stringent environmental regulations and the need to achieve the United Nations Sustainable Development Goals (SDG 6 — Clean Water and Sanitation) [6]. This requires the development of efficient, affordable and environmentally friendly cleaning methods.

Most dyes found in wastewater are hydrophilic and have low biodegradability, making them resistant to traditional purification methods [7]. The consumption of water or food contaminated with toxic dyes can lead to their bioaccumulation in the body and long-term health problems, including disruption of the endocrine system, the appearance of malignant tumors, and decreased immunity. Additionally, many dyes have a negative impact on aquatic organisms, leading to decreased reproductive capacity, slower growth, and increased mortality [8].

Dyes are classified into three main categories based on the type of charge they carry in an aqueous solution: cationic, anionic, and non-ionic. Among them, cationic dyes such as methylene blue (MB), malachite green and crystalline violet are widely used in the textile, printing and dyeing industries, as well as in paper production. Methylene blue, with the chemical formula $C_{16}H_{18}N_3ClS$, is a common cationic dye from the family of phenothiazine compounds. It is characterized by stable structure, high water solubility and toxicity. Studies have found that wastewater containing methylene blue can lead to the death of aquatic plants and a decrease in dissolved oxygen levels in water bodies [9]. While methylene blue may have some medical applications when used safely, these benefits do not apply when it enters the human body through contaminated water. Instead, it can cause a range of health problems, including cyanosis, tissue necrosis, jaundice, and tachycardia. The negative effects of methylene blue on plants have been well documented. Its presence leads to growth inhibition and reduction in the content of pigments and proteins in microalgae such as *Chlorella vulgaris* and *Spirulina platensis*. Therefore, the adverse consequences of methylene blue in wastewater necessitate its proper disposal before discharge [10].

Various processing methods are used to remove dyes from the environment. Biological methods are environmentally friendly and economical, but they have a number of limitations: low process speed, dependence on environmental conditions (temperature, pH), and limited effectiveness against resistant synthetic dyes. Chemical methods such as advanced oxidation using ozone, chlorine, or persulfates can effectively destroy dyes, but require expensive reagents and are can often produce toxic byproducts. Chemical cleaning is also associated with high energy costs and expensive equipment, limiting its practical use. Adsorption is an efficient and environmentally sustainable method of water purification due to its cost-effectiveness and the potential for sorbent regeneration. The development of inexpensive, highly efficient adsorbents is a pressing challenge [10, 11].

Materials such as rice husks, biochar from plant waste, activated banana peel waste, and others are used as sorbents [12]. In [13], a geopolymer based on partially dealuminated metakaolin was proposed for removing methylene blue. It was shown that adsorption depended on pH, contact time, and dye concentration, and the maximum sorption capacity was 8 mg/g. The process was described by the Freundlich model, indicating a heterogeneous surface and a multilayer nature of adsorption. Although these materials are environmentally friendly and inexpensive, they have a relatively low sorption capacity. It is demonstrated in [14] that activated carbon synthesized from *Denolix Regia* pods and activated with zinc chloride and phosphoric acid provides a degree of methylene blue removal of up to 99.9%. Adsorption proceeds mainly by a physical mechanism and is described by the Langmuir and Freundlich models. Despite its high efficiency, the production of this material requires complex chemical processes and significant energy input.

Clays are used as an efficient alternative for removing anionic and cationic dyes due to their high ion exchange capacity, specific surface area, low cost and environmental friendliness. However, clay materials have some disadvantages, such as difficult regeneration, limited selectivity, and the need for chemical modification to optimize the removal efficiency.

Numerous studies on organomineral materials for the adsorption of dyes have confirmed their high sorption capacity due to their unique structural properties. These materials are promising as inexpensive and environmentally friendly adsorbents [15]. Special attention has been paid to modifying clay sorbents. In study [16], activation of bentonite using NaOH solution increased the specific surface area to 74.15 m^2/g and achieved a sorption capacity of 22.131 mg/g. At the same time, adsorption is a spontaneous process, and sorbents remain operational for several regeneration cycles. However, the use of concentrated alkaline solutions complicates the technology and increases its cost.

A composite of activated bentonite and rapeseed straw was synthesized by hydrothermal carbonation followed by $FeCl_3$ impregnation and NaOH treatment. The composite showed excellent adsorption capacity (425.65 mg/g at neutral pH) compared to the original biochar. Despite the high rates, the multi-stage synthesis (hydrothermal treatment, chemical modification) limits scalability and increases the cost of production [17].

An analysis of modern research suggests that, despite the high effectiveness of a number of adsorbents, their widespread use is limited due to their high cost and difficulty of preparation and activation [18]. Therefore, organomineral materials based on clay and clay waste hold promise for obtaining inexpensive and efficient sorbents for the removal of dyes from aqueous media. One such clay sludge waste is the residue from the regeneration of engine oil. In developed countries, processing of used oil at specialized plants amounts to 20–100 thousand tons per year. For every ton of refined used oil processed, 80–140 kilograms of spent mineral sorbent are generated [19, 20].

Despite encouraging research results on the use of clay sorbents, the use of clay sludge waste from the regeneration of engine oils to remove cationic dyes, in particular methylene blue, remains poorly understood. Therefore, the aim of this work was to obtain and experimentally study sorption properties of a material based on clay sludge waste during the removal of methylene blue.

To achieve this goal, the following tasks have been set:

– to obtain sorption material by heat treatment of oiled clay at various temperatures and to determine the optimal conditions for its formation;

– to characterize the porous structure of the resulting sorbent;

– to evaluate sorption properties of the materials obtained at different temperatures and to select the optimal sample;

– to determine the maximum sorption capacity of the selected sorbent, to construct adsorption isotherms and to evaluate the nature of the adsorption process.

Materials and Methods. The initial clay, which formed the basis of the clay sludge, according to the supplier's documentation, contained up to 30% clinoptilolite, up to 30% montmorillonite, approximately 20% calcite, 11% quartz, and 9% other impurities. The bulk density was 0.65–0.75 g/cm^3 and a true density was 2.3 g/cm^3.

The sorption material was obtained through thermal modification of oiled clay sludge formed during sorption regeneration of used engine oils. Heat treatment was conducted in a muffle furnace "Liop LF-7/13-G2" (RF).

At a temperature of 200°C, the clay waste retained a strong odor and lumpy structure. However, at 400°C, the original paste-like substance transformed into a loose, black material (GSH_{400} sample). An increase in temperature to 500°C caused the color to lighten due to the burning out of organic components.

Methods of mathematical processing of adsorption isotherms were used to evaluate the features of the pore distribution and determine the specific surface area. The porous structure of the samples was studied by low-temperature nitrogen adsorption on a TriStar 3020 instrument (Micromeritics, USA). The specific surface area was calculated using the Brunauer–Emmett–Teller (BET) method. The volume of micropores was determined by the t-Plot method of statistical thickness of the adsorption layer, based on a comparison of experimental and reference adsorption isotherms [21]. The volume of mesopores was determined by the Barrett–Joyner–Halenda (BJH) method. Working solutions of methylene blue were prepared by dissolving an exact amount of dye in distilled water. The dye concentration in the solutions before and after purification was determined photocolorimetrically at a wavelength of 670 nm using a “KFK-3-01” photocolorimeter (Russia). The purification of the model solutions was conducted as follows: 100 cm^3 of the solution was placed in a conical flask with a capacity of 250 cm^3, a calculated amount of sorbent was added, the mixture was stirred for a certain time, and then allowed to settle.

Cleaning efficiency was calculated by formula (1)

$$Э = \frac{C_{нач} - C_{кон}}{C_{нач}} \cdot 100\ \%, \qquad (1)$$

where Э — efficiency, %; $C_{нач}$ and $C_{кон}$ — concentrations of pollutants in the system, mg/dm, before and after purification, respectively.

Adsorption capacity was calculated by formula (2):

$$A = \frac{C_н - C_р \cdot V}{G}, \qquad (2)$$

where A — sorption capacity, mg/g; $C_н$ — concentration of dye before purification, mg/dm; $C_р$ — equilibrium concentration of dye in the solution, mg/dm; V — MB solution volume, dm; G — mass of the sorption material, g.

Results. Figure 1 demonstrates the isotherm of low-temperature nitrogen sorption.

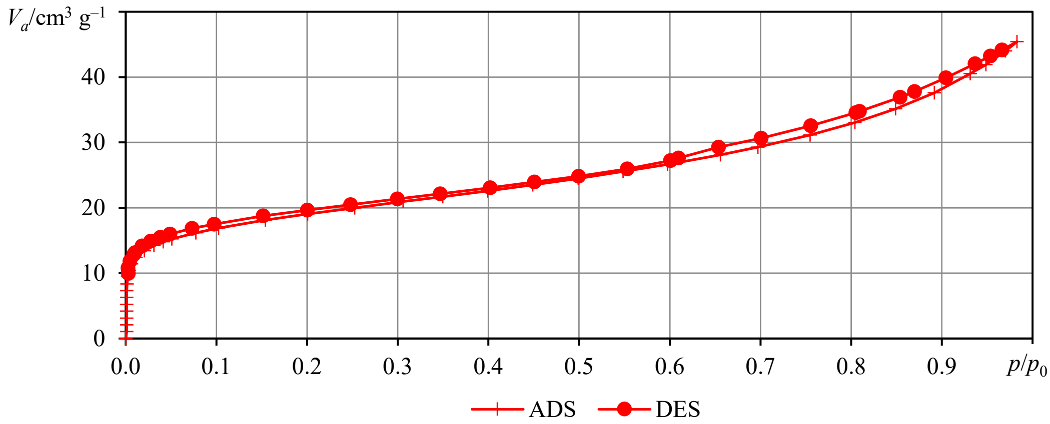

Рис. 1. Isotherm of low-temperature nitrogen sorption on the GSH400 surface:
ADS — isotherm of adsorption; DES — isotherm of desorption

As follows from Figure 1, the shape of the isotherm corresponded to type IV according to the classification [22], which indicated the occurrence of polymolecular sorption in mesopores, complicated by the phenomena of capillary condensation, since a small hysteresis loop was observed. The specific surface area was 69.148 m^2/g, and the total pore volume was 0.159 cm^3/g. According to the data obtained, there were no micropores in the sorbent under study.

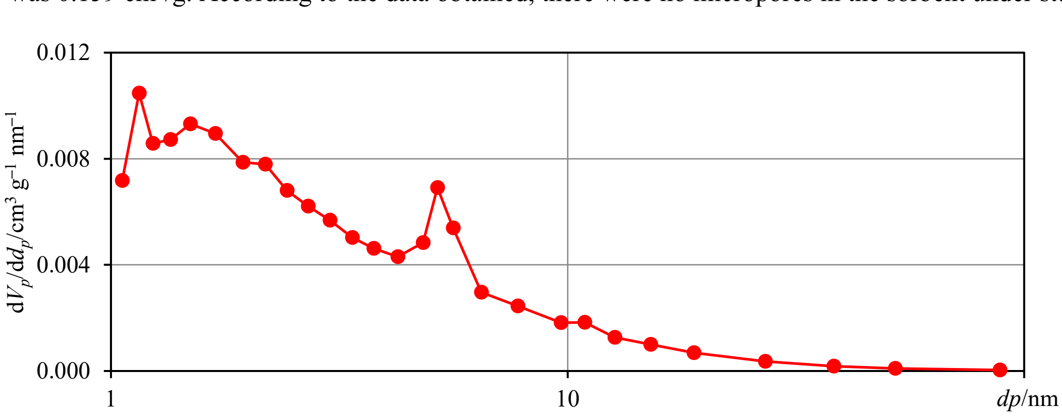

Fig. 2. Nitrogen adsorption isotherm calculated by the BJH method

As can be seen from Figure 2, the mesopore volume was 0.061 cm^3/g, the average pore diameter was 4.22 nm, and the median was 5.69 nm. This indicated the formation of a fairly homogeneous mesoporous structure, favorable for the processes of sorption of organic compounds.

To evaluate the GSH_{400} sorption properties, experiments were conducted with a model dye, methylene blue. Figure 3 shows the experimental results. The initial concentration of the dye was 30 mg/dm^3, the amount of the sorption material additive was 1 g/100 cm^3 of the solution.

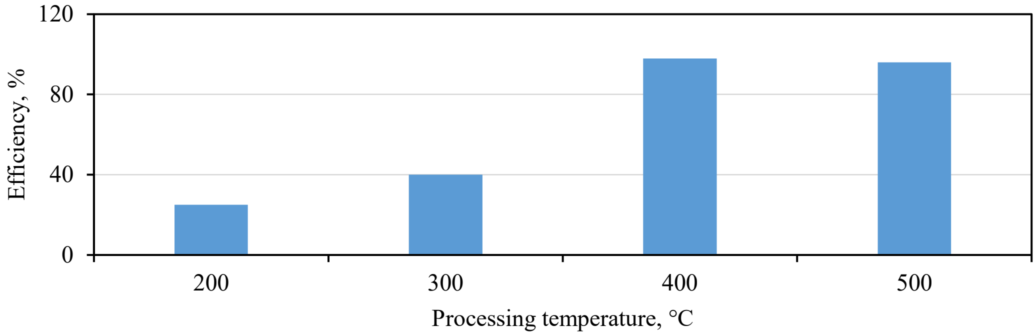

Fig. 3. Discoloration of solutions of methylene blue dye by sorption materials

As can be seen from Figure 3, the studied material demonstrated a pronounced ability to discolor the solution, indicating its high sorption activity with respect to organic dyes.

For a more detailed study of sorption processes, an adsorption isotherm was constructed on the surface of the GSH_{400} material (Fig. 4). The initial dye concentration was 0.313 $mmol/dm^3$, the sorbent mass varied from 1 to 8 g/dm^3.

According to the data in Figure 4, the maximum sorption capacity of the material reached 0.139 mmol/g, which characterized GSH_{400} as an effective sorbent for removing methylene blue from aqueous solutions.

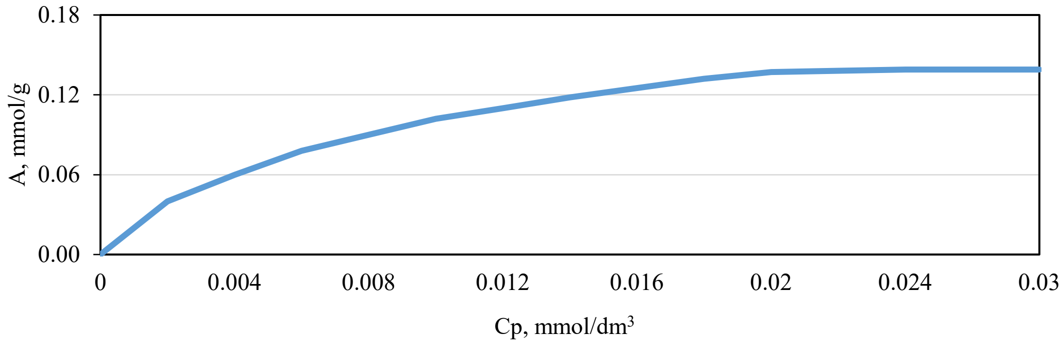

Fig. 4. Isotherm of adsorption of methylene blue on GSH_{400}

According to the data in Figure 4, the maximum sorption capacity of the material reached 0.139 mmol/g.

The experimental isotherm was approximated by the Langmuir, Freundlich, and Dubinin–Radushkevich models. Based on this processing, the isotherms were constructed (Fig. 5–7), the regression equations were determined, and the corresponding coefficients were calculated (Tables 1–3).

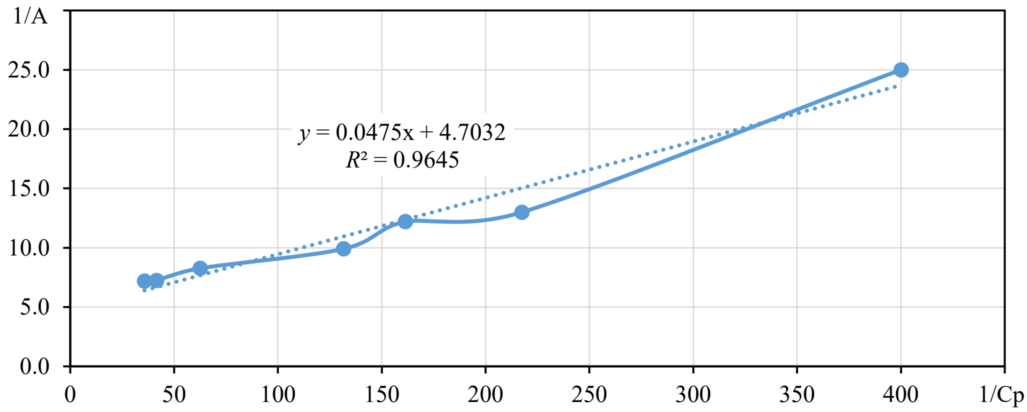

Fig. 5. Langmuir isotherm for methylene blue sorption on GSH_{400}

Table 1

Langmuir constants

$1/A = 1/A_\infty + 1/(K_L A_\infty C_p)$			
$y = 4.7032 + 0.0475x$			
$1/A_\infty =$	4.70316	$1/(K_L A_\infty) =$	0.04749
$A_\infty =$	0.21262	$K_L =$	99.02873

Langmuir model correlation coefficient R = 0.982097293.

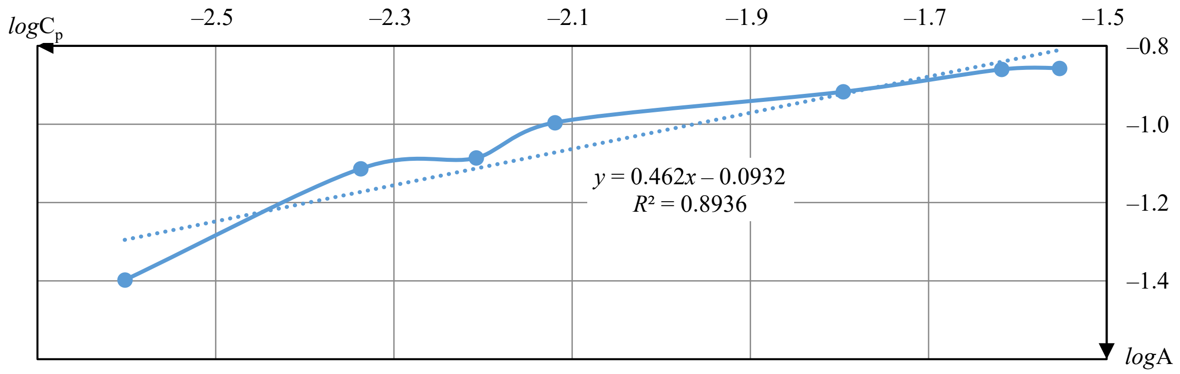

Fig. 6. Freundlich isotherm for methylene blue sorption on GSH_{400}

Table 2

Freundlich constants

$logA = logK_F + 1/n\ logC_p$			
$y = –0.0932 + 0.462x$			
$logK_F =$	–0.09316	1/n =	0.46200
$K_F =$	0.80695	n =	2.16450

Freundlich model correlation coefficient *R* = 0.945298744.

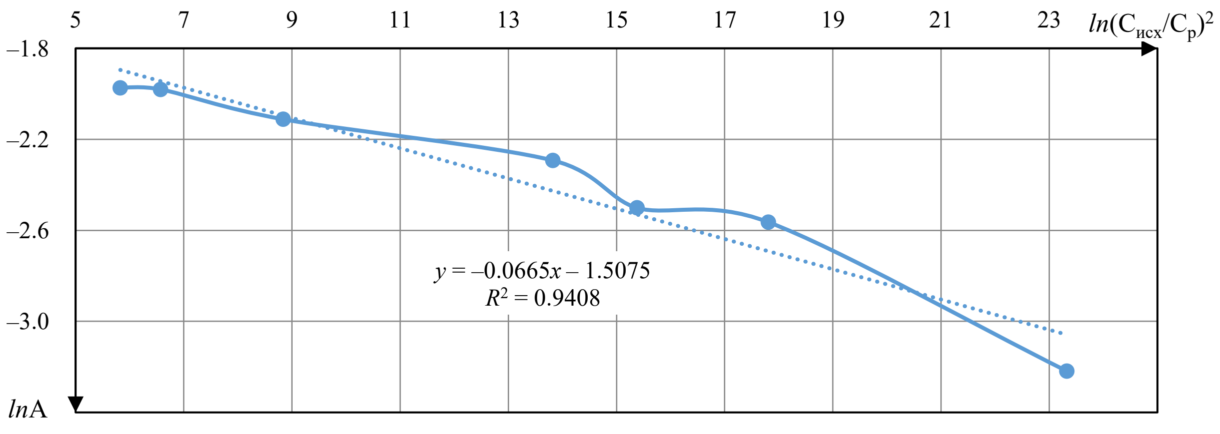

Fig. 7. Dubinin–Radushkevich isotherm for methylene blue sorption on GSH_{400}

Table 3

Dubinin–Radushkevich constants

$lnA = lnA_\infty – (R*T/E)^2 * (ln(C_{исх}/C_p))^2$			
$y = –1.5075 – 0.0665x$			
$lnA\infty =$	–1.50751	$(R*T/E)^2 =$	0.06649
$A\infty =$	0.22146	E =	9608.69551

Dubinin–Radushkevich model correlation coefficient *R* = –0.969934818.

Of the various models of isotherms of equilibrium and nonequilibrium sorption or sorption characteristics, the Langmuir and Freundlich models were the most popular.

As can be seen from the data obtained, the process of sorption of methylene blue on $GSH_{40}0$ was best described by the Langmuir model (approximation coefficient $R^2 = 0.9645$). Consequently, sorption had a monolayer character. The Langmuir constant was 99.02873, hence the R_L value could be determined by formula (3):

$$R_{\mathrm{L}} = \frac{1}{1 + K_{\mathrm{L}} C_0}, \tag{3}$$

where K_L — Langmuir constant, C_0 — initial concentration of adsorbate.

The calculated parameter value was 0.031, which confirmed the favorable nature of the process. The value of 1/n in the Freundlich model (0.462) indicated effective adsorption. Adsorption energy E, calculated using the Dubinin–Radushkevich model (9.608 kJ/mol), corresponded to the physical mechanism of sorption.

Discussion. The results show that clay sludge treated at a temperature of 400°C demonstrated high efficiency of cleaning solutions. The material obtained at elevated temperatures also exhibited significant sorption activity, but its synthesis required additional energy consumption. The optimal heat treatment temperature ensured a balance between the effectiveness of the process and economic feasibility.

High sorption activity of the GSH_{400} sample was due to the peculiarities of its porous structure. The predominance of mesopores with a diameter of ~4–6 nm ensured the availability of the active surface for methylene blue molecules, which corresponded to the well-known ideas about the mechanism of adsorption of organic dyes on mesoporous materials.

The isotherm classification as type IV and the presence of a hysteresis loop indicated capillary condensation, which helped to retain sorbate molecules in the pores. Similar patterns were found in the literature for clay and aluminosilicate sorbents, where the developed mesoporous structure was considered as a determining factor in increasing purification efficiency [23, 24].

The analysis of the adsorption models showed that the process was most fully described by the Langmuir equation, indicating the monolayer nature of adsorption on the sorbent surface. This may be due to the relative uniformity of the active centers formed during heat treatment.

The calculated value of the adsorption energy (9.608 kJ/mol) allowed us to attribute the process to physical adsorption, which meant the interaction between the sorbate and the sorbent through weak intermolecular forces (van der Waals), typical for organic dye – mineral sorbent systems.

The limitations of the research included the use of a single model pollutant and the lack of the analysis of the related factors influence (pH of the medium, ionic strength of the solution, the presence of competing substances). These parameters could significantly affect the mechanism and effectiveness of adsorption in real conditions.

Conclusion. Thus, the possibility of using waste from the regeneration of machine oils (clay sludge) as an effective sorbent for the extraction of the dye “Methylene Blue” has been demonstrated.

It has been found that the heat-treated clay sludge (GSH_{400}) has a well-developed mesoporous structure (with a volume of 0.159 cm^3/g) and demonstrates a sorption capacity of 0.139 mmol/g (44.8 mg/g) for methylene blue.

It has been shown that the sorption process is monolayer in nature and is mainly described by the Langmuir model, and the adsorption energy (9.608 kJ/mol) indicates a physical mechanism of interaction.

The practical significance of the results obtained lies in the fact that GSH_{400} can serve as an affordable and effective sorbent for wastewater treatment from organic dyes. Of particular importance is its application in the context of waste disposal, which increases the environmental and economic feasibility of the proposed approach.

The results obtained can be used in the development of water treatment technologies, as well as in further research aimed at optimizing heat treatment conditions and expanding the range of pollutants to be removed.

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