

ISSN 2181-8622

Manufacturing technology problems



Scientific and Technical Journal Namangan Institute of Engineering and Technology

INDEX  COPERNICUS
INTERNATIONAL

**Volume 11
Issue 1
2026**



NamMTI ILMIY-TEXNIKA JURNALI TAHRIR HAY'ATI A'ZOLARI

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ADSORPTION ACTIVITY OF BENTONITE CLAYS TOWARD DYES

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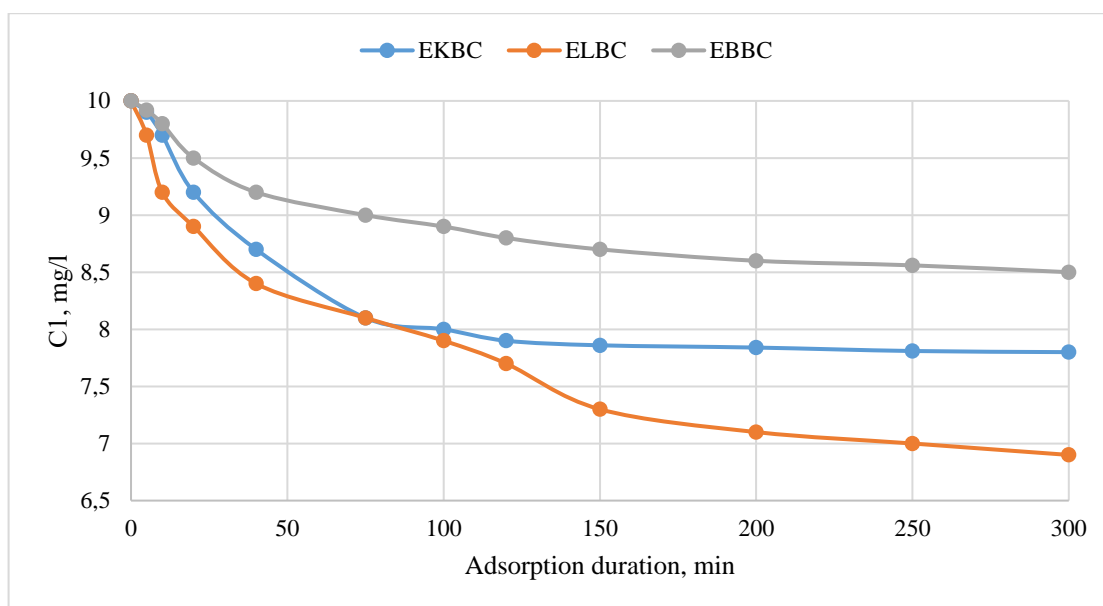
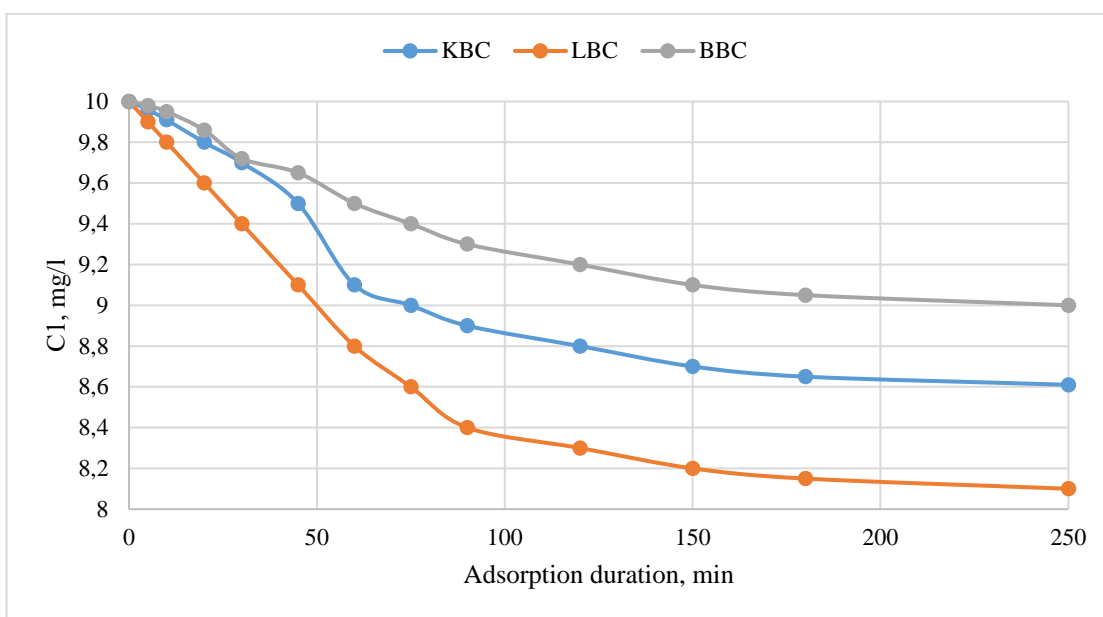
Abstract: This study examines the adsorption activity of bentonite clays toward various synthetic dyes in aqueous solutions. Bentonite, a naturally occurring aluminosilicate material with a layered structure and high specific surface area, demonstrates significant potential as an efficient and low-cost adsorbent for wastewater treatment. The adsorption capacity of bentonite depends on factors such as pH of the medium, initial dye concentration, contact time, temperature, and surface modification of the clay. Experimental results indicate that bentonite effectively removes both cationic and anionic dyes due to ion exchange, electrostatic interactions, and surface complexation mechanisms. The adsorption process was evaluated using equilibrium isotherm models and kinetic studies to determine the efficiency and mechanism of dye removal. The findings confirm that bentonite is a promising material for environmental purification technologies and can be applied in industrial wastewater treatment systems to reduce pollution caused by textile and chemical industries.

Keywords: Bentonite, adsorption, dyes, wastewater treatment, sorption capacity, clay minerals, isotherms, kinetics, ion exchange, surface modification, environmental protection, aluminosilicates, textile effluents, pH influence, adsorption mechanism.

Introduction. The relevance of investigating the adsorption of organic dyes by bentonites is обусловлена the widespread use of dyes in the textile, paper, and food industries and the high toxicity of their wastewater discharges into natural water bodies. Dyes are resistant to biodegradation, produce colored effluents, and disrupt photosynthesis in aquatic ecosystems; therefore, the development of inexpensive and efficient sorbents, in particular modified bentonites, represents an important task of environmental chemistry. High specific surface area, a developed pore structure, and the possibility of functional modification make bentonites promising materials for the removal of toxic dyes from water, thereby ensuring environmental protection and meeting modern wastewater treatment requirements.

Methodology & empirical analysis. Cationic methylene blue and anionic methyl orange were selected as model dyes to evaluate the sorption activity of bentonites. Methylene blue ($\lambda_{\max} = 664 \text{ nm}$) is widely used in textile and biological studies. Methyl orange ($\lambda_{\max} = 464 \text{ nm}$) belongs to anionic dyes and exhibits high resistance to photodegradation, which makes it suitable for analyzing its interaction with positively charged centers of bentonite after modification.

To study the sorption activity, a fixed mass of dry adsorbent (0.050g) and 50 ml of an aqueous dye solution of known concentration (10–100 mg/l) were placed into crystalline cuvettes or beakers. The suspensions were shaken on a shaker at 150rpm and room temperature for 2 h until adsorption equilibrium was reached. After centrifugation (3000rpm, 10 min), the supernatant was diluted if necessary, and the optical density was measured at λ_{max} of each dye. The concentrations before and after contact were determined using previously constructed calibration curves, and the amount of adsorbed dye was calculated from the difference in concentrations normalized to the mass of the adsorbent.



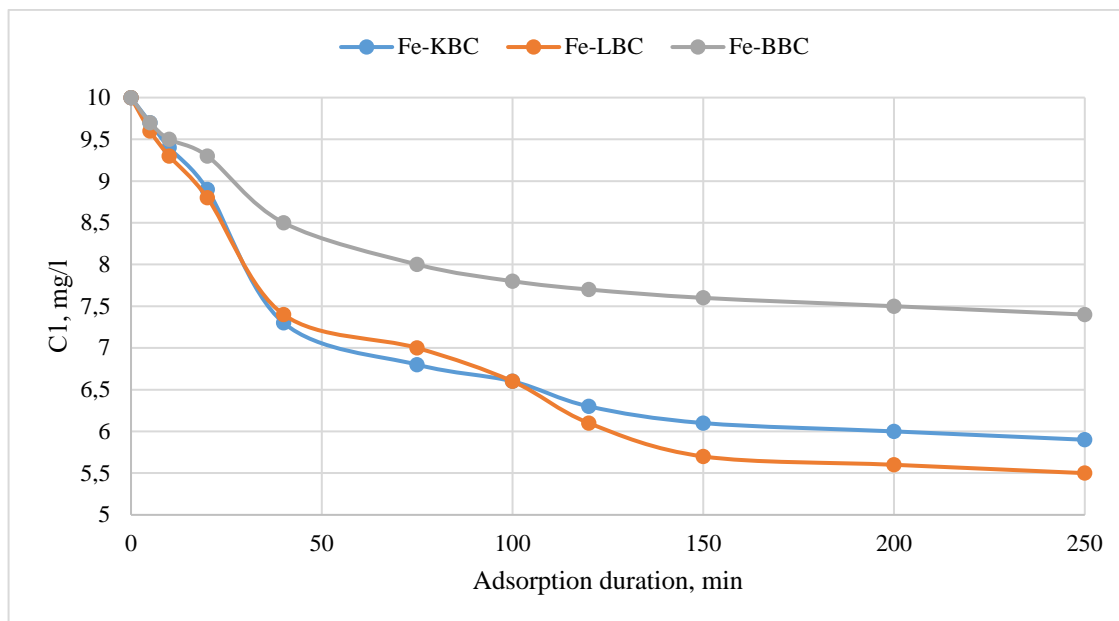


Figure 1. Kinetics of adsorption-induced decrease in the concentration of MB ions

In the adsorption kinetics of MB on the initial bentonites, a key role is played by electrostatic attraction between the cationic dye and the negatively charged basal surfaces of montmorillonite. In LBC, the more developed porous structure and higher cation exchange capacity (CEC) provide a more rapid decrease in C_1 , and within 20 min the MB concentration decreases to 9.6 mg/l, whereas for KBC and BBC this value is only 9.8 and 9.86 mg/l, respectively. During the subsequent 150–300 min, adsorption slows down due to the saturation of the main charge centers, and C_1 reaches a plateau (~8.2 mg/l for LBC, ~8.6 mg/l for KBC, and ~9.1 mg/l for BBC).

After enrichment, i.e., removal of coarse impurities and conversion to the sodium form, more open slit-like pores are formed and the number of available Na^+ exchange sites increases. In ELBC, within 40 min C_1 decreases to 8.4 mg/l (compared to 9.4 mg/l for the initial LBC), and by 100 min to 7.9 mg/l. Similar improvements are observed for EKBC and EBBC: $C_1 \approx 8.7$ and 9.2 mg/l at 40 min, with plateau values of 7.8 and 8.5 mg/l, respectively.

Upon Fe modification, nanoparticles of iron oxides/hydroxides are formed on the surfaces and in the interlayers, bearing strongly acidic (Fe–OH) groups. These groups create new electron-donor centers capable of forming donor–acceptor interactions with the π -electron system of MB. In Fe-LBC, C_1 decreases to 7.8 mg/l within 40 min and to 5.4 mg/l by 300 min, which is 30% more effective than the enriched analogue. Fe-KBC reaches $C_1 \approx 5.75$ mg/l, while Fe-BBC reaches 7.0 mg/l, although the latter remains inferior to the other samples due to its lower initial CEC.

Results. Thus, the differences in the kinetics and extent of MB removal are associated with three factors:

- 1) the CEC and porosity determine the rate of the initial phase of diffusion-controlled ion exchange;

- 2) removal of impurities provides access to exchange sites;
- 3) the presence of surface Fe–OH centers ensures specific π – π and donor–acceptor interactions with the dye, which accelerate and deepen the adsorption process.

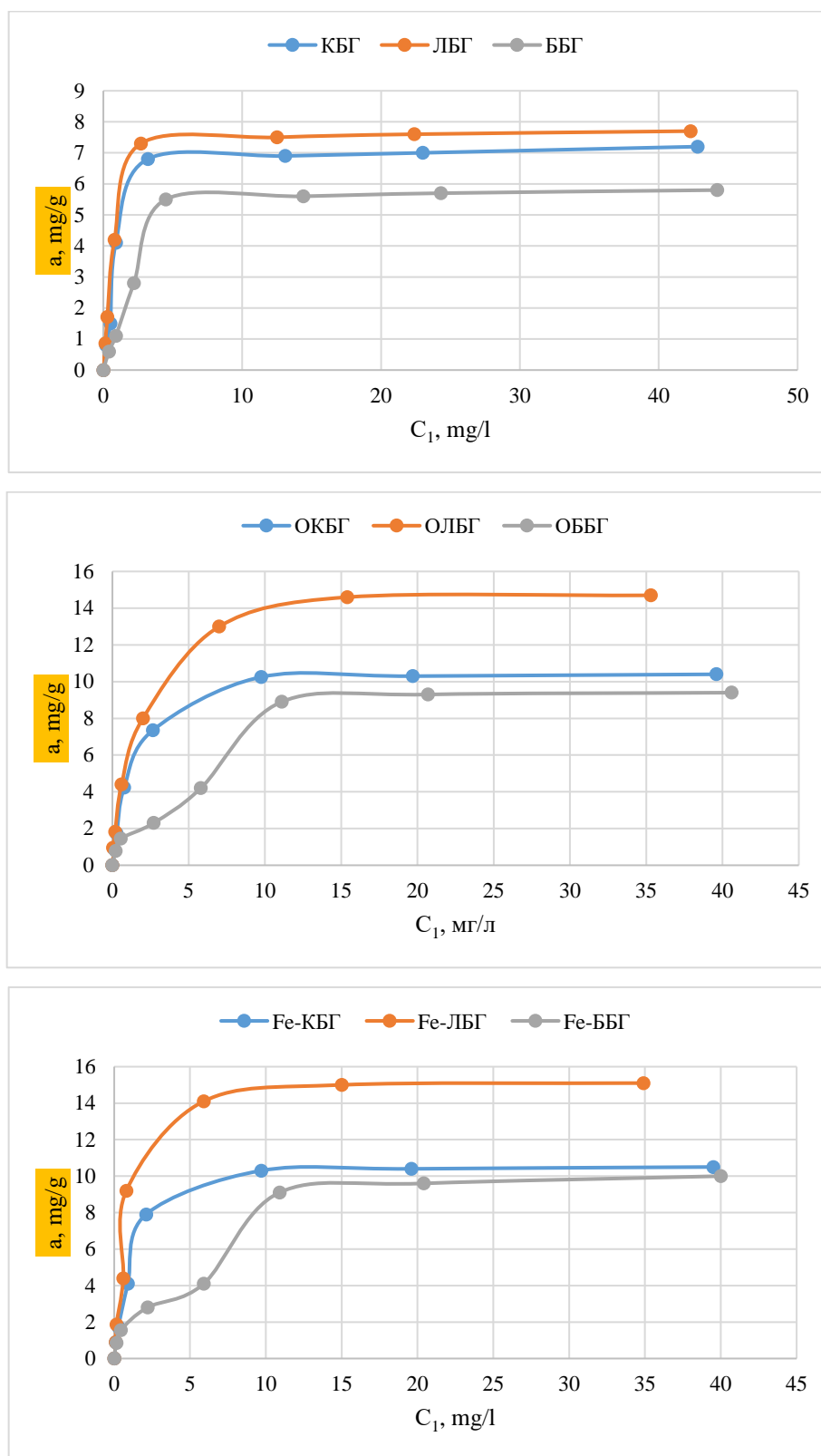


Fig. 2. Adsorption isotherms of MB from aqueous solutions (pH = 7, 25 °C)

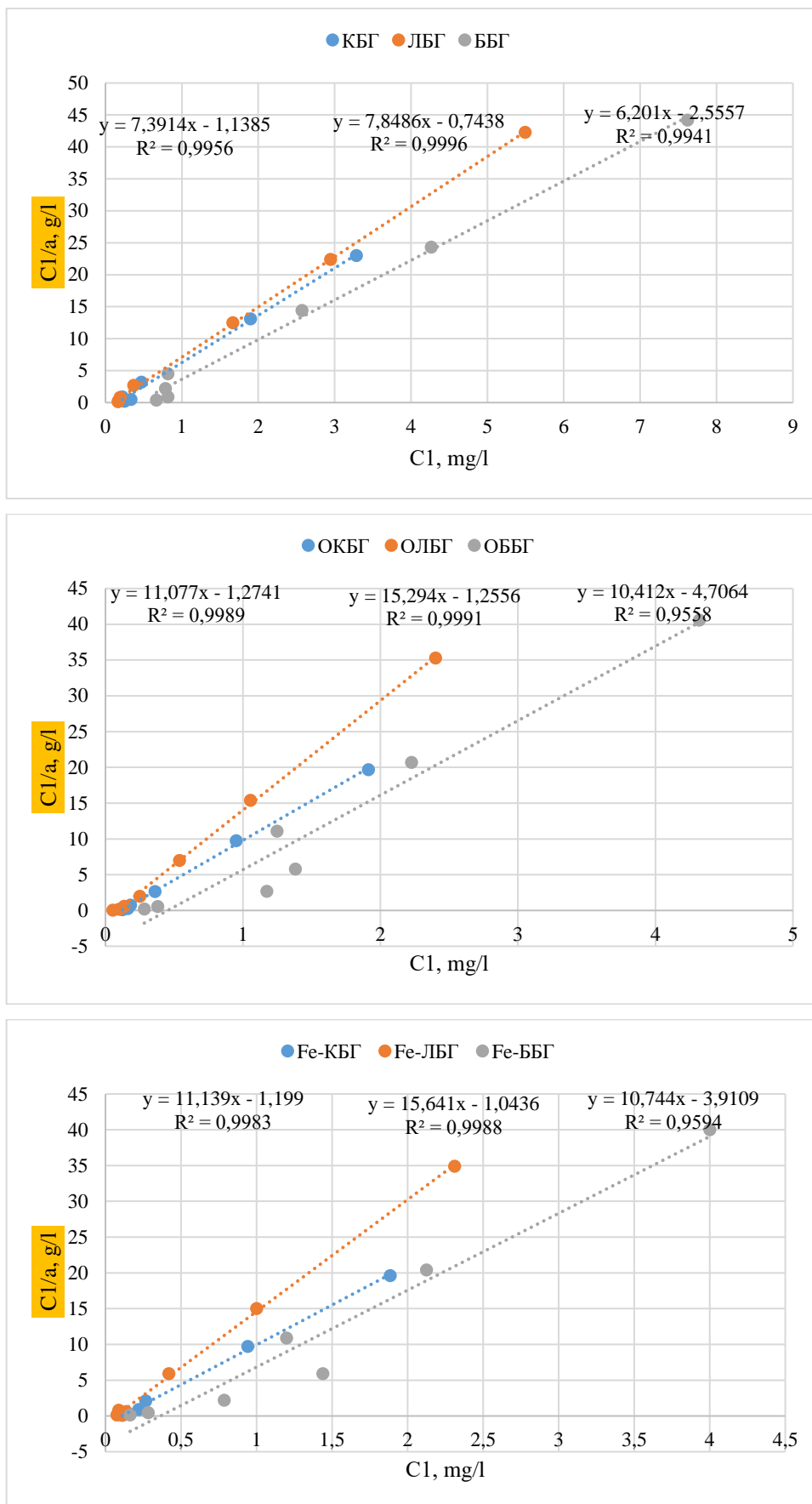


Fig. 3. Linear form of the Langmuir isotherm for MB adsorption.

Table 1. Porous structure parameters derived from MB adsorption isotherms according to the Langmuir equation

Sample	A_0 , mmol/g	K	S_{sp} , m ² /g	ΣV , cm ³ /g	R ²
KBC	0,090	-9,29	56,747	0,09	99,56
LBC	0,064	-14,988	40,413	0,064	99,96
BBC	0,093	-2,747	58,833	0,093	99,41
EKBC	0,091	-8,694	57,064	0,090	99,89
ELBC	0,065	-12,181	41,330	0,065	99,91
EBBC	0,096	-2,212	60,709	0,096	95,58
Fe-KBC	0,135	-6,492	85,518	0,135	99,83
Fe-LBC	0,161	-2,426	101,935	0,161	99,88
Fe-BBC	0,127	-10,552	80,537	0,127	95,94

The Langmuir isotherm parameters presented in Table 1. indicate that the maximum adsorption capacity A_0 and the porous structure characteristics increase consistently from the natural samples to the beneficiated and further to the Fe-modified ones. Thus, Fe-LBC reaches $A_0 = 0.161$ mmol/g at $S_{sp} = 101.9$ m²/g and $\Sigma V = 0.161$ cm³/g, whereas the initial LBC exhibits only 0.064 mmol/g, 40.4 m²/g, and 0.064 cm³/g, respectively. A similar trend is observed for KBC and BBC, indicating the key role of mechanical beneficiation and iron hydroxide intercalation in the expansion of the basal spacing and the formation of new meso- and micropores.

The affinity constant b (reciprocal of K in the table) reflects the strength of interaction between the MB cation and the adsorption sites. The highest absolute b values for the beneficiated LBC indicate strong affinity at a relatively low q_m , whereas after Fe modification b decreases (corresponding to a lower absolute K value), suggesting a shift of equilibrium toward an increased number of available sites (increase in q_m) accompanied by a somewhat weaker affinity per unit concentration. The high coefficients of determination $R^2 (> 0.95)$ confirm the applicability of the Langmuir model and the homogeneity of the sorption sites at all treatment stages.

Subsequently, the adsorption processes of MO (Methylene orange) on the studied samples were investigated. The obtained isotherms are presented in Fig. 4.

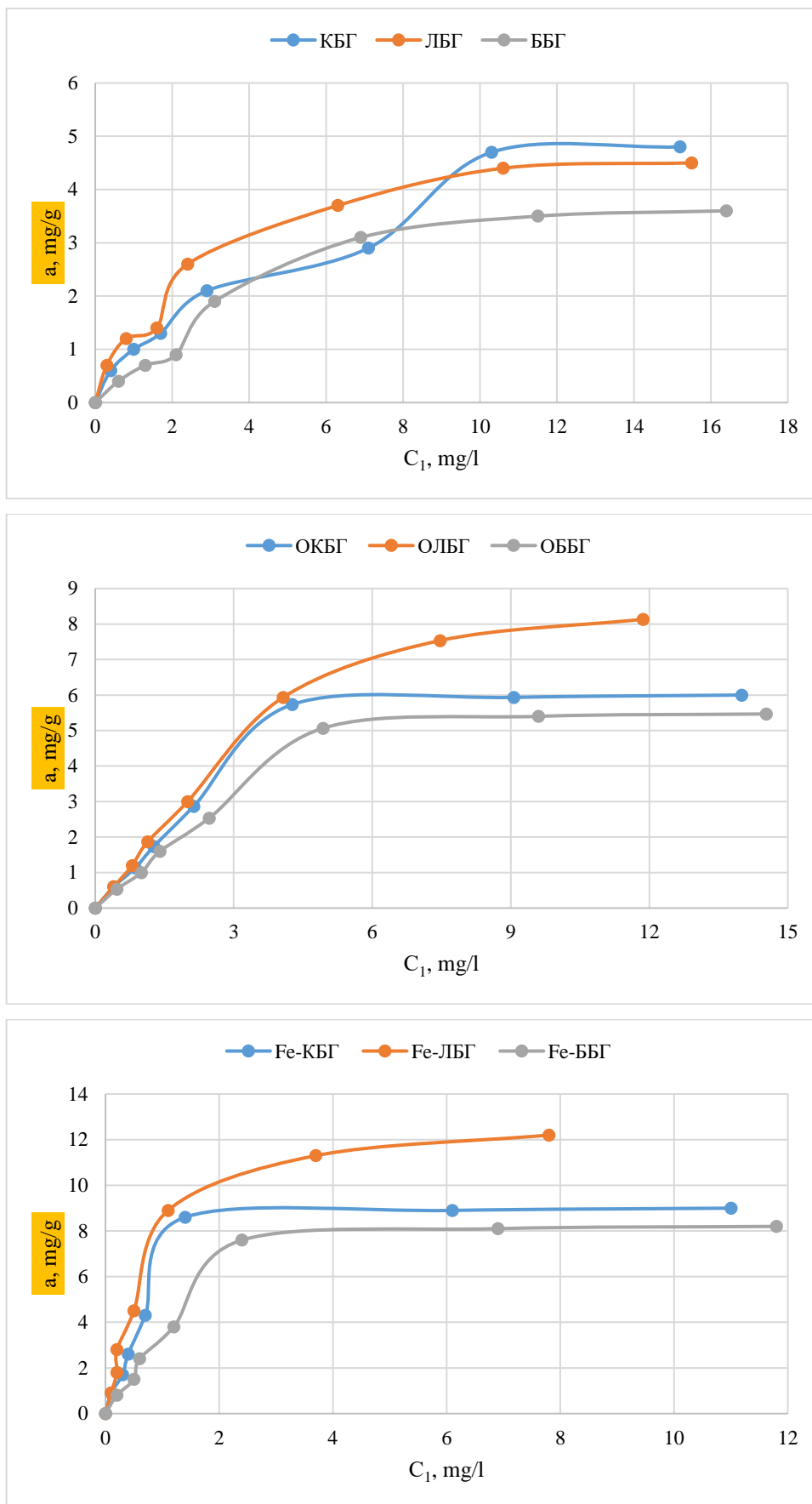


Fig. 4. Adsorption isotherms of MO from aqueous solutions (pH = 7, 25 °C)

The adsorption isotherms of MO on the initial bentonites demonstrate significantly different kinetics of monolayer filling and plateau attainment. For KBG, adsorption begins relatively slowly: at $C_1 = 0.4$ mg/l, $a = 0.6$ mg/g, and only at $C_1 = 1.7$ mg/l does it reach 1.3 mg/g. This is followed by a sharp increase to $a \approx 4.7$ mg/g at $C_1 = 10.3$ mg/l, after which saturation is completed at 4.8 mg/g at $C_1 = 15.2$ mg/l. Such an S-shaped profile indicates that in KBC the most accessible sites are involved first, followed by the more “hidden” regions of the porous structure.

For LBC, the curve is smoother and shifted to the left. Already at $C_1 = 0.8$ mg/l, $a = 1.2$ mg/g, and at $C_1 = 2.4$ mg/l the value reaches $a = 2.6$ mg/g. The main increase in adsorption occurs in the C_1 range 0.3–6.3 mg/l, and the plateau (~ 4.5 mg/g) is established at $C_1 \approx 10.6$ –15.5 mg/l. BBC exhibits the slowest and most gradual isotherm. At $C_1 = 0.6$ mg/l, a is only 0.4 mg/g; further growth proceeds uniformly to $a = 3.6$ mg/g at $C_1 = 16.4$ mg/l, without a pronounced stage of accelerated increase.

The enriched samples demonstrate a more distinct rapid-rise phase and a higher saturation level. In ELBC, already at $C_1 \approx 1.3$ mg/l adsorption reaches $a \approx 1.87$ mg/g, whereas in the initial LBC at the same C_1 the value of a was only 1.4 mg/g. Further, in the C_1 range ≈ 2 –5 mg/l, the enriched samples show a sharp increase of a to 2.9–3.0 mg/g (EKBC) and to 3.0–3.0 mg/g (ELBC), while EBBC in the same region increases only to 2.53–2.47 mg/g. This difference emphasizes that the effect of enrichment is most pronounced for KBC and LBC due to their higher initial CEC and the removal of coarse non-adsorbing inclusions.

At residual concentrations $C_1 > 10$ mg/l, a plateau is observed for the enriched samples: ELBC reaches $a \approx 7.5$ mg/g at $C_1 = 7.5$ –11.9 mg/l, and EKBC reaches $a \approx 6.0$ mg/g at $C_1 = 9$ –14 mg/l. EBBC attains a plateau of $a \approx 5.4$ mg/g at $C_1 \approx 9$ –15 mg/l. These levels are noticeably higher than those of the natural samples and reflect an increase in the number of available monolayer adsorption sites after fractional purification.

Thus, beneficiation of bentonites improves sorption kinetics and increases the maximum adsorption capacity toward methyl orange due to pore opening, an increase in specific surface area, and a higher concentration of exchangeable Na^+ centers, with the most pronounced effect observed for LBC.

For the Fe-modified samples, the MO isotherms exhibit an even steeper initial rise and significantly higher plateau levels compared with the enriched forms. Fe-LBC demonstrates the highest maximum adsorption and the lowest C_1 plateau value, whereas Fe-BBC shows the most broadened curve with the highest C_1 plateau, reflecting differences in the degree of iron incorporation and the initial porous structure of each sample.

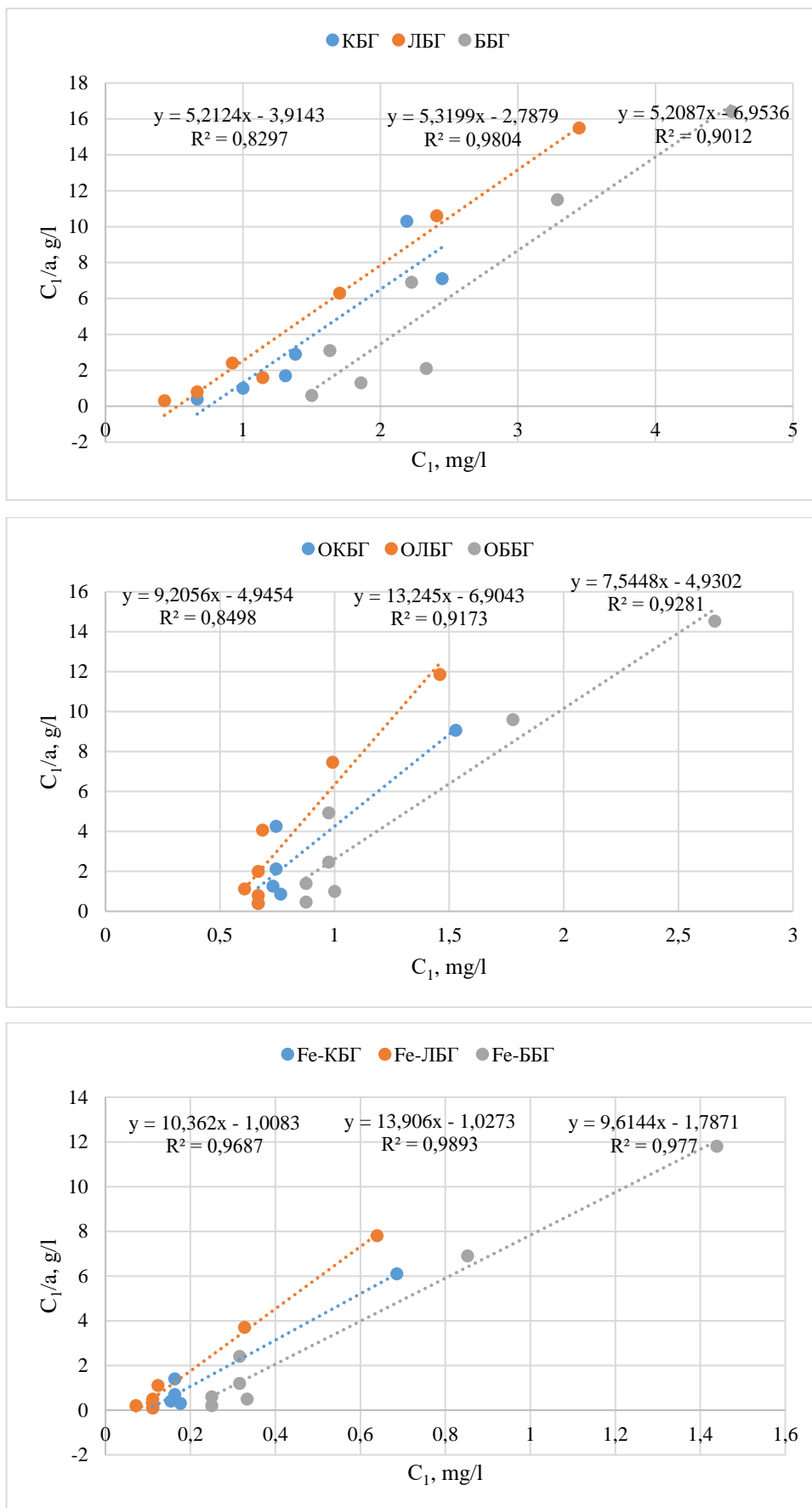


Fig. 5. Linear form of the Langmuir isotherm for MO adsorption

Table 2. Porous structure parameters derived from MO adsorption isotherms according to the Langmuir equation

Sample	A ₀ , mmol/g	K	S _{sp} , m ² /g	ΣV, cm ³ /g	R ²
KBC	0,076	-1,332	21,043	0,033	82,97
LBC	0,074	-1,908	20,489	0,031	98,04
BBC	0,072	-0,749	19,936	0,032	90,12
EKBC	0,097	-1,861	26,858	0,038	84,98
ELBC	0,092	-1,918	25,473	0,035	91,73
EBBC	0,076	-1,53	21,043	0,033	92,81
Fe-KBC	0,188	-10,28	52,054	0,042	96,87
Fe-LBC	0,192	-13,54	53,168	0,044	98,93
Fe-BBC	0,156	-5,38	43,199	0,039	97,70

The Langmuir isotherm parameters for MO adsorption on bentonites demonstrate a clear relationship between textural structure and sorbent capacity. In the initial samples (KBC, LBC, BBC), the specific surface area S_{sp} is only 19–21 m²/g, and the total pore volume ΣV is 0.031–0.033 cm³/g. The affinity constant b (expressed through the negative value K = -0.75...-1.91) indicates a moderate strength of interaction between MO and the pure clay.

Enrichment leads to an increase in S_{sp} to 25–27 m²/g and ΣV to 0.035–0.038 cm³/g. The growth in the number of slit-like pores and the opening of interlayer spaces in the enriched samples enhance the accessibility of adsorption sites; however, the affinity (K ≈ 1.53–1.92) remains at a comparable level, since the chemical nature of the active centers does not change.

The most significant changes are observed after Fe modification. S_{sp} doubles (43–53 m²/g), ΣV increases to 0.042–0.044 cm³/g, and A₀ reaches 0.156–0.192 mmol/g. The formation of new Fe–OH groups on the surface and in the interlayer space in the form of iron oxides creates highly active sites with strong affinity toward methyl orange (K = 5.38–13.54), confirming the synergistic effect of ion exchange and complex formation.

High R² values (>0.84) for all samples indicate the adequacy of the Langmuir model, while the substantial changes in textural parameters and A₀ emphasize that Fe modification is a key approach for enhancing the sorption capacity of clays toward anionic dyes.

For a more detailed characterization of adsorption, a multistage analysis based on the Dubinin–Radushkevich equation was applied, allowing estimation of the mean adsorption energy E and differentiation between physisorption and chemisorption. In its linear form:

$$\ln a = \ln a_0 - K\varepsilon^2,$$

$$\varepsilon = RT \ln \left(1 + \frac{1}{C_1} \right),$$

the coefficient K is related to the mean energy of transfer of an ion into the adsorption “site” through:

$$E = \frac{1}{\sqrt{2K}}.$$

The E values obtained from the Dubinin–Radushkevich equation are presented in Table 3.

Table 3. Characteristic adsorption energy values of MB and MO on bentonite samples

Sample	E, kJ/mol	Sample	E, kJ/mol
For MB adsorption		For MO adsorption	
KBC	7,8	KBC	10,3
LBC	11,1	LBC	12,2
BBC	8,3	BBC	13,6
EKBC	8,1	EKBC	11,4
ELBC	10,3	ELBC	9,1
EBBC	8,9	EBBC	9,3
Fe-KBC	15,6	Fe-KBC	18,3
Fe-LBC	16,1	Fe-LBC	19,1
Fe-BBC	14,7	Fe-BBC	20,6

The calculated values of the mean adsorption energy E for MB and MO at different stages of bentonite treatment allow assessment of the prevailing sorption mechanism. In the initial samples, for MB adsorption, E lies in the range 7.8–11.1 kJ/mol. At E < 8 kJ/mol (KBC, 7.8), physical adsorption predominates, whereas at 8 ≤ E ≤ 16 kJ/mol (LBC, 11.1; BBC, 8.3, etc.), the ion-exchange mechanism dominates.

For MO, the initial E values of 9.1–13.6 kJ/mol clearly fall within the “ion-exchange” range, which is consistent with electrostatic binding of the anion to positively charged centers of the clay. Fractional enrichment only slightly changes E (by 0.2–0.6 kJ/mol), confirming that the sorption mechanism remains the same, while the number of accessible exchange sites increases (as evidenced by the growth of A₀ and the textural parameters).

After Fe modification, the E value for MB increases to 14.7–16.1 kJ/mol, indicating an enhanced ion-exchange mechanism with a noticeable contribution from affinity to the newly formed Fe–OH centers. For MO, the modified samples exhibit even higher E values of 18.3–20.6 kJ/mol: Fe-KBC and Fe-LBC still fall within the upper ion-exchange range (≈19 kJ/mol), whereas Fe-BBC (E = 20.6 kJ/mol) exceeds the chemisorption threshold, indicating predominance of specific complex formation on Fe oxide surfaces.

Thus, the transition from physical adsorption to ion exchange and further to partial chemisorption upon Fe modification is confirmed by the increase in E. Untreated bentonites operate mainly via ion exchange (or even purely physical adsorption), whereas Fe-modified materials involve high-energy chemisorption processes.

Conclusions. The investigation of the adsorption activity of bentonites of different origin toward dyes has shown that even without any chemical treatment, natural clays exhibit relatively high adsorption activity toward the cationic dye methylene blue, not less than 0.07–0.10 mmol/g, primarily due to ion exchange between positively charged dye molecules and balancing $\text{Na}^+/\text{Ca}^{2+}$ cations in the interlayer positions of montmorillonite.

Enrichment further opens the pore network and increases the accessibility of exchange sites, leading to an approximately 15–30 % increase in A_0 for both types of dyes. However, the most significant effect is observed after Fe modification. The incorporation of iron oxides forms new positively charged Fe–OH groups on the surface and within the interlayer niches, capable of specific chemisorption binding of the anionic dye. As a result, the maximum A_0 values for methyl orange increase to 0.156–0.192 mmol/g, which is 2–3 times higher than those of the initial samples.

Thus, the combination of ion exchange in natural bentonite with complex formation at Fe oxide centers provides a synergistic increase in adsorption activity toward both cationic and anionic dyes, making Fe-modified bentonites universal sorbents for the removal of organic pollutants of different nature.

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