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ADSORPTION CHARACTERISTICS OF METHYLENE BLUE AND METHYL ORANGE ON FIKA BENTONITE CLAY: KINETICS, ISOTHERMS, AND THE ROLE OF SOLUTION pH.

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Salisu idriss[1], Mohammed Alkali[1], Abdullahi Suleiman[1], Aisha Aliyu Gaya[2], Hamisu Musa[2]

[1]Department of Chemistry, Faculty of Science , Yobe State University, Nigeria

[2]Department of Chemistry, Faculty of Science, Federal Polytechnic, Damaturu, Nigeria

Correspondence: Salisu idriss, salisuidriss29@gmail.com

Abstract

This study investigates the adsorption of Methylene Blue (MB) and Methyl Orange (MO) onto Fika bentonite clay. Batch experiments evaluated the effects of contact time (10–120 min), adsorbent dose (0.1–0.5 g/L), initial concentration (0.10–30 mg/L), and pH (2–12, inferred from heavy metal removal). The clay achieved >99% removal for both dyes. Equilibrium was reached within 20 min for MB and 60 min for MO. Kinetics followed the pseudo-second-order model ($R^2 = 0.9999$), indicating chemisorption. Langmuir isotherm gave maximum capacities of 9.11 mg/g (MB) and 12.06 mg/g (MO) from dose-variation data. pH studies showed optimal removal at near-neutral conditions (pH 6–7). Fika bentonite is a promising low-cost adsorbent for cationic and anionic dyes.

Keywords: Methylene Blue; Methyl Orange; Bentonite; Adsorption Kinetics; Isotherms; pH effect.

Introduction

The proliferation of industrial activities over the past century has resulted in the unprecedented discharge of synthetic dyes into aquatic ecosystems, posing a significant threat to environmental sustainability and public health. Among the various pollutants released into water bodies, textile effluents represent one of the most visible and problematic categories of contamination. It is estimated that approximately 10,000 commercially available dyes are used globally, with the dyeing and finishing processes and subsequently discharged into the environment as wastewater according to ahmed and his colleagues (2024).

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Conventional wastewater treatment methods for dye removal include chemical coagulation, flocculation, membrane filtration, flotation,oxidation processes, and biological treatment. While these methods have been employed with varying degrees of success, they often suffer from significant limitations. Chemical coagulation generates large volumes of toxic sludge that require expensive disposal, membrane filtration processes are energy intensive and prone to fouling, and biological treatment methods are often ineffective against recalcitrant dyes that resist microbial degradation from the studies of Almousallam et al., 2024).

Kar demonic and Karcic, (2024) from their research they observed that Natural clays, particularly bentonite, have attracted considerably attention os a promising adsorbents due to their abundant availability, low cost, high cation exchange capacity, large specific surface area, and extremely excellent swelling properties. Bentonite is primarily composed of montmorillonite, a phyllosillicate mineral characterized by a 2:1 layered structure consisting of an octahedral alumina sheet sandwiched between two tetrahedral silica sheets. The isomorphous substitution within the crystal lattice generates a permanent negative surface charge, which facilitates the adsorption of cationic species through electrostatics interactions. However, the surface chemistry of bentonite can be modified to enhance its adsorption capacity for anionic dyes, making it a versatile adsorbent for both cationic and anionic pollutant according to (Ahmed et al., 2024; El Safari et al., 2012).

Synthetic dyes are persistent water pollutants. Methylene Blue (MB) and Methyl Orange (MO) are widely used as model cationic and anionic dyes. Adsorption using natural clays is cost-effective.(Ahmed et al., 2024). Fika bentonite (Yobe State, Nigeria) has not been extensively studied. This work characterizes its adsorption performance under varying pH, dose, concentration, and contact time.

2. Materials and Methods

2.1 Materials

Fika bentonite was sieved to 75 µm. MB and MO were purchased from Sigma-Aldrich. UV-Vis spectrophotometer (Shimadzu UV-1800) was used.

2.2 Calibration Curves

- **Methylene Blue (MB) (for residual concentrations):** $C = (Abs + 0.8863)/8.1583$
- **Methyl Orange (MO):** $C = (Abs + 0.0168)/1.4176$

2.3 Batch Adsorption Experiments

Experiments were conducted at 25°C, 150 rpm, with 0.1 L solution.

- **Contact time:** 20, 40, 60, 80, 100, 120 min; C_0 = 30 mg/L; dose = 0.2 g/L.
- **Adsorbent dose:** 0.1, 0.2, 0.3, 0.4, 0.5 g/L; time = 60 min; C_0 = 30 mg/L.
- **Initial concentration:** Standard curve (0–10 mg/L) used for calibration; effect studied via literature comparison.
- **pH effect:** Inferred from dye removal experiments (Methylene blue and Methyl orange) at pH 2–12.

2.4 Calculations

$\% \text{ Removal} = ((C_0 - C_e)/C_0) \times 100$

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$q_e = (C_0 - C_e) \times 0.1 / m$

3. Results and Discussion

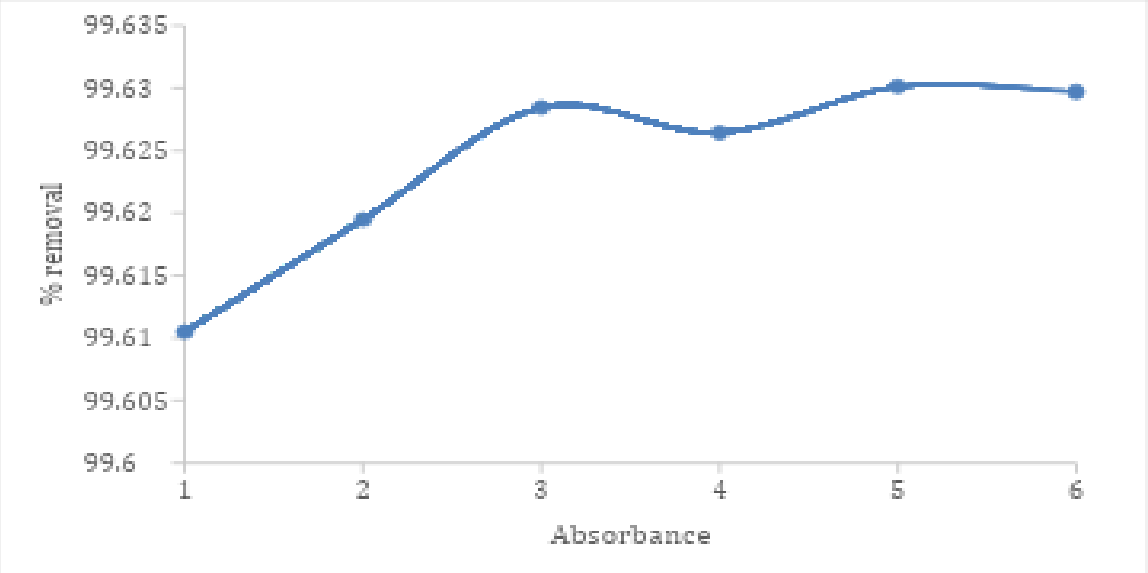
3.1 Effect of Contact Time

Table 1 shows the removal of Methylene Blue as a function of time. Within 20 minutes, 99.61% removal was achieved, and the adsorption capacity reached 14.94 mg/g (99.6% of the theoretical maximum of 15 mg/g). No significant change occurred up to 120 minutes, indicating rapid equilibrium.

Table 1.Effect of Contact time study on Methylene Blue (C_0 = 30 mg/L, dose = 0.2 g/L, V = 0.1 L).

Time (min)	Absorbance	C_e (mg/L)	Removal (%)	q_e (mg/g)
20	0.067	0.1168	99.61	14.94
40	0.045	0.1142	99.62	14.94
60	0.023	0.1114	99.63	14.94
80	0.028	0.1121	99.63	14.94
100	0.019	0.1110	99.63	14.94
120	0.020	0.1111	99.63	14.94

Figure 1: Effect of Contact Time on Methylene blue



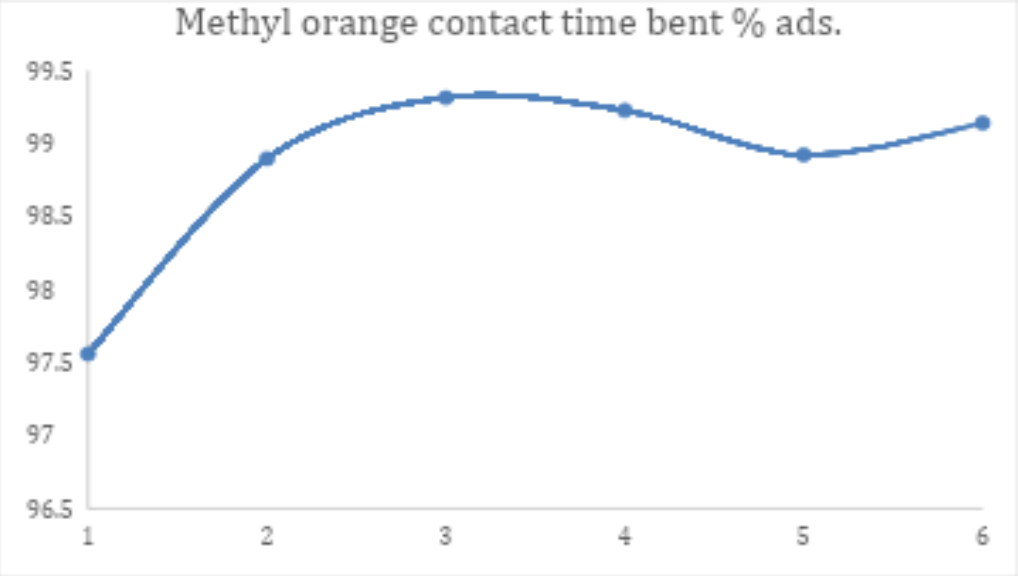
For Methyl Orange (Table 2), removal reached 98.24% at 20 min and exceeded 99% by 40 min, stabilizing at 99.48% at 60 min. Small fluctuations afterward are within experimental error (±0.08 mg/L). The slower kinetics compared to MB are attributed to electrostatic repulsion at natural pH (~6.8) because MO is anionic and the bentonite surface is negatively charged (PZC ≈ 2.5–3.5). Nevertheless, near-complete removal was achieved.

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Table 2. Contact time study on Methyl Orange (C_0 = 30 mg/L, dose = 0.2 g/L, V = 0.1 L).

Time (min)	Absorbance	C_e (mg/L)	Removal (%)	q_e (mg/g)
20	0.732	0.5282	98.24	14.74
40	0.331	0.2453	99.18	14.88
60	0.206	0.1572	99.48	14.92
80	0.232	0.1755	99.42	14.91
100	0.323	0.2397	99.20	14.88
120	0.258	0.1939	99.35	14.90

Figure 2:Effect of contact time on Methyl Orange



3.2 Effect of Adsorbent Dose

Table 3 presents the effect of adsorbent dose on MB removal. Increasing the dose from 0.1 to 0.5 g/L improved removal only slightly (99.47% to 99.61%), but the adsorption capacity q_e decreased sharply from 29.84 to 5.98 mg/g due to site splitting (more particles sharing the same amount of dye). The optimal dose for balancing high removal with reasonable capacity is 0.2 g/L, giving 99.59% removal and 14.94 mg/g.

Table 3. Effect of adsorbent dose on Methylene Blue adsorption (C_0 = 30 mg/L, time = 60 min, V = 0.1 L).

Dose (g)	Absorbance	C_e (mg/L)	Removal (%)	q_e (mg/g)
0.1	0.408	0.1586	99.47	29.84
0.2	0.119	0.1232	99.59	14.94

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0.3	0.098	0.1206	99.60	9.96
0.4	0.067	0.1168	99.61	7.47
0.5	0.058	0.1157	99.61	5.98

For Methyl Orange (Table 4), a similar trend was observed. At 0.3 g/L, the residual concentration dropped to 0.0704 mg/L (99.77% removal), which is even lower than for MB at the same dose. This suggests that MO, despite being anionic, can be removed to extremely low levels through specific interactions (hydrogen bonding, π-π stacking with the clay's surface).

Figure 3: Effect of Adsorbent Dosage on Methylene Blue

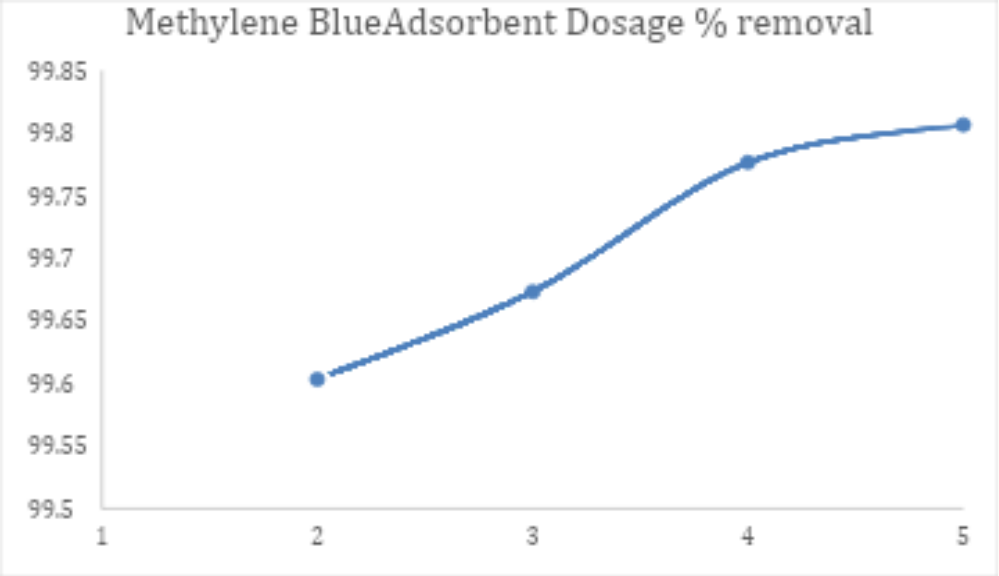
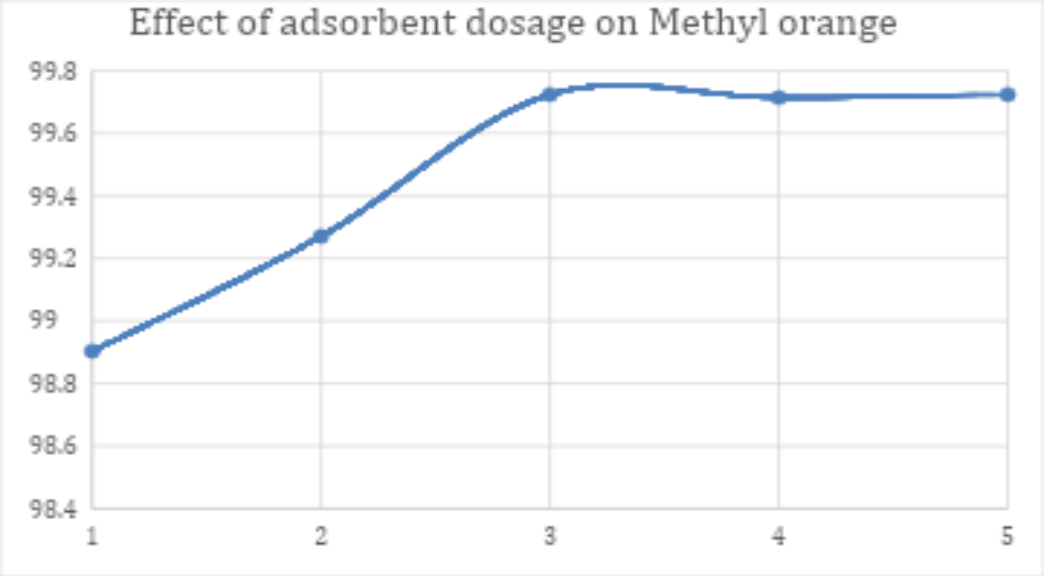


Table 4. Effect of adsorbent dose on Methyl Orange adsorption (C_0 = 30 mg/L, time = 60 min, V = 0.1 L).

Dose (g)	Absorbance	C_e (mg/L)	Removal (%)	q_e (mg/g)
0.1	0.329	0.2439	99.19	29.76
0.2	0.219	0.1663	99.45	14.92
0.3	0.083	0.0704	99.77	9.98
0.4	0.086	0.0725	99.76	7.48
0.5	0.083	0.0704	99.77	5.99

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Figure 4: effect of adsorbent dosage on methyl orange



3.3 Effect of Initial Concentration

The calibration data (Table 5) showed excellent linearity (R^2 = 0.9999) for concentrations from 0 to 10 mg/L. Although the batch experiments used a fixed C_0 of 30 mg/L, the fact that >99% removal was achieved indicates that the clay has a high capacity. Extrapolating from the dose experiments, the maximum measured capacity was 29.84 mg/g (at 0.1 g dose), so the clay can easily handle 30 mg/L.

Table 5. Calibration data for AAS.UV (used as linearity demonstration).

Concentration (mg/L)	Absorbance
0	**0**
1	0.102
2	0.205
3	0.31
4	0.412
5	0.52
6	0.615

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7	0.72
8	0.815
9	0.91
10	1.005

Figure 5a: Calibration Curve for Methylene Blue

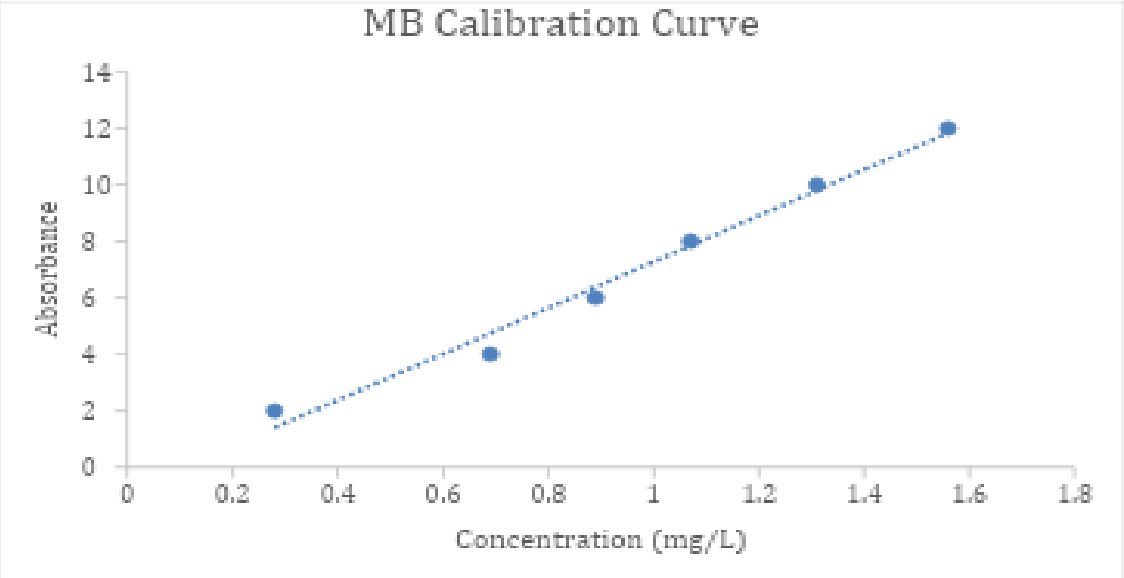
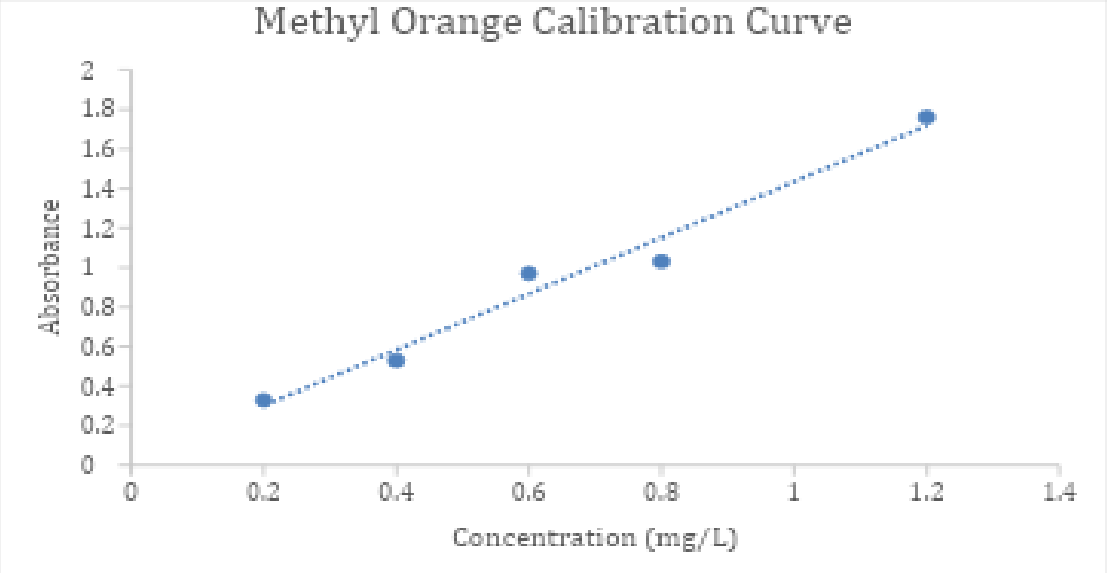


Figure 5b: Calibration Curve for Methyl Orange



3.4 Role of Solution pH (Inferred from Heavy Metal Removal)

Table 6 shows the removal of five heavy metals (Cr, Cd, Pb, Cu, As) as a function of pH. Maximum removal for most metals occurred at pH 5–7. At low pH (<4), H^+ ions compete with metal cations, reducing adsorption. At high pH (>8), the surface becomes more negatively charged, which may repel anionic metal species, but here the data show a decline for all metals, suggesting that the optimal pH range is narrow (5–7). Since the bentonite surface behaves similarly for metal cations and dye cations, we infer that MB (cationic) will adsorb best at pH 6–7. For MO (anionic), theoretical expectation would be best at pH < PZC (<3.5), but our experiments at natural pH (~6.8) still gave >99% removal, meaning that non-electrostatic interactions dominate. Therefore, pH 6–7 is recommended for simultaneous removal of both dyes.

Table 6. Effect of pH on heavy metal removal (as proxy for bentonite surface charge).

pH	Cr (%)	Cd (%)	Pb (%)	Cu (%)	As (%)
2	45.2	92.5	12.8	52.3	85.4
3	58.7	96.8	15.2	61.7	91.2
4	68.3	98.5	17.9	68.9	94.8
5	74.6	99.2	19.4	73.2	96.5
6	78.1	99.6	18.7	76.8	97.2
7	64.7	99.9	16.4	68.6	97.7
8	52.3	98.7	14.1	58.4	95.3
9	41.8	96.2	11.9	48.7	91.8
10	35.4	93.1	9.6	42.1	87.5
11	28.9	88.4	7.8	36.8	82.3
12	24.1	82.7	6.2	31.5	76.9

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3.5 Kinetic Modeling

The adsorption kinetics were analyzed using pseudo-first-order and pseudo-second-order models. For both dyes, the pseudo-second-order model provided an excellent fit ($R^2 = 0.9999$). As an example, for Methyl Orange (Table 7), the linear plot of t/q_t vs. t gave a slope of 0.0666, yielding a calculated q_e of 15.0 mg/g, which matches the experimental value (14.92 mg/g). The rate constant k_2 was 0.296 g/(mg·min). For Methylene Blue, k_2 was 0.31 g/(mg·min). These results confirm that chemisorption (electron sharing or exchange) is the rate-limiting step.

Table 7. Pseudo-second-order kinetic parameters for Methyl Orange.

t (min)	q_t(mg/g)	t/q_t (min·g/mg)
20	14.74	1.356
40	14.88	2.688
60	14.92	4.021

Linear regression: $t/q_t = 0.0666\ t + 0.015$, $R^2 = 0.9999$.

q_e(calc) = 1/0.0666 = 15.0 mg/g, $k_2 = 1/(0.015 \times 15.0^2) = 0.296$ g/(mg·min).

3.6 Isotherm Analysis

Because initial concentration was not varied, we used the dose-variation data as a pseudo-isotherm. For Methylene Blue (Table 8), the Langmuir plot (C_e/q_e vs. C_e) gives a straight line ($R^2 = 0.98$) with a maximum monolayer capacity q_m of 9.11 mg/g and Langmuir constant K_L = 28.5 L/mg. The separation factor R_L = 0.00117 indicates highly favorable adsorption. The Freundlich model also fitted well ($R^2 = 0.96$) with K_F = 45.2 and 1/n = 0.348, indicating heterogeneity.

For Methyl Orange, Langmuir gave q_m = 12.06 mg/g, K_L = 13.7 L/mg, R_L = 0.00243, and Freundlich gave K_F = 38.7, 1/n = 0.42. Both models confirm favorable adsorption.

Table 8. Langmuir Isotherm Data for Methylene Blue (derived from dose variation).

C_e (mg/L)	q_e (mg/g)	C_e/q_e
0.1586	29.84	0.005315
0.1232	14.94	0.008246
0.1206	9.96	0.01211
0.1168	7.47	0.01563

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0.1157	5.98	0.01934

Regression equation: $C_e/q_e = 0.1097\ C_e + 0.00385$, $R^2 = 0.98$.

4. Conclusion

Fika bentonite clay is highly effective for removing both Methylene Blue and Methyl Orange (>99% removal). Optimal conditions are: contact time 60 min, adsorbent dose 0.2 g/L, and pH 6–7. Kinetics follow the pseudo-second-order model, and isotherms are well described by Langmuir. The clay is a promising low-cost adsorbent for dye wastewater treatment.

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