



Adsorption of Methylene Blue by a Carboxymethyl Cellulose-Guar Gum Adsorbent: Freundlich and Langmuir Isotherm Analysis

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ABSTRACT

A carboxymethyl cellulose-guar gum (CMC-GG) adsorbent cross-linked with aluminum sulfate octadecahydrate and boric acid was evaluated for methylene blue (MB) adsorption. The study determined the adsorption equilibrium time, adsorption capacity at different MB concentrations, and the isotherm model that best described the adsorption process. The adsorbent was characterized by water absorption capacity (WAC), degree of cross-linking, Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). Contact times of 15, 30, 45, 60, and 75 min were examined, and equilibrium was reached at 45 min. Adsorption experiments were then conducted at initial MB concentrations of 20, 40, 60, 80, and 100 mg/L. The corresponding adsorption capacities were 3.7017, 7.4113, 10.9718, 14.9653, and 18.6531 mg/g. The adsorption capacity increased with the initial MB concentration. Isotherm analysis showed that the Langmuir model provided the best fit, with $R^2 = 0.9912$ and a calculated maximum adsorption capacity of 158.73 mg/g. These results indicate that the CMC-GG-Al-BA adsorbent has potential for the adsorption of cationic methylene blue from aqueous solution.

Keywords: Adsorbent, Adsorption Isotherm, Carboxymethyl Cellulose, Guar Gum, Methylene Blue

ABSTRAK

Adsorben karboksimetil selulosa-guar gum (CMC-GG) yang terdiri atas 10 g CMC dan 0,6 g guar gum serta diikat silang dengan 0,2 g aluminium sulfat oktadekahidrat dan 0,2 g asam borat (CMC-GG-Al-BA) dievaluasi untuk adsorpsi metilen biru (MB). Penelitian ini menentukan waktu kontak, kapasitas adsorpsi pada berbagai konsentrasi awal MB, dan model isoterm yang paling sesuai. Adsorben dikarakterisasi melalui kapasitas penyerapan air, derajat ikat silang, FTIR, dan SEM. Variasi waktu kontak 15, 30, 45, 60, dan 75 menit menunjukkan kapasitas adsorpsi terukur tertinggi pada 45 menit. Adsorpsi pada konsentrasi awal 20, 40, 60, 80, dan 100 mg/L menghasilkan kapasitas berturut-turut 3,7017; 7,4113; 10,9718; 14,9653; dan 18,6531 mg/g. Model Langmuir memberikan kecocokan lebih baik ($R^2 = 0,9912$) dibandingkan model Freundlich ($R^2 = 0,9721$), dengan kapasitas maksimum hasil perhitungan model sebesar 158,73 mg/g.

Kata Kunci: Adsorben, Isoterm Adsorpsi, Karboksimetil Selulosa, Guar Gum, Metilen biru

1. Introduction

Water is an irreplaceable resource that supports ecosystem sustainability, normal human activities, and life. However, the discharge of pollutants into aquatic systems has caused serious water contamination and presents risks to environmental and human health [1]. Inadequate wastewater management in the textile industry is a major contributor because textile effluents commonly contain persistent organic dyes that can adversely affect aquatic ecosystems [2].

Methylene blue (MB) is a water-soluble cationic dye widely used in textile and printing applications. In aquatic systems, dye contamination can hinder light penetration, reduce photosynthetic activity, and disturb aquatic biota [3]. Previous studies describe toxic effects of MB exposure, including serotonin toxicity at



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sufficiently high doses and a range of adverse physiological responses [4,5]. Consequently, effective treatment of MB-containing wastewater remains important.

Conventional treatment methods for synthetic dyes include chemical precipitation, ultrafiltration, coagulation, ozonation, oxidation processes, ion exchange, and reverse osmosis. These methods can be associated with high operating costs, hazardous by-products, or substantial energy demand [6]. Adsorption is therefore widely investigated because it is comparatively simple, effective, and potentially efficient for removing dissolved dyes [7]. Adsorption involves the accumulation of an adsorbate at the surface of a solid adsorbent through interactions between the adsorbate and active sites on the solid surface [8].

Biodegradable polymers such as chitin, alginate, guar gum (GG), and cellulose have attracted attention as low-cost adsorbent matrices. Guar gum cross-linked with boric acid has been reported to absorb 36.50 g/g of water [9], while a CMC system cross-linked with aluminum sulfate showed a water absorption capacity of 58 g/g [10]. High water uptake can support dye adsorption by facilitating access of dye molecules to the polymer network [11].

Carboxymethyl cellulose (CMC) is a cellulose derivative in which some hydroxyl groups of the glucopyranose units are substituted by carboxymethyl groups (-CH₂-COOH). It is an anionic, water-soluble, biodegradable polymer that is colorless, odorless, and non-toxic [12,13]. Guar gum is a renewable polysaccharide obtained from *Cyamopsis tetragonolobus* and is increasingly used in polysaccharide hydrogels because of its biodegradability, hydration ability, hydrogen-bonding capability, pH stability, and porous structure [14].

Previous studies have demonstrated the potential of GG- and CMC-based materials for cationic dye removal. Guar gum-acrylic acid graft copolymers have been used for methylene blue adsorption [15]. A CMC/GG/graphene oxide biopolymer nanocomposite removed malachite green with a reported maximum adsorption capacity of 17.6 mg/g and 88.2% removal efficiency [16]. A CMC-graphene oxide composite cross-linked with glutaraldehyde achieved 93-98% methylene blue removal and followed the Langmuir model with a maximum monolayer adsorption capacity of 76.92 mg/g [5].

Based on these considerations, this study prepared a CMC-GG adsorbent cross-linked with aluminum sulfate octadecahydrate and boric acid (CMC-GG-Al-BA) for methylene blue adsorption. Unlike previously reported systems that used guar gum graft copolymers, CMC/graphene oxide composites, or glutaraldehyde-cross-linked CMC-based materials, the present formulation combines CMC and guar gum with an inorganic aluminum sulfate cross-linker and boric acid in a simple film-forming system without graphene oxide or synthetic grafting. The study therefore provides adsorption and isotherm data for this specific CMC-GG-Al-BA formulation. The objectives were to determine the adsorption equilibrium time, evaluate adsorption capacity over a range of initial MB concentrations, and identify the adsorption isotherm model that best describes the system.

2. Methods

2.1. Materials and Equipment

The materials used were carboxymethyl cellulose, guar gum, aluminum sulfate octadecahydrate (Al₂(SO₄)₃·18H₂O), boric acid (H₃BO₃), distilled water, and methylene blue. The main equipment included standard laboratory glassware, an analytical balance, blender, oven, hot-plate magnetic stirrer, filtration equipment, Fourier-transform infrared (FTIR) spectrometer, scanning electron microscope (SEM), and UV-Visible spectrophotometer.

2.2. Preparation of the CMC-GG-Al-BA Adsorbent

CMC (10 g) was dissolved in 900 mL of distilled water under magnetic stirring. Separately, 0.6 g of guar gum was dissolved in 50 mL of distilled water. The two solutions were mixed and stirred for 1 h until homogeneous. Aluminum sulfate octadecahydrate (0.2 g) and boric acid (0.2 g) were then added as cross-linking agents. The available experimental record does not specify the stirring speed or the additional stirring duration after addition of the cross-linking agents; these parameters are therefore reported as unavailable rather than assigned retrospectively. The mixture was cast onto a Teflon-lined tray and dried in an oven at 60 °C until a dry film was obtained. The resulting film was subsequently characterized and used as the adsorbent.

2.3. Characterization

2.3.1. Degree of Substitution of CMC

The FTIR spectrum of commercial CMC was used to estimate an FTIR absorbance-ratio parameter associated with the relative intensities of the hydroxyl and carboxylate bands. Absorbance was calculated from transmittance using the expression below. Because this approach is based on band-intensity ratios rather than a direct chemical titration, the resulting value is treated as a semi-quantitative FTIR-based estimate and should not be interpreted as an independently validated absolute degree of substitution as presented in Equation 1 and 2.

$$Abs = \log \left(\frac{I}{I_0} \right) \quad (1)$$

$$FTIR \text{ absorbance ratio} = Abs \frac{-OH}{-COO-} \quad (2)$$

2.3.2. Water Absorption Capacity

A 0.25 g portion of the adsorbent was wrapped in filter paper and immersed in distilled water for 7 h. After removal and drainage of excess water, the hydrated adsorbent was weighed. Water absorption capacity (WAC) was calculated as shown in Equation 3.

$$WAC = \frac{(W_1 - W_0)}{W_0} \quad (3)$$

where W_0 is the initial dry mass and W_1 is the mass after water absorption.

2.3.3. Degree of Cross-Linking

Approximately 0.53 g of dried adsorbent was immersed in water for 24 h, filtered, and dried at 60 °C for 3 h. The dry mass after immersion was recorded and the degree of cross-linking was calculated as demonstrated in Equation 4.

$$\text{Cross - linking degree (\%)} = (W_g - W_0) \times 100 \quad (4)$$

where W_g is the dry mass after immersion and W_0 is the initial dry mass.

2.3.4. FTIR and SEM Analysis

FTIR analysis was performed over 4000-400 cm^{-1} to identify the functional groups of the synthesized adsorbent. SEM was used to observe the adsorbent surface morphology; the sample was coated with gold prior to electron-beam imaging, as described in the source manuscript.

2.4. Preparation of Methylene Blue Solutions and Calibration

A 1000 mg/L MB stock solution was prepared by dissolving 1 g of MB and diluting to 1 L with distilled water. A 100 mg/L solution was prepared by diluting 100 mL of the stock solution to 1 L. Working solutions of 80, 60, 40, and 20 mg/L were prepared by dilution. For the calibration curve, 5, 10, 15, 20, and 25 mL aliquots of the 100 mg/L solution were diluted to 100 mL to obtain 5-25 mg/L standards. Absorbance was measured at 664.1 nm.

2.5. Adsorption Experiments

2.5.1. Effect of Contact Time

A 100 mL portion of 20 mg/L MB solution was contacted with approximately 0.5 g of adsorbent at 250 rpm for 15, 30, 45, 60, or 75 min. The available experimental record does not document a controlled adsorption temperature or the initial solution pH, and no pH adjustment procedure was reported. The mixture was filtered and the residual MB concentration in the filtrate was determined by UV-Visible spectrophotometry at 664.1 nm. The number of independent experimental replicates was also not documented in the available record; consequently, the values are presented descriptively without standard deviations or inferential statistics.

2.5.2. Effect of Initial Concentration and Isotherm Analysis

Approximately 0.5 g of adsorbent was added to 100 mL MB solutions with initial concentrations of 20, 40, 60, 80, and 100 mg/L. Each mixture was stirred at 250 rpm for the selected contact time and filtered. As noted above, adsorption temperature, initial pH, and the number of independent replicates were not documented in

the available experimental record. The equilibrium concentration (C_e) was determined spectrophotometrically. The adsorption capacity was calculated using Equation 5.

$$Q_e = \frac{(C_0 - C_e) \times V}{m} \quad (5)$$

where Q_e is the equilibrium adsorption capacity (mg/g), C_0 and C_e are the initial and equilibrium concentrations (mg/L), V is the solution volume (L), and m is the adsorbent mass (g). The Freundlich and Langmuir linear equations used in the source study were Equation 6 and 7:

$$\log Q_e = \log Kf + \left(\frac{1}{n}\right) \log C_e \quad (6)$$

$$\frac{1}{Q_e} = \left(\frac{1}{(KL - Q_m)}\right) \left(\frac{1}{C_e}\right) + \left(\frac{1}{Q_m}\right) \quad (7)$$

where Kf and n are Freundlich constants, KL is the Langmuir constant (L/mg), and Q_m is the maximum monolayer adsorption capacity (mg/g). The Langmuir separation factor was calculated as $RL = 1/(1 + KL C_0)$.

3. Results and Discussion

3.1. Preparation and Characterization of the Adsorbent

The CMC-GG-Al-BA adsorbent was obtained as a dried film after cross-linking CMC and guar gum with aluminum sulfate octadecahydrate and boric acid and drying the cast mixture at 60 °C. The composition used was 10 g CMC, 0.6 g guar gum, 0.2 g $Al_2(SO_4)_3 \cdot 18H_2O$, and 0.2 g H_3BO_3 . The obtained adsorbent is shown in Figure 1.



Figure 1. CMC-GG-Al-BA adsorbent obtained after drying.

The water absorption capacity of the adsorbent was 60.47 g/g. The source study attributes this high uptake to an open polymer structure that can facilitate water penetration and increase the accessible area for adsorbent-dye interaction [11]. The dry adsorbent mass before immersion was 0.5360 g and the dry mass after the 24 h immersion/drying procedure was 0.3571 g, corresponding to a cross-linking degree of 66.62%. Cross-linking can influence adsorption by changing the number and accessibility of interaction sites within the hydrogel matrix [17].

3.2. FTIR analysis

FTIR spectra of CMC, guar gum, and the CMC-GG-Al-BA adsorbent are presented in Figure 2. The main bands reported are summarized in Table 1.

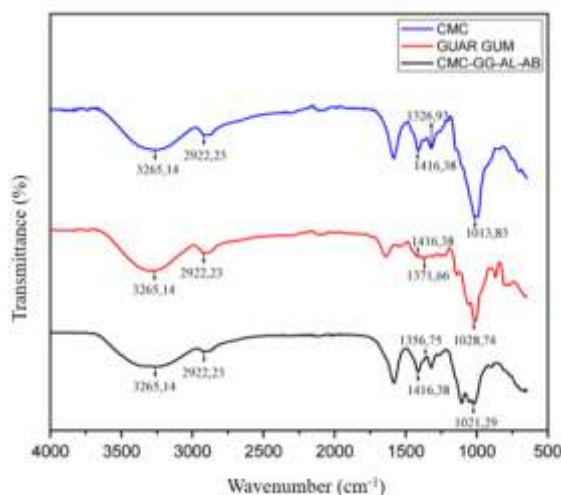


Figure 2. FTIR spectra of CMC, guar gum, and CMC-GG-Al-BA adsorbent.

Table 1. Main FTIR bands of CMC, guar gum, and CMC-GG-Al-BA adsorbent.

Assignment	CMC (cm ⁻¹)	Guar gum (cm ⁻¹)	CMC-GG-Al-BA (cm ⁻¹)
O-H stretching	3265.14	3265.14	3265.14
C-H stretching	2922.23	2922.23	2922.23
O-H bending	1326.93	1371.66	1356.75
COO- stretching	1416.38	1416.38	1416.38
C-O-C stretching	1013.83	1028.74	1021.29

The three spectra show similar major functional groups. A broad O-H stretching band occurs at 3265.14 cm⁻¹. C-H stretching is observed at 2922.23 cm⁻¹. O-H bending bands occur at 1326.93 cm⁻¹ for CMC, 1371.66 cm⁻¹ for guar gum, and 1356.75 cm⁻¹ for CMC-GG-Al-BA. A carboxylate (COO-) band appears at 1416.38 cm⁻¹, while C-O-C stretching bands occur at 1013.83, 1028.74, and 1021.29 cm⁻¹ for CMC, guar gum, and the adsorbent, respectively. These hydroxyl and carboxylate groups are chemically plausible interaction sites for cationic MB molecules [18]. However, FTIR spectra after MB adsorption were not available in the experimental record; therefore, direct spectral evidence for the proposed adsorbent-MB interaction could not be evaluated in the present study and should be addressed in future work.

3.3. SEM Morphology

SEM micrographs of the cross-linked adsorbent at 1000× and 1500× magnification are shown in Figure 3. The surface appears relatively smooth and homogeneous at the observed magnifications. The observed this morphology to the cross-linked structure, which promotes a more uniform polymer mixture. SEM provides surface and topographical information through the interaction of an electron beam with the specimen [19]. Furthermore, while post-adsorption SEM characterization would further enrich the mechanistic insights, it is reserved for comprehensive follow-up investigations.

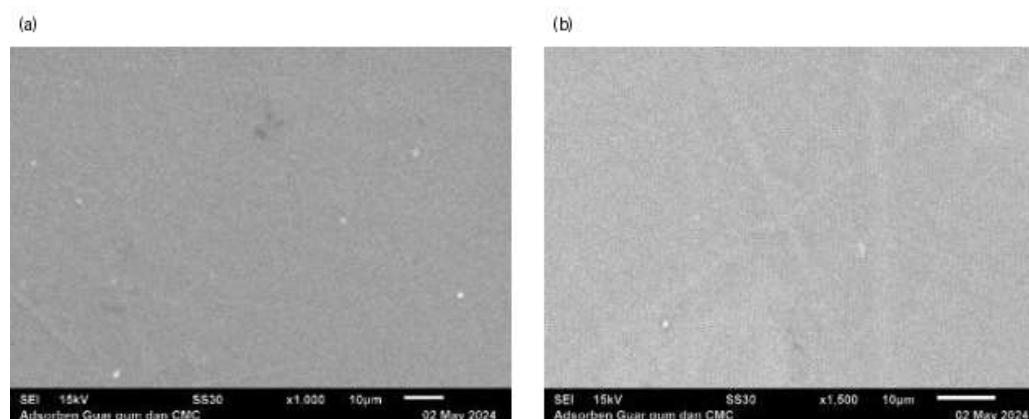


Figure 3. SEM micrographs of the CMC-GG-Al-BA adsorbent at (a) 1000× and (b) 1500× magnification.

3.4. Degree of Substitution of CMC

The FTIR spectrum used for evaluating the commercial CMC is shown in Figure 4. The absorbance values at 3265.14 cm^{-1} (-OH) and 1416.38 cm^{-1} (-COO-) were 0.1045 and 0.1049, respectively. The absorbance-ratio calculation gave a value of 0.9961. This value is reported here as an FTIR-based estimate derived from the operational equation used in the experimental record. Because the method relies on spectral band intensities and was not independently validated by a direct substitution-analysis method, the value should be interpreted cautiously.

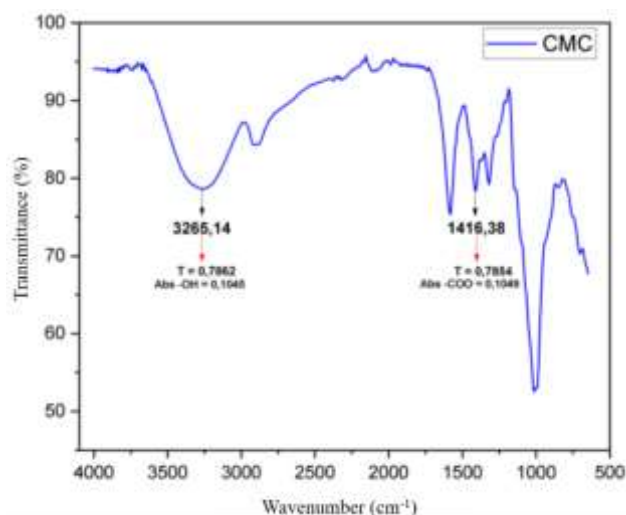


Figure 4. FTIR spectrum of commercial CMC used to estimate the degree of substitution.

3.5. Methylene blue calibration curve

The absorbance of the 5-25 mg/L MB standards increased proportionally with concentration. The measured values are listed in Table 2. Linear regression gave $y = 0.0468x - 0.027$ with $R^2 = 0.9997$, as shown in Figure 5.

Table 2. UV-Visible calibration data for methylene blue at 664.1 nm.

No.	MB concentration (mg/L)	Absorbance
1	5	0.2067
2	10	0.4480
3	15	0.6642
4	20	0.9102
5	25	1.1454

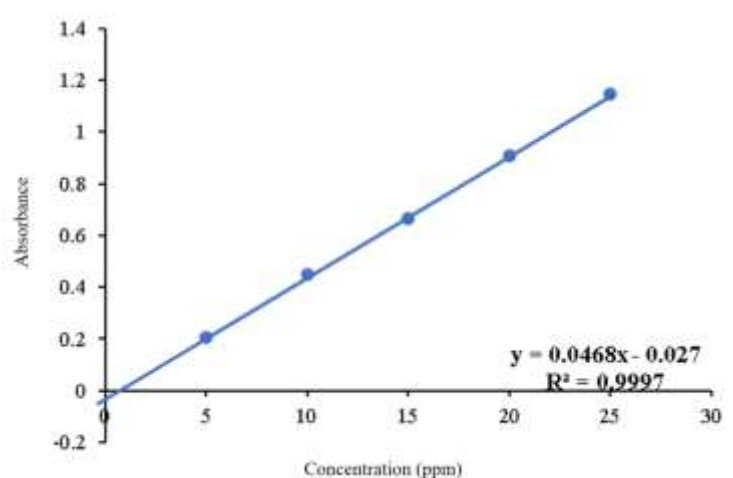


Figure 5. Calibration curve of methylene blue at 664.1 nm.

3.6. Effect of Contact Time

Contact time influenced the measured MB adsorption capacity. Table 3 shows that the adsorption capacity increased from 3.44685 mg/g at 15 min to the highest measured value of 3.69451 mg/g at 45 min, after which it slightly decreased to 3.6517 and 3.64125 mg/g at 60 and 75 min, respectively. Therefore, 45 min was selected as the operational contact time for the subsequent concentration experiment. Because replicate measurements were not documented, the small differences among 45, 60, and 75 min are interpreted descriptively rather than as statistically significant differences.

Table 3. Effect of contact time on methylene blue adsorption.

Time (min)	Adsorbent mass (g)	C0 (mg/L)	Absorbance	Ce (mg/L)	Qe (mg/g)	Adsorbed MB (mg/L)
15	0.5089	20	0.0881	2.4590	3.44685	17.5410
30	0.5058	20	0.0740	2.1581	3.52746	17.8419
45	0.5041	20	0.0374	1.3760	3.69451	18.6240
60	0.5048	20	0.0463	1.5662	3.6517	18.4338
75	0.5093	20	0.0411	1.4551	3.64125	18.5449

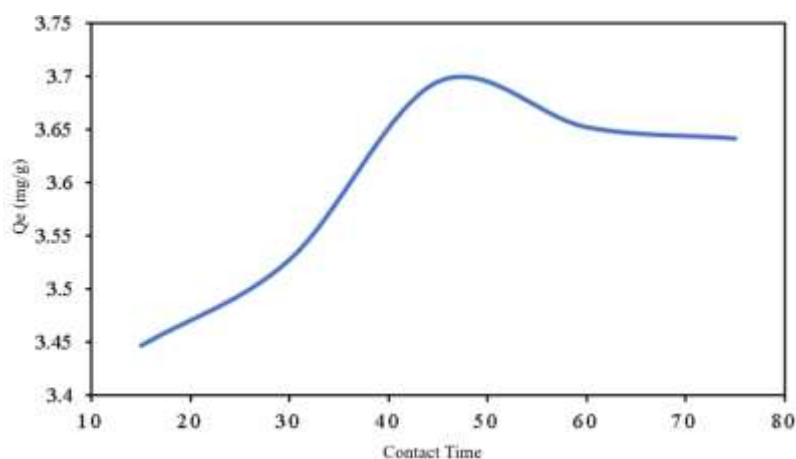


Figure 6. Effect of contact time on the adsorption capacity of methylene blue.

The initial increase in adsorption capacity is consistent with progressive occupation of accessible adsorption sites. As contact proceeds, the net adsorption rate is expected to decrease as the system approaches equilibrium. The slight decrease in the measured capacity after 45 min may reflect desorption, redistribution, or normal experimental variability; without documented replicate measurements, a specific mechanism cannot be established from these data alone [22].

3.7. Effect of Initial Methylene Blue Concentration

The effect of initial MB concentration is summarized in Table 4. As the initial concentration increased from 20 to 100 mg/L, the adsorption capacity increased from 3.7017 to 18.6531 mg/g. The concentration of MB removed from solution likewise increased from 18.6603 to 94.1795 mg/L. This trend indicates that a larger concentration gradient increases the frequency of interactions between MB molecules and adsorption sites [21,23].

Table 4. Effect of initial methylene blue concentration on adsorption.

Time (min)	Adsorbent mass (g)	C0 (mg/L)	Absorbance	Ce (mg/L)	Qe (mg/g)	Adsorbed MB (mg/L)
45	0.5041	20	0.0357	1.3397	3.7017	18.6603
45	0.5046	40	0.0948	2.6026	7.4113	37.3974
45	0.5066	60	0.1797	4.4167	10.9718	55.5833
45	0.5042	80	0.1857	4.5449	14.9653	75.4551
45	0.5049	100	0.2454	5.8205	18.6531	94.1795

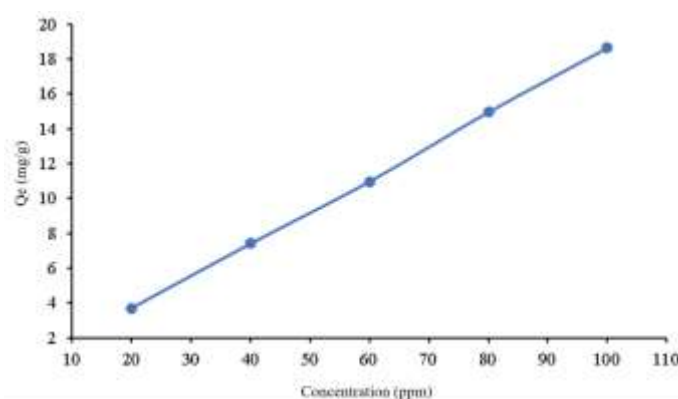


Figure 7. Effect of initial methylene blue concentration on adsorption capacity.

3.8. Adsorption isotherms

Adsorption isotherms describe the relationship between the amount adsorbed and the equilibrium concentration. The source study evaluated the Freundlich model, which is commonly used for heterogeneous adsorption, and the Langmuir model, which represents adsorption at equivalent sites with monolayer coverage in Tabel 5 [24].

3.8.1. Freundlich isotherm

Table 5. Freundlich isotherm data.

C0 (mg/L)	log Ce	log Qe
20	0.1270	0.5684
40	0.4154	0.8699
60	0.6451	1.0403
80	0.6575	1.1751
100	0.7649	1.2707

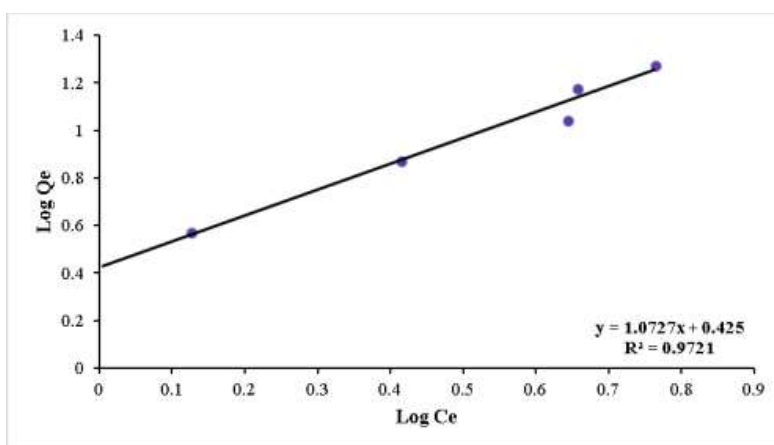


Figure 8. Freundlich plot of log Ce versus log Qe.

The Freundlich plot yielded $y = 1.0727x + 0.425$ with $R^2 = 0.9721$. Because $1/n = 1.0727$, n was calculated as 0.9322. In the interpretation adopted by the source study, $n < 1$ indicates a chemisorption contribution [25]. The intercept corresponded to $\log K_f = 0.425$, giving $K_f = 2.6607$ mg/g [26].

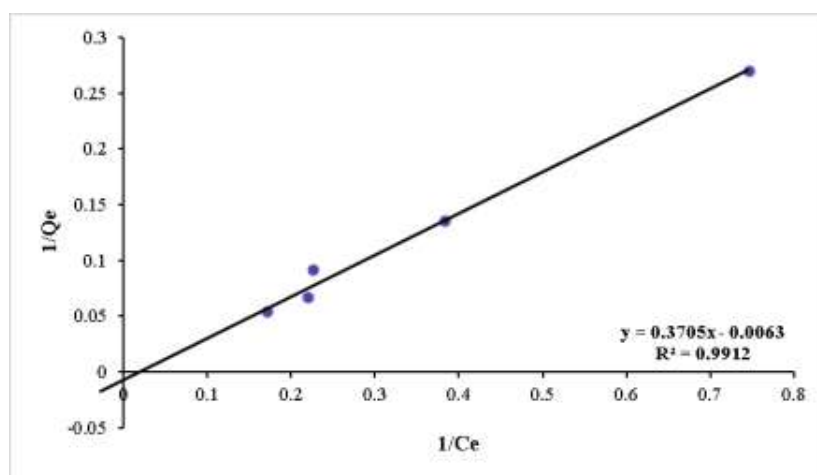
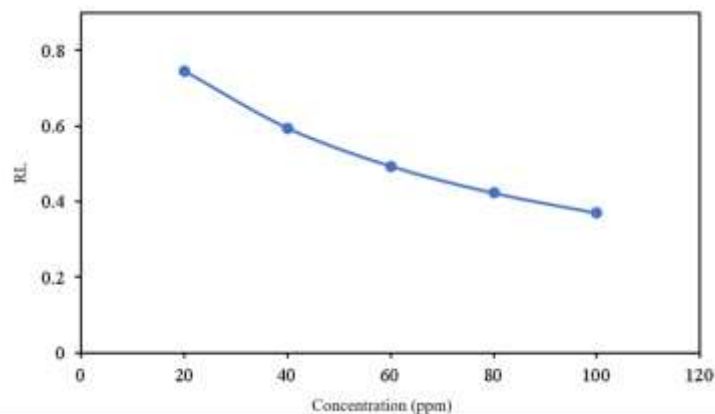
3.8.2. Langmuir Isotherm

The Langmuir plot gave $R^2 = 0.9912$. The calculated Langmuir constant K_L was 0.017004 L/mg and the model-derived maximum adsorption capacity Q_m was 158.7302 mg/g. The higher R^2 value relative to the Freundlich model indicates that the Langmuir equation described the experimental data more closely within the investigated concentration range. The Q_m value is a model-predicted parameter and should not be interpreted as an experimentally achieved capacity, because the highest measured Q_e in this study was 18.6531 mg/g at 100 mg/L initial MB concentration.

The Langmuir separation factor RL was 0.746, 0.595, 0.494, 0.423, and 0.370 for initial MB concentrations of 20, 40, 60, 80, and 100 mg/L, respectively (Figure 10). The decrease in RL with increasing initial concentration indicates increasingly favorable adsorption according to the interpretation used in the source manuscript [27]. Because all RL values were between 0 and 1, the adsorption was classified as favorable [24].

Table 6. Langmuir isotherm data.

C_0 (mg/L)	$1/C_e$	$1/Q_e$
20	0.7464	0.2701
40	0.3842	0.1349
60	0.2264	0.0911
80	0.2200	0.0668
100	0.1718	0.0536

Figure 9. Langmuir plot of $1/C_e$ versus $1/Q_e$.Figure 10. Langmuir separation factor (RL) as a function of initial methylene blue concentration.

3.8.3. Comparison of the Isotherm Models

Isotherm parameters reflect adsorbent capacity and surface characteristics (Table 7) [28]. Although both models produced relatively high R^2 values, the Langmuir fit ($R^2 = 0.9912$) was closer than the Freundlich fit ($R^2 = 0.9721$). Accordingly, the Langmuir equation provides the better empirical description of the present data within the tested concentration range. This comparison does not by itself prove an exclusively homogeneous monolayer adsorption mechanism. The calculated Q_{max} of 158.73 mg/g is a model-derived parameter rather than an experimentally observed capacity.

Table 7. Comparison of Langmuir and Freundlich isotherm parameters.

Sample	K_L (L/mg)	Q_{max} (mg/g)	Langmuir R^2	K_f (mg/g)	n	Freundlich R^2
CMC-GG-Al-BA	0.017004	158.7302	0.9912	2.6607	0.9322	0.9721

4. Conclusion

A CMC-GG adsorbent cross-linked with aluminum sulfate octadecahydrate and boric acid was prepared and evaluated for methylene blue adsorption. The highest measured capacity in the contact-time experiment was obtained at 45 min, with $Q_e = 3.69451$ mg/g for a 20 mg/L MB solution. For initial concentrations of 20, 40, 60, 80, and 100 mg/L, the adsorption capacities were 3.7017, 7.4113, 10.9718, 14.9653, and 18.6531 mg/g, respectively. The Langmuir model provided the closer fit ($R^2 = 0.9912$) than the Freundlich model ($R^2 = 0.9721$), with a model-calculated Q_{max} of 158.73 mg/g. Future work should evaluate the effects of solution pH and temperature, establish adsorption kinetics using replicated experiments, characterize the adsorbent before and after MB uptake, and assess regeneration and reuse.

5. Acknowledgements

None declared in the available manuscript record.

6. Conflict of Interest

The authors declare no conflict of interest.

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