

Methylene Blue Adsorption Using the Metal-Organic Framework of Calcium Alginate (CaA)

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Abstract

Methylene blue (MB) is one of the most commonly used dyes in the printing and textile industries. This research aimed to synthesize MOF CaA (Metal-Organic Framework Calcium Alginate) as an adsorbent for MB dye waste and investigate the effects of contact time and MB concentration on adsorption efficiency. MOF CaA was synthesized through ion exchange between sodium alginate (NaA) and calcium chloride (CaCl₂) solution to produce calcium alginate (CaA). The obtained CaA adsorbent was then calcined at 800 °C for two hours to produce MOF CaA. Characterization was carried out using FTIR and SEM-EDX. FTIR confirmed the presence of characteristic alginate functional groups. SEM-EDX showed C, O, Ca, Na, and Al in the CaA adsorbent. The results showed the highest MB reduction at 60 minutes of contact time, with MOF CaA concentrations of 0.02 g/L and MB concentrations of 0.005 g/L. The adsorption efficiency was 36.3549%. The adsorption kinetics of MB on CaA followed the intraparticle diffusion model, which showed the highest correlation coefficient (R^2) of 0.9683 among the kinetic models evaluated. These findings demonstrate the potential of technical-grade CaA as an economical adsorbent for methylene blue removal from aqueous solutions.

Keywords *Bio-MOF, Brown Algae, Degradation, Dye, Methylene Blue*

INTRODUCTION

Synthetic dyes, such as methylene blue (MB), are widely used in various industries and pose environmental risks due to their complex aromatic structures and carcinogenic and mutagenic properties (Alegbe & Uthman, 2024; Lellis et al., 2019). The textile industry is a major user of MB, with only approximately 5% of the dye binding to products while the remaining 95% is discharged as dye waste (Wafiroh et al., 2020). Exposure to MB can cause adverse effects on aquatic organisms, with chronic effects reported at concentrations above 4.7 µg/L (Li et al., 2023). For this reason, it is important to treat wastewater containing MB before discharging it into the environment.

Adsorption is an efficient method for removing dyes from wastewater due to its simplicity and ease of operation. Adsorption employs an adsorbent, a solid with pores, to remove components from gas or liquid fluids (Shabir et al., 2022). The adsorbent should be effective, environmentally friendly, and cost-effective to manufacture. In this study, the bio-adsorbent was selected from residual alginate production waste derived from brown algae, particularly *Sargassum* sp., which remains underutilized (Monagail et al., 2017).

Alginate, a polysaccharide found in brown algae, is composed of mannuronic and guluronic

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acids. It is extracted from algae such as *Sargassum* sp. and is crucial for algal tissue structure. Based on previous research ([Mahreni et al., 2022](#)), the synthesis of MOF CaA (Metal-Organic Framework Calcium Alginate) to adsorb Malachite green dye using pure-grade precipitating solutions (pro-analysis) required high costs. Thus, this study used a technical-grade precipitating solution to produce calcium alginate adsorbents, which cost much less, and it was expected that the resulting adsorbents would be more affordable and economical for use. This research aimed to investigate the effects of contact time and MB concentration on adsorption efficiency and to model the adsorption kinetics using technical-grade calcium alginate.

LITERATURE REVIEW

Metal-Organic Framework (MOF)

Metal-Organic Framework (MOF) is a porous crystalline material consisting of metal centers or clusters linked by ionic or metal-organic bonds, typically formed with N-containing carboxylic acids and metals such as zinc, copper, chromium, aluminum, zirconium, and others. MOFs are widely used in gas adsorption and storage processes, separation processes, catalysis, etc., due to their large surface area, flexible structure, and adjustable pore size ([Soni et al., 2018](#)). MOFs have been used as adsorbents, catalysts, and separation materials because of their high surface area, porosity, and tunable structure ([Ihsanullah, 2022](#); [van Velthoven et al., 2019](#)).

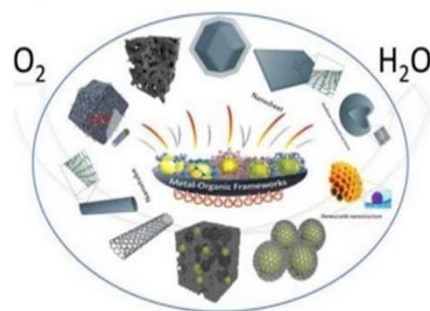


Figure 1. Metal-Organic Framework (MOF) Structure ([Shah et al., 2020](#))

Synthesis of MOF CaA

Alginate can undergo ionic gelation in the presence of divalent cations. Ca^{2+} is commonly used as a crosslinking ion and interacts with G-block regions through the egg-box mechanism, forming a three-dimensional network ([Sutirman et al., 2021](#)). In alginate-based materials, metal cations can be trapped within the alginate matrix to form a stable structure ([Mahreni & Ristianingsih, 2020](#)).

RESEARCH METHOD

Material and Method

Material

The materials used consisted of brown algae (*Sargassum* sp.) as a source of alginate. Sodium carbonate (Na_2CO_3) 2% by weight as a solvent. Technical calcium chloride (96% CaCl_2) was used as the precipitating solution.

Experimental Equipment

(1) Condenser, (2) 500-mL three-neck flask, (3) electric heater, (4) thermometer, (5) stirrer motor. Atmospheric ovens and furnaces serve as auxiliary equipment for drying and crystal formation.

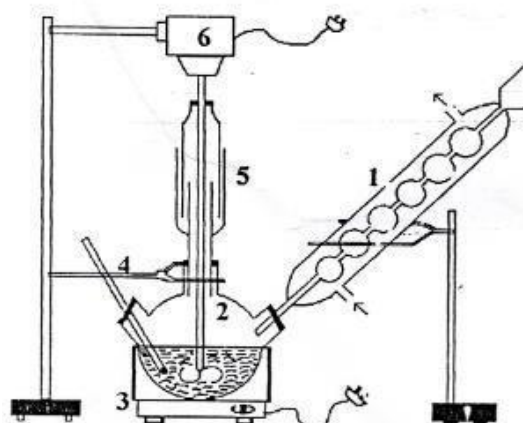


Figure 2. Set of Experimental Apparatus

Method

Extraction of Alginate from Brown algae

Alginate was created by extracting alginate from brown algae using sodium carbonate as a solvent. The brown algae were washed thoroughly and then drained until no water dripped. Next, aquadest was added at a weight ratio of brown algae to aquadest of 1:6 (w/w), and the mixture was left for two hours and then blended until it was shaped like mush. The brown algae slurry was mixed with a 2 wt% Na_2CO_3 solution at a ratio of 1:20 (w/v), then extracted at 70°C for two hours. After cooling, the mixture was filtered using filter paper. The resulting filtrate was a sodium alginate (NaA) solution, which was used as the raw material for synthesizing MOF CaA.

Synthesis of MOF CaA

At this stage, ion exchange between the extracted NaA and CaCl_2 was carried out. The NaA solution was dripped into 1 M CaCl_2 solution to form a precipitate. Dropping was stopped when no more precipitate formed. Next, it was allowed to stand for 24 hours in a closed glass beaker. The precipitate was then separated using filter paper, and the filtrate was disposed of as waste. The precipitate was dried in an oven at 104°C to a constant weight. The dry precipitate was calcined using a furnace at 800°C to produce MOF CaA.

Determining the MB maximum wavelength

The maximum absorbance wavelength of MB was determined using UV-Vis spectroscopy over 500–700 nm, based on the reported wavelength of 663 nm ([Khan et al., 2022](#)).

Determination of the Calibration Curve

A calibration curve was prepared using MB solutions of various concentrations at 663.4 nm to establish the relationship between MB concentration and absorbance

Characterization of C-A adsorbent

FTIR Characterization

In this study, the adsorbent activity was tested by the adsorption process using MB dye. The standard FTIR characterization can occur at a $4000\text{--}400\text{ cm}^{-1}$ wavelength.

SEM-EDX

SEM-EDX analysis was performed to characterize the surface morphology and elemental

composition of the CaA adsorbent.

Experiment to Determine the Adsorption Equilibrium Time

Prepare an MB solution with a concentration of 0.005 g/L by weighing 0.005 g of dye powder and dissolving it in distilled water in a 1 L volumetric flask. Next, weigh the adsorbent as much as 0.002 g, put it into 100 mL of the dye solution that had been made, and then determine the optimum contact time with a stirring speed of 600 rpm for one minute. Then repeat the experiment with different contact times: 2, 3, 4, 5, 7, 10, 12, 15, 17, 20, 25, 30, 35, 40, 45, 50, 55, 60, 75, and 90 minutes.

Experiment to determine the optimum MB concentration

Prepare an MB solution at 0.001 g/L by weighing 0.001 g of MB powder and dissolving it in distilled water in a 1000 mL volumetric flask. Then weigh the adsorbent as much as 0.002 g and put it into 100 mL of the dye solution that has been made. Determine the optimum MB concentration by stirring at 600 rpm for 60 minutes (optimum contact time), then repeat the experiment with different adsorbent concentrations, namely 0.002, 0.003, 0.004, 0.005, and 0.006 g/L.

FINDINGS AND DISCUSSION

Analyzing the morphological surface of CaA adsorbent

The adsorbent synthesized by the precipitation method was then characterized using SEM-EDX. This characterization was carried out to obtain morphological data of the surface and the composition of the MOF C-A adsorbent.

Analyzing the functional groups of the CaA adsorbent

The adsorbent synthesized by the ion exchange method was then characterized using FTIR. This characterization was carried out to obtain functional group data contained in CaA.

Analyzing the absorbed percent of adsorbent

The percent adsorption analysis was carried out using a spectrophotometer. The wavelength of the MB solution was measured, and the solution absorbance was obtained. The solution concentration of the dye was then calculated, then it was used to calculate the percentage absorbed by the dye, using the following formula:

$$\% \text{ MB absorbed} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (1)$$

The following formula was used to calculate the adsorption capacity of an adsorbent

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (2)$$

Where C_0 is the initial concentration of MB (mg/L), C_e is the concentration of the solution at equilibrium (mg/L), q_e is the adsorption capacity of MB on the surface of MOF Ca-alginate adsorbent, m is the mass of MOF CaA adsorbent (g), and V is the volume of the solution during the process.

Result and Discussion

Maximum Wavelength for MB

MB in solution can be analyzed using a UV-VIS Spectrophotometer at 664 nm ([Shahinyan](#)

et al., 2019). The blue color in MB is a complementary compound that can be seen in the visible area with a light spectrum at a wavelength of 500-700 nm. Therefore, the wavelength of MB falls within this range (Khan et al., 2022). The maximum wavelength was determined in the 500-700nm range using a UV-Vis spectrophotometer, as shown in Figure 3.

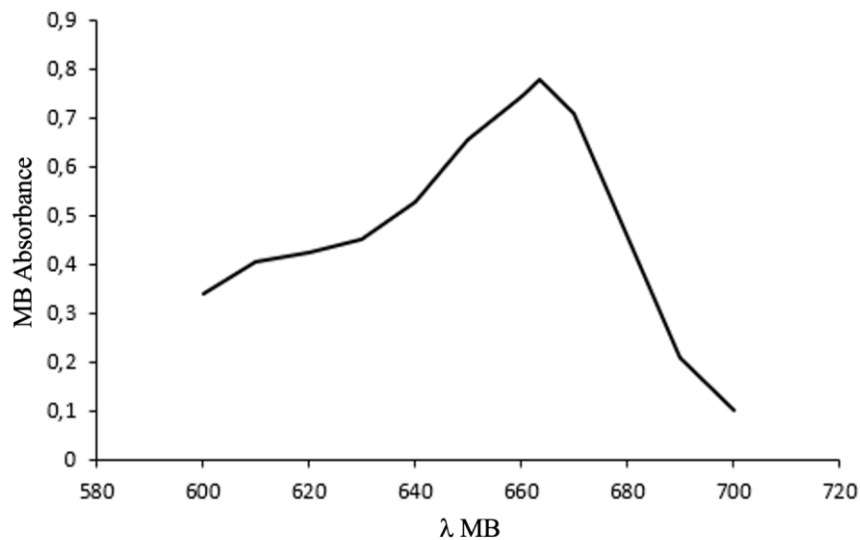


Figure 3. Relationship Between Wavelength and MB Absorbance

Calibration Curve

The calibration curve was based on measuring MB adsorption at concentrations of 0.001, 0.002, 0.004, 0.007, and 0.01 g/L. The adsorption values from the measurements were used to create a calibration curve, as shown in Figure 4, where the x-axis is the concentration and the y-axis is the adsorption. The curve has a regression value of $R^2 = 0.9983$ and a linear equation of $y = 131.66x - 0.0055$. The linear equation is used to calculate the optimum contact time.

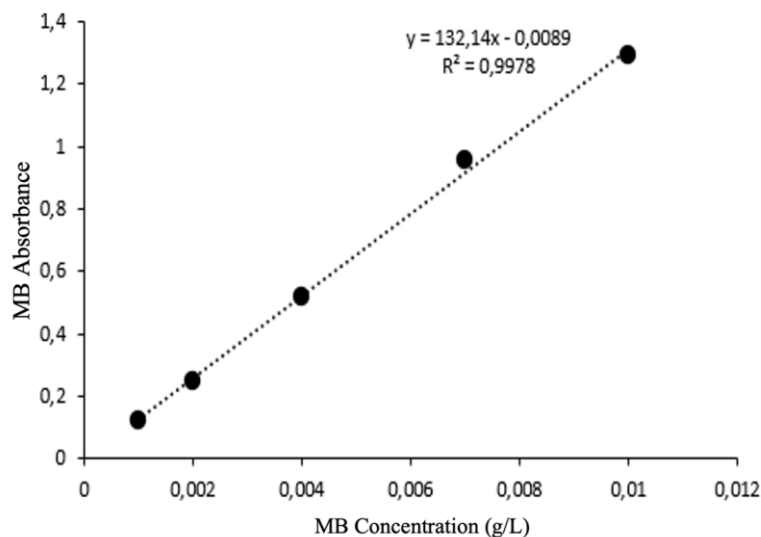


Figure 4. Relationship Between MB Concentration and Absorbance

Adsorbent Analysis

1. FTIR Analysis

FTIR analysis confirmed the presence of calcium alginate in the powder over the 4000–400 cm^{-1} range. The analysis results showed absorption peaks at specific wavenumbers. Typical absorption found in the spectra, presented in Figure 5, shows the hydroxyl group (-OH) at wave number 3639.26 cm^{-1} , the carbonyl group (C=O) at wave number 1641.54 cm^{-1} , the carboxyl group (COO-) at a wave number of 1409.71 cm^{-1} , CO-C bond at wave number of 1111.46 cm^{-1} , alkane group (C-H) at a wave number of 711.95 cm^{-1} , and a characteristic mannuronate fingerprint region at a wave number of 873.29 cm^{-1} . The alginate functional group consists of hydroxyl (OH), carbonyl (C=O), and carboxyl (C-O) groups. Based on the infrared absorption peaks presented in Figure 5, the resulting compound of the analysis is calcium alginate.

The functional group analysis results in this study are consistent with previous studies (Papageorgiou et al., 2010; Pathak et al., 2008; Pathak et al., 2010a; Sai'Aan et al., 2016). The stretching of the hydroxyl group (-OH) was observed at 3,448.87 cm^{-1} , the carbonyl group (C=O) at 1,605.81 cm^{-1} , the -C-OH bond at 1,418.71 cm^{-1} , and the C-O-C and -COOH bonds at 1,026.17 cm^{-1} .

Prior studies (Papageorgiou et al., 2010; Pathak et al., 2008; Pathak et al., 2010a) show that NA has three specific peaks: the hydroxyl bond, asymmetric COO-, and symmetric COO-. There is a hydroxyl function (-OH) at a wavelength of 3000 – 3600 cm^{-1} (Sai'Aan et al., 2016) with an absorption peak at 3425.54 cm^{-1} . A carboxyl group was observed at wavenumbers of 1789.00 cm^{-1} for the asymmetric COO group and 1419.22 cm^{-1} for the symmetric COO group. Absorption peaks at 873.65 cm^{-1} and 711.37 cm^{-1} indicated the presence of mannuronic and guluronic fingerprint groups (Aoyagi et al., 2025; Sai'Aan et al., 2016).

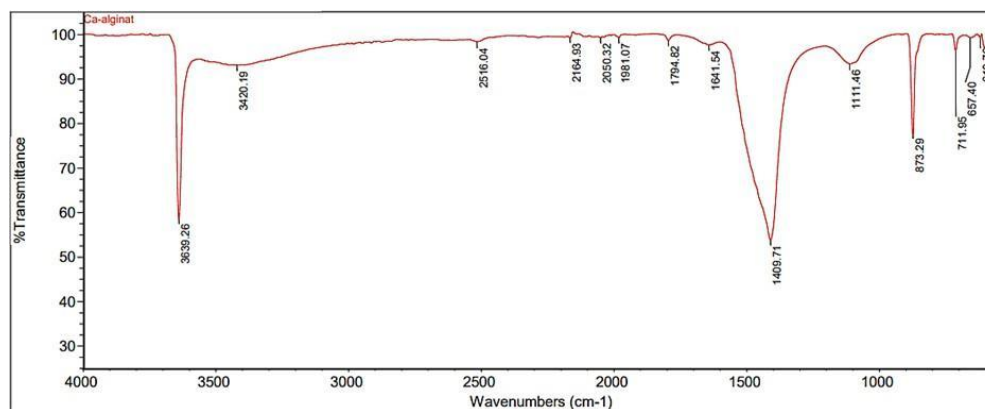


Figure 5. Transmission Pattern of FTIR of C-A Adsorbent Analysis

2. SEM-EDX Analysis

Figure 6 shows the results of the SEM-EDX scan of C-A before MB adsorption. It provides an overview of C-A surface morphology. The surface morphology of C-A appears to have pores with different shapes and sizes. The SEM-EDX also shows cavities on C-A, which directly indicates the high surface area of the adsorbent. Tests were carried out on the C-A adsorbent with 2500×, 5000×, 7500×, and 10000× magnifications.

SEM image in Figure 6(a), obtained at a magnification of 2500 $\times$, showed the surface of the C-A adsorbent with an irregular structure, which was in line with previous studies by [Abdin et al. \(2021\)](#), [Báez et al. \(2017\)](#), [He et al. \(2016\)](#), [Kashima et al. \(2021\)](#), and [Pathak et al. \(2010b\)](#). C-A has a porous structure (many cavities), characterized by an open structure of imperfect grains with a less homogeneous particle distribution. The figure also shows that the monomer, cross-linker, and initiator that did not react during the polymerization process have been successfully removed from the adsorbent material.

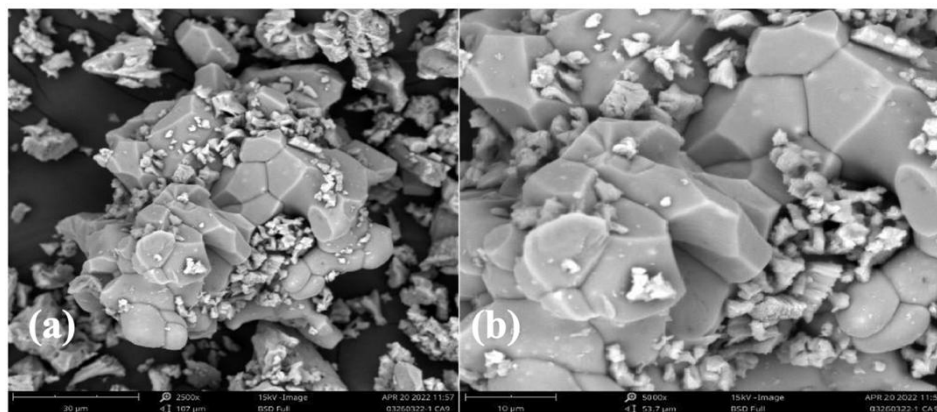


Figure 6. SEM C-A results after calcination: (a) 2500 $\times$ magnification; (b) 5000 $\times$ magnification

The EDX analysis revealed that the primary elements in C-A are O, Ca, C, Na, and Al. Table 1 presents the elemental composition of the analyzed C-A adsorbent.

Table 1. Chemical composition of C-A after calcination

Elements	Mass Percentage (%)
O	61.11
Ca	19.97
C	18.68
Na	0.15
Al	0.09

Table 1 shows that the largest composition in C-A is oxygen (O), at 61.11%. Based on the element concentrations, C and O in C-A likely come from the functional group Na_2CO_3 , where Na_2CO_3 contains oxygen functional groups such as hydroxyl and carbonyl in the carbon structure. This functional group forms during polymerization due to the interaction of surface free radicals with oxygen or nitrogen from the atmosphere. It makes the surface of the adsorbent chemically reactive and can affect its adsorption properties.

Determining the Optimum Contact Time

Contact time plays an essential role in determining an adsorbent's ability to adsorb the adsorbate. Figure 7 shows the effect of contact time on the adsorption efficiency of MB.

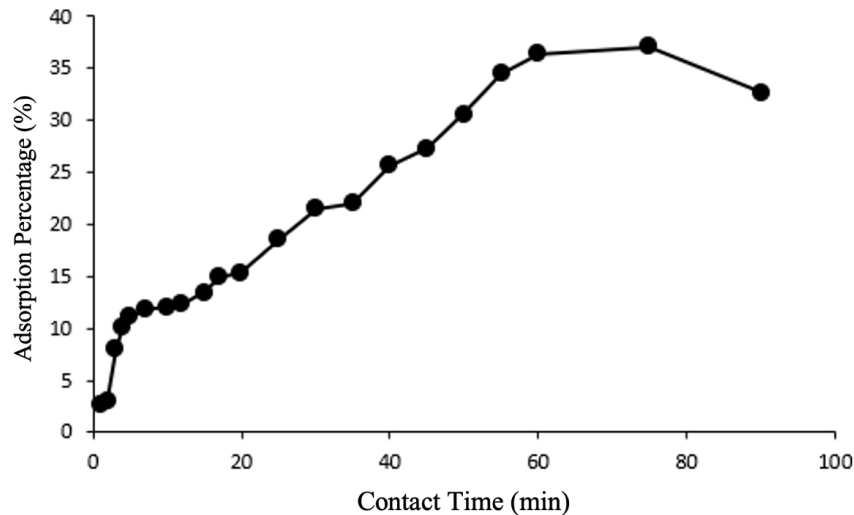


Figure 7. The correlation between contact time in the MB adsorption process and the MOF C-A adsorbent

Figure 7 shows an increase in adsorption efficiency because the adsorbent can still adsorb the adsorbate in solution. The adsorption process had not reached its optimum point because the adsorbent's active sites were not yet saturated, allowing adsorption to continue increasing (Al-Othman et al., 2013; Eyupoglu et al., 2025; Puchongkawarin et al., 2021). The optimum contact time obtained in this study was 60 minutes, with an adsorption efficiency of 36.3549%. This means that at 60 minutes, the adsorbent and adsorbate reach a balance, and all negatively charged active sites are covered by MB, which is positively charged (Al-Othman et al., 2013; Bulut & Aydin, 2006; Turp et al., 2020).

After reaching the optimum point, the adsorption efficiency decreased at 90 minutes due to desorption as the adsorbent approached saturation and equilibrium (Al-Othman et al., 2013). Prolonged contact may promote the gradual release of adsorbed MB into the solution, resulting in decreased adsorption capacity.

Determining the Optimum MB Concentration

This study conducted a concentration variation test to determine the concentration required to achieve optimum adsorption of MB dye. MB adsorption results with various concentrations are shown in Figure 8.

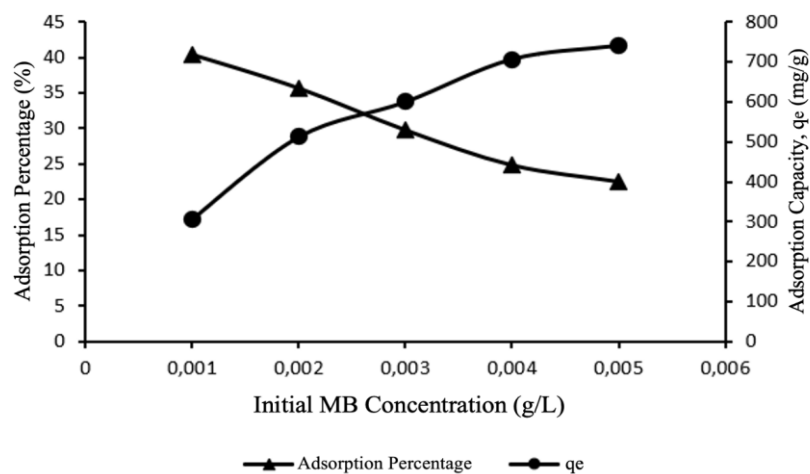


Figure 8. Effect of initial MB concentration on the adsorption capacity of MOF C-A

Figure 8 shows that the adsorption capacity of MB using MOF C-A increases with increasing initial MB concentration. The optimal adsorption capacity of the adsorbent was obtained at an initial MB concentration of 0.005 g/L, which was 740.5438 mg/g. It is assumed that the increase in the adsorption capacity of MB dyes using an adsorbent is due to the higher concentration of MB, which results in an increasing amount of MB in the solution (Al-Othman et al., 2013; Huang et al., 2011; Jia et al., 2017). The amount of MB adsorbed is proportional to the active groups in the adsorbent. If the adsorbent's active groups are not saturated, MB adsorption capacity will be higher (Al-Othman et al., 2013; Huang et al., 2011). If the active groups of the adsorbent have been saturated, the increase in the active groups of the adsorbate will decrease. However, if the adsorbent is not saturated by the adsorbate when the groups are active, increasing adsorbate concentration increases the amount of adsorbate adsorbed (Atun et al., 2003; Huang et al., 2011).

Adsorption Kinetics

The parameter used to determine the adsorption kinetics of MB was the variation of contact time or adsorption time. Adsorption kinetics data were obtained empirically using pseudo-first-order and pseudo-second-order models, Elovich's equation, and the intraparticle diffusion equation. Table 2 presents the MB adsorption kinetics results.

Table 2. Data of MB adsorption kinetics using MOF C-A

Kinetic Models	Parameters
PFO	$R^2 = 0.7963$ $K1 = -0.0443 \text{ second}^{-1}$ $q_e = 1469.6947 \text{ mg}\cdot\text{g}^{-1}$
PSO Type I	$R^2 = 0.8375$ $K1 = 2.5531 \cdot 10^{-5} \text{ second}^{-1}$ $q_e = 1666.6667 \text{ mg}\cdot\text{g}^{-1}$
PSO Type II	$R^2 = 0.5437$ $K1 = 3.5994 \cdot 10^{-5}$ $q_e = 1553.0232 \text{ mg}\cdot\text{g}^{-1}$
PSO Type III	$R^2 = 0.5437$ $K1 = 8.8758 \cdot 10^{-5}$ $q_e = 1158.9000 \text{ mg}\cdot\text{g}^{-1}$
PSO Type IV	$R^2 = 0.8999$ $K1 = 8.3500 \cdot 10^{-5}$ $q_e = 1111.1111 \text{ mg}\cdot\text{g}^{-1}$
Elovich	$R^2 = 0.8509$ $\alpha = 3.3583 \times 10^{-3}$ $\beta = 220.5497$
Intraparticle Diffusion	$R^2 = 0.9683$ $Kt = 39.6840$

Based on the calculations and the regression equation, the adsorption kinetics model for each variation was determined by the highest R^2 value among the evaluated models (Acemioğlu, 2005; Kumar, 2006; Ton-That et al., 2023; Xiao & He, 2013). Table 2 shows that MB adsorption using the C-A adsorbent followed the intraparticle diffusion kinetic model, with an R^2 value of 0.9683.

The intraparticle diffusion model can explain the adsorption mechanism. The adsorption process consists of several stages, including the movement of particles from outside the adsorbent

to the adsorbent surface, followed by the diffusion of molecules into the pores. Intraparticle diffusion is usually a slow, rate-determining step in the adsorption process (Chaurasia, 2007). This indicates that the adsorption process occurs in two stages: macropore and micropore diffusion. In the macropore diffusion stage, MB molecules are adsorbed on the surface of the adsorbent. Meanwhile, during the micropore diffusion stage, MB molecules are adsorbed on the inner surface of the adsorbent material (Acemioğlu et al., 2014; Doğan et al., 2009).

CONCLUSIONS

MOF-based CaA was successfully synthesized using the ion-exchange method and characterized by SEM-EDX and FTIR. The optimum adsorption condition was obtained at a contact time of 60 minutes, with an adsorption efficiency of 36.3549%. The greatest adsorption capacity of the adsorbent was obtained at an initial MB concentration of 0.005 g/L, which was 740.5438 mg/g. The adsorption kinetics followed the intraparticle diffusion model, with the highest R^2 of 0.9683 among the models evaluated.

LIMITATIONS & FURTHER RESEARCH

The study was limited to the effects of contact time and initial MB concentration under controlled laboratory conditions using an aqueous MB solution. The adsorption performance was not evaluated using actual wastewater, and the reusability and regeneration of the CaA adsorbent were not investigated. Further research should investigate the reusability and regeneration of the CaA adsorbent, as well as its adsorption performance under different wastewater conditions and in repeated adsorption cycles, to assess its practical applicability.

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