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Adsorption of Congo Red Dye From Aqueous Solution Using *Mesembryanthemum Crystallinum*-Based Bioadsorbent

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ABSTRACT: Adsorption technique is a simple and inexpensive separation process compared to other separation methods, which have significant contributions in water treatment and pollutant removal. Batch adsorption processes are good for studies on a small scale, whereas continuous adsorption processes are preferred for processing a large scale, especially in industrial applications. In this context, a fixed bed adsorption column was fabricated and the leaves of *Mesembryanthemum crystallinum* biomass (MC-BM) was used as bioadsorbent for water wastewater treatment applications. Congo red dye (CRD) was used as a pollutant model in the wastewater. The effects of different operating conditions were verified on the CRD adsorption efficiency, which includes the bed depth, the initial concentration of the dye, pH, flow rate, and operating temperature. The results showed that the derived bioadsorbent material showed a high potential for adsorption of CRD from aqueous solution. The maximum adsorption efficiency of 85.83 % was achieved. The results also indicated that the increase in the bed depth and the operating temperature enhance the adsorption efficiency. However, the increase in the flow rate and initial concentration of the CRD decrease the CRD adsorption efficiency. The investigations showed that the adsorption of CRD onto MC-BM was endothermic process.
Keywords: **Biomass; Wastewater treatment; Adsorption; Fixed bed column.**

الملخص: عمليات الامتزاز من عمليات الفصل البسيطة الغير مكلفة مقارنة بطرق الفصل الاخرى الأمر الذي جعلها ذات أهمية خصوصا في معالجة المياه وإزالة الملوثات. تعتبر عمليات الامتزاز الدفعية جيدة عند إجراء الدراسات أو في المعامل أو عند العمل على نطاق صغير ولكن في التطبيقات الصناعية وعند العمل على نطاق واسع يتم استخدام عمليات الامتزاز المستمرة. في هذا البحث، تم صناعة مادة مازة من أوراق النبات الثلجي (نبات الغسول) وذلك لاستخدامه في امتزاز الملوثات العضوية من المحاليل المائية باستخدام عمود امتزاز ذو طبقة ثابته. وقد استخدمت صبغة الكونغو الحمراء كنموذج لهذه الملوثات في الماء. وتم التحقق من تأثير الظروف التشغيلية المختلفة على كفاءة الامتزاز وهي تتضمن عمق الطبقة المازة، التركيز الابتدائي للصبغة، درجة الحموضة، معدل التدفق، ودرجة الحرارة .أوضحت النتائج أن المادة المازة المستخدمة أظهرت إمكانية عالية في إزالة الملوثات حيث تم تحقيق نسبة إزالة وصلت لحوالي (85.83%) كما أشارت النتائج إلى أن زيادة عمق الطبقة المازة ودرجة الحرارة تعزز من كفاءة الامتزاز كما أن تقليل معدل التدفق والتركيز الابتدائي للصبغة تعزز من كفاءة الامتزاز أيضا. أظهرت دراسة تأثير درجة الحرارة أن امتزاز صبغة الكونغو الحمراء على المادة المازة المستخدمة عملية ماصة للحرارة.

الكلمات المفتاحية: المخلفات النباتية، معالجة المياه، الأمتزاز، عمود الفصل الثابت.

I. INTRODUCTION

Nowadays, water environmental pollution is the most critical issue that have been received tremendous attention worldwide; therefore, various techniques have been developed for removal of pollutants from water, including distillation, adsorption, extraction, coagulation, filtration, ion exchange, advanced oxidation, electrochemical, and membrane process (Ali and Gupta, 2006; Santhosh, Velmurugan, Jacob, Jeong, Grace, and Bhatnagar, 2016). Among of them, adsorption is the most commonly applied method for removal or reducing concentrations of non-degradable compounds from groundwater and wastewaters (Ahmad, Mujtaba, Zubair, Shah, Daud, Mu'azu, and Al-Harthi, 2025; Ali and Gupta, 2006; El Meziani, Agnaou, El Haddaj, Boumya, Barka, and Elhalil, 2025; Santhosh, Velmurugan, Jacob, Jeong, Grace, and Bhatnagar, 2016). Adsorption technique is physicochemical interactions of adsorbate molecules with active sites on the surface of the adsorbent, which possesses many advantages such as simple and easy process, cost-effective with high efficiency, fast kinetics, environmentally benign, and possibility for regeneration as well as recycling for several times (Ali and Gupta, 2006; Duran, Ozdes, Gundogdu, and Senturk, 2011; Kumar, Reddy, Parashar, and Ngila, 2017). Additionally, it is working with wide range of operating conditions, and appropriate technique for various application. Moreover, it is a capable technique for removal of soluble, insoluble, and biological pollutants from wastewaters. Furthermore, the adsorption technique can be carried out using batch and column systems in laboratory scale. However, continuous processes using large columns and contractors that filled with suitable adsorbent materials for removal pollutants from water in industrial operations (Ali and Gupta, 2006; Patel, 2022).

In modern research work, development of eco-friendly sorbents from eco-friendly materials has been received tremendous attention (Hlihor and Gavrilescu, 2009; Salleh, Mahmoud, Abdul Karim, and Idris, 2011). Therefore, abundant residual of natural materials, including coconut shells, sunflower seed hull, maize cob waste, almond shells, wood sawdust, and olive stone have been used for fabrication of biomass-based sorbents for water purification. However, the development of eco-friendly and inexpensive sorbents from available natural materials is still in challenge and under investigation (Hlihor and Gavrilescu, 2009).

Dyes are essential to many industries, which can cause various health issues, including kidneys, damage to vital organs like the heart, liver, spleen, lungs, and eyes, and more (Ahmad, Mujtaba, Zubair, Shah, Daud, Mu'azu, and Al-Harthi, 2025). However, dye wastewater is one of the most difficult industrial wastewaters to treat (Srinivasan and Viraraghavan, 2010). Among dyes, Congo red dye (CRD), which is an anionic organic toxic dye that belongs to a group of azo dyes were derived from benzidine (Mall, Srivastava, Agarwal, and Mishra, 2005; Salleh, Mahmoud, Abdul Karim, and Idris, 2011; Zhao, Shang, Xiao, Dou, and Han, 2014). CRD has several applications such as textile, printing, and dyeing industries. In addition, it widely used in histology to stain tissues for microscopic examination and to serve as an acid-base indicator, which turns blue when exposed to acidic medium and changes to red in the presence of alkali. However, CRD is among of undegradable dyes, which is more resistant to light and to washing. Also, it notoriously difficult to dispose of in an environmentally benign way (Salleh, Mahmoud, Abdul Karim, and Idris, 2011). Therefore, various derived sorbents have been tested for CRD removal from water streams (El Messaoudi, El Khomri, Dbik, Bentahar, Lacherai, and Bakiz, 2016; Mall, Srivastava, Agarwal, and Mishra, 2005; Zhao, Shang, Xiao, Dou, and Han, 2014).

Currently, agricultural-based sustainable materials have been considered as effective-cost sorbents (El Meziani, Agnaou, El Haddaj, Boumya, Barka, and Elhalil, 2025; Salleh, Mahmoud, Abdul Karim, and Idris, 2011). These sorbents showed excellent efficiencies because they contain polysaccharides and proteins, which have various functional groups, including carboxyl and hydroxyl surface groups that are the most attractive sites toward many pollutants (Ahmad, Mujtaba, Zubair, Shah, Daud, Mu'azu, and Al-Harthi, 2025). *Mesembryanthemum crystallinum* (*Cryophytum crystallinum*) is ice plant that belongs to the family Aizoaceae, which is a creeping plant with succulent leaves (See Fig. 1) (Abushaina, Sultan, Elhrari, and Alhwaige, 2018; Alhwaige, Al-Drogi, Yahiya, and Elhrari, 2025). Evan though, researches have explored fabrication of various promising renewable sorbents from biomass materials, challenges is still for development of promising sorbents from new natural materials with improve sorption efficiency and environmental concerns associated with synthesis procedures. Besides, a limited number of studies reported on using Mesembryanthemum crystallinum (MC-BM) based sorbents for wastewater treatment applications. Abushaina et al. (2018) reported fast kinetics of methylene blue dye removal suing a batch system. In addition, mixed matrix membrane derived from composed of polyethylene/MC-BM for methylene blue dye rejection from aqueous solution (Alhwaige, Al-Drogi, Yahiya, and Elhrari, 2025). Therefore, the present study introduces an innovative approach of using cost-effective MC-BM as an abundant local material in Libya that to be used in the first time for removal of CRD in fixed-bed system. The characteristics and adsorption parameters have also been studied in details.

Fig. 1 Optical image of *Mesembryanthemum crystallinum*.

II. EXPERIMENTAL SECTION

A) Materials

Leaves of the *Mesembryanthemum crystallinum* as biomass (MC-BM) were collected from their native biotope in Zliten, Libya. Congo red dye (CRD), HCl, and NaOH were purchased from Merck Chemical (analytical grade). The distillate water was obtained from desalination station, Zliten, Libya. All substances were used as received without any additional purifications.

B) Preparation of the Sorbent

An appropriate amount of *Mesembryanthemum crystallinum* biomass (MC-BM) was cleaned and dried. Fig. 2 illustrates the steps for preparation of the MC-BM adsorbent. Firstly, the leaves were separated from the stems. After that, the leaves were cleaned using distilled water to remove any available dust and other impurities, and then it was dried in an oven at 60 ºC for 24 h. The obtained dried leaves were ground into a fine powder using a mortar and pestle. The produced powder was washed using distilled water to eliminate any tiny powders to avoid a change in the color of the aqueous solutions during the adsorption experiments. Finally, the obtained powder of MC-BM was dried in a drying oven at120 °C for 2 h. The obtained powder of MC-BM was kept for use as bioadsorbent (see Fig. 2).

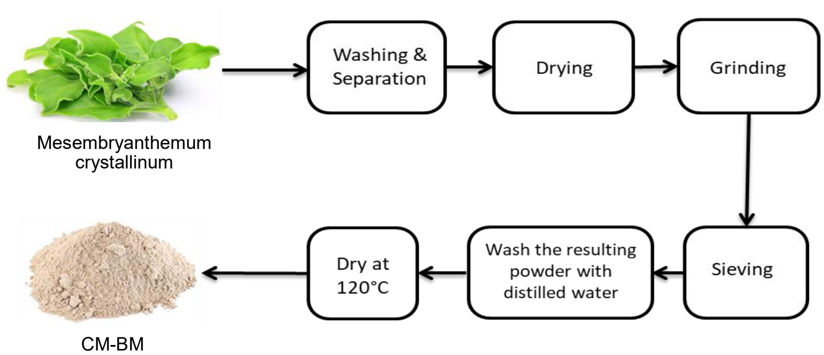


Fig. 2 Schematic diagram of MC-BM adsorbent preparation.

C) Characterizations

Fourier transform infrared (FT-IR) spectroscopy was used for investigation of the chemical structure of MC-BM sorbent. The PerkinElmer FT-IR Spectrometer, Model Frontier equipped with a one-bounce diamond-zinc selenide ATR accessory was used in this study. The FT-IR spectra were obtained using the KBr pellet method.

The apparent bulk density (ρ_b) of MC-BM sorbent was obtained from the membrane mass and its volume. A sample of MC-BM powder is placed in the density tester until fully filled. The mass of the filled simple of powder was measured. Therefore, the unknown density of the sorbent sample was obtained using Equation (1) (Alhwaige, Ishida, and Qutubuddin, 2020). All the experiments were repeated 3 times and the average value was considered to reduce the experimental deviation.

$$\rho_b = \frac{m}{V} \tag{1}$$

where: m is the membrane mass (g) and V is the membrane volume (cm).

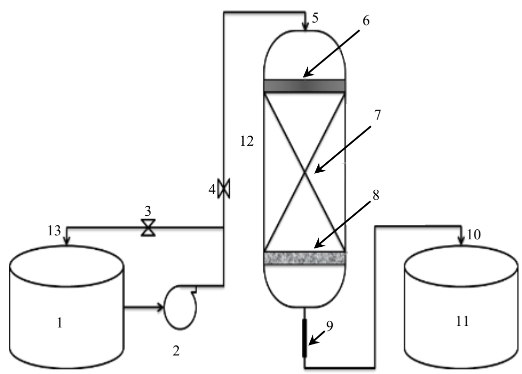
D) Preparation of Congo Red Dye Stock Solution

A concentrated solution of feed stock was prepared at a concentration of 1000 ppm, by addition of 1 g of Congo red dye (CRD) powder in 1 L of distilled water. The mixture was stirred using magnetic stirring for 1 h, and then the mixture was kept at room temperature for 48 h to ensure the equilibrium state of CRD distribution in the distilled water was achieved. Finally, the CRD stock solution was kept in dark medium for the use in adsorption experiments. Therefore, the concentration of the stock solution was diluted to needed concentrations for each adsorption experiments.

Calibration curve was used to obtain the unknown concentrations of CRD solutions after adsorption experiments. The unknown concentration of sample can be obtained from a plot of absorption intensity of the substance solution versus known concentrations. Therefore, various samples of CRD was prepared using known concentrations and the absorption intensity was measured using UV-vis spectrophotometer at a maximum wavelength (λ_{max} = 496 nm). A plot of the absorption intensity versus concentration was prepared.

E) Adsorption Experiment

Column experiments were performed in transparent a plastic column with an internal diameter of 2.5 cm and a length of 12.5 cm. A schematic diagram of the column experimental setup used for the continuous adsorption experiments is shown in Fig. 3. The column was packed with adsorbent between two supporting layers of cloth and glass beads (Patel, 2022). The experiments were carried out with CRD solution that was passed through the column bed at a certain concentrations in a downward direction. The exit stream of purified water was collected from bottom of the column at atmospheric pressure.



1: Feed solution tank; **2:** Electric pump; **3** and **4:** Valves; **5:** Feed zone; **6:** Distributor (glass beads); **7:** Adsorbent; **8:** Cloth; **9:** Sample area; **10:** Exit zone; **11:** Storage tank of the treated water; **12:** Adsorption column; **13:** Recycle line.

Fig. 3 The experimental setup of the adsorption system.

Fig. 3 illustrates the continuous process of CRD adsorption using an adsorption column. The CRD aqueous solution was fed at the top of the column and the flow direction operated from top to bottom. At the top of the column, a distributor was placed to ensure that the effluent flow was uniformly distributed as it enters the packed bed of the sorbents. To avoid exiting the sorbents in the fluent stream from the bottom of the column, a circular piece of cloth with a diameter of 2.8 cm (equivalent to the column inner diameter) was placed at the bottom of the column. To study the effect of the processing parameters, several adsorption experiments were carried out with different ranging values for each parameter, including CRD initial concentration (2 to 10 ppm), flow rate (0.3 to 30 mL min^{-1}), bed depth (0.25 to 1 cm), and pH value (2 to 10). For each experiment, during the processing of the adsorption system, various samples were taken from the exit stream at different intervals of time. The CRD concentrations in the feed (C_f) and the exit effluent solutions concentrations at different processing time (C_t) were measured using a UV–vis spectrophotometer and the previously prepared the calibration curve, while the absorbency of the CRD solution was analyzed at wavelength of 496 nm using JENWAY 7305 UV–Vis Spectrophotometer (Abushaina, Sultan, Elhrari, and Alhwaige, 2018). The residual concentration of CRD in outlet solution (C_t) at different time intervals was obtained in the form of the adsorption efficiency (R%) of CRD using Equation (2) (Ekrayem, Alhwaige, Elhrari, and Amer, 2021).

$$R\% = \left(\frac{C_f - C_t}{C_f}\right) \times 100 \quad (2)$$

where R% is the rejection efficiency of the CRD, C_f and C_t are the feed and permeate concentrations of the CRD in mg L^{-1}, respectively.

III. RESULTS AND DISCUSSION

A) Figures and Tables

Fig. 4 illustrates of the FTIR spectrum of the used MC-BM. The strong peak at 3426 cm^{-1} is ascribed to the stretching band of –OH Groups. The absorption bands located around 2935 and 2848 cm^{-1} are due to the stretching vibration of the methyl $–CH_3$ and $–CH_2$, respectively (Gabris, Periyasamy, and Tehrani-Bagha, 2025). The peaks at 1634 cm^{-1} is due to stretching vibration of the carbonyl groups functional groups (–C=O). Therefore, the MC-BM is rich with active sites for cationic water pollutants. Previously, the studies indicated that biomass sorbents rich with –OH functional groups, which are important active site for removal dyes from water (Abushaina, Sultan, Elhrari, and Alhwaige, 2018; Ahmad, Mujtaba, Zubair, Shah, Daud, Mu'azu, and Al-Harthi, 2025; Alhwaige, Al-Drogi, Yahiya, and Elhrari, 2025; El Messaoudi, El Khomri, Dbik, Bentahar, Lacherai, and Bakiz, 2016; Hua, Pana, and Hong, 2023; Ekrayem, Alhwaige, Elhrari, and Amer, 2021).

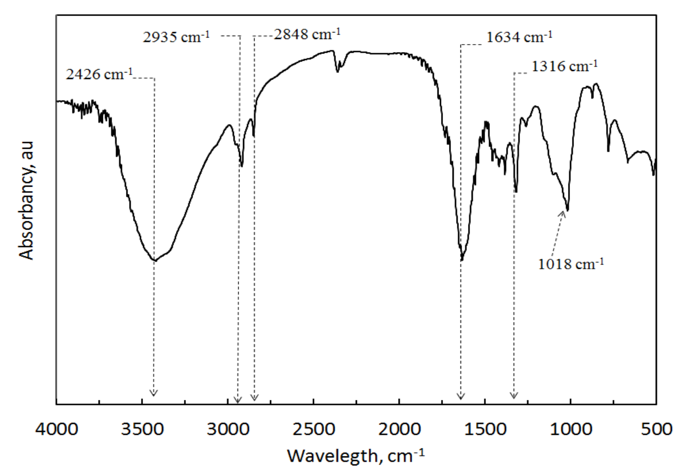


Fig. 4 FTIR of Mesembryanthemum Crystallinum (MC-BM).

B) Calibration Curve

UV spectrophotometer was used to identify the CRD concentrations by finding out the absorbance characteristic wavelength. To find the wavelength corresponding to maximum absorbance (λ_{max}), the absorbance of solution of the CRD with known concentration was measured at different wavelengths. The wavelength corresponding to maximum absorbance (λ_{max}) was obtained from the plot of absorbance versus wavelength (Fig. 5a). The results indicated that the λ_{max} for CRD was found to be 496 nm.

Fig. 5b represents the calibration curve of a plot of absorbance against concentration of the CRD solution. The previously prepared stock solution with a concentration of 1000 ppm was diluted different concentration of CRD (2, 5, 7, and 10 ppm). The UV-vis absorbency for each known concentration of CRD solution was measured at wavelength, λ_{max}, of 496 nm (Mall, Srivastava, Agarwal, and Mishra, 2005). Finally, the calibration curve was plotted of absorbance against concentration of the CRD solution.

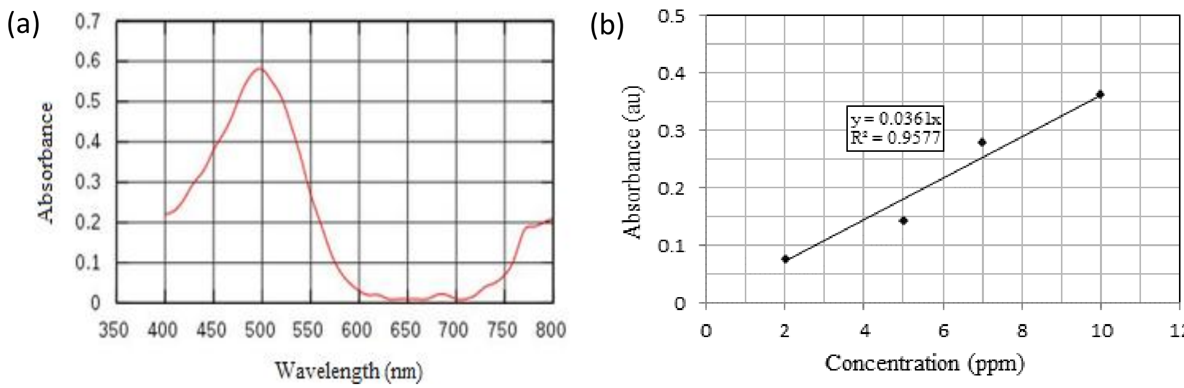


Fig. 5 Absorption of CRD: **(a)** Absorption spectrum for measurement of λ_{max}, **(b)** A plot of UV-vis absorbency versus the concentration of CRD.

C) Analysis of CRD Adsorption

To understand the adsorption behavior of CRD using the derived bioadsorbent from MC-BM leaf powder with density of 0.265 g cm-3, effect several processing parameters, including parameters include the change in bed depth, flow rate, initial pH, initial concentration and operating temperature on adsorption of CRD were investigated. The following are the results analysis of the adsorption experiments.

D) Effect of Bed Depth

From economic point of view, the appropriate amount of adsorbent is one of the important factors for practical adsorption processes in industry (Hua, Pana, and Hong, 2023). Therefore, the effect of the adsorbent amount on CRD adsorption was studied using various bed depth (0.25, 0.5 and 1 cm) of the adsorption column, while the other operating conditions were kept fixed. Various samples were taken at different time intervals and the CRD concentrations were obtained using UV-vis spectroscopy and the calibration curve (Fig. 5b). The adsorption efficiency was obtained using Equation (2) and the results are shown in Fig. 6a. The results indicated that the CRD adsorption efficiency increases with an increase in the bed depth for all samples. For example, after 20 minutes, the removal efficiency increased from 63.87 to 85.83 % with an increase in the bed depth from 0.25 to 1 cm. The reason for this increase in the CRD adsorption efficiency with the bed depth is due to the increase in the adsorbent amount, which provide more active adsorption sites on the surface (Mall, Srivastava, Agarwal, and Mishra, 2005).

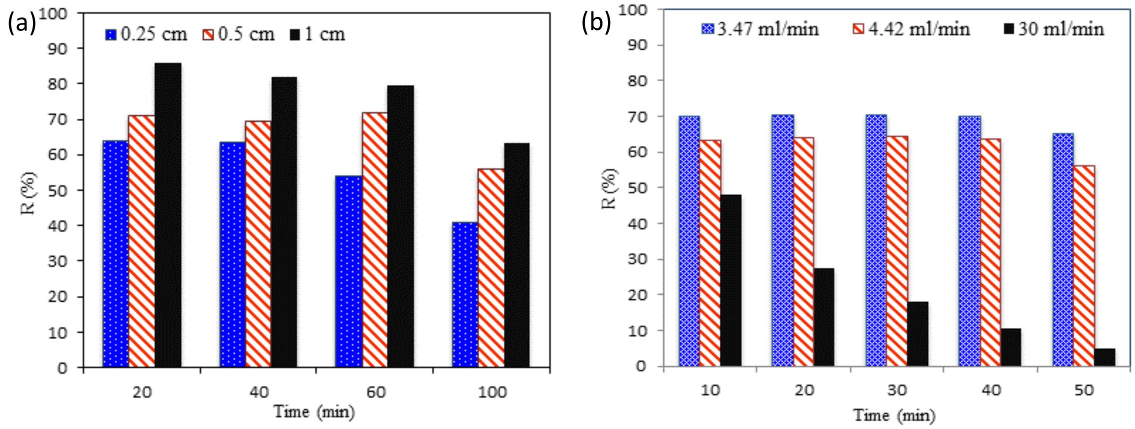


Fig. 6 Absorption of CRD: **(a)** Effect of bed depth on removal CRD **(b)** Effect of flow rate on removal CRD.

E) Effect of Flow Rate

The effect of flow rate on CRD adsorption was studied using a CRD solution at a concentration of 5 ppm using continuous adsorption column with various flow rates of (3.47, 4.42 and 30 mL min^{-1}) at a bed depth of 0.25 cm. Fig. 6b illustrates the effect of flow rate of the CRD solution on the adsorption efficiency. All samples showed similar adsorption behavior. The results indicated that the CRD removal efficiency decreases with an increase in the flow rate at fixed processing time. For instance, after 20 min, the CRD adsorption efficiency of 70.30% and 55.26% were achieved during operating the column with flow rates of 3.47 mL min^{-1} and 4.42 mL min^{-1}, respectively. In addition, low flow rate (3.47 and 4.42 mL min^{-1}) showed stable adsorption efficiency for first 40 min, and then a decline in the adoption efficiency was achieved; however, the high flow rate showed a reduction in adsorption efficiency from the beginning time. The decrease in CRD removal efficiency with an increase in the feed flow rate is suggested to the decrease in contact time of CRD molecules with the sorbent active sites. Furthermore, the decrease in the adsorption efficiency after certain operating time is ascribed to the saturation of the adsorbent with the CRD molecules (El Messaoudi, El Khomri, Dbik, Bentahar, Lacherai, and Bakiz, 2016; Zhao, Shang, Xiao, Dou, and Han, 2014).

F) Effect of Initial pH Value

The pH of initial feed aqueous solution is one of the important parameter on adsorption experiments (Ekrayem, Alhwaige, Elhrari and Amer, 2021). Therefore, the effect of pH value of initial feed CRD aqueous solution was evaluated using CRD with varying pH values (5, 8.6 and 10) and the other adsorption parameters were kept fixed. Each experiment was performed in a batch system, which carried out by agitating the 10 mL of dye solution (10 ppm) containing fixed amount of adsorbent (0.8 g) at room temperature. Fig. 7a displays the relationship between pH of initial CRD solution and adsorption efficiency. The results indicated the adsorption efficiency remained constant at low pH (acidic medium), but decreases with an increase of pH value higher that 8.6 (alkaline medium). For example, the pH values of 5, 8.6, and 10 showed adsorption efficiency of 58.1, 55.9, and 23.6, respectively. The decrease in CRD adsorption efficiency with an increase in the pH value may be attributed to the polarization of adsorbent decreases in alkaline medium (Hua, Pana, and Hong, 2023; Gabris, Periyasamy, and Tehrani-Bagha, 2025).

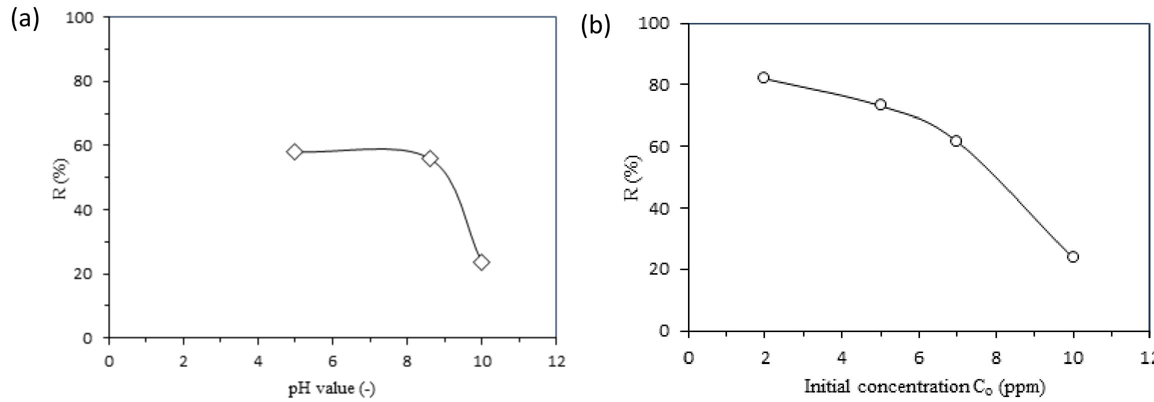


Fig. 7 Adsorption of CRD: **(a)** Effect of pH value, **(b)** Effect of initial concentration.

G) Effect of Initial Concentration of the CRD solution

The adsorbate initial concentration is one key of adsorption experiments because it provides an important driving force to transport the adsorbate molecules from bulk aqueous solution to active sites (Zhao, Shang, Xiao, Dou, and Han, 2014). Besides, the effect of the CRD concentration in the feed stream on adsorption efficiency of CRD was investigated using various CRD solution at initial concentrations of 2, 5, 7, and 10 ppm at fixed the other operating conditions and the obtained results are shown in Fig. 7b. The adsorption efficiency reduced with a rise in the initial concentration of the feed CRD solution for all samples. For example, the adsorption efficiency decreased from 82.07% to 23.64% with an increase in the CRD initial concentration from 2 to 10 ppm, which is ascribed to the saturation state of the adsorption sites on the surface of the adsorbent was achieved during processing feed aqueous solution with high CRD initial concentration faster than the solution with low CRD initial concentration. Similar observations have been previously reported (El Messaoudi, El Khomri, Dbik, Bentahar, Lacherai, and Bakiz, 2016; Zhao, Shang, Xiao, Dou, and Han, 2014).

H) Effect of Operating Temperature

The effect of operating temperature on the adsorption of CRD was studied using a 10 ppm initial concentration of CRD solution at different temperatures (20, 40, and 60 °C) in an adsorption column and the obtained results are shown in Fig. 8. The results indicated that the adsorption efficiency of CRD as a proportional relationship with operating temperature for all samples. For example, after 10 min of adsorption time, the adsorption efficiency increased from 57.14 to 73.56% with a rise in the adsorption temperature from 20 to 60 °C, which is attributed to the enhance in the mobility of the CRD molecules from the buck aqueous solution into the sorbent surface (Ekrayem, Alhwaige, Elhrari; and Amer, 2021). In addition, the increase in the adsorption efficiency with temperature indicated that the CRD adsorption using MC-BM is endothermic process. Generally, it is kwon that the diffusion of molecules in fluids are temperature dependently. However, after a certain of adsorption period, a reduction in the adsorption efficiencies were observed for all adsorption temperatures. For instance, the CRD adsorption at operating temperatures of 20, 40, and 60 ºC showed adsorption efficiencies of 57.14, 65.71, and 73.56 %, respectively, after 10 min, which decreased after 40 min to 22.16, 50.91, and 66.04 %, respectively.

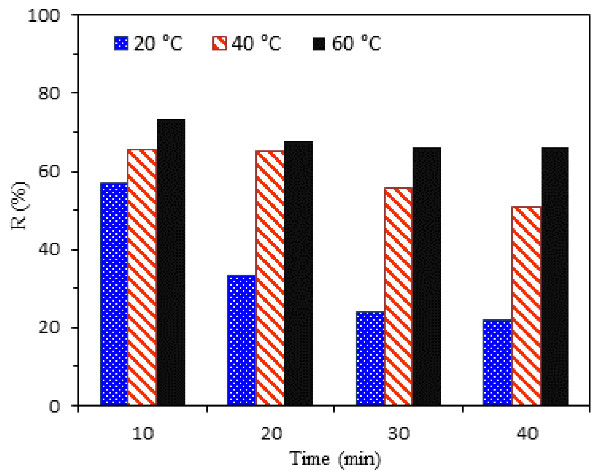


Fig. 8 Effect of the operating temperature on CRD removal.

IV. CONCLUSIONS

In this research work, powdered biosorbent was derived from *Mesembryanthemum crystallinum* biomass (MC-BM). In addition, the efficiency of these biosorbents for removal of toxic Congo red dye (CRD) from an aqueous solution has been studied. The derived biosorbent offers many attractive features such as outstanding CRD adsorption efficiency as well as it is considered low-cost, non-toxic, and biocompatible material. Adsorption parameters including, bed depth, flow rate, feed pH, temperature, and initial concentration were found to significantly affect the CRD removal efficiency. Therefore, the results demonstrated that the adsorption efficiency of CRD is better at higher bed depths, higher temperatures, lower initial concentrations, and lower feed effluents. The highest adsorption efficiency of 85.83% was achieved using the bed depth of 1 cm, the flow rate of 6 mL min^{-1}, the initial concentration of 5 ppm, at room temperature. This high value of adsorption efficiency is mainly attributed to available active sites on the surface of the derived sorbent which was confirmed using FT-IR. It was also found that after a period of time from operating the system, the removal efficiency begins to decline gradually as a result of the saturation of the adsorbent material over time. In addition, the adsorption at various temperatures indicated that the adsorption process of CRD using MC-BM was endothermic process.

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