

Hydrogels in the removal of industrial pollution: Adsorption characteristics for the removal of a toxic dye from aqueous solutions

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ABSTRACT

Pollution of water resources with various organic and inorganic pollutants is one of the biggest challenges of the present age. These infections seriously threaten human health and the lives of plants and animals. To minimize the mischievous effects of polluted water, technology-based smart treatment methods are necessary for sustainable equipping of drinking water. Application of (AAC-co-AM)-g-MCC hydrogels for the percentage removal of dye Methyl volute MV, from aqueous solutions have been studied. The synthesized (AAC-co-AM)-g-MCC hydrogels was described via FTIR, Thermogravimetric analysis (TGA), and UV-visible spectroscopy. The experiments held out in batch method effect of the factor like the solution of pH, initial concentration dye, and solution of temperature on the removal percentage of dye was studied. Contact time was done in 120 min. The data appear that an amount of 0.08 g (AAC-co-AM)-g-MCC hydrogels showed maximum removal percentage capacity of dye (100 mg L⁻¹). Maximum adsorption of MV dye was attained at pH 6.2. It was further proven the adsorption dye onto (AAC-co-AM)-g-MCC hydrogels followed the isotherm Langmuir while the kinetics studies detect that each adsorption method was pseudo-second-order. The thermodynamic factors like enthalpy (ΔH°), change in free energy (ΔG°), and entropy (ΔS°) were determined, and the values negative of ΔG° indicated that the method of removal was spontaneous at wholly values of temperatures. Further, the values of ΔH° indicated the endothermic nature of the method of removal.

Keywords: Water-pollution, Human health, Dyes, Adsorption, Equilibrium isotherm; Thermodynamics, Kinetics; hydrogel.

Article type: Research Article.

INTRODUCTION

From later little years, many developing countries are improved quarrel in provided that sustainable equipping of drinking water of the people, as their natural water resources are heavily polluted from agricultural effluents /industrial and man-made contamination threats large industrial method utilizes a set of synthetic chemical dyes for different utilize (Banerjee & Chattopadhyaya 2017; Saleh *et al.* 2019; Sawadogo-Ilboudo *et al.* 2021). Pollution of water resources with various organic and inorganic pollutants is one of the biggest challenges of the present age. These contaminants pose a serious threat to human health and the life of plants and animals. As shown in Fig. 1, various physical, chemical, and biological methods have been used to treat and reuse contaminated water resources (Mahmood *et al.* 2005). Industrial dyes are one of the most important causes of water pollution. Dyes have compound aromatic molecular structures and are mostly resistant to temperature, light, and oxidizers. This characteristic advantage makes the dye non-degradable and thus causes bioaccumulation in living creatures, leading to severe illness and defect. Several quantities of clean water are either consumed in chemical methods and/or products connecting dyes (e.g., leather, textile, paper, paints, cloth dyeing, pulp manufacturing, leather treatment, and printing).

Depending on chromophores, there are about 25-35 dye groups, among that phthalocyanine, azo, triarylmethane, and anthraquinones are the largest contributor of dyes (Barragán *et al.* 2007; Gamoudi & Srasra 2019; Olasehinde *et al.* 2020). Azo (around 80%) composes the major class of dyes. Several kinds of both synthetic and natural adsorbents have been estimated for the dye's removal and other contaminants from wastewater (Aljeboree & Alshirifi 2018; Liu *et al.* 2019; Aljeboree *et al.* 2019). However, these materials are perfect for removing pollutants but usually have some abuse, like poor strengths of mechanical, difficulty in separating the natural adsorbent powder (except via high-speed centrifugation from the treated effluent), and nonresistance against acid solutions (Al-Hayder & Al-Hussainawy 2016; Aljeboree & Abbas 2019a).

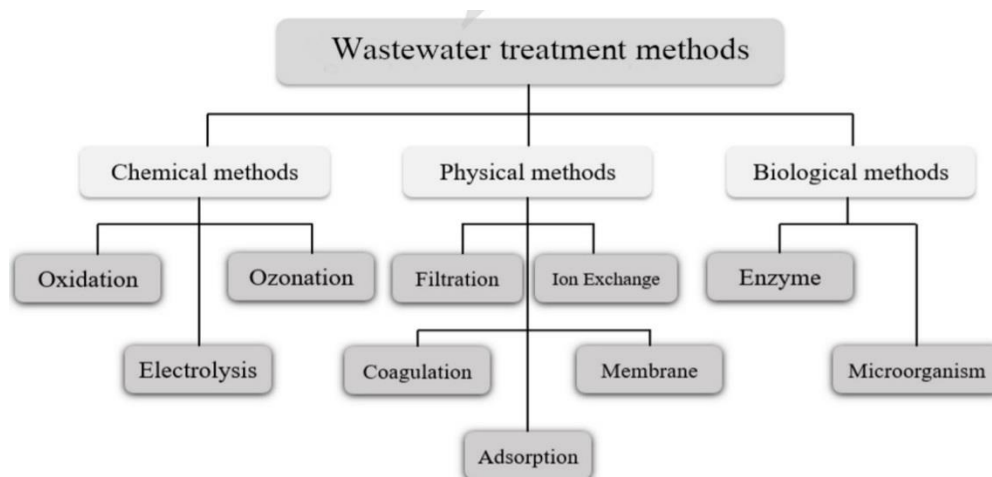


Fig. 1. The conventional wastewater treatment methods.

Therefore, several investigators should have involved their efforts to recover adsorption techniques and develop low-cost, efficient novel substitutional adsorbents with high power adsorptive. In this respect, great attention has lately been focused on the benefit of the method connecting hydrogel. At present, a hydrogel is receiving important interest to their utilized as adsorbents as these have been utilized for the treatment of contaminants environmental and removal dyes. A number of new studies have reported the successful utilization of hydrogel for several contaminants handling and treatment (Momeni 2018; Alshamusi *et al.* 2019; Aljeboree 2019a; Aljeboree 2019b; Adejoke 2019; Hosseinpour *et al.* 2020; Naderi 2021; Saadatmand 2021).

METHODOLOGY

Adsorbent

The hydrogel was prepared via MCC (1.5 g) is dissolved in 30-mL deionized water through magnetic stirring (150 rpm) for 15 min in a 250-mL three-lines flask equipped with the reflux condenser, and a thermometer and the nitrogen line with stirring at 50°C, KPS (0.3 g) was slowly added into solution MCC with continuous stirring for 5 min. Thereafter, mixture was placed in a water bath at 50 °C for 2 h to complete the polymerization reaction (Fig. 2). The resulting samples were washed excessively with deionized water to get rid of unreacted parts of reagent, and then dried in an oven at 60 °C under the reduced pressure for a period of 48 h. The dried samples were ground and screened for further analyses.



Fig. 2. A photograph of the (AAC-co-AM)-g-MCC hydrogels after washing.

Adsorbant

Methyl violet is a family of organic complexes that are mostly utilized as dyes. Relying on the number of linked methyl groups, the dye of the color can be changed. Its major application is as a purple dye for textiles and to give deep violet colors in ink and paint. Fig. 3 depicts the chemical structure of Methyl Violet (MV) which was prepared a 1000-mg L⁻¹ stander solution by dissolving 1 gm in 1000 mL double distilled water. The natural pH of MV was 6.4.

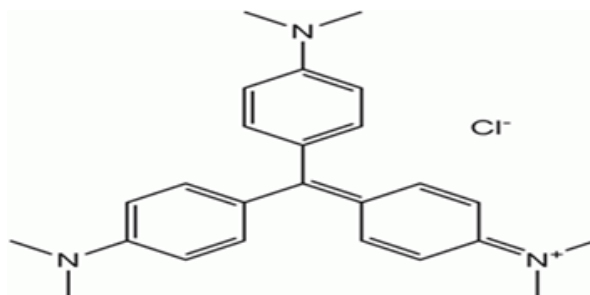


Fig. 3. Chemical structure of methyl violet (MV).

Preparation of calibration curve

The stander solution of MV that range about 5-250 mg L⁻¹ that obey Lambert - Beer law (Fig. 4) and the maximum wavelength (λ max) of MV dissolved in an aqueous solution found at 572 nm.

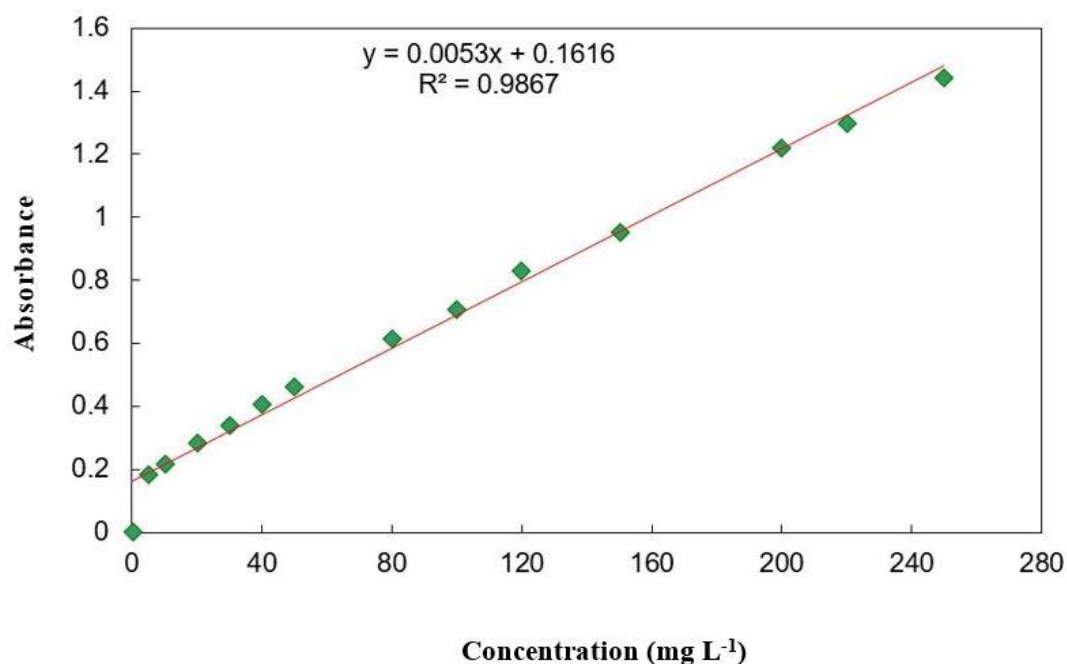


Fig. 4. Calibration curve of methyl violet (MV).

Adsorption kinetics and equilibrium studies

The effect of several parameters was studied to remove the dye from its aqueous solution, like contact time, adsorbent mass, solution pH, solution temperature, primary dye concentration, whereas studying the adsorption model and kinetics reactions. Dye solutions by the known concentration were prepared and thereafter, the amount required (AAC-co-AM)-g-MCC hydrogels was added to the shaking water bath for the predefined time about 120 min. The necessary time to achieve equilibrium conditions was determined via primary measurements kinetic. Then the solutions were filtered and remaining concentration of MV in the filtrate was determined via monitoring the absorbance at 572 nm utilizing UV-Vis spectrophotometer. The effect of pH was too study via controlling the pH of the solution by utilizing HCl/NaOH. Several primary concentrations (5-250 mg L⁻¹). The samples were

agitated on a water bath at 160 rpm at 283, 293, 298, and 303 K for 120 min. The adsorption kinetics was estimated by analyzing the adsorption efficiency of the MV solution at several contact times. To study the adsorption model, MV solutions of several concentrations were agitated with the adsorbent until attaining the equilibrium. The removal percentage (E%) of MV via (AAC-co-AM)-g-MCC hydrogels was calculated by Equation 1 where C_o is the primary MV concentration and C_e is its concentration at equilibrium.

$$\% \text{ dye removal} = (C_o - C_e)100C_o \quad (1)$$

MV dye adsorbed quantity q_t , and dye removal percent was calculated via these equations:

$$Q_e = \frac{(C_o - C_t)V}{W} \quad (2)$$

The equilibrium adsorption, Q_e (mg g^{-1}), was designed using the following formula:

$$Q_e = \frac{(C_o - C_e)V}{W} \quad (3)$$

RESULTS AND DISCUSSION

Characterization

Thermogravimetric analysis technique was employed to study the thermal stability of the prepared composite, and the sample was heated under an atmosphere with a rate of $10 \text{ }^\circ\text{C min}^{-1}$ from 40-900 $^\circ\text{C}$, where heating the (AAC-co-AM)-g-MCC composite leads to a weight loss of up to 3.75 % in the range of 80-220 $^\circ\text{C}$ due to the presence of moisture. Heating the composite in the range of 220-351 $^\circ\text{C}$ causes 43.59% mass loss which can be due to the pyrolysis, leading to the breaking of the chemical bonds inside the polymeric chains and consequently a decreased degree of polymerization along with removing the groups of hydroxyl and carboxyl in the form of CO_2 and CO , as well as removing the groups of amine and amide. Thereafter, the composite loses 18.14% of its weight within the thermal range (351-640 $^\circ\text{C}$) which is due to the thermal breakage of the interlocking polymeric chains of the hydrogel composite as appear in Fig. 5 (Karim & Jasim 2019).

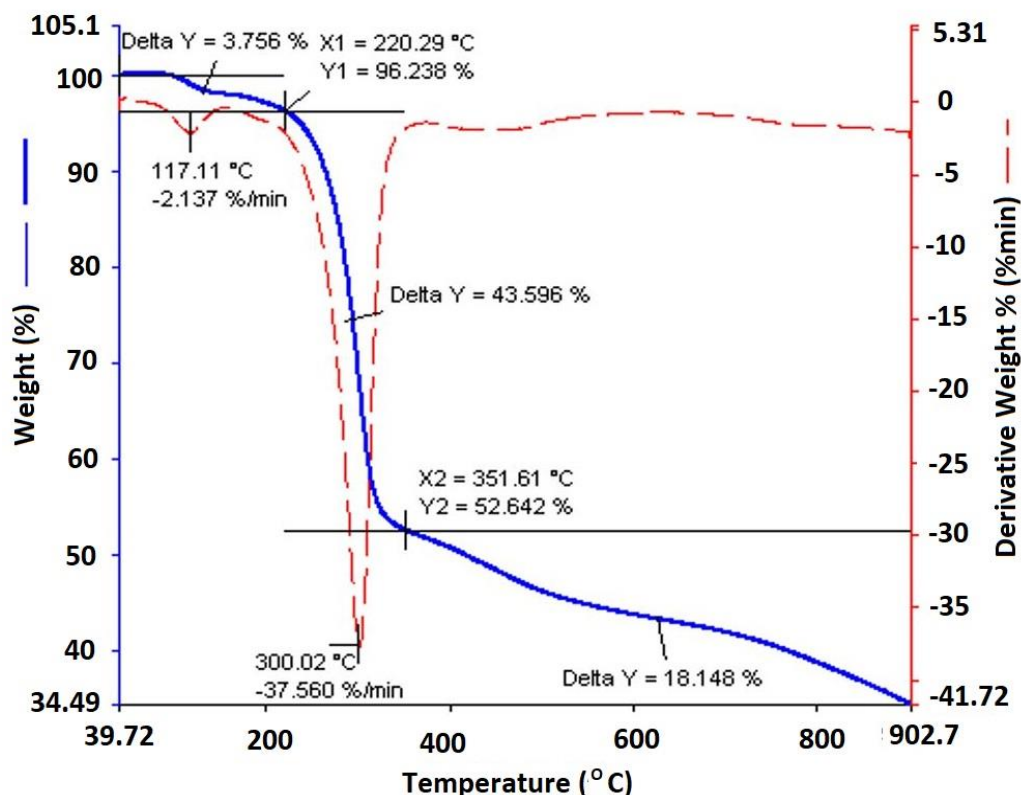


Fig. 5. TGA analysis of (AAC-co-AM)-g-MCC composite.

FT-IR characterization for adsorbent/adsorbate

The (AAC-co-AM)-g-MCC hydrogels were characterized via FT-IR spectroscopy. FT-IR spectra was collected in the mid-IR range from 4000 to 400 cm^{-1} with a resolution of 1 cm^{-1} . The FT-IR spectra of (AAC-co-AM)-g-MCC hydrogels before and after MV adsorption are illustrated in Fig. 4. The FT-IR pattern appears reduced in the intensity of bands next to the adsorption.

In addition, there is a real difference among (AAC-co-AM)-g-MCC hydrogels before and after the interaction by MV, suggesting that a phy-sorption phenomenon happens as a data of attractive forces among the (AAC-co-AM)-g-MCC hydrogel surface and MV under investigation (Alkaim & Alqaraguly 2013; Alrobayi *et al.* 2017).

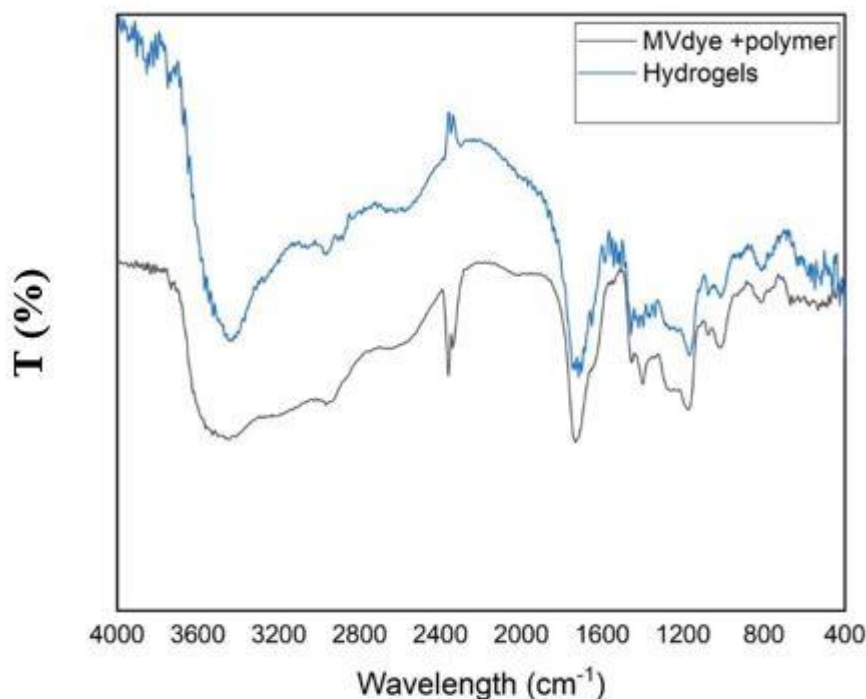


Fig. 6. FT-IR spectra of (AAC-co-AM)-g-MCC hydrogels (a) before and (b) after adsorption of MV dye.

Effect of (AAC-co-AM)-g-MCC hydrogels quantity

The adsorbent quantity is also one of the utmost significant parameters that present the efficiency of the adsorbent for a given primary quantity of the adsorbate. To investigate the correlation of removal MV on (AAC-co-AM)-g-MCC hydrogels quantity, several quantities (0.01–0.125 g) of (AAC-co-AM)-g-MCC hydrogels at pH 6.2 and 25 °C were added into 100-mL of 150 mg L^{-1} MV solutions. The data appeared in Fig. 6 illustrate that the adsorption capacity of MV decreases by elevating the (AAC-co-AM)-g-MCC hydrogels quantity. As the (AAC-co-AM)-g-MCC hydrogels quantity increased from 0.01 g to 0.15 g, the adsorption capacity of MV decreases significantly from 574.717 to 91.414 mg g^{-1} . In addition, the large quantity of (AAC-co-AM)-g-MCC hydrogels give higher removal percentage (del Mar Orta *et al.* 2017; Jasim *et al.* 2018). On the other hand, at greater adsorbent quantities, the accessibility of adsorbent active sites with higher energy decreases, and a larger fraction of the active sites with lower energy become occupied, leading to a decrease in the adsorption capacity. Thus, a 0.08 g (AAC-co-AM)-g-MCC hydrogels quantity was chosen as the optimal amount for further study (Aljeboree *et al.* 2019a; Aljeboree *et al.* 2019b).

Effect of solution pH on dye adsorption

The pH is a major control standard in the adsorption method. Not only affect the ionization degree for adsorbate but to the surface of adsorbent found in the solution effect of the pH on adsorption using 150 mg L^{-1} dye concentration, pH 2-12 at 25 °C (Fig. 6). Dye absorption (Q_e), was found to elevate by rising pH. The pH 6 give higher adsorption capacity (Q_e) after decreasing Q_e by upraised pH. Low adsorption MV occur by acidic pH

possibly because excessive existence H^+ ions contending with positive sets on the dye of adsorption locations (Als 2019; Altaa *et al.* 2018). At top pH, the surface of (AAC-co-AM)-g-MCC hydrogels may acquire negatively charged, that reinforce dye ions positively charged during electrostatic attraction forces (Jasim *et al.* 2018; Aljeboree & Alkaim 2019b).

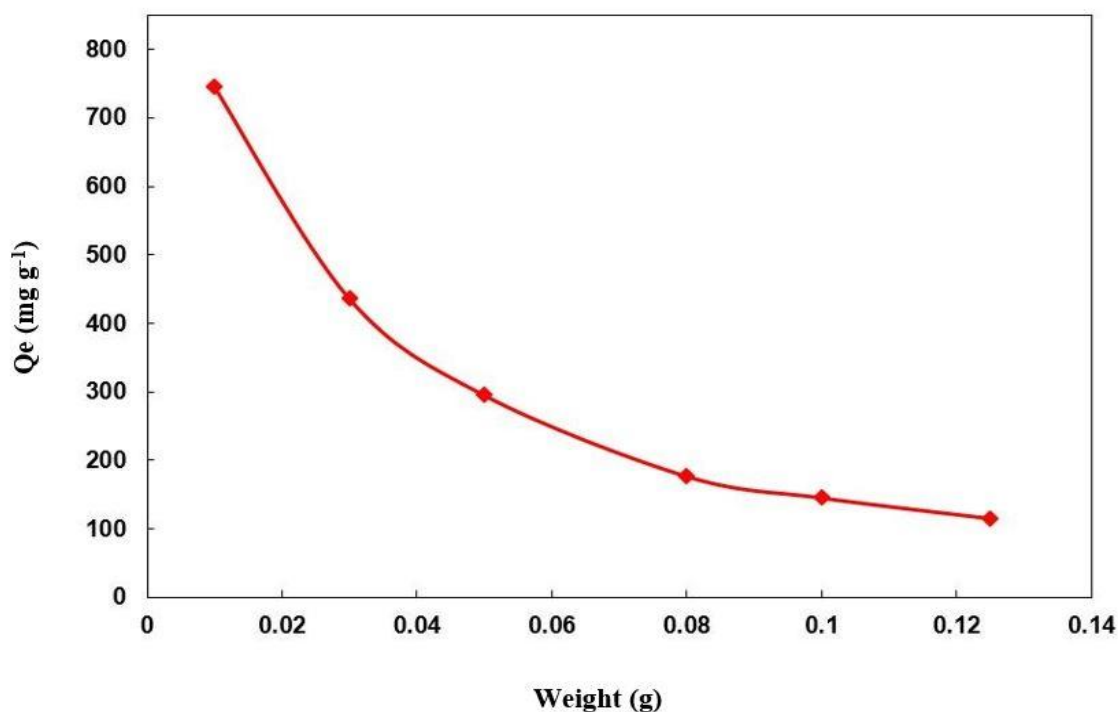


Fig. 7. Effect of mass adsorbent on adsorption dye MV: (25 °C, 120 min, and pH 6.2).

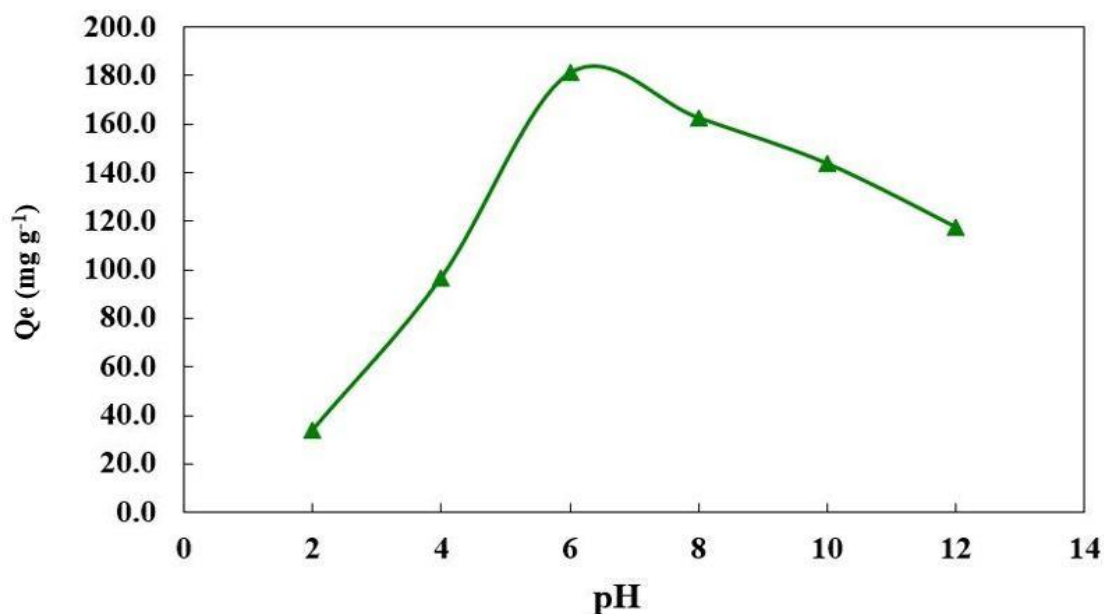


Fig. 8. Effect of the solution pH on adsorption of MV above the surface (AAC-co-AM)-g-MCC hydrogels at 25°C, 120 min, and 0.08 g in 150 mg L⁻¹ inside the 100-mL water.

Effects of temperature and thermodynamic function calculations

The effect of temperature (10, 20, 25, and 30 °C) on dye adsorption is illustrated in Fig. 9. Temperature elevation is accompanied by adsorption efficiency increase of (AAC-co-AM)-g-MCC hydrogels (the great the temperature

the best the adsorption). The data in Fig. 9 displayed that the method of adsorption was endothermic because MV percentage removal rises by elevating the temperature. The highest percentage of removal occurred at 25 °C after 120 min. Thermodynamic parameters of MV, such as ΔH° , ΔG° , and ΔS° assessed from the variation of the thermodynamic equilibrium constant (K) with temperature are depicted in Table 2.

The heat of adsorption (ΔH) can be determined from the following equation:

$$\ln X_m = -\frac{\Delta H}{RT} + \text{constant} \quad (4)$$

$$\Delta G = -RT \ln K \quad (5)$$

ΔS (change in entropy) can be obtained from the Gibbs equation: Substituting ΔG from the equation, then:

$$\Delta G = \Delta H - T\Delta S \quad (6)$$

$$\ln K = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \quad (7)$$

If $\ln K$ plotted versus $1/T$, we would find a straight line having a slope of $-\Delta H/R$ and an intercept $\Delta S/R$.

Fig. 9 and Table 1 demonstrate these factors, whereas Table 2 appear the thermodynamic values of calculations of MV adsorption onto (AAC-co-AM)-g-MCC hydrogels (Chiban *et al.* 2016).

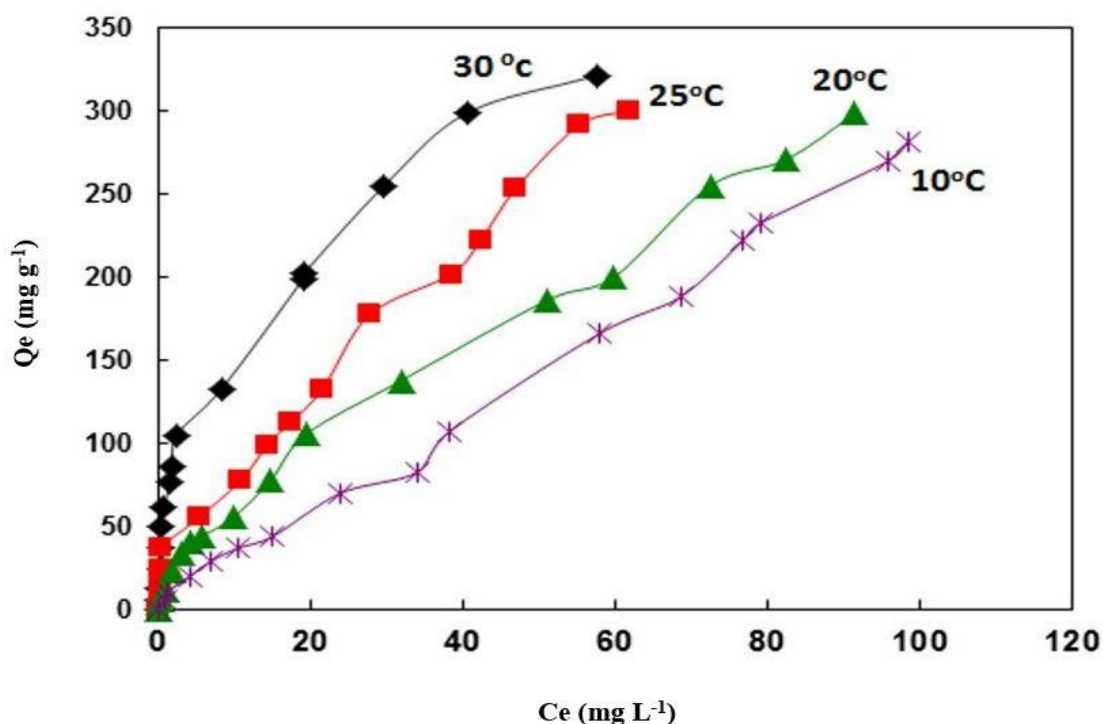


Fig. 9. Adsorption isotherms of MV dye onto (AAC-co-AM)-g-MCC hydrogel at several temperatures.

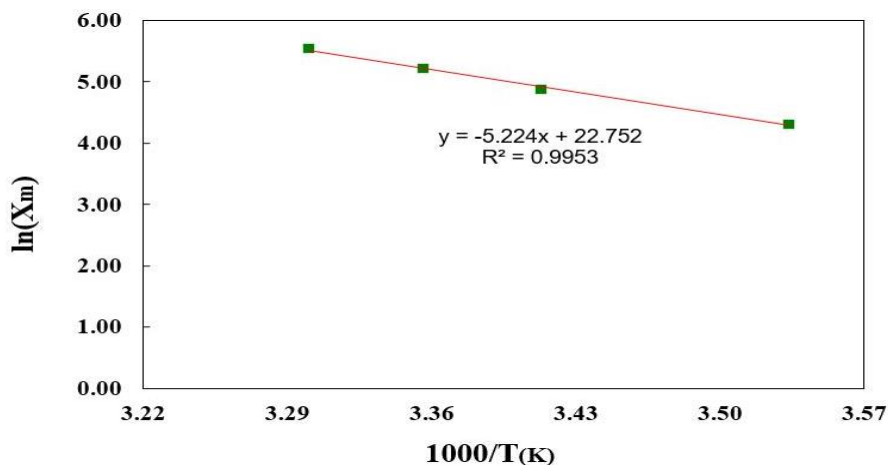


Fig. 10. Plot $\ln X_m$ against the absolute temperature of the adsorption (MV dye) onto (AAC-co-AM)-g-MCC hydrogels.



Table 1. Effect of temperature on highest adsorption of (MV dye) onto (AAC-co-AM)-g-MCC hydrogels.

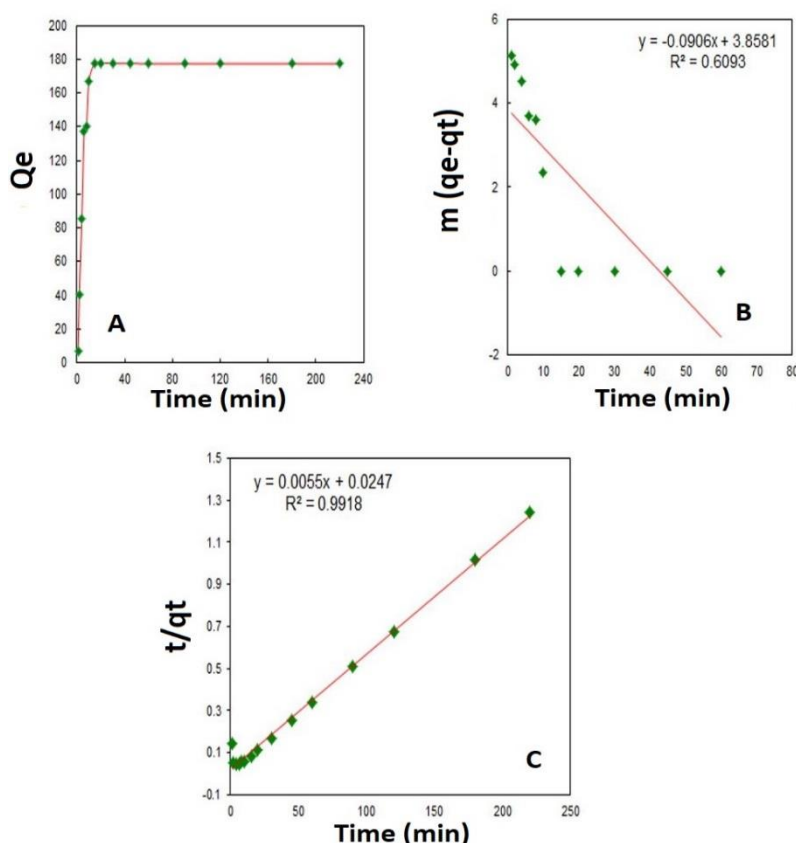
T (°C)	Tk	1000/T(K ⁻¹)	Ce = 28.5	
			Xm	lnXm
10	283	3.53357	75	4.31748
20	293	3.41297	131	4.87519
25	298	3.3557	184	5.21493
30	303	3.3003	255	5.54126

Table 2. Thermodynamic factors of adsorption for MV dye on the surface.

ΔH (kJ mol ⁻¹)	ΔG (kJ mol ⁻¹)	ΔS (J mol ⁻¹ K ⁻¹)	Equilibrium constant
43.4323	-5.4041	166.677	3.67719

Adsorption kinetics study and equilibrium time effect

After the steadiness of wholly other factors, the MV dye adsorption was studied at several times, 1 to 180 min. In Fig. 10, the adsorption capacity of MV dye was augmented, as the time elongated, until reaching utmost value (saturation state). Thereafter, the adsorption efficiency lowering by rising time due to a desorption method (Ghaedi *et al.* 2012). The models of kinetics of the adsorption method that define the experimental result, elected for adsorption of MV on the (AAC-co-AM)-g-MCC hydrogels (Fig. 8). Experimental data investigation was done utilizing the pseudo-first- and second-orders. Table 3 displays adsorption of MV via the (AAC-co-AM)-g-MCC hydrogels is an achievable method, because the value of R² rises by the model of pseudo-second-order compared to the pseudo-first-one (Mahde *et al.* 2018).

**Fig. 11.** Effects of reaction time (A), pseudo first-order (B), and the pseudo-second order (C) on MV adsorption.**Table 3.** Adsorption kinetic parameters of MV on (AAC-co-AM)-g-MCC hydrogel.

Pseudo- first order					
Slope	Intercept	k1(min ⁻¹)	qe (mg g ⁻¹)	R ²	
-0.0906	3.8581	0.0906	47.37525	0.6093	
Pseudo- second order					
Slope	Intercept	K2 (g mg ⁻¹ min ⁻¹)	qe (mg g ⁻¹)	R ²	h (mg g ⁻¹ min ⁻¹)
0.0055	0.0247	0.001225	181.812	0.9918	40.48583

Adsorption isotherm

Numerous isotherm equations are existing for investigating sorption equilibrium parameters, the most public being is the three isotherms. This kind is characterized via the particles of the adsorbate substance being perpendicularly or diagonal on the surface of the adsorbent. The surface is not homogeneous. The Freundlech, Langmuir and Timkein equations were employed, as Fig. 10 explains the data from the adsorption method acquired at 25 °C. The data appear the adsorption process of the MV on the surface of the adsorbent is analogical to the Langmuir isotherm. Table 4 depicts the correlation coefficients and constants of isotherm of Langmuir, Friendlech, and Timkein (Choy *et al.* 2000; 2019).

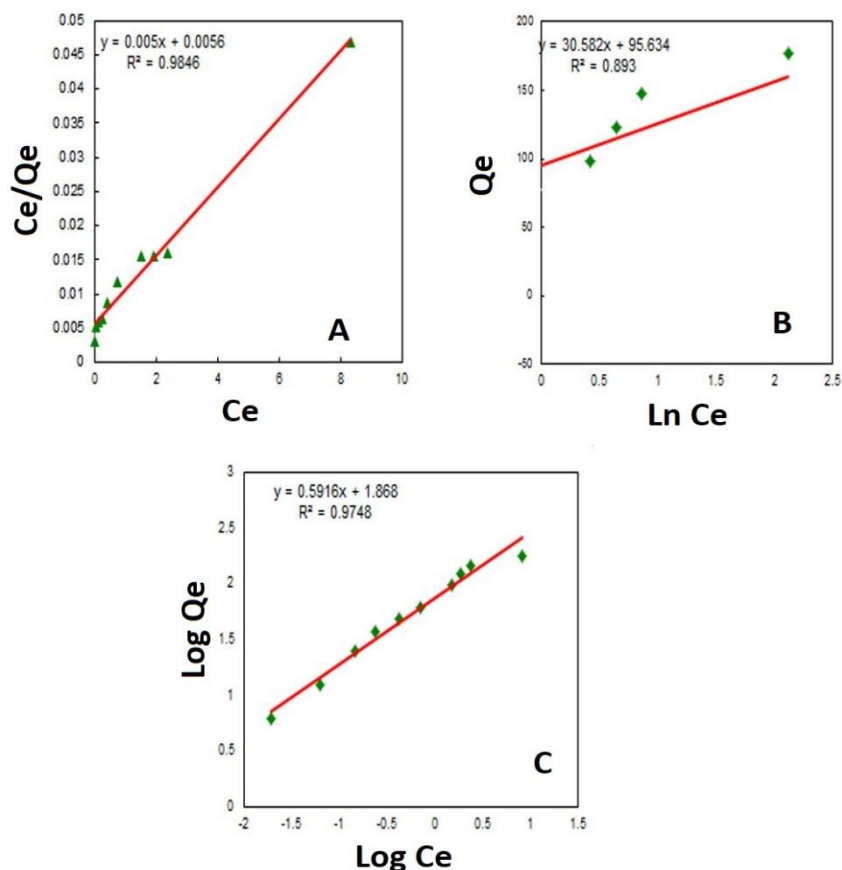


Fig. 12. Langmuir (A), Freundlech (B), and Temkin (C) isotherms of MV dye adsorption on (AAC-co-AM)-g-MCC hydrogels.

Table 4. The correlation coefficients and constants of model of Langmuir, Friendlich and Timken of adsorption MV dye adsorbed on to surface of (AAC-co-AM)-g-MCC hydrogels at 25 °C.

Langmuir equation			Freundlich equation			Timken equation		
K_L	qm	R^2	K_F	N	R^2	K_T	b	R^2
0.8928	200	0.9846	4.312	0.03269	0.8930	23.5126	0.5916	0.9748

CONCLUSION

In this study to remove the textile dye Methyl Violet (MV) we employed (AAC-co-AM)-g-MCC hydrogels as an alternative adsorbent surface. These results reveal that MV canister effectively removed from aqueous solution employing (AAC-co-AM)-g-MCC hydrogels as inexpensive adsorbent. In addition, this study show that the adsorption confirms on mechanism of decomposition [(AAC-co-AM)-g-MCC hydrogels] utilized as alternative, economical and environmentally-harmless condenser for water purification.

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