

Cellulose-Based Hydrogel for Sustainable Dye Removal from Industrial Wastewater

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Abstract: This study examines the efficiency of a biodegradable carboxymethyl cellulose (CMC)-citric acid hydrogel for adsorbing Methylene Blue (MB) from water. The hydrogel was created utilizing solution casting and thermal crosslinking with citric acid as a green crosslinker. Batch adsorption tests were carried out at MB concentrations of 5, 50, and 100 ppm, with equilibrium reached within 60 minutes and maximum removal efficiencies of 99.35%, 98.97%, and 92%, respectively. Adsorption increased under alkaline circumstances as carboxylate groups deprotonated more effectively. FTIR analysis revealed electrostatic and hydrogen-bonding interactions, while kinetic modeling yielded the best agreement with the pseudo-second-order model, showing chemisorption-controlled adsorption. The findings demonstrate the hydrogel's ability to remove dyes from wastewater in a sustainable manner.

Keywords: Carboxymethyl Cellulose; Citric Acid; Methylene Blue; Hydrogel Adsorption; pH-Responsive Behavior; Adsorption Kinetics.

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I. INTRODUCTION

The increasing growth of the textile, leather, paper, pharmaceutical, and chemical industries has made the release of synthetic dyes into aquatic habitats a critical global environmental concern [1-3]. Among industrial sectors, the textile dyeing and finishing business is one of the most polluting, accounting for 17-20% of total industrial water pollution globally [4]. A considerable fraction of dyes used in dyeing procedures fail to bond permanently to fibers; depending on dye chemistry, fabric type, and process conditions, 10-50% of applied dyes are discharged straight into wastewater streams [5]. Even at low concentrations, these dye-laden effluents cause vivid pigmentation of water bodies, restrict light penetration, impair photosynthesis, and harm aquatic ecosystems [6].

A significant amount of dye pollution eventually reaches marine habitats. In expanding and increasingly industrializing areas, up to 70-80% of industrial wastewater is discharged without proper treatment, allowing dyes to infiltrate rivers, estuaries, and coastal waters [7]. Dyes may survive in the water because of their chemical stability and resistance to biodegradation. Furthermore, textile-derived microfibers, which account for roughly 9% of total microplastic imports to the seas, can transport dye molecules and auxiliary

compounds, prolonging dye residence time in marine systems [8]. This cumulative intake causes localized coastal discoloration and long-term ecological damage.

Beyond industrial sources, household dye pollution is a major but generally neglected vector. Domestic laundry of dyed textiles discharges residual dyes, colorants, and microfiber-associated contaminants into municipal wastewater systems [9]. According to research, household washing adds significantly to urban dye loads, especially in areas without advanced wastewater treatment infrastructure [10]. Similarly, residential usage of hair colors, fabric dyes, and colored cleaning products adds dye residues to sewage streams, which may eventually reach natural water bodies [11].

Methylene Blue (MB) is a popular model cationic dye because to its widespread industrial use, high solubility, and strong chromophoric structure [12]. MB is known to have negative health consequences, such as oxidative stress and neurological problems, and it is extremely resistant to natural degradation mechanisms [13]. Conventional dye removal procedures, such as coagulation, chemical oxidation, membrane filtering, and biological treatment, are frequently associated with high operational costs, secondary

contamination, and low efficacy for stable dyes such as MB [14,15].

Adsorption with biopolymer-based hydrogels has emerged as a promising, long-term dye removal option [16]. Carboxymethyl cellulose (CMC), a renewable and biodegradable cellulose derivative high in hydroxyl and carboxyl functional groups, has a great affinity for cationic dyes [17]. Crosslinking CMC with citric acid, a non-toxic and environmentally friendly crosslinker, improves structural stability while being eco-friendly [18]. However, there have been few comprehensive research on concentration- and pH-dependent MB adsorption utilizing CMC-citric acid hydrogels. As a result, the current study focuses on creating and testing a sustainable CMC-based hydrogel for the efficient removal of Methylene Blue from aqueous solutions, with an emphasis on adsorption kinetics, pH responsiveness, and interaction processes.

II. MATERIALS AND METHODS

A. Materials

Various substances and apparatus were employed in the experimental research. Carboxymethyl cellulose (CMC) was used as the primary polymer for hydrogel production, with citric acid (CA) acting as a non-toxic crosslinking agent to construct a stable three-dimensional hydrogel network via heat esterification. Methylene Blue (MB) dye of analytical grade was used as the model cationic dye for the adsorption tests. To eliminate interference from dissolved pollutants, the tests employed distilled water for hydrogel synthesis, dye solution preparation, cleaning, and dilution. For pH-dependent adsorption investigations, sodium hydroxide (NaOH) and hydrochloric acid (HCl) solutions with a minimum purity of 99% were used to alter the solution pH to acidic, neutral, and alkaline conditions.

At ambient temperature, batch adsorption studies were conducted using an orbital shaker to achieve equal mixing of the hydrogel and dye solution. A UV-Visible spectrophotometer with quartz cuvettes of 1 cm path length and Methylene Blue's maximum absorption wavelength was used to quantify dye concentration. FTIR spectroscopy was used to detect functional groups and validate chemical interactions between the hydrogel matrix and dye molecules. The spectra were acquired in the region of 4000-400 cm^{-1} . Accurate pH values were obtained using a calibrated digital pH meter. To ensure experimental precision and repeatability, all glassware used in the experiments was properly cleaned, acid washed, and rinsed with distilled water before being used.

B. Methods

➤ Experimental Procedure

The experimental procedure involved six primary stages:

- Synthesis of CMC–Citric Acid Hydrogel
- Preparation of Methylene Blue Dye Solutions
- Batch Adsorption Experiments
- pH-Dependent Adsorption Mechanism

- Characterization
- Adsorption Kinetic Analysis

➤ Synthesis of CMC–Citric Acid Hydrogel

The hydrogel was created utilizing a solution casting process followed by thermal crosslinking, which was chosen for its simplicity and usefulness in biodegradable polymer systems. At room temperature, a specified amount of carboxymethyl cellulose (CMC) was progressively added to distilled water while magnetic stirring was continuously maintained. Stirring was continued for several hours until a clear, homogenous, and viscous polymer solution was formed, ensuring complete hydration and chain disentanglement of CMC molecules.

Separately, citric acid (CA) was dissolved in distilled water and gradually added to the CMC solution while stirring continuously. The mixture was further agitated to ensure homogeneous citric acid dispersion and intermolecular hydrogen bonding between CMC's hydroxyl (-OH) and citric acid's carboxyl (-COOH) groups. The resulting homogenous solution was poured into clean glass Petri dishes to form uniform thickness films, which were then dried in a hot air oven at around 60 °C overnight.

During thermal treatment, citric acid dehydrates to create cyclic anhydride intermediates that react with CMC's hydroxyl groups to form ester crosslinks, resulting in a stable three-dimensional hydrogel network. The crosslinking reaction can be represented as:



After thermal crosslinking and drying, the CMC-citric acid hydrogel films were carefully removed from the glass Petri plates and washed several times with distilled water to remove any unreacted citric acid that could affect adsorption behaviour. The washed hydrogels were then dried again until they reached a consistent weight. The dried hydrogel films were cut into uniform rectangular sheets which were then utilized for batch adsorption tests to assure experimental uniformity and reproducibility.

➤ Preparation of Methylene Blue Dye Solutions

An carefully weighed amount of dye was dissolved in distilled water to make a stock solution of 100 ppm Methylene Blue (MB). Working solutions of 50 ppm and 5 ppm were produced from the stock following the dilution equation:

$$C_1V_1 = C_2V_2$$

To conduct pH-dependent adsorption investigations, the 100 ppm MB solution was changed to acidic (pH ≈ 4), neutral (pH ≈ 7), and alkaline (pH ≈ 8) conditions using 0.1 N HCl or 0.1 N NaOH, and the pH was monitored with a calibrated digital pH meter.

➤ Batch Adsorption Experiments

Batch adsorption tests were carried out at room temperature, using a constant mass of dried hydrogel sheet and

a known volume of MB solution in beaker. An orbital shaker was used to stir the beaker and guarantee consistent mixing while minimizing external mass transfer. The samples were withdrawn at predefined contact times (0, 10, 30, and 60 minutes).

To remove hydrogel fragments, the extracted samples were filtered or allowed to settle. The residual dye concentration was quantified using a UV-Visible spectrophotometer at Methylene Blue's maximum absorption wavelength (λ_{\max}). A calibration curve was created prior to analysis to convert absorbance values into concentration.

The adsorption capacity at time t (q_t) and percentage of removal were estimated using:

$$q_t = \frac{(C_0 - C_t) \times v}{m}$$

$$\text{Adsorption Efficiency (\%)} = \left(\frac{C_0 - C_t}{C_0} \right) \times 100$$

Where:

C_0 = Initial concentration(mg/L)

C_t = Final concentration(mg/L)

V = solution volume(L)

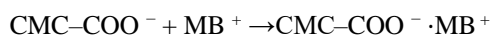
m = mass of hydrogel(g)

➤ pH-Dependent Adsorption Mechanism

The adsorption of MB onto the CMC–citric acid hydrogel is strongly influenced by solution pH. Under alkaline conditions, carboxyl groups on the hydrogel surface undergo deprotonation:



The negatively charged hydrogel surface interacts strongly with cationic MB molecules through electrostatic attraction:



At acidic pH, protonation of functional groups reduces surface charge density, leading to lower adsorption efficiency.

➤ Characterization

• UV-Visible Spectroscopic Analysis

UV-Visible spectrophotometry was used to measure the concentration of Methylene Blue (MB) in aqueous solutions before to and after adsorption, as well as to analyze adsorption kinetics. A UV-Visible spectrophotometer with quartz cuvettes of 1 cm in length was used for the analysis. Prior to adsorption tests, a calibration curve was created by measuring the absorbance of standard MB solutions at known concentrations (5, 50, and 100 ppm) at the maximum absorption wavelength (λ_{\max} = 665 nm). The calibration curve

showed strong linearity over the concentration range examined, allowing for reliable conversion of absorbance measurements to dye concentration.

During the batch adsorption studies, samples were taken at predefined intervals (0, 10, 30, and 60 minutes). To eliminate any suspended hydrogel pieces, the samples were filtered or allowed to settle before being analyzed. The decrease in absorbance intensity with increasing contact time was utilized to determine dye removal efficiency and adsorption capacity. The UV-Visible spectra also gave qualitative evidence of dye elimination by gradually decreasing the typical MB absorption peak, indicating a successful interaction between the hydrogel surface and dye molecules.

• FTIR Spectroscopic Analysis

Fourier Transform Infrared (FTIR) spectroscopy was utilized to identify the functional groups in the CMC-citric acid hydrogel, as well as to explore chemical interactions between the hydrogel matrix and Methylene Blue dye. The hydrogel's FTIR spectra were collected in the 4000-400 cm^{-1} region before and after dye adsorption at various pH settings.

The virgin hydrogel's FTIR spectra confirmed efficient crosslinking between carboxymethyl cellulose and citric acid, with characteristic absorption bands corresponding to hydroxyl (-OH), carboxyl (-COOH/-COO⁻), and ester (-COO-) groups. To establish dye-hydrogel interactions, variations in peak intensity and small shifts in characteristic bands were evaluated following adsorption. The presence or amplification of bands associated with Methylene Blue's aromatic rings and sulfonate groups showed efficient dye adsorption on the hydrogel surface.

Comparative FTIR measurement before and after adsorption revealed electrostatic interactions, hydrogen bonding, and π - π interactions between the functional groups of the hydrogel and MB molecules, corroborating the hypothesized process.

• Adsorption Kinetic Analysis

Adsorption kinetic studies were conducted to determine the rate of Methylene Blue (MB) uptake and to better understand the regulatory mechanism of the adsorption process onto the CMC-citric acid hydrogel sheets. Batch adsorption studies were carried out at room temperature with a constant mass of hydrogel sheet and a known volume of MB solution. The studies were conducted with various beginning dye concentrations, and samples were collected at predefined contact durations of 0, 10, 30, and 60 minutes.

At each time interval, the residual dye content in the solution was measured using UV-Visible spectrophotometry at Methylene Blue's maximum absorption wavelength. The mass balance equation was used to compute the amount of dye adsorbed at time t (q_t , mg/g^{-1}).

$$q_t = \frac{(C_0 - C_t)V}{m}$$

Where C_0 and C_t are the initial and time-dependent dye concentrations (mg L^{-1}), V is the volume of the dye solution (L), and m is the mass of the hydrogel sheet (g).

The pseudo-first-order model is expressed as:

$$\text{Log } (q_e - q_t) = \log q_e - \frac{k_1 t}{2.303}$$

Where q_e (mg g^{-1}) is the adsorption capacity at equilibrium and k_1 (min^{-1}) is the pseudo-first-order rate constant.

The pseudo-second-order model is given by:

$$\frac{t}{q_e} = \frac{1}{k_1 q_e^2} + \frac{t}{q_e}$$

Where k_2 ($\text{g mg}^{-1} \text{min}^{-1}$) is the pseudo-second-order rate constant.

The kinetic parameters were determined using linear plots of $\log(q_e - q_t)$ and $\log(q_e - q_t)$ versus t . Each model's appropriateness was evaluated based on the correlation coefficient (R^2) and agreement between experimental and computed equilibrium adsorption capabilities. The kinetic analysis revealed whether the adsorption process was dominated by physical adsorption or chemisorption mechanisms.

III. RESULTS AND DISCUSSIONS

This section provides a detailed analysis of the experimental data obtained for the adsorption of Methylene Blue (MB) with the produced CMC-citric acid hydrogel. Batch adsorption tests were carried out at initial dye concentrations of 5, 50, and 100 ppm, with adsorption behavior measured as a function of contact time (0-60 min). The hydrogel demonstrated quick adsorption kinetics, reaching equilibrium within 60 minutes for all concentrations, with maximum removal efficiencies of 99.35% (5 ppm), 98.97% (50 ppm), and 92% (100 ppm). UV-Visible spectroscopy data were used to assess dye elimination, whereas FTIR analysis revealed details about hydrogel production and dye-hydrogel interactions. The effect of pH on adsorption efficacy, particularly at greater dye concentrations, is also explored to better understand the adsorption mechanism. These findings collectively show the efficiency, pH responsiveness, and adsorption potential of the CMC – citric acid hydrogel for dye-contaminated wastewater treatment.

➤ Batch Adsorption Experiments

The result of the batch adsorption experiments, presented in Table 2, Time-Dependent Batch Adsorption Results for Methylene Blue at 5 ppm and 50 ppm and in table 2 for pH-Dependent Batch Adsorption Results for 100 ppm Methylene Blue.

Table 1 Time-Dependent Batch Adsorption Results for Methylene Blue at 5 ppm and 50 ppm Using Hydrogel

Conc. (ppm)	Time (min)	Initial Abs. (A_0)	Final Abs. (A_t)	% Removal
5 ppm	0	0.462	0.462	0.00
	10	0.462	0.120	74.03
	30	0.462	0.018	96.10
	60	0.462	0.003	99.35
50 ppm	0	0.618	0.618	0.00
	10	0.618	0.207	66.47
	30	0.618	0.038	93.92
	60	0.618	0.006	98.97



Fig 1 Progressive Reduction in Color Intensity of 5 ppm Methylene Blue Solution During Batch Adsorption

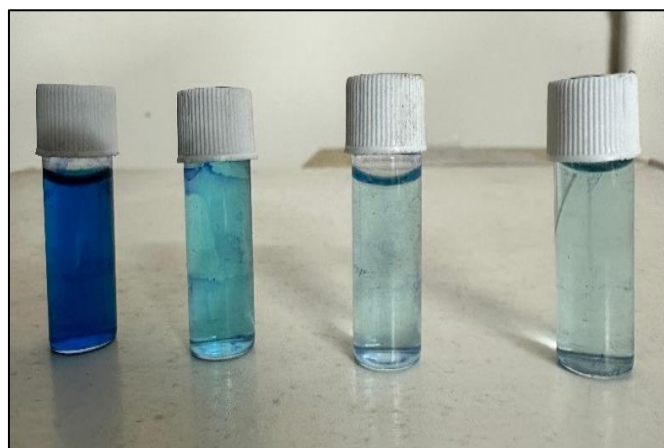


Fig 2 Progressive Discoloration of 50 ppm Methylene Blue Solution During Batch Adsorption

➤ UV-Visible Spectroscopic Analysis

Figure 3 shows the UV-visible spectra of Methylene Blue (MB) adsorption onto the CMC-citric acid hydrogel over time. The initial MB solution has a high distinctive absorption peak at about 665 nm, which corresponds to the chromophoric structure of MB. As the contact duration increases, the peak

intensity decreases in a systematic manner, showing that dye molecules are gradually being removed from the solution. The significant drop in absorbance after 60 minutes demonstrates quick adsorption kinetics and an effective interaction between the hydrogel surface and MB molecules.

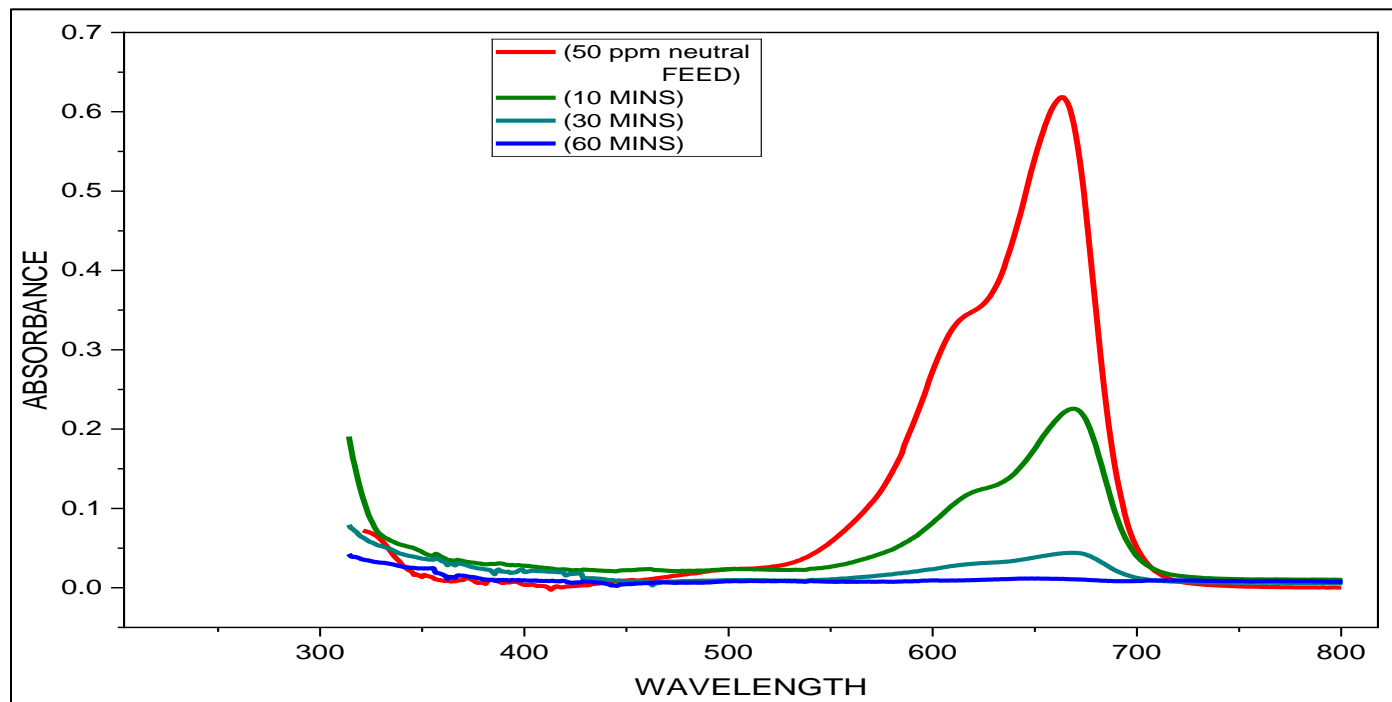


Fig 3 Time-Dependent UV-Visible Spectra of Methylene Blue During Hydrogel Adsorption.

Despite changes in initial peak intensity, the attenuation patterns for 5 ppm and 50 ppm MB solutions are nearly identical. While the 50 ppm solution has a larger initial absorbance due to the higher dye concentration, its overall spectral evolution over time is similar to that of the 5 ppm system. This commonality demonstrates that the adsorption process and kinetics remain constant at low and moderate concentrations. The near-complete suppression of the MB absorption peak after 60 minutes is consistent with batch adsorption data, verifying the high removal efficiencies obtained experimentally. As a result, the UV-Visible study supports the choice of 50 ppm as a typical concentration for detailed discussion, since it exhibits adsorption behaviour comparable to 5 ppm while better approximating practical wastewater conditions.

➤ pH-Dependent Adsorption

Table 2 shows the influence of solution pH on the adsorption of 100 ppm Methylene Blue with the CMC-citric acid hydrogel. The adsorption efficiency is clearly dependent on pH, with dye removal rising from acidic to alkaline settings. At pH 4, the hydrogel removed 89.11% after 60 minutes, showing a decreased adsorption due to carboxyl group protonation, which lessens electrostatic attraction to cationic MB molecules. At neutral pH (6.7), adsorption efficiency increased to 92.02% due to partial deprotonation of surface functional groups and improved dye-hydrogel interaction.

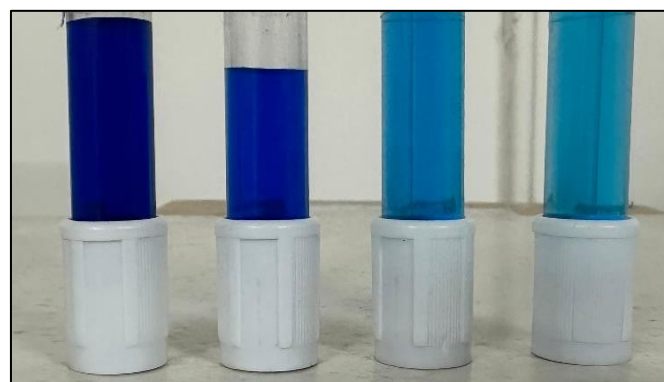


Fig 4 Time-Dependent Color Change of 100 ppm Dye at pH Condition

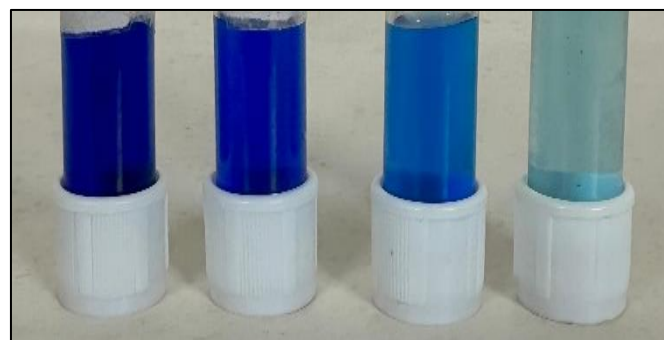


Fig 5 Time-Dependent Color Change of 100 ppm Dye at pH 6.7 Condition

Table 2 pH-Dependent Batch Adsorption Results for 100 ppm Methylene Blue

Conc.(ppm)	Time (min)	Initial Abs. (A_0)	Final Abs. (A_t)	% Removal
100ppm(4pH)	0	1.240	1.240	0.00%
	10	1.240	0.620	50.00%
	30	1.240	0.380	69.35%
	60	1.240	0.135	89.11%
100ppm(pH 6.7)	0	1.240	1.240	0.00%
	10	1.240	0.645	48.00%
	30	1.240	0.347	72.02%
	60	1.240	0.099	92.02%
100ppm (pH 8)	0	1.240	1.240	0.00%
	10	1.240	0.420	66.13%
	30	1.240	0.180	85.48%
	60	1.240	0.050	95.97%



Fig 6 Time-Dependent Color Change of 100 ppm Dye at pH 8 Condition

The best adsorption efficiency was found at alkaline pH (8), where color removal was 95.97% in 60 minutes. Under alkaline conditions, removal increases significantly due to

considerable deprotonation of $-\text{COOH}$ groups to $-\text{COO}^-$, resulting in stronger electrostatic attraction between negatively charged hydrogel surface and MB^+ ions. The gradual increase in removal effectiveness over time across all pH settings validates quick adsorption kinetics, whereas the overall trend indicates that alkaline circumstances favor maximum dye removal at greater dye concentrations.

➤ FTIR Analysis

The FTIR spectra of the CMC-citric acid hydrogel before and after adsorption of 100 ppm Methylene Blue (MB) under acidic, neutral, and alkaline conditions show clear dye-hydrogel interactions. The pristine hydrogel spectrum shows broad bands in the region of $3200\text{--}3500\text{ cm}^{-1}$ corresponding to $-\text{OH}$ stretching vibrations, whereas peaks at $1700\text{--}1730\text{ cm}^{-1}$ and $1600\text{--}1650\text{ cm}^{-1}$ are attributed to ester carbonyl ($-\text{C}=\text{O}$) and carboxylate ($-\text{COO}^-$) groups formed after citric acid crosslinking.

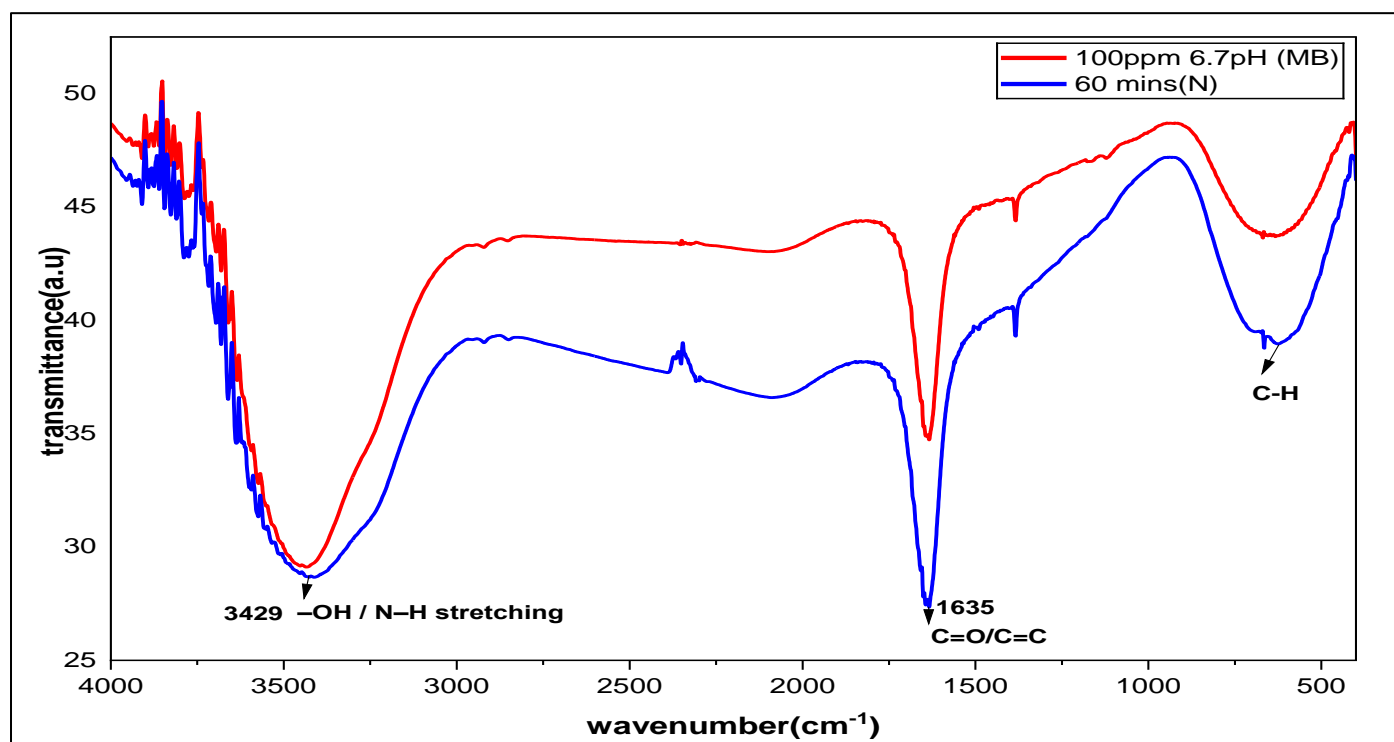


Fig 7 FTIR Spectra of before and after Adsorption of 100 ppm Methylene Blue Under Neutral pH Conditions.

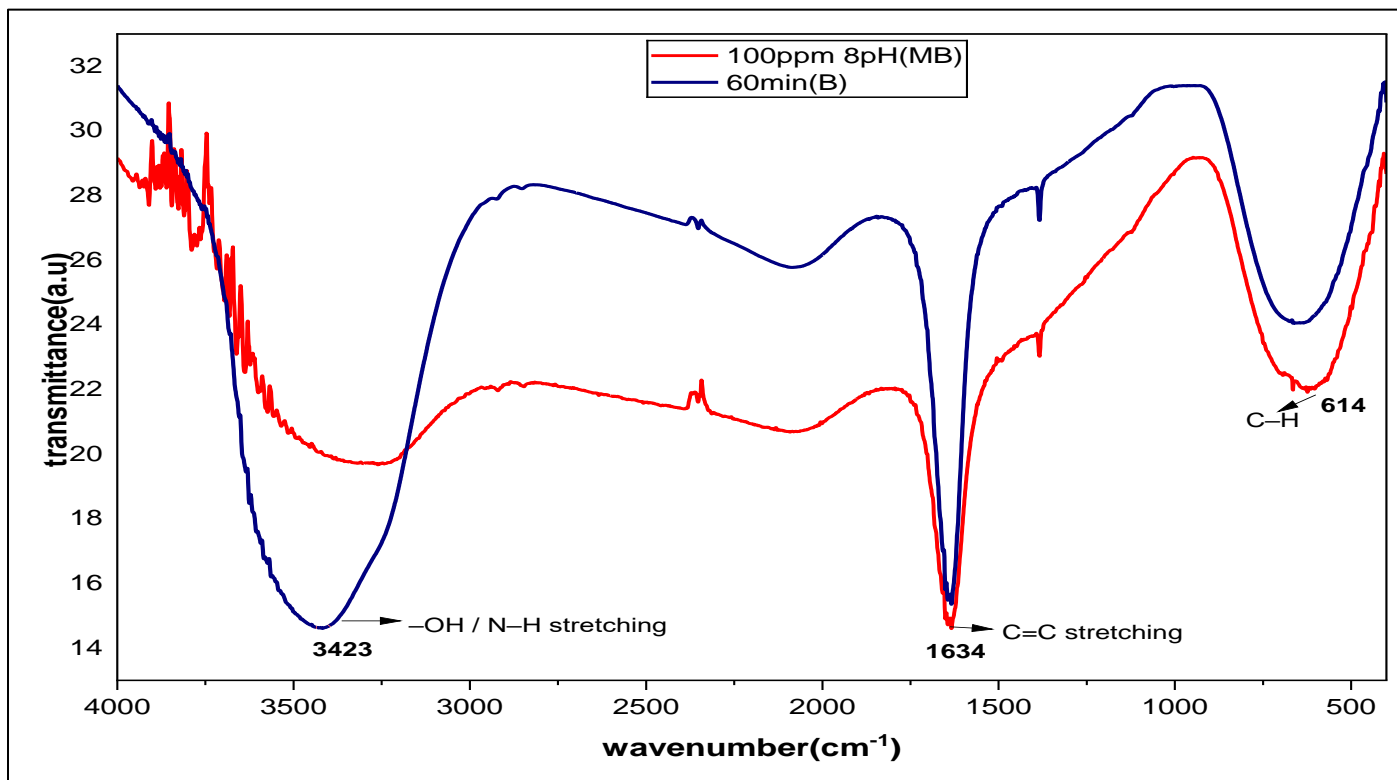


Fig 8 FTIR Spectra of before and after Adsorption of 100 ppm Methylene Blue Under Alkaline (Basic) pH Conditions.

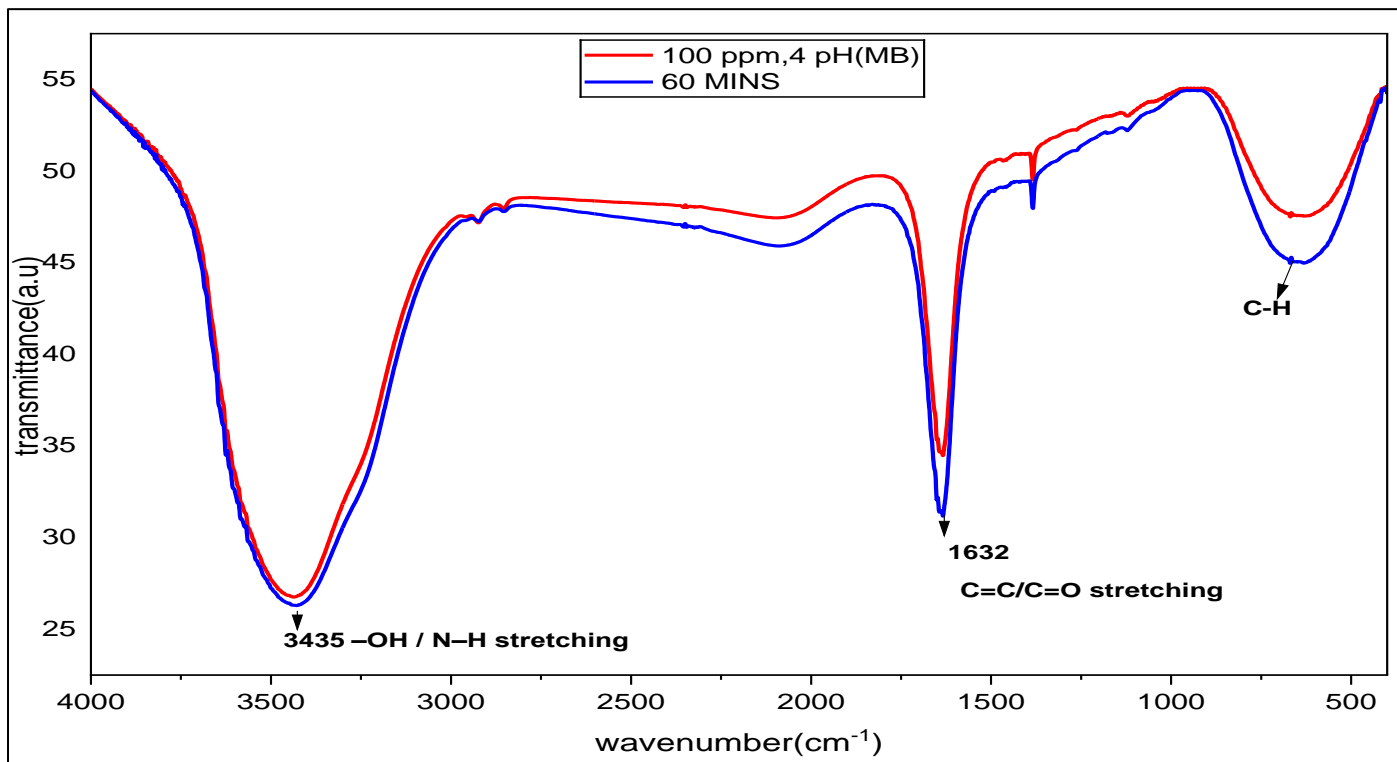


Fig 9 FTIR Spectra of before and after Adsorption of 100 ppm Methylene Blue Under Acidic Conditions (pH 4).

MB adsorption caused considerable changes in peak intensity and small shifts, especially in the -OH and -COO⁻ regions. These alterations demonstrate the presence of hydrogen bonding and electrostatic interactions between the hydrogel functional groups and cationic MB molecules. The degree of spectrum alteration increased from acidic to alkaline

pH, which is precisely proportional to the adsorption efficiencies provided in the 100 ppm pH-dependent adsorption table. Under acidic circumstances (pH 4), smaller spectrum shifts were detected, which is consistent with decreased dye loss (89.11%) due to carboxyl group protonation. At neutral

pH (6.7), moderate peak shifts were associated with increased adsorption (92.02%).

Under alkaline conditions (pH 8), carboxylate-related bands show stronger electrostatic attraction between deprotonated -COO^- groups and MB^+ ions. This observation is consistent with the maximum dye removal efficiency (95.97%) reported at alkaline pH. Overall, the FTIR results substantially validate the pH-dependent adsorption mechanism deduced from batch adsorption data, demonstrating that electrostatic interaction is the primary

factor in the adsorption of 100 ppm Methylene Blue onto the CMC-citric acid hydrogel.

➤ Adsorption Kinetics Study

The kinetics of Methylene Blue (MB) adsorption onto the CMC-citric acid hydrogel were studied to better understand the rate-controlling processes and adsorption mechanism. Figure X depicts the non-linear analysis of experimental adsorption data (q_t against time) using pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models. These models are extensively used to differentiate between physical and chemical adsorption processes.

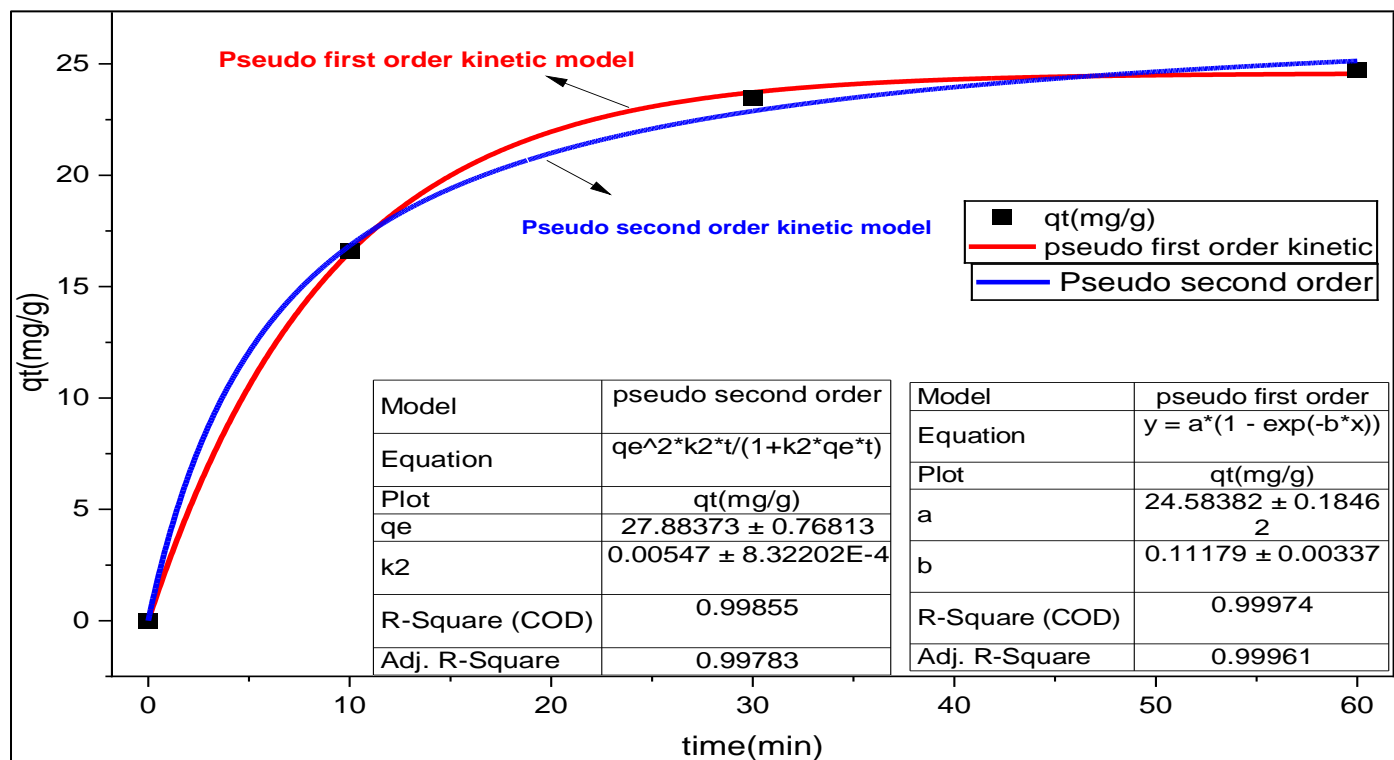


Fig 10 Kinetic Modeling of Methylene Blue Adsorption Using Pseudo-First- and Pseudo-Second-Order Models

The pseudo-first-order kinetic model posits that the rate of adsorption is proportional to the number of available adsorption sites. In this investigation, the PFO model provided a high correlation coefficient ($R^2 = 0.99974$), demonstrating an excellent mathematical match to the experimental data. The equilibrium adsorption capacity predicted by this model differed slightly from the experimentally measured q_e value, indicating that the PFO model alone does not adequately represent the adsorption mechanism.

In contrast, the pseudo-second-order model performed admirably with experimental data. The equilibrium adsorption capacity ($q_e = 27.88 \text{ mg g}^{-1}$) closely matched the experimental result. The model produced a strong correlation coefficient ($R^2 = 0.99855$) with an adjusted R^2 of 0.99783. The rate constant ($k_2 = 0.00547 \text{ g mg}^{-1} \text{ min}^{-1}$) suggests a rapid adsorption process. The PSO model indicates that adsorption is primarily driven by chemisorption, which involves electron sharing or exchange between the hydrogel's functional groups (-COO^- and -OH) and the cationic MB molecules.

Furthermore, the rapid initial adsorption observed in experiments, followed by a slower approach to equilibrium, lends support to the kinetic view. Initially, there are many active sites on the hydrogel surface, resulting in rapid dye uptake. As adsorption advances, site saturation and intraparticle diffusion effects limit the rate, resulting in equilibrium being reached within 60 minutes. Overall, the kinetic analysis demonstrates that the CMC-citric acid hydrogel has quick adsorption kinetics and strong dye-hydrogel interactions, making it an effective adsorbent for dye removal from aqueous solutions.

IV. CONCLUSION

A biodegradable CMC-citric acid hydrogel was effectively manufactured and utilized to the removal of Methylene Blue (MB) from aqueous solutions. The hydrogel demonstrated rapid adsorption kinetics, reaching equilibrium in 60 minutes. Under neutral circumstances, high removal efficiencies of 99.35% (5 ppm), 98.97% (50 ppm), and 92.02% (100 ppm) were obtained, indicating robust adsorption

performance over a wide concentration range. pH-dependent tests at 100 ppm revealed increased adsorption in alkaline circumstances, with a maximum removal efficiency of 95.97% at pH 8, confirming the role of electrostatic interactions.

UV-Visible and FTIR tests indicated that the dye was effectively removed, as well as the chemical interactions between MB molecules and hydrogel functional groups. Kinetic study shows that the adsorption process follows the pseudo-second-order model ($R^2 = 0.998$), indicating a chemisorption-dominated mechanism. Overall, the CMC-citric acid hydrogel exhibits high efficiency, sustainability, and practical applicability, indicating a significant potential for environmentally friendly wastewater treatment applications.

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