

Evaluation of the Adsorption Efficiency of Biopolymer Hydrogel Nanocomposite/Nanoclay in Wastewater Dye Removal

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Abstract: A new type of hybrid biopolymer nanocomposite hydrogel was created using sodium alginate and carrageenan combined with N-isopropyl acrylamide and bentonite nanoclay. The hydrogel was synthesised through free radical polymerisation. The purpose was to test its ability to remove the dye crystal violet (CV) from an aqueous solution. The structure and morphology of the synthesised nanocomposite hydrogel were analysed using Fourier transform infrared spectroscopy, scanning electron microscopy, and X-ray diffraction. Key parameters affecting the efficiency of crystal violet removal were explored, including pH, initial dye concentration, contact time, and amount of adsorbent. Optimal values for each parameter were identified. The removal mechanism was highly pH-dependent, with the dye removal rate increasing when more nanoclay was added in an acidic environment. Using an initial dye concentration of 30 mg/L and 0.05 g of the nanocomposite hydrogel, the equilibrium adsorption time was 3 hours. The adsorption matched the Langmuir isotherm model and followed pseudo-second order kinetics. Overall, the nanocomposite hydrogel was an effective adsorbent for eliminating crystal violet dye from water.

Key words: Nanocomposite hydrogel, carrageenin, crystal violet, adsorption.

Introduction

Currently, environmental pollution is recognised as a global problem and challenge. Dyes represent one of the most hazardous groups of chemical compounds found in industrial wastewaters. Therefore, the removal of these various materials is a human necessity. Textile industries, paint manufacturing, paper mills, metal plating factories, food processing plants, etc. are among the sources that discharge coloured wastewater into the environment. Conducted research indicates that in most cases, colour wastes also contain a spectrum of heavy metals and

other toxic pollutants (Abu Elella et al., 2024; Ritter et al., 2024). In general, methods used for removing dyes from industrial wastewater can be divided into three groups: chemical methods including oxidation, electrolysis and ozonation, physical methods including adsorption, coagulation or flocculation, filtration and reverse osmosis, biological methods including Enzymes and Microorganisms. In this research work, the physical method of adsorption was utilised. Basic dyes are chemically referred to as cationic dyes and are in the form of salts. CV dye is one of the strong cationic dyes that is used in textile industries and fish farming, with a

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maximum absorption wavelength of 590 nm. In fact, CV is a dyeing material from the triphenyl methane group that is widely used for dyeing purposes in industries such as silk, leather and paper. Among the conventional methods for removing pollutants, adsorption on resins has been of particular importance because these materials are easily separated from water (Khan et al., 2024). Among these resins, hydrogels can be mentioned which, due to functional groups, can absorb pollutants from aqueous environments. The hydrogel material can hardly give up this water even under pressure. Hydrogels are three-dimensional network polymers that can absorb and retain much larger amounts of water compared to water-absorbing materials. These materials also swell in the presence of water due to crosslinks resulting from the insoluble network builder, superabsorbents can have synthetic or natural bases. Free radical polymerisation of acrylamide, acrylic acid and other hydrophilic monomers in the presence of crosslinkers such as methylene bisacrylamide can lead to synthetic superabsorbents (Khan et al., 2024). In recent years, natural-based superabsorbents have attracted researchers' attention due to their low cost, biodegradability and environmental compatibility. So superabsorbents based on polysaccharides such as starch, chitosan, chitin, sodium alginate, carrageenan, cellulose and cellulose derivatives have been reported. These hydrogels do not have sufficient strength, and nanoclays can be used to overcome this problem. In recent years, nanocomposite hydrogel superabsorbents have attracted the attention of researchers. Among these biological polymers, sodium alginate has been widely used due to the presence of many carboxylate anionic groups. synthesised a nanocomposite superabsorbent hydrogel using sodium acrylate polymerised in a solution containing the biopolymers carrageenan and bentonite nanoclay (Karam et al., 2015). They determined the optimal swelling for this nanocomposite hydrogel occurred with 8 wt% of bentonite nanoclay. The addition of nanoclay to the carrageenan-based superabsorbent increased both the rate of dye adsorption and the removal efficiency by 11% compared to without nanoclay. The adsorption data aligned with the Freundlich isotherm model, rather than the Langmuir model as described for the previous nanocomposite hydrogel (Aljeboree et al., 2023). This demonstrated that incorporating nanoclay into carrageenan-based superabsorbents can enhance their performance for dye removal through increased adsorption rate and capacity. (investigated the removal of CV using agricultural waste barley hulls. They concluded that the equilibrium

adsorption time was 100 min and the optimum pH was 3. With increasing initial CV concentration, the dye adsorption capacity decreases, which is due to the repulsion between the negative charge of CV and graphene oxide. The process is endothermic and follows the Langmuir isotherm. Alginate hydrogels have low cationic dye removal capacity in acidic media due to their pKa of 4 (Algubili et al., 2015; Huerta-Angeles et al., 2024; Vahid et al., 2023;) Therefore, in this study, use a mix of the biopolymer carrageenan and sodium alginate. Because carrageenan is a biopolymer containing sulphate anion groups that do not protonate in acidic pH. Also, the presence of nanoclay in hydrogels increases the removal rate of cationic dyes, which in this study we tried to investigate the influence of bentonite nanoclay on the removal percentage of CV cationic dye by sodium alginate-carrageenan hydrogel nanocomposite. Also, the adsorption capacity of CV was fully investigated under different conditions such as adsorbent concentration, CV concentration, and pH. After obtaining the results of the experiments, the conformity of adsorption isotherms such as Freundlich and Langmuir with the obtained results and the pseudo-first order kinetic model and pseudo-second order kinetics were considered and investigated.

Experimental

Chemicals

Kappa-carrageenan and sodium alginate were purchased from Macklin, China. and N-isopropyl acrylamide monomers was obtained from HiMedia, India. Methylene bis acrylamide as a crosslinked and potassium persulfate initiator were bought from Merck, Germany. Bentonite clay particles are smaller than 2 μm and CV dye with a molecular weight of 407.979 g/mol was provided by CDH, India.

Instruments

Scanning electron microscopy (SEM) using a Vega-Tescan model was utilised to study the surface morphology of synthesised materials. To investigate the nanostructure of nanocomposites, an XRD instrument with a Siemens D-500 model, Cu-K α wavelength ($\lambda=1.54\text{\AA}$), tube voltage 35 KV, and tube current 31 mA was employed. Fourier transform infrared spectroscopy (FTIR) with a Bruker model was also used to study the spectra and functional groups and bonds present in the nanocomposite structures. To homogenise, a shaker platform made by Fan AzmaGostar, Iran was applied.

The pH was adjusted using a Metrohm 827 pH meter made in Switzerland.

Methods

Synthesis of Hydrogel Nanocomposite

Varying amounts of bentonite clay were dissolved in 30 mL of distilled water and the solution stirred for 24 hours using a magnetic stirrer heated to 60°C to control the temperature. Then 1.0 g of kappa-carrageenan and 1.0 g of sodium alginate were added to the clay solution and stirred for 2 hours to fully dissolve. Next, 1.0 g of N-isopropyl acrylamide monomer and 0.05 g methylene bisacrylamide dissolved in 5 mL distilled water were added at once and stirred for 1 hour. Finally, 0.05 g potassium persulfate dissolved in 2 mL distilled water was added as an initiator and the solution was stirred for 3 hours to obtain the nanocomposite hydrogel and then washing with ethanol was replaced every 12 h, 2 to 3 times. The dehydrated hydrogel nanocomposite, after being cut with scissors, was placed on a glass clock in a drying oven at a temperature of 50°C for 24 h to dry completely. Finally, the obtained nanocomposite hydrogel was ground into fine particles using a mill. It was then sieved using mesh sizes of 20, 40 and 60. Mesh size 20 was used for water absorption studies, 40 for CV adsorption studies, and 60 for obtaining FTIR spectrum.

Batch Adsorption Experiments

A CV dye stock solution (1000 mg/L) was prepared by dissolving a specific amount of CV in double distilled water. The required dilute solutions (10-100 mg/L) for experiments were prepared by diluting the stock solution. This concentration range was selected based on previous studies and dye concentrations in some industrial waste waters.

Batch removal experiments of CV by the synthesised nanocomposite hydrogel were carried out in 250 mL Erlenmeyer flasks with mixing using a shaker. Each experiment involved the preparation of a 100 mL solution with a specific initial concentration and pH (adjusted to 5 ± 0.2 using HCl or NaOH solutions). A known mass of 0.05 g of nano-samples sieved through 40 mesh was then added to the solution for CV removal. The resulting suspension was immediately placed on a shaker at 120 rpm. At different time intervals, the removal of CV by the nanocomposite hydrogel was studied by measuring absorbance at 590 nm wavelength using a spectrophotometer.

To study the adsorption isotherm curves of CV removal, solutions with concentrations of 10, 20, 30, 50, 70, 100 and 150 mg/L were used. The equilibrium adsorption capacity was calculated using Equation 1:

$$q_e = \frac{C_0 - C_e}{m} \times V \quad (1)$$

where q_e is the amount of dye adsorption (mg/g), C_0 and C_e are the initial and equilibrium CV concentrations in the liquid phase in mg/L, V is the solution volume in litres, and m is the mass of adsorbent used for removal in g.

Results and Discussion

The structure and functional groups were examined using FTIR spectral analysis. In the spectrum of clay (Figure 1), the stretching vibration bands of OH groups in the clay structure and water molecules appeared at 3634 and 3338 cm^{-1} . Also, the bending vibrations of clay OH and the stretching vibration band related to Si-O-Si bonds in the clay structure appeared at 1636 and 1031 cm^{-1} , respectively (Al-Mashhadani, Aljeboree et al., 2021). To better study the nanocomposite structure, first, the FTIR spectrum of the hydrogel was studied and its structure was compared with the nanocomposite hydrogel containing 0.5 g of bentonite nanoclay. The results are shown in Figure 1. In Figure 1, the resulting hydrogel contains carrageenan chains and amide and carboxylate functional groups which are indicated by peaks at 1720 cm^{-1} related to amide and 1401 cm^{-1} related to carboxylate, respectively. The presence of carrageenan in this structure is shown by peaks at 1227 and 849 cm^{-1} . In the spectrum of the nanocomposite hydrogel, the peaks related to nanoclay at 3634 cm^{-1} are omitted, indicating the interaction between nanoclay

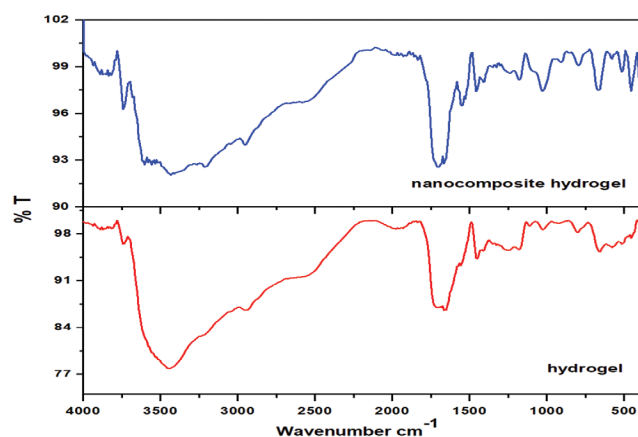


Figure 1: FTIR spectrum of hydrogel and nanocomposites hydrogel.

and hydrogel functional groups. The presence of carrageenan, clay, amide and carboxylate groups in the nanocomposite is proven by peaks at 928, 1031, 1701, and 1401 cm^{-1} , respectively. The only difference is that the peaks related to carrageenan and amide groups have shifted compared to the hydrogel without clay, indicating the interaction between these functional groups and nanoclay (Tainara, 2021).

XRD patterns were used to study the bentonite clay dispersion in the polymer matrix. For this purpose, samples with different amounts of nanoclay were used for the study. The results are shown in Figure 2, the XRD pattern belongs to hydrogel broad peak indicating it is amorphous, while nanocomposite hydrogel and a pure bentonite nanoclay are sharp. As shown in this figure, the peak at $2\theta=7.08^\circ$, 19.94° and 26.65° indicates the interlayer spacing of nanoclay sheets. In the presence of nanoclay for nanocomposites hydrogel containing 0.75 g of bentonite nanoclay, the intensity of the peak present in the nanoclay sample is reduced, indicating the exfoliation of the sheets in the prepared nanocomposite hydrogel. This result shows that in these samples, the nanoclay is exfoliated in the polymer matrix (Mosaa et al., 2019).

Scanning electron microscopy (SEM) was used to study the surface of hydrogels and nanocomposite hydrogels. In fact, the surface structure of hydrogel was compared with nanocomposite hydrogels containing different amounts of nanoclay, and the related results are shown in Figure 3. Figure 3a belongs to the hydrogel which has a relatively smooth surface (Radia et al., 2022). With the addition of nanoclay to the hydrogel composition, the surface structure changed as shown in Figure 3b. As is evident, the surface of this nanocomposite has become rough instead of smooth, which could be due to the presence of nanoclay in the hydrogel composition. In Figure 3c and d, energy dispersive X-ray spectroscopy (EDS) analysis showed the percentage of carbon, oxygen, and other elements in both the hydrogel and nanocomposite hydrogels, respectively. The presence of elements such as Si, Ca, Na, Al, Mg, and K was observed, indicating the dispersion of nanoclay layers within the polymer matrix (Bader et al., 2019).

Effect of Contact Time and Adsorbent Dosage

In this study, the effect of contact time on the removal percentage of CV dye at an initial concentration of 30 mg/L, dose of sample (0.02, 0.05 and 0.1 g), pH=3 and temperature of 298 K was investigated. As shown in Figure 4, the removal efficiency rapidly increases

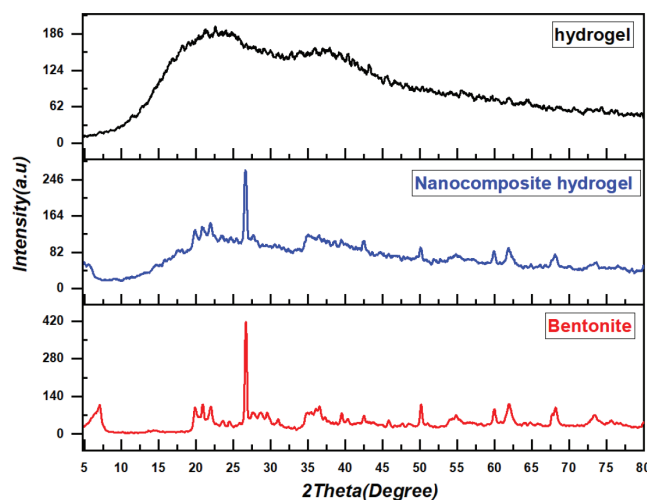


Figure 2: XRD pattern of hydrogel, nanocomposites hydrogel and bentonite.

within the first 90 min, which is attributed to the high amount of available active absorption sites and the high dye concentration gradient between the pollutant solution and adsorbent surface. Then, the removal efficiency reaches an approximately constant value as the concentration gradient decreases and active sites on the adsorbent become saturated, and increasing the contact time does not affect dye removal. In this study, the optimum contact time for dye absorption by both adsorbents was considered to be 300 min (Kareem et al., 2016; Maktouf, 2023; Zhuqing 2019).

Effect of pH

Figure 5 shows the amount of equilibrium absorption after 300 min. The results indicate that in acidic environments, the adsorption rate is higher compared to alkaline environments. This suggests that carrageenan is more active in acidic media since CV becomes protonated in acidic conditions and is more readily removed. This nanocomposite has a very good ability to absorb CV and the absorption process is very fast. The absorption of CV is due to the presence of sulphonate, and carboxylate groups in the nanocomposite structure as well as the groups present in the nanoclay structure (Radia, et al., 2022; Zhao, et al., 2017).

Adsorption Isotherms

As shown in Figure 6 and Table 1, the adsorption isotherm data was fitted to both the Langmuir and Freundlich models under isothermal conditions at pH 3 for crystal violet removal by the nanocomposite. Based on the correlation coefficient (R^2) values, the Langmuir model provided the best fit for the data. This suggests

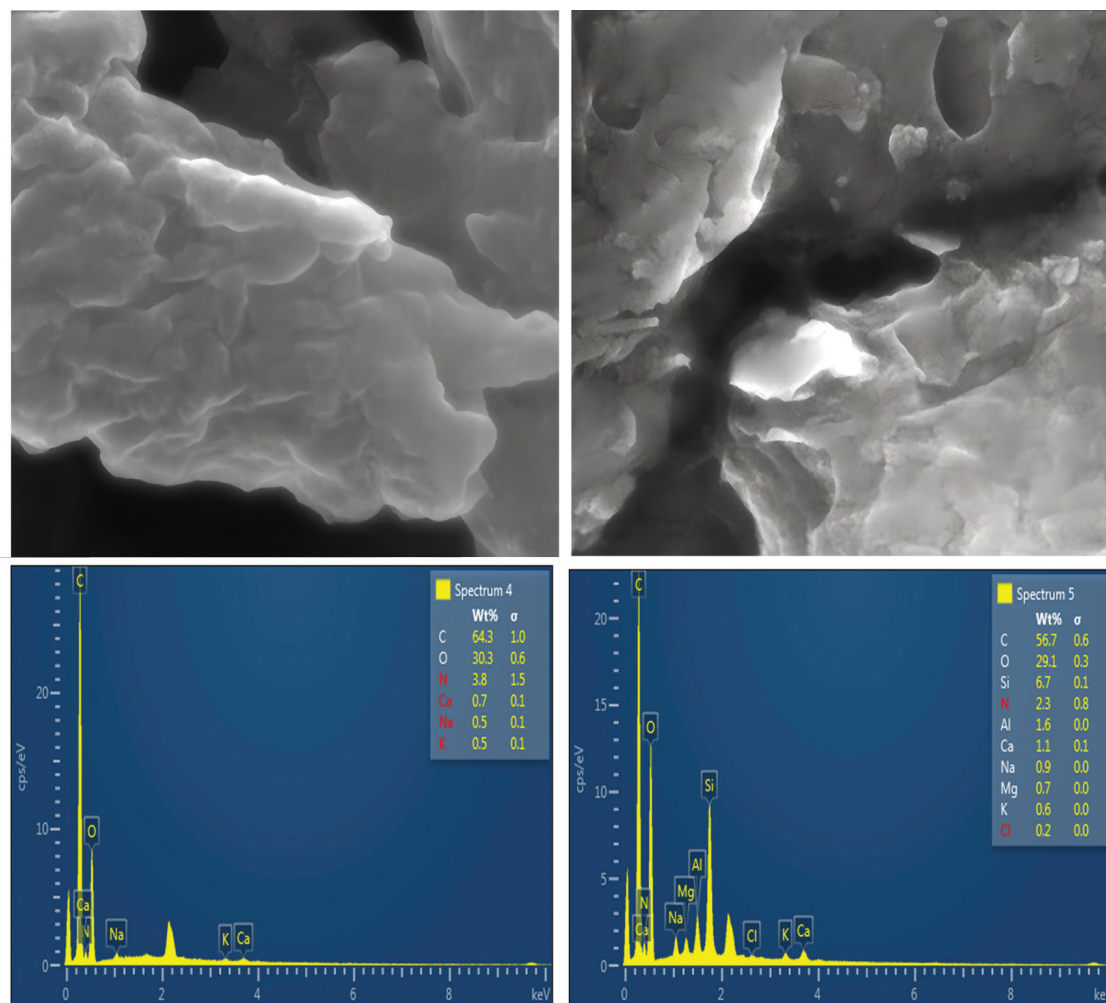


Figure 3: SEM image of a) hydrogel and b) nanocomposites hydrogel c) EDS of hydrogel and d) EDS of nanocomposites hydrogel.

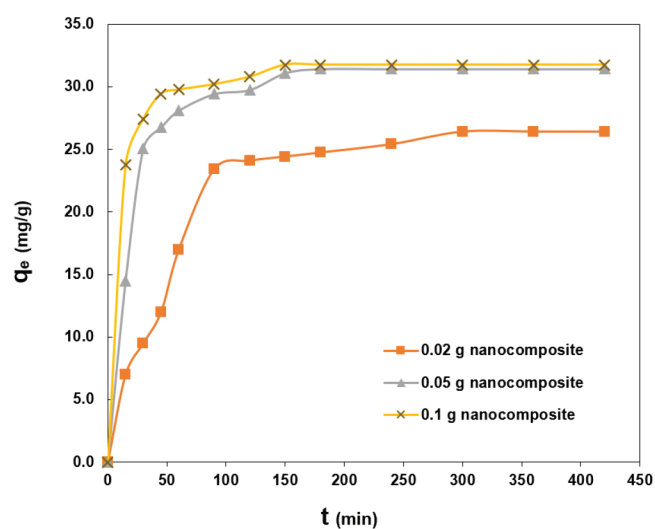


Figure 4: Contact time and adsorbent dosage of the adsorption capacity of the nanocomposite hydrogel.

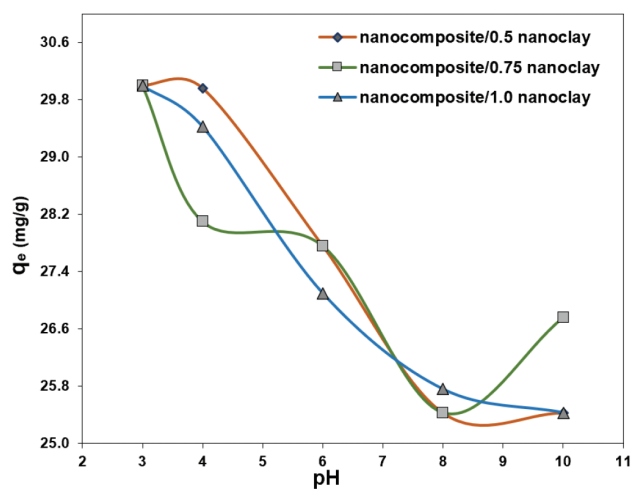


Figure 5: Effect of pH on adsorption capacity in different amount nanoclay nanocomposite.

the adsorption sites on the prepared nanocomposite are homogeneously distributed and the surface adsorption of the dye occurred uniformly. The higher R^2 value for the Langmuir model compared to the Freundlich model supports the conclusion that the Langmuir model is more suitable for describing the crystal violet adsorption behaviour of this nanocomposite hydrogel (Sadoq, 2024).

Table 1: Coefficients of Langmuir and Freundlich adsorption isotherms

<i>Langmuir equation</i>			<i>Freundlich equation</i>		
K_L	q_m	R^2	K_F	n	R^2
1.637	67.114	0.9930	0.9899	1.676	0.9753

Adsorption Kinetic Models

Figure 7 shows the pseudo-first and pseudo-second order kinetic plot for the adsorption process of CV dye by nanocomposite hydrogel. The pseudo-first order kinetic model provides a poor description of the CV dye adsorption kinetic data compared to the pseudo-second

order model. As shown in Figure 7, the pseudo-second order model exhibits a very good fit with this adsorption system and its equation fits the experimental data well. Conformity with the pseudo-second order adsorption kinetics has also been reported for many other dye adsorption systems based on hydrogels (Alhattab et al., 2023). The kinetic data obtained from this study are given in Table 2. Also the characteristics of certain adsorbents to the adsorption of CV dye are shown in Table 3.

Conclusion

This research was conducted with the aim of removing the cationic dye CV from industrial wastewater using a nanocomposite hydrogel of the biopolymer carrageenan and sodium alginate in the presence of bentonite nanoclay with exfoliated dispersion type. Based on XRD studies, the clay was exfoliated in the nanocomposite matrix. The SEM results also showed that incorporating nanoclay made the surface more porous. The CV dye removal rate increased with increasing the amount of

Table 2: Kinetic data for the adsorption of CV dye by nanocomposite hydrogel

<i>Pseudo first order</i>			<i>Pseudo second order</i>			
K_1 (min^{-1})	q_e (mg/g)	R^2	K_2 ($\text{g.mg}^{-1}\text{min}^{-1}$)	q_e (mg/g)	H ($\text{mg.g}^{-1}\text{min}^{-1}$)	R^2
0.8927	11.62549	0.0191	0.002551	33.0033	2.77855	0.9989

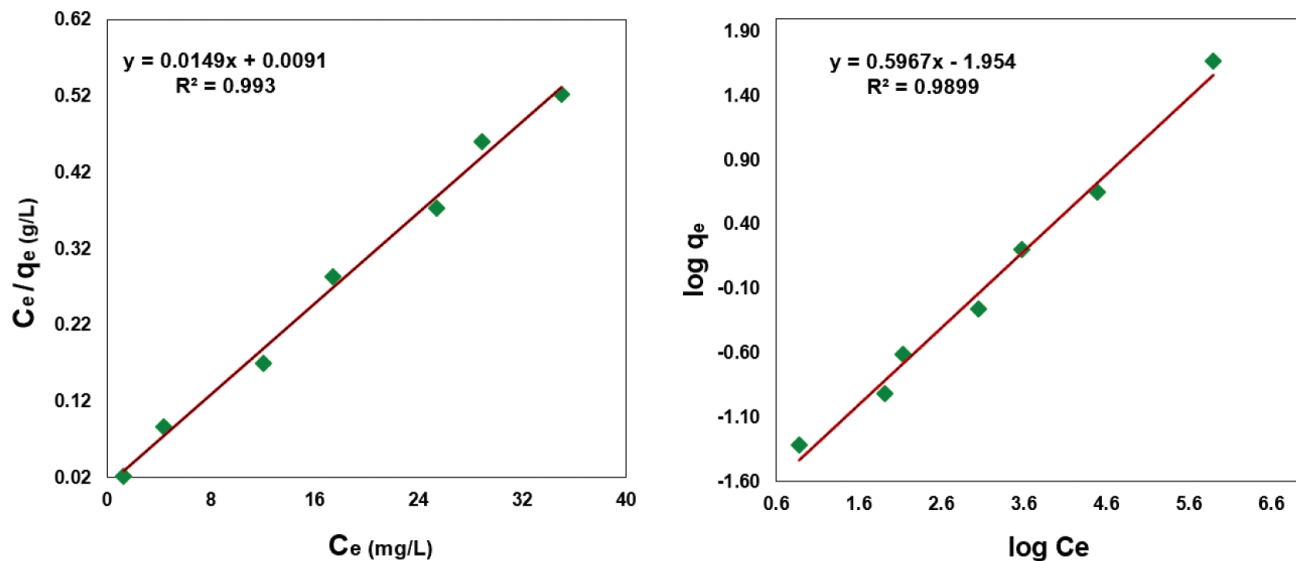


Figure 6: (a) Langmuir and (b) Freundlich models isothermal for the removal of CV dye.

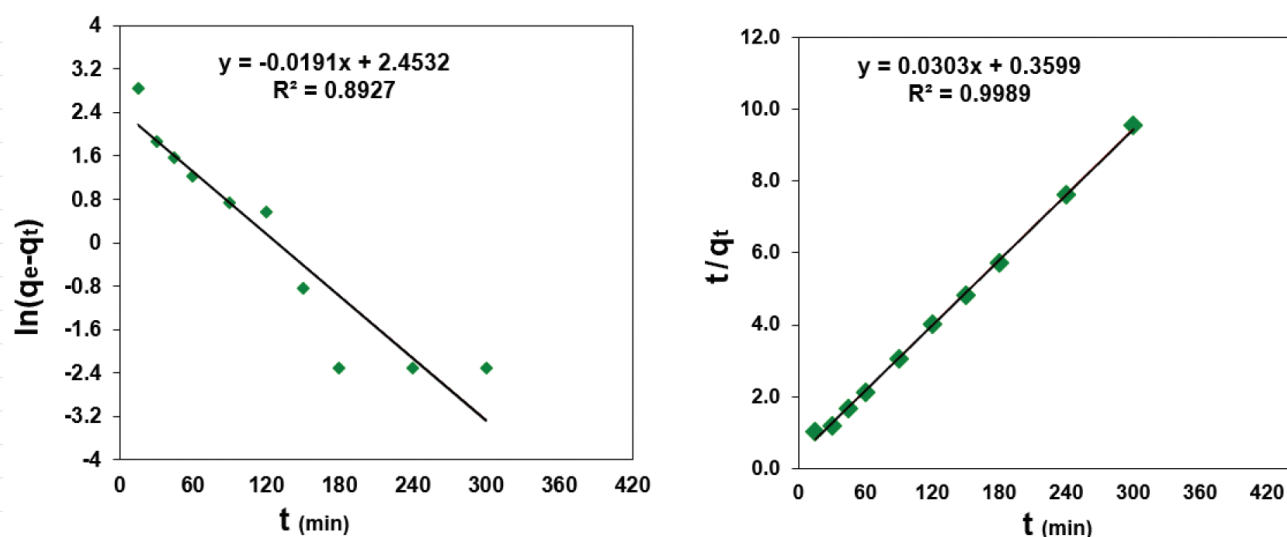


Figure 7: Pseudo-first and pseudo-second order kinetic plot for the adsorption process of CV dye nanocomposite hydrogel.

Table 3: Characteristics of certain adsorbents to the adsorption of CV dye

Adsorbents material	$q_m(\text{mg/g})$	$C_0(\text{mg/L})$	pH	References
Chitosan/PVA Hydrogels	2.7	12	7	Alfuraydi, Al-Harby et al. (2023)
Living roots of the <i>Eichhorniacrassipes</i>	20.84	100	7	Patil (2020)
Zeolite from fly ash	19.6	100	7	Bertolini 2013)
Soil-Ag NP	1.918	10	7	Satapathy 2014)
Hydrogel Nanocomposite/Nanoclay	26	30	7	This work

nanoclay. The optimum pH for removing CV was 3.0, so the removal rate in acidic environments was higher than in alkaline environments and the absorption rate increased over time. Then, after 3 hours, it reached an equilibrium state. The results obtained from this study demonstrate good compliance with the pseudo-second order kinetic model, and the equilibrium data also follow the Langmuir model. In conclusion, it can be inferred that the biopolymer blend of carrageenan and sodium alginate in the presence of bentonite nanoclay is a suitable adsorbent for dye removal from aqueous solutions.

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