

RESEARCH PAPER

Synthesis, Characterization and Adsorption of Crystal Violet (CV) Dye from Aqueous Solutions by Modified Bentonite/Polymethacrylic Acid/Sodium Carboxymethyl Cellulose Hydrogel Nano/Micro Surface

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ABSTRACT

Herein, the synthesis of a novel eco-friendly hydrogel composite was carried out using sodium carboxymethyl cellulose (SCMC) and methacrylic acid (MAA) as major monomers to produce free radical polymerization combined with modified bentonite. Both PMAA added functional groups and increased the density of functional groups on the surface for more efficient reaction kinetics with AAP, while sodium-modified bentonite also significantly improved BET surface area, cation-exchange capacity (CEC), and structural integrity – all contributing to a clearly defined porous structure containing abundant active sites. FTIR, XRD, BET, FE-SEM and TGA were used to characterize the composite and confirm its structural, chemical and morphological properties. The influence of varying adsorbent dosage, contact time, solution pH and temperature were evaluated through batch adsorption tests for its removal on the crystal violet (CV) dye. Through optimization of parameters such as pH (pH = 8) and equilibrium time ($t = 72$ h), a maximum adsorption capacity of the hydrogel was reached, equal to $140.56 \text{ mg}\cdot\text{g}^{-1}$, whereas the optimal removal efficiencies towards toxic metals were computed at $T = 5^\circ\text{C}$. Results obtained from adsorption data fitted the Langmuir isotherm and suggested monolayer chemisorption on a homogenous surface. Thermodynamic calculation indicated $\Delta S^\circ = -9.108 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$, $\Delta G^\circ = +2.523 \text{ kJ}\cdot\text{mol}^{-1}$ and $\Delta H^\circ = -10.485 \text{ kJ}\cdot\text{mol}^{-1}$ that presented the moderate spontaneity and exothermic reaction stage during the crystallization process. The PMAA/SCMC/modified bentonite hydrogel shows a good structural property and an exceptional adsorption stability for CV dye, as proved by above mentioned findings. In summary, this composite exhibited a potential clean adsorbent for high performance wastewater treatment.

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INTRODUCTION

Water pollution by means of industrial activities is still one of the biggest environmental issues globally. Among other potential sources, wastewater from textile, printing and dyeing

industries is particularly problematic due to the high levels of synthetic dyes it may carry (of which many are chemically stable and resistant in degradation). They are released into the aquatic

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systems, often without sufficient treatment that aims to minimize negative ecological implications such as attenuating light penetration, interfering with photosynthesis processes, and demonstrating toxic effects on aquatic organisms. Additionally, the persistent nature of such pollutants in water bodies represents a direct danger to human health through bioaccumulation and chronic exposure [1]. Crystal violet (CV) Fig. 1(A), a frequently applied cationic dye in the textile, biological, and medical fields, has been regarded as one of the most hazardous dye pollutants. Its chemical structure confers high stability with respect to light, heat and microbial degradation and as a result there is significant limitation on its natural removal from the environment. Its occurrence in water systems is of significant concern due to its reported toxic, mutagenic and carcinogenic effects even at low concentrations. The potential for CV to endanger human health in this way underscores the need for effective and sustainable treatment options, as the continuous build-up of CV in water sources places an added burden on ecological systems [2]. Physicochemical and biological techniques including membrane filtration, advanced oxidation processes, ion exchange, coagulation–flocculation, and biodegradation have thus been proposed to facilitate the removal of dyes from wastewater. Techniques for pollutant removal that have been proposed include adsorption on active carbon, ozonation or chlorination, and low-pressure ultraviolet treatment. Although these techniques can be effective in controlling pollution, most of them have limitations such as high operational cost and generation of secondary pollutants, complex processing conditions, or limited efficiency at low contaminant concentrations. In this context, one notable technique that has garnered attention for dye removal is adsorption; due to its simplicity, high efficiency and cost-effectiveness, as well as adaptability to a variety of pollutants [3]. The adsorption process can be affected by the characteristics of adsorbent material and its surface area, porosity, and functional groups. Polymers hydrogels have received great attention as advanced adsorbent materials for wastewater treatment in recent years. Hydrogels are three-dimensional crosslinked polymer networks that can hold a considerable amount of water without dissolving due to hydrophilic functional groups and ordered structures. These materials have several advantages, including tunable porosity and

high swelling capacity, as well as many functional groups (like hydroxyl (-OH) and carboxy (-COOH)) that allow strong interaction with the pollutants [4]. Specific adsorption mechanism in hydrogels often includes electrostatic attraction, hydrogen bond, ion exchange and coordination interactions in certain cases. These unique traits make hydrogels highly suitable for the removal of ionic dyes such as crystal violet. Nevertheless, some drawbacks still exist in the pure polymeric hydrogels such as low mechanical properties or adsorption capacities in certain cases. To address the limitations, large amounts of research have been focused on developing composite hydrogels through combination of inorganic materials into polymer matrix. Of these, bentonite is one type of naturally existing clay mineral that has drawn much interest owing to its specific structural and physicochemical properties [5]. Bentonite is primarily composed of montmorillonite and is characterized by a layered structure, high specific surface area, and excellent cation exchange capacity. These features enable it to effectively adsorb cationic pollutants, including dyes and heavy metal ions. However, the direct application of bentonite in wastewater treatment is often hindered by issues such as particle aggregation, low mechanical stability, and difficulty in separation after adsorption. To address these challenges, bentonite is frequently modified and incorporated into polymer matrices to form composite hydrogels. This approach enhances the dispersion of clay particles within the polymer network, improves mechanical strength, and increases the number of available active adsorption sites [6]. This synergism of interaction between the polymeric chains and clay layers leads to a marked improvement in the adsorption property of the composite. The most essential process that occurs is the transformation of bentonite from its natural calcium form into sodium bentonite using acids. Sodium-assimilated bentonite has a greater swelling volume, superior diffusive capacity in water medium, and a higher ion-exchanging capability than calcium-based that are either similar or type. Such improvements lead to better interaction of the dye molecules and facilitate their removal from aqueous solutions. On the polymer side, acrylic-based materials have been widely applied in hydrogel synthesis owing to their availability and functional versatility. In contrast, Fig. 1 (B) polymethacrylic acid (PMAA) emerges as a promising substitute of regular

polyacrylic acid (PAA), recently acknowledged through several studies. PMAA has more methyl groups than AA, and therefore achieves an altered polymer chain configuration that enables changes in porosity, surface reactivity, and overall network structure. Moreover, the rich carboxylic groups existing in PMAA is beneficial for interacting with various positively charged pollutants through electrostatic attraction and ion exchange mechanisms [7,8]. The addition of sodium carboxymethyl cellulose (SCMC), a biodegradable and hydrophilic polysaccharide, enhances the property of hydrogel composites. SCMC also leads to improved water retention, higher flexibility and the occurrence of additional functional groups taking a part in physicochemical adsorption processes [9]. The interaction between SCMC, PMAA and sodium-modified bentonite results in a highly interconnected network with favorable mechanical properties, even distribution of active sites and high adsorption efficiency. In this respect, the present paper describes design and synthesis of novel bentonite-based hydrogel composites for adsorption of crystal violet dye from aqueous media. This clearly explains the synergistic interaction of sodium-modified bentonite, SCMC and different acrylic monomers (e.g. methyl acrylic acid, methacrylic acid) to produce materials of enhanced adsorptive ability. In this work systematic study on adsorption behaviour under various operating conditions such as contact time, influence of initial dye concentration and temperature specifically is focused to establish a clear perspective regarding adsorption mechanism and efficiency for the fabricated composites. The new structures are

contrasted against more traditional approaches used in designing and altering ionic acrylic polymer networks wherein modern ION based polymer compositions are substituted with alternative, more suitable monomers. The findings will pave the way for new and inexpensive, sustainable and eco-friendly materials to treat dye-laden waste water, which is an urgent requirement in modern day environmental engineering.

MATERIALS AND METHODS

Materials

Sodium carboxymethyl cellulose (SCMC) and N, N'-methylene bisacrylamide (MBA) were supplied by China National Pharmaceutical Group Chemical Reagent Co., Ltd., while calcium-based bentonite (industrial grade) was obtained from Guangxi Tiandong Weiye Bentonite Co., Ltd. . Anhydrous ethanol and potassium persulfate (KPS) were provided by Guangdong Guanghua Chemical Technology Co., Ltd., and Methacrylic Acid (MAA) was purchased from Tianjin Damao Co., Ltd. Sodium hydroxide (NaOH, M.wt = 40 g·mol⁻¹, purity > 98%) was supplied by Loba Chemicals, India, and hydrochloric acid (HCl) was obtained from BDH. The adsorbate, crystal violet (CV, C₂₅H₃₀N₃Cl, 99.9%), was supplied by Merck Pharmaceutical Company, and deionized water was used to prepare all solutions. Except for bentonite, all reagents were of analytical grade and used without further purification.

Synthesis of the Bent-SCMC-poly (methacrylic acid) hydrogel

Pretreatment of Bentonite

Calcium-based bentonite (20 g) was first

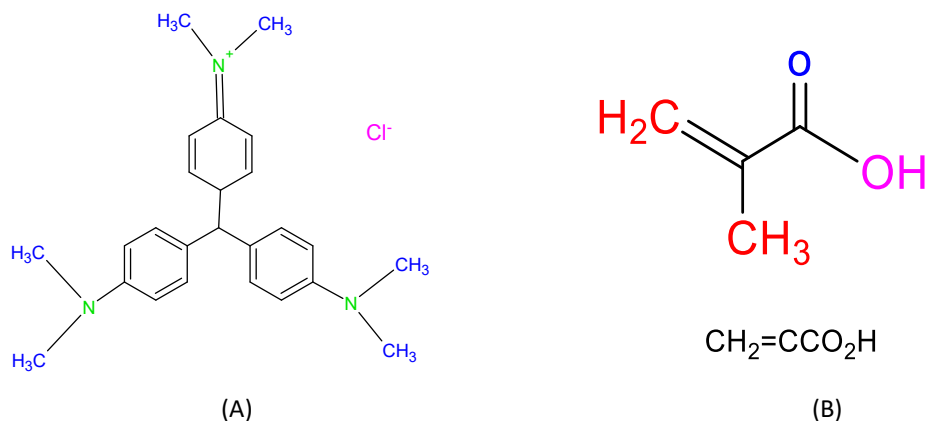


Fig. 1. (A) Crystal violet (CV) dye structure, (B) Methacrylic acid structure

treated with 100 mL of hydrogen peroxide for 24 hours to remove contaminants. The bentonite was then heated at 70 °C for 2 hours to decompose any remaining peroxide, followed by filtration, drying, grinding, and sieving to obtain pure bentonite. For modification, 15 g of the purified bentonite was combined with 0.6 g of sodium carbonate and 80 mL of deionized water. The suspension was stirred in a water bath at 50 °C for 1.5 hours, centrifuged at 500 rpm for 10 minutes, and washed repeatedly with deionized water. The final product was dried at 70 °C until constant weight, ground, and passed through a 250-mesh sieve to obtain uniformly sized modified bentonite suitable for hydrogel incorporation [10].

Synthesis of Hydrogel

For hydrogel synthesis, 10 g of methacrylic acid (MAA) and 0.2 g of sodium carboxymethyl cellulose (SCMC) were initially introduced into a beaker, followed by the addition of 50 mL of distilled water. The solution was placed in a water bath of 80 °C temperature for stirring until complete dissolution. Then, the degree of neutralization was adjusted to 70% using slow addition of 40 wt% sodium hydroxide solution under ice-water bath conditions. Then, 2 g of the pretreated bentonite, 0.004 g N, N'-methylene bisacrylamide (MBA)

as a crosslinking agent, and 0.08 g potassium persulfate (KPS) as an initiator were poured into the reaction system one after another. The mixed solution was sonicated to make all the ingredients uniformly dispersed. Finally, the solution was transferred into a microwave synthesis reactor and submitted to microwave irradiation at 75 °C with 70 W power for 1 h to continue polymerization and form hydrogel. After completion, the hydrogel was cooled to room temperature, washed several times with deionized water, cut into pieces, and dried in an oven at 60 °C until a constant weight was reached [10]. The overall synthesis process is illustrated in Fig. 2.

Characterization

The functional groups and chemical bonding of the Bent-SCMC-PMAA hydrogel were characterized by Fourier Transform Infrared Spectroscopy (FTIR) using a (Tensor II spectrometer, Bruker, Germany). Spectra were measured in the 500–4000 cm^{-1} range using KBr as a reference background. X-ray Diffraction (XRD) with a PANalytical diffractometer using Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$), at 40 kV and 30 mA was used to study the crystalline character of the composite hydrogel and its structural organization. X-ray diffraction data were collected at room temperature over the 2θ range of 5–80°

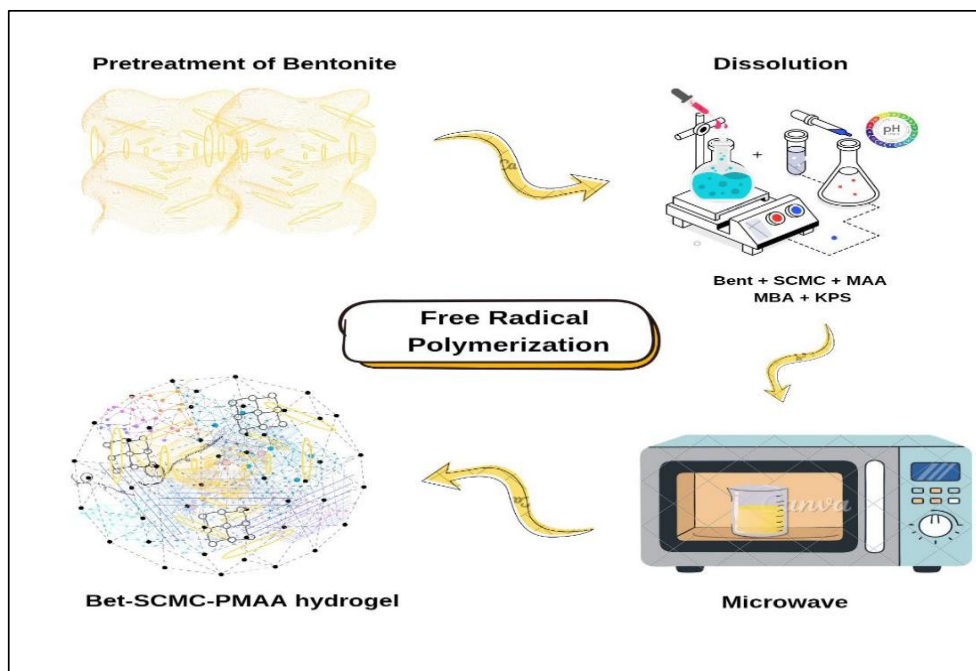


Fig. 2. Synthesis pathway of the Bent-CMC-poly (methacrylic acid) hydrogel.

with a step increment of 0.05° and a scanning time of 1 s per step. The textural properties, such as the surface area and pore structures, were obtained by N₂ adsorption–desorption measurement with a Tristar II 3020 analyzer (Micromeritics, USA). The BET method was employed to calculate the specific surface area of the hydrogel. The surface morphology and microstructural features were analyzed by Field Emission Scanning Electron Microscopy (FE-SEM) (MIRA, TESCAN). Imaging was conducted at 15 kV with a magnification of 150,000×, a working distance of 4.353 mm, and a scale bar of 200 nm, using an in-beam detector. Thermal properties were investigated by Thermogravimetric Analysis (TGA) using an SDT Q600 instrument (TA Instruments). Approximately 3.256 mg of the sample was heated from 25 to 800

°C at a heating rate of 20 °C·min⁻¹ under a nitrogen atmosphere to evaluate its thermal stability.

Batch adsorption experiment

Batch adsorption experiments for the methacrylic acid–based hydrogel composite was conducted by adding 0.02 g of the adsorbent to 50 mL of a crystal violet (CV) aqueous solution with an initial concentration of 50 ppm. The experiments were carried out over a pH range of 4–10 in a temperature-controlled water-bath shaker at temperatures of 5–45 °C, with a fixed shaking speed of 150 rpm. The solution pH was adjusted to the desired value using 0.01 mol·L⁻¹ HCl or NaOH solutions before initiating the adsorption experiments. Following adsorption, the remaining concentration of CV was measured using a dual-

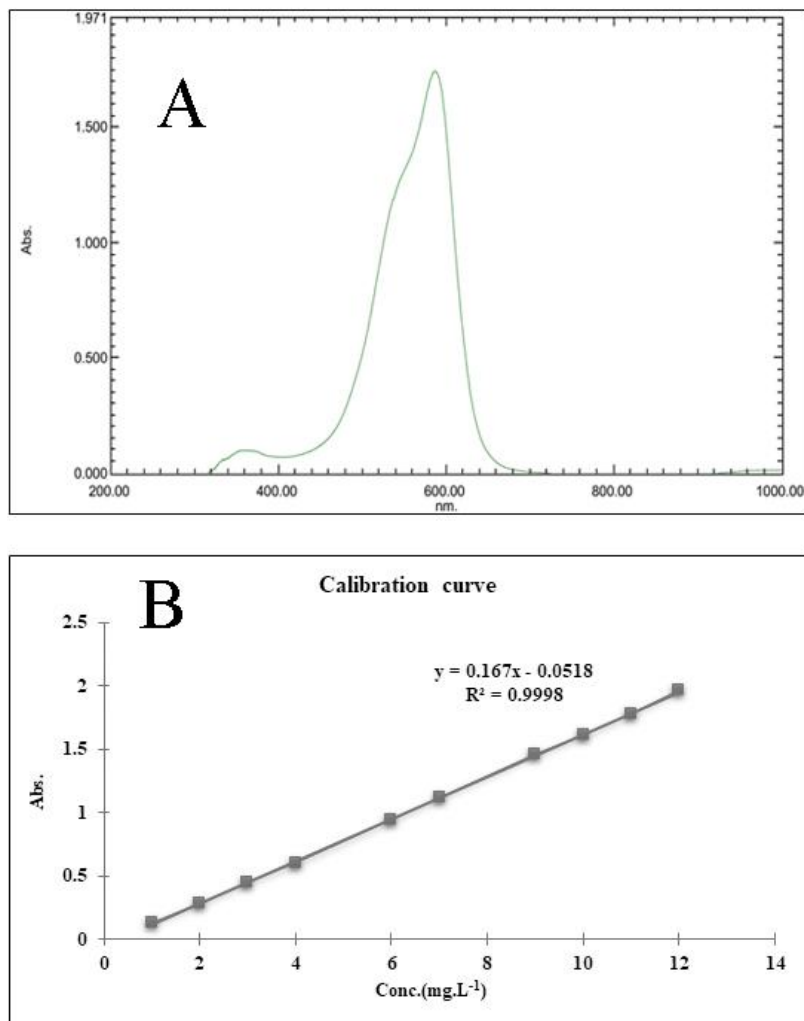


Fig. 3. A) Calculating the CV dye’s maximum wavelength (λ_{max}), B) CV calibration curve.

beam UV-Vis spectrophotometer (TU-1901, Beijing Purkinje General Instrument Co., Ltd.) at a wavelength of 587 nm. The adsorption capacity (Qe) of the methacrylic acid-based hydrogel composite was determined according to Eq. 1.

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

Here, V is the volume of the solution employed (L), Qe corresponds to the adsorption capacity at reaction time t (mg·g⁻¹), C₀ and C_e represent the initial concentration and concentration at reaction time t of crystal violet (CV) dye (mg·L⁻¹), respectively, and m is the mass of the adsorbent used (g).

The removal efficiency of Crystal Violet (CV) dye using the Bent-SCMC-PMAA hydrogel composite was calculated in accordance with Eq. 2.

$$\text{Removal \%} = \left(\frac{C_0 - C_e}{C_0} \right) * 100 \quad (2)$$

The adsorption behavior of the composite and its effectiveness in decolorizing water solutions containing Crystal Violet dye were investigated using the equation.

2.5 Calculating the maximum wavelength (λ_{max})

To evaluate the λ_{max} of CV for methacrylic acid-based composite, we also performed a similar experiment and found the maximum absorption wavelength (λ_{max}) was determined subsequently using 12 mg·L⁻¹ dye solution. As exhibited in Fig. 3A, UV-Vis spectra were acquired in the region of 200–800 nm, which showed an intense absorption peak at 587 nm. This wavelength was used for all later CV concentration determinations in the adsorption studies.

Calibration curve of CV dye

The CV calibration curves were similarly constructed with the methacrylic acid-based composite using standard solutions ranging from 1 to 12 mg·L⁻¹. UV-Visible spectroscopy was used to measure the absorbance at λ_{max} = 587 nm. The calibration curve Fig. 3B was obtained from the linear relationship between absorbance and concentration, which was later employed to quantify CV removal in adsorption assays of methacrylic acid-based hydrogel.

Adsorption isotherms

Adsorption isotherms are crucial to determine

the affinity and distribution of adsorbate molecules across adsorbent surfaces. These models give a logical way to handle the procedure of whether adsorption is helpful for one monolayer coverage or formation of multilayers and will also help in estimating maximum adsorption ability from isotherm models like Langmuir, Freundlich and Temkin. Isotherm analysis offers significant insight into the role of surface functional groups and textural properties in the dye-adsorbent interactions and saturation behavior for Bent-SCMC-PMAA hydrogel composites. This knowledge is crucial for the rational design and optimization of effective adsorbents for wastewater treatment. Consequently, the suitable model (Eqs. 3-5) might be used to represent quantitatively the corresponding adsorption process provided [11,12].

$$\text{Langmuir: } \frac{C_e}{Q_e} = \frac{1}{Q_m} C_e + \frac{1}{Q_m * K_L} \quad (3)$$

$$\text{Freundlich: } \log Q_e = \log K_f + \frac{1}{n} \log C_e \quad (4)$$

$$\text{Temkin: } Q_e = \frac{RT}{b} \ln K_T + \frac{RT}{b} \ln C_e \quad (5)$$

Adsorption kinetics

Kinetic studies for Bent-SCMC-PMAA hydrogel are thus helpful in better understanding the adsorption rate, processes and time required to achieve equilibrium. This kind of study is specifically relevant to the effect of PMAA functional groups and it's combined with sodium-modified bentonite action in respect to the dye uptake behavior. The adsorption kinetics was well described by the classical kinetic models summarized in Eqs. 6 and 7 [13,14].

Pseudo-first order:

$$\text{Log } (q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (6)$$

Pseudo-second order:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (7)$$

The thermodynamics of the adsorption of Crystal Violet onto the Bent-SCMC-PMAA hydrogel were also performed according to Eqs. 8-10 (as shown by The Gibbs free energy (ΔG⁰),

enthalpy(ΔH^0), entropy(ΔS^0). The thermodynamic data offered a comprehensive characterization of the adsorption through spontaneous degree, energetic costs and molecular disorder transition on the hydrogel-solution interface. Study findings confirmed and demonstrated the applicability as well as goodness of adsorption process and the efficiency of methacrylic acid functional group in addition with bentonite synergistic effect on Crystal Violet removal [15].

$$\Delta G^0 = -RT \ln K_c \quad (8)$$

$$K_c = \frac{C_o}{C_e} \quad (9)$$

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \quad (10)$$

where T is the absolute temperature, (R) is the general gas constant ($8.314 \text{ J.K}^{-1}.\text{mol}^{-1}$), (K_c) is the equilibrium constant, and (ΔG) is the change in free energy.

RESULTS AND DISCUSSION

FTIR Analysis

The successful synthesis of the Bent-SCMC-PMAA composite hydrogel (the FTIR spectrum of which is displayed in Fig. 4A) was verified by carrying out Fourier transform infrared spectroscopy (FTIR). construction of a coherent organic-inorganic framework. An intense broad absorption band at 3432.91 cm^{-1} is due to O-H stretching vibrations of the carboxymethyl cellulose hydroxyl groups, -OH structural intermolecular hydrogen bonded water molecules, suggesting far more extensive intermolecular hydrogen bonding within the hydrogel matrix [16]. 2925.76 and 2855.01 cm^{-1} peaks are related with asymmetric and symmetric stretching vibrations of aliphatic C-H groups derived from the polymer backbone, respectively. The presence of a weak band at 2221.19 cm^{-1} can be attributed to overtone or combination bands that are sometimes observed in crosslinked polymer systems [17]. The peaks at 1567.02 , 1445.55 , 1413.97 , and 1385.49 cm^{-1} are attributed to the asymmetric and symmetric stretching vibrations of carboxylate ($-\text{COO}^-$) groups from methacrylic acid units and partially ionized carboxylic functionalities of SCMC confirming successful chemical attachment of PMAA unit on the SCMC chain resulting in crosslinked polymer network formation [18]. The absorption at the bands 1266.93 - 1116.51 cm^{-1} assigned to C-O

stretching vibrational modes of SCMC indicated the structure of polymeric network specific to them. The prominent peak observed at 1042.89 cm^{-1} and other peaks appeared to be observed at 939.25 , 860.94 , and 775.01 cm^{-1} , which are all attributed to Si-O-Si and Si-O-Al stretching vibrations that are characteristic of unaltered bentonite layers in the composite before incorporation indicating that bentonite did not lose its structural functionality after addition. Lastly, the bands at 659.69 , 619.13 , 528.32 and 472.07 cm^{-1} correspond to Si-O vibration deformation bond and the lattice vibration of bentonite [19], serving as an additional indicator that clay is incorporated into the hydrogel matrix. In sum, the FTIR is indicative of successful preparation of a methacrylic-acid-based composite hydrogel with expected functional groups.

XRD Analysis

As it can be seen in the XRD pattern of Bent-SCMC-PMAA composite hydrogel, its structure is semi-crystalline which can be attributed to the presence of both amorphous polymeric network and crystalline bentonite domains. Fig. 4B the basal reflection observed at $2\theta = 9.15^\circ$ ($d = 9.67 \text{ \AA}$) is characteristic of bentonite layers and indicates an expansion of the interlayer spacing, suggesting effective intercalation of polymethacrylic acid and SCMC chains within the clay galleries [20]. The broad diffraction peak centered at $2\theta = 19.80^\circ$ ($d = 4.48 \text{ \AA}$) is associated with the amorphous nature of the grafted polymer matrix [21]. The most intense peak at $2\theta = 26.83^\circ$ ($d = 3.32 \text{ \AA}$) corresponds to silicate structures and residual crystalline domains of cellulose, confirming the structural contribution of both bentonite and SCMC [22]. Additional weaker reflections in the range 44.41 - 62.17° , with d-spacings between 2.04 and 1.49 \AA , are attributed to aluminosilicate phases of bentonite [23]. With this broadening of the diffraction peaks (see enhanced FWHM) a decrease in crystallite size as well as an increase structural disorder can be deduced following graft polymerization. Overall, the XRD results confirm the preparation of an organically-inorganic integrated hydrogel composite with meet expectation for adsorption applications.

FESEM Analysis

The FESEM micrograph of the Bent-SCMC-PMAA hydrogel composite (Fig. 4C) also

manifest a porous, rough, and non-homogenous surface morphologies with an irregularly agglomerated structure. Such surface feature reflects the efficient loading and uniform distribution of bentonite particles into the polymeric hydrogel matrix, leading to an increased roughness and structural diversity [24]. The presence of connected porous regions indicates the formation of a 3D network structure, which is beneficial to enhance the availability of active adsorption sites [25]. In addition, no smooth or dense surface feature are present and interconnected pore structure indicates micro- to nano-scale porosity in the composite matrix [26]. Such porous structure of the hydrogel allows the easy access and deeper diffusion of dye molecules through the interior of the hydrogel leading to improved mass transfer and adsorption kinetics [27]. Accordingly, the synergistic effect of the ion-exchange ability of bentonite and functional groups such as $-\text{COOH}$, $-\text{OH}$ in the polymer network could greatly improve the adsorptive efficiency for cationic dyes [28]. Clearly, the FESEM results confirm that this Bent-SCMC-PMAA hydrogel composite was nanoparticles with highly porous and heterogeneous morphology, which could support for its potential prospects as a good adsorbent material in wastewater treatment.

TG Analysis

Three characteristic decomposition stages are observed in the thermogram of the Bent-SCMC-

PMAA composite (Fig. 5A). Stage I (25–220 °C) is a mass loss of (21.97%, 1.504 mg), ascribing to the physically adsorbed water and interlayer water in hydrogel–clay matrix [29]. Stage II (220–550 °C) feature the largest mass loss (38.95%, 2.666mg), which is attributed to decarboxylation of PMAA/SCMC side groups in sequence with its polymer backbones decomposition [30]. Stage III (550–800 °C) displays an incremental weight loss (%) dominated by (17.34%, 1.187mg), corresponding to the carbon organic matter oxidation and further dehydroxylation reaction of bentonite layers [31]. At 800 °C, the composite remains about (78.26%, 5.356mg) of its weight, which confirms that bentonite plays an important role in promoting thermal stability in a heat-resistant skeleton of the composites core-shell structure.

BET Analysis

As shown in [Fig. 5B], the isotherms of the Bent-SCMC-PMAA hydrogel composite belong to type IV “I”-shape with hysteresis loop, typical of those observed for mesoporous substance [32,33]. This BET linear range, obtained in the relative pressure interval $P/P_0 = 0.05\text{--}0.30$, is clear evidence of the validity of the surface area measurement. The specific surface area of the composite is also higher than that of the acrylic acid–based hydrogel ($53.87\text{ m}^2\cdot\text{g}^{-1}$), indicating enhanced accessibility to more pores. According to the result of [Fig. 5C], the total pore volume was determined $0.412\text{ cm}^3\cdot\text{g}^{-1}$ and the average pore size was around 31.2nm, also

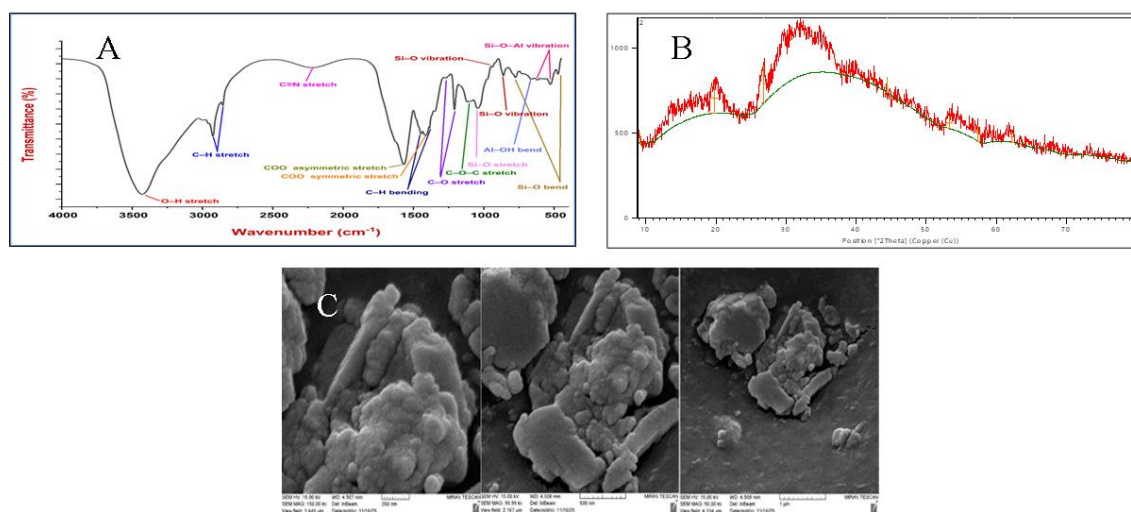


Fig. 4. A) Bent-SCMC-PMAA hydrogel Fourier-transform infrared spectra, B) X-ray diffraction patterns of the Bent-SCMC-PMAA hydrogel, C) Scanning electron microscopy for the Bent-SCMC-PMAA hydrogel.

demonstrating that predominant of mesoporous structures. Also, a broader hysteresis loop indicates better developed and connected pores that could be due to the presence of methyl groups on the polymethacrylic acid backbone [34,35]. These $-CH_3$ groups decrease the packing density of polymer chains, increase the free volume and facilitate to create a more open porous network, and hence better adsorption toward bulky dye

molecules such as Crystal Violet.

The adsorption capability of the Bent-CMC-PMAA hydrogel

Effect of the Adsorbent Mass

The surface morphology and functional groups available for the adsorption of CV dye are crucial in Bent-SCMC-PMAA hydrogel. The pH effects The effect of adsorbent dose was

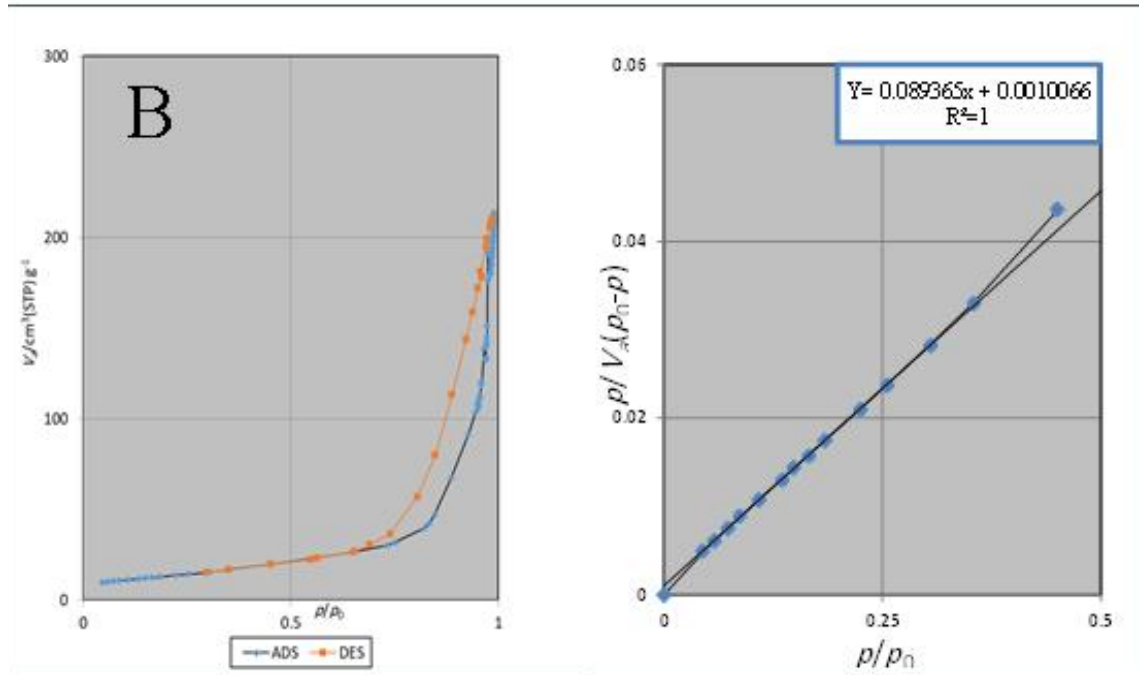
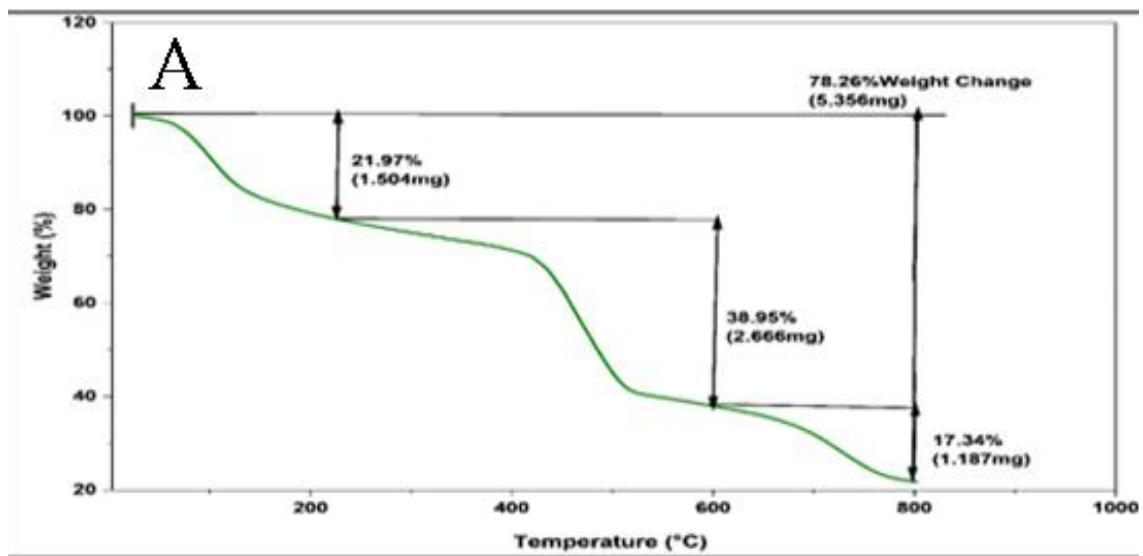


Fig. 5. A) Thermogram of the Bent-SCMC-PMAA hydrogel, B) DES, (C) BET of the Bent-SCMC-PMAA hydrogel.

studied by using different quantities (0.02-0.1 g) of the prepared hydrogel keeping initial dye concentration constant. The removal efficiency was slightly improved from 96.6% to 97.2%, as shown in Figs. 6A and B. The enhancement is explained by an increase in the total surface area and thus adsorption site number. On the contrary, with an increase of dosage, the Q_e decreased. This phenomenon is generally attributed to the high amount of active sites covered and functional groups partially exposed due to particles aggregation at higher adsorbent loading. This aggregation reduces the effective surface area per unit mass and restricts access to internal porosity which in turn leads to lower adsorption capacities. In lower dose (0.02g) operation, the high ratio of dye molecules to binding sites causes an effective occupation of the sites resulting in higher value of Q_e values [36-38].

Effect of the Contact Time of CV

The effect of the contact time for Crystal Violet (CV) adsorption was studied using a range from

1 to 76 h with an initial dye concentration of 20 mg·L⁻¹, temperature at 25 °C, and pH at 7 (Fig. 6C). The adsorption was fast in the beginning due to the high surface area of Bent-SCMC-PMAA hydrogel, which offered plenty potential active adsorption sites for dye molecules uptake [39,40]. Moreover, because of the existing functional groups on hydrogel face, interacting with cationic dye was effectively resulted in improved removing action. With time, the rate of adsorption decreased and equilibrium was achieved at about 72 h with $Q_e = 92.46$ mg·g⁻¹. Overall, the contact time increased adsorption capacity until equilibrium values were attained [41].

Effect of pH

The pH of the solution is an important parameter affecting adsorption performance, it determines surface charge of adsorbent and ionization state of discolored molecules. The present work illustrated the pH influence on adsorption of Crystal Violet (CV) onto Bent-SCMC-PMAA hydrogel (Fig. 7A); for those different values of pH=4,6,7,8 and 10. at

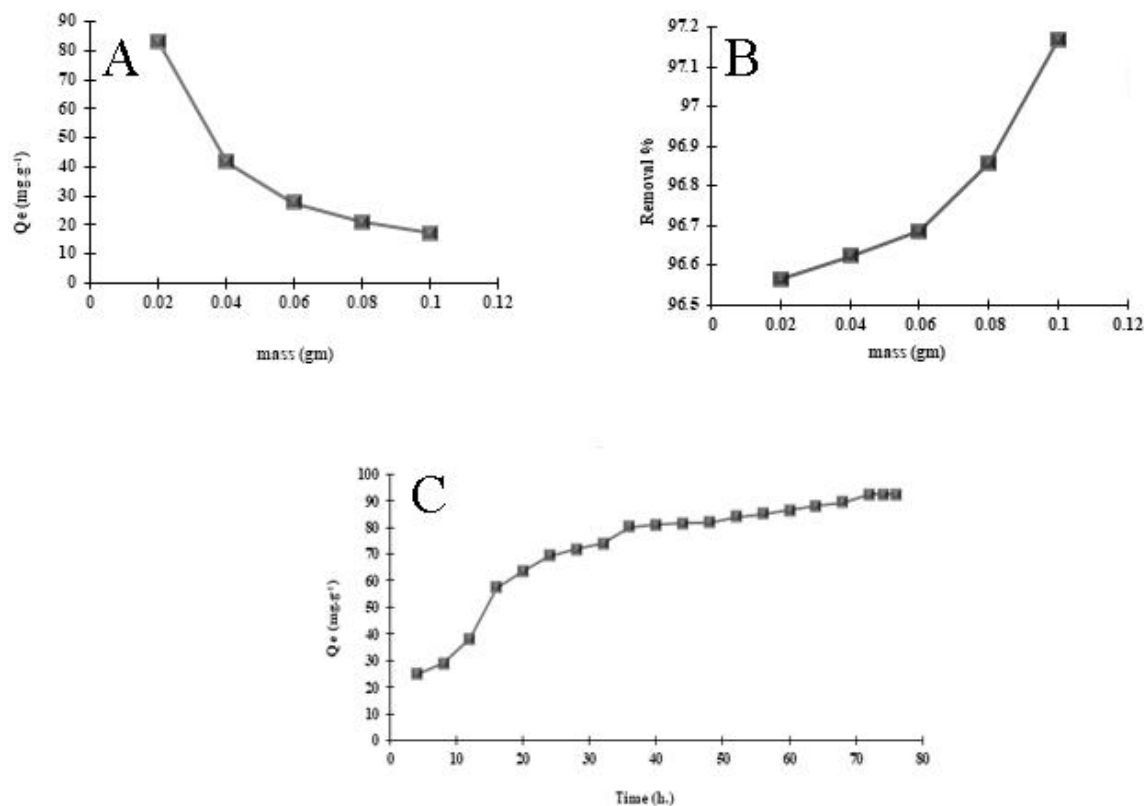


Fig. 6. (A) Effect of mass of the Bent-SCMC-PMAA hydrogel on removal of Crystal Violet dye. (B) Removal rate of crystal violet dye, (C) Effect of CV dye contact time.

pH 8, respectively, followed by pH 7 > pH 6 > pH 10 > pH 4. It is likely that the enhanced electrostatic interaction between negatively charged hydrogel surfaces and positively charged CV molecules in a slightly alkaline to near-neutral environment would further lead to more active adsorption sites available, promote the enhanced adsorption phenomenon [42,43]. At low pH (pH 4), there is protonation of carboxyl and hydroxyl groups, decreasing a net negative load on the surface, causing decreased electrostatic attraction and lower adsorption potential. However, at highly competitive adsorption (pH 10) with hydroxide ions and possibly changes in the swelling behavior of hydrogel may limit dye uptake. This observation also accounts the critical role of pH in regulating surface charge and adsorption behavior in polymer-clay hydrogel system [44,45].

Effect of the Temperature's

The temperature effect of the adsorption of Crystal Violet (CV) on Bent-SCMC-PMAA hydrogel was studied in the 5–45 °C region, with pH at optimum value of 8. As the temperature rose, the adsorption capacity decreased; and maximum removal efficiency was found at 5 °C (Fig. 7B), indicating that the adsorption was an exothermic reaction. It was concluded that thermodynamically this type of reaction is spontaneous as evidenced by its negative enthalpy charge ($\Delta H^{\circ} = -10.48 \text{ kJ. mol}^{-1}$) confirm the exothermic character of the interaction. Moreover, the adsorption process was energetically favorable with a negative entropy change ($\Delta S^{\circ} = -9.11 \text{ J. mol}^{-1. K}^{-1}$), which indicated a decline in the randomness at the solid-liquid interface due to an increase in orderliness of dye molecules upon adsorption on hydrogels

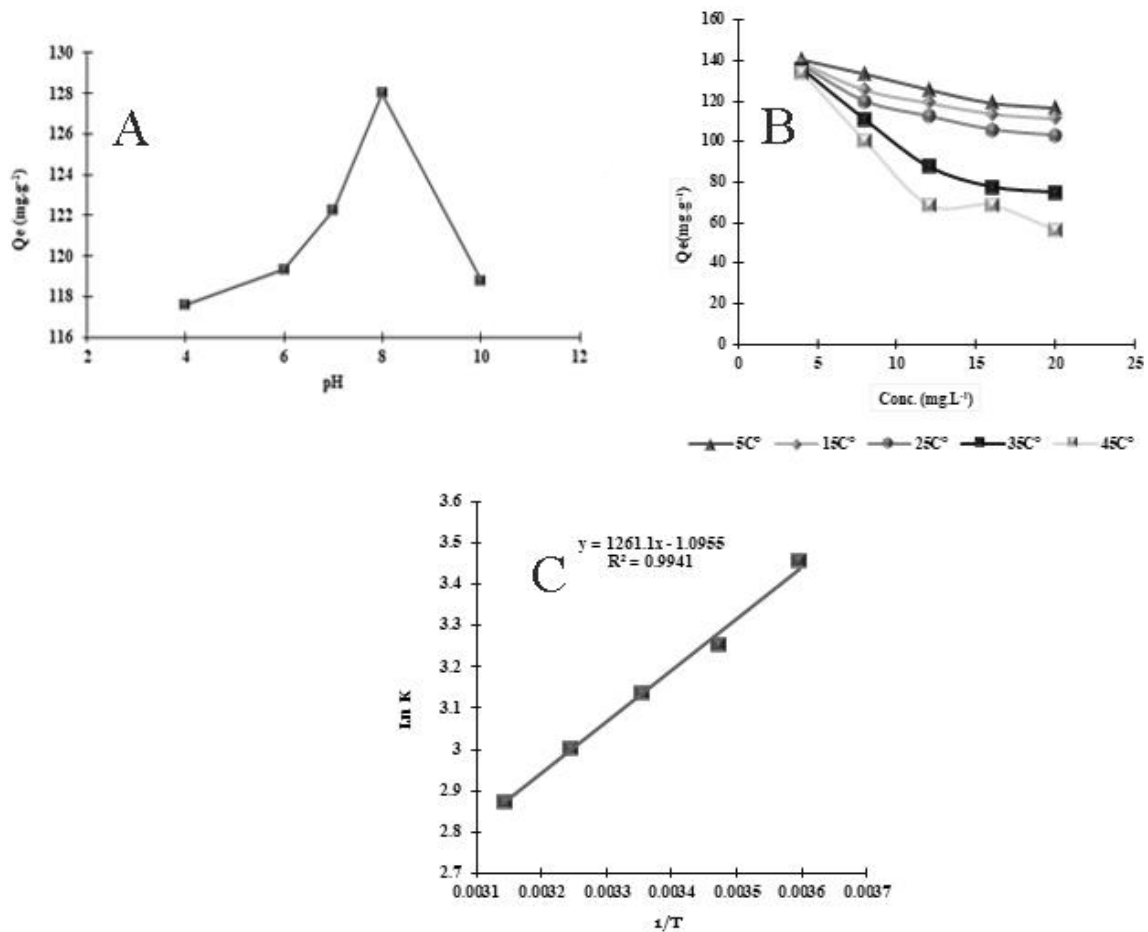


Fig. 7. A) The effect of pH on the removal of CV dye, B) Effect of temperature on CV adsorption, (C) Plot of $\ln K$ against reciprocal absolute temperature for adsorption of CV.

[46]. The positive values of Gibbs free energies ($\Delta G^\circ = +2.52 \text{ kJ. mol}^{-1}$) in all range of temperature considered, which indicate that adsorption is non-spontaneous at the experimental conditions, meaning that an external driving force such as mixing or agitation should be considered to assist in this process. The thermodynamic parameters were determined in $\ln K$ versus $1/T$ linear plot (Fig. 7C) showed that we critical temperature for that of the adsorption dependent on mechanism [47]. In summary, these results confirm the fact that the at low temperatures, Bent-SCMC-PMAA hydrogel has a better capacity for dye removal as in accordance with along with an exothermic physisorption.

Effect of the Ionic strength

Of these, the ionic strength is an important parameter that affects the adsorption performance of CV on Bent-SCMC-PMAA hydrogel because dissolved ions can alter electrostatic interactions and accessibility of active sites. The ionic strength effect was furthermore studied under the following constant conditions: temperature of 5 °C, adsorbent dosage of 0.02 g, pH at 8 and contact time of 72 h; sodium chloride (NaCl) and calcium carbonate (CaCO₃) were selected as representatives mono- and divalent salts respectively [48]. As shown in Fig. 8 (e.g. both salts), the presence of these two solutes produced slight inhibition on CV adsorption.

For NaCl, this observed decrease in adsorption efficiency with increasing salt concentration arises from the competitive interactions between Na⁺ ions and CV molecules for negatively charged sites on the hydrogel surface. Likewise, the presence of CaCO₃ showed a weak inhibitory effect; whereas an increase in Q_e was noted at higher salt concentrations and this may be due to compression of the electrical double layer and therefore increase of dye diffusion toward hydrogel network [49]. All in all, the data show that Bent-SCMC-PMAA hydrogel possesses good tolerance to ionic strength and simultaneous addition of both monovalent or divalent ions only slightly suppresses adsorption maintaining relatively high values of Q_e within the tested range of concentration.

Adsorption isotherms

Crystal violet (CV) adsorption onto the surface of hydrogel at constant temperature is characterized by an adsorption isotherm, which relates the amount adsorbed to the equilibrium concentration in aqueous solution. In order to understand the adsorption behaviour, the experimental equilibrium data were fitted with respect to Langmuir, Temkin and Freundlich isotherm models [50]. From (Table 1), It can be seen that the Langmuir model provided the best fit for the experimental adsorption data

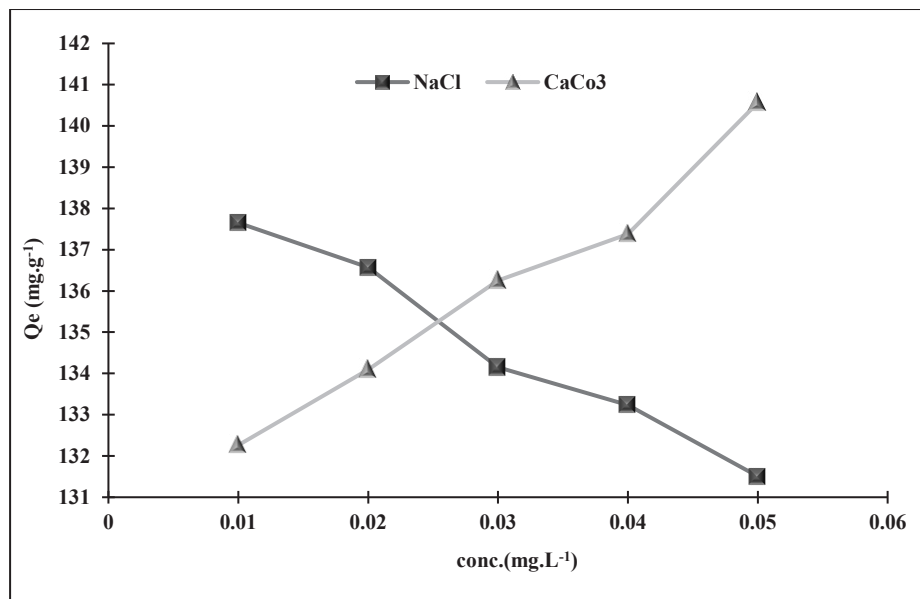


Fig. 8. The effect of ionic strength.



with a coefficient of determination R^2 value of ($R^2= 0.9976$). This indicates that CV adsorption takes place principally as monolayer coverage on a relatively homogeneous surface in which adsorption sites are independent and of equal energy. The Temkin model provided a lower correlation ($R^2 = 0.9720$), suggesting the existence of moderate adsorbate–adsorbent interactions and that the adsorption heat decreases with surface coverage. On the contrary, the Freundlich model has yielded a lower correlation coefficient

($R^2= 0.9635$), indicating less strength of surface heterogeneity in adsorption process. The isotherm plots in Figs. 9 A–C further confirm that monolayer adsorption as described by the Langmuir model is the main mechanism at play controlling CV uptake under the conditions explored [51,52].

Adsorption kinetics

The kinetic study of the crystal violet (CV) absorption on this hydrogel indicates that CV takes a significant time to reach at equilibrium, with

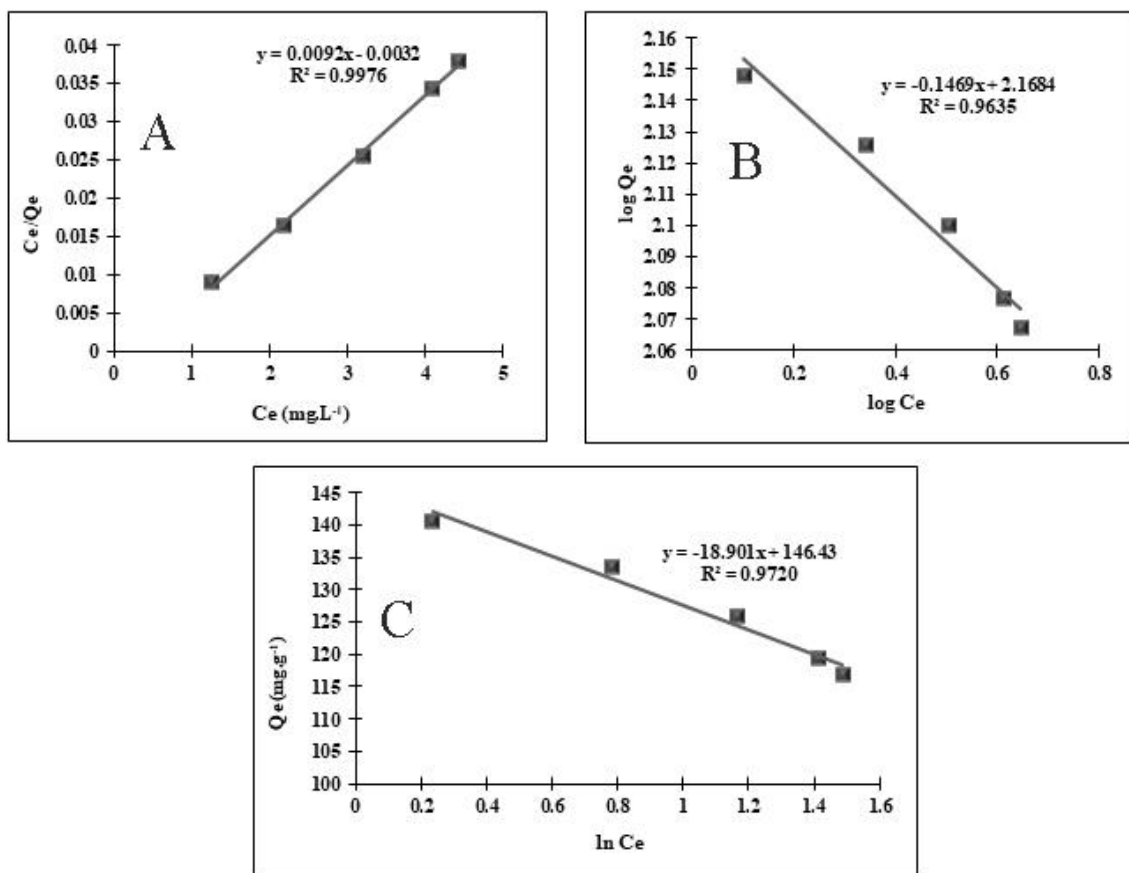


Fig. 9: Adsorption isotherms for Crystal Violet dye adsorption under ideal conditions on the surface of a composite Bent-SCMC-PMAA hydrogel A) Langmuir isotherm, B) Freundlich isotherm, C). Temkin isotherm.

Table 1. Using the parameters of the Langmuir, Freundlich, and Timken equations, the composite Bent-SCMC-PMAA hydrogel was shown to absorb crystal violet dye.

Langmuir equation			Freundlich equation			Timken equation		
K_L	Q_m	R^2	K_f	n	R^2	K_T	b	R^2
-2.875	108.696	0.9976	147.367	-6.807	0.9635	0.000432	-131.0815	0.9720

most CV being taken up during the initial period. The rapid adsorption seen in the beginning can be explained by a large number of active sites available on the adsorbent surface, and most of these active sites become occupied with the passage of time [53]. The pseudo-first-order and pseudo-second-order kinetic models were employed to analyze the experimental kinetic data in order to clarify the controlling adsorption mechanism. Linear relationships were achieved for both kinetic models as seen in Figs. 10A and B. The R^2 value of the pseudo-second-order model ($R^2 = 0.9890$)

was significantly higher than that of the pseudo-first-order model ($R^2 = 0.9356$), confirming that CV dye adsorption onto the hydrogel nanocomposite is best described by a second order kinetic process [54]. From the comparison between pseudo-first order and pseudo second order, it shows that the process of adsorption is mainly controlled by active sites available for adsorption and the interaction between dye molecules and functional groups on hydrogel. These interactions may include electrostatic attraction as well as ion-exchange mechanisms. Thus, the pseudo-second-order

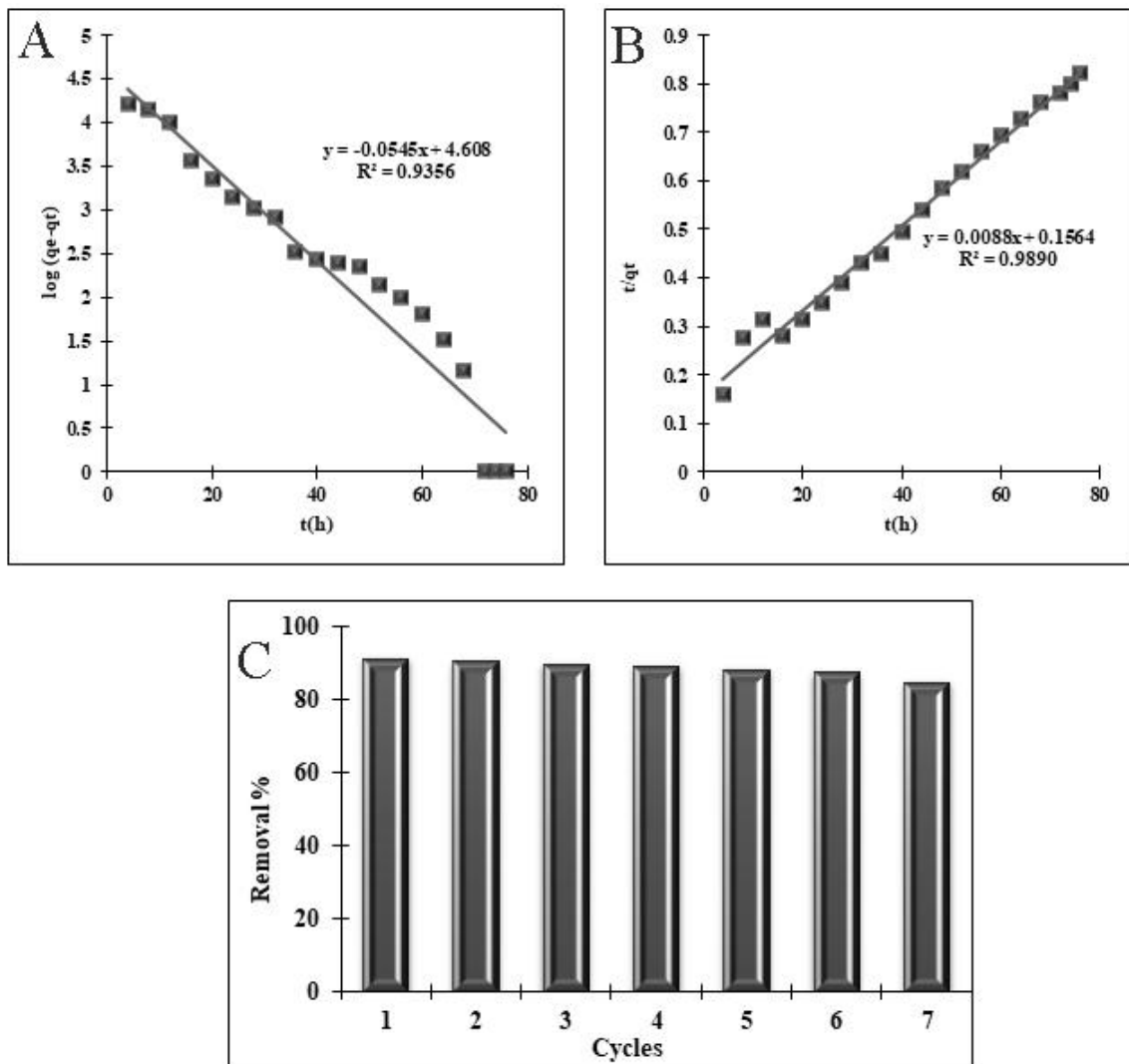


Fig. 10. A) Kinetic model for the adsorption of Crystal Violet dye due to the surface composite Bent-SCMC-PMAA hydrogel at optimum condition (temp 5°C, pH = 8, Adsorbing surface weight 0.02 g and concentration of the dye solution 20 mg. L⁻¹). Pseudo-first-order kinetic model B) pseudo-second-order kinetic model C) Bent-SCMC-PMAA hydrogel reusability research up to seven desorption/adsorption cycles at (adsorption condition: Time: 72 h, pH: 8, concentration: 20 mg L⁻¹, temperature: 5 °C.

kinetic model can be concluded as most suitable to describe the adsorption process of crystal violet on synthesized hydrogel nanocomposite confirming efficient adsorption behavior and stable kinetics [55].

Reusability of regenerated Bent-SCMC-PMAA hydrogel

As shown in [Fig. 10C], the reusability of the Bent-SCMC-PMAA hydrogel was evaluated over seven successive adsorption-desorption cycles for crystal violet removal. The results indicate a gradual decline in removal efficiency from 91.24% in the first cycle to 84.71% in the seventh cycle. This slight decrease suggests that the hydrogel retains a high proportion of its active binding sites and maintains acceptable structural stability even after repeated regeneration processes. The reduction in performance may be attributed to partial blockage or limited loss of active functional groups during successive cycles. Overall, the Bent-SCMC-PMAA hydrogel demonstrates good reusability and stability, highlighting its potential applicability as a sustainable and cost-effective adsorbent for wastewater treatment applications [56-58].

CONCLUSION

A new kind of hydrogel composite based on Bent-SCMC-PMAA was synthesized in this study, and was studied as an efficient adsorbent for the removal of Crystal Violet (CV) dye from aqueous solutions. This composite was formed from sodium carboxymethyl cellulose (Na-SCMC) and poly (methacrylic acid) (PMAA) arranged within the framework of a crosslinked hydrogel network with modified bentonite. This combination was aimed to take advantage of the complementary characteristics of inorganic clay and functional polymers for improving adsorptive performance. The achieved results indicate definitively that the synthesized composite is an effective adsorbent material with appropriate structural stability and ready adaptability with variable operating conditions thus can be exploited for waste water treatment. The adsorption efficiency of Bent-SCMC-PMAA hydrogel toward Crystal Violet reached an impressive 97.2% removal under optimum conditions (i.e., contact time, pH 8, and temperatures of 5 °C with a slurry concentration of adsorbent dosage = 0.02 g, and CV initial concentration = 20 mg·L⁻¹ for a period up to 72 h).

The improved adsorption performance is mainly ascribed to the synergistic effect of composite constituents: On one hand, due to PMAA will produce a large number of carboxylic groups (–COOH/–COO⁻), these strong electrostatic interaction with cationic dye molecules. Na-SCMC improved the hydrophilicity and swelling properties of the hydrogel, implying this could help facilitate dye molecules diffusion to internal adsorption sites. These traits, alongside the advent of new energetic sites as a result of the addition of modified bentonite that improve surface area and porosity or mechanical strength finally increase comprehensive sorption capacities. Adsorption for this composite was relatively less affected by NaCl and CaCO₃, showing good ionic strength tolerance, thus suggesting the suitability of the composite in real wastewater systems. Thermodynamic parameters indicated that the adsorption process is exothermic ($\Delta H^{\circ} < 0$) and associated with a loss in randomness at the solid-liquid interface ($\Delta S^{\circ} < 0$), but positive ΔG° values implied that the process is non-spontaneous under studied conditions. Isotherm and kinetic analyses revealed that the adsorption process conformed to both Langmuir ($R^2 = 0.9976$) model with monolayer adsorption on a relatively homogeneous surface, best describe the kinetic behavior was pseudo-second-order model supporting chemisorption via interactions of active sites. Longer studies are suggested from these implications to address the practical deployment of this hydrogel composite. Future studies should be aimed towards assessing regeneration and reusability of the adsorbent for several adsorption-desorption cycles to evaluate long-term performance and economic viability. Moreover, the adsorption behavior towards a diverse range of contaminants, especially heavy metals and multicolor dyes system, facilitates understanding its effectiveness in complex wastewater mixture matrices. It is also necessary to test the composite under real industrial wastewater conditions, to ensure that its efficiency is not affected by competing ions and organic matter. Structural modifications including the introduction of nanomaterials or functional dopants can also enhance their adsorption capacity and selectivity. Ultimately, scale-up of synthesis and cost-benefit analyses will be important steps towards bringing this material from lab-based research to actual environmental applications.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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