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Synthesis and Characterization of Oxidized Starch-Montmorillonite Composite for Efficient Removal of Crystal Violet Dye from Aqueous Solutions.

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ABSTRACT

An effective adsorbent was synthesized by modifying Montmorillonite (MMT) clay with oxidized starch, aiming to remove crystal violet (CV) dye from aqueous solutions. The chemical modifications in the MMT-oxidized starch composite were characterized using Fourier-transform infrared spectroscopy (FTIR), confirming the successful integration of oxidized starch with MMT. The adsorption performance was evaluated under varying conditions, including initial dye concentration, adsorbent dosage, pH, and contact time. Kinetic analysis revealed that the adsorption process followed a pseudo-second-order model, while equilibrium data were fitted by the Langmuir isotherm, indicating monolayer adsorption. The adsorbent demonstrated a high adsorption capacity of 25.4 mg/g at pH 7 for CV dye. Thermogravimetric analysis (TGA) confirmed the thermal stability of the synthesized material and these findings suggest that oxidized starch-MMT is a promising alternative for the efficient removal of cationic dyes like crystal violet from wastewater, with significant implications for scalable water treatment applications and broader environmental remediation efforts in both industrial and municipal settings.

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1. Introduction

The development of sustainable materials derived from natural resources has gained significant attention in recent years due to the increasing demand for environmentally friendly alternatives to synthetic polymers. Among these materials, starch-based biopolymers represent promising alternatives to synthetic materials due to their cost-effectiveness, biodegradability, and renewable nature. Produced from abundant natural resources, starch offers a sustainable option for developing eco-friendly materials. To achieve the desired functional properties for various applications, starch is typically modified into an amorphous form and combined with synthetic additives. However, a major challenge in this approach is the limited compatibility between starch and synthetic components, which can hinder the performance and uniformity of the resulting materials. This incompatibility remains a key obstacle in the development of truly sustainable starch-based composites [1]-[5]. Furthermore, the high-water solubility and poor mechanical properties of native starch presents limitations of this approach to environment friendly commodity materials, which restrict its applications in advanced materials. To overcome these drawbacks, starch can be chemically modified through oxidation, introducing functional groups that improve its film-forming abilities and interaction with other materials. Oxidized starch, characterized by the presence of carbonyl and carboxyl groups, offers improved reactivity and compatibility with inorganic fillers, enabling the design of hybrid materials with enhanced functional properties [6]-[8].

Montmorillonite (MMT) is a clay mineral widely utilized as an adsorbent across various fields, including catalysis and coatings, due to its large surface area, high cation exchange capacity, availability, and cost-effectiveness [9]-[11]. These features allow MMT to interact with polymer matrices at the

molecular level, potentially reinforcing the mechanical strength, thermal stability, and barrier properties of the resulting composites [12]-[14]. The combination of oxidized starch and Montmorillonite has the promising results to create biodegradable and high-performance materials suitable for adsorption technique for industrial water. Indeed, oxidized starch was selected purposely as a potentially reactive additive. A detailed understanding of the chemical effects of synthesis on the MMT-oxidized starch material is a prerequisite to any attempt to optimize the material properties.

In this general context, the present investigation aimed to develop an effective adsorbent with the following objectives (i) to monitor the progress of starch oxidation using Fourier-transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA), (ii) to assess the influence of intial conditions on the synthesis of the MMT-oxidized starch composite using X-ray diffraction (XRD) and (iii) to quantitatively evaluate the effects of pH, contact time, adsorbent dosage, and initial dye concentration on adsorption performance. The adsorption kinetics were analyzed using pseudo-first-order and pseudo-second-order models, while the equilibrium adsorption behavior was described by fitting the data to the Langmuir and Freundlich isotherm models.

2. Materials and Methods

2.1. Reagents and apparatus

Montmorillonite (MMT) with a purity greater than 95% was purified from raw bentonite sourced in Lam Dong Province, Vietnam. Cassava starch, with an amylose content exceeding 80 % by weight, and crystal violet (CV) dye (CAS 548-62-9, λ_{max} = 592 nm, molecular formula $C_{52}H_{30}N_3Cl$) were obtained from Sigma-Aldrich. A stock solution of CV dye (1000 mg/L) was prepared by dissolving 1 g of the dye powder in 1 L of distilled water, which was further diluted to obtain working solutions with concentrations ranging from 20 to 200 mg/L.

The morphology of the MMT-oxidized starch composite was characterized using X-ray diffraction (PANalytical X'pert, Nertherlands). The chemical structure of the modified material was analyzed over the range of 400 - 4000 cm^{-1} using Fourier-transform infrared spectroscopy (FT-IR, Nicolet iS 50, Thermo, USA). Thermal stability was assessed with a thermogravimetric analyzer (TGA 55, USA) at a heating rate of 20 °C/min, from 38 °C to 900 °C.

2.2. Synthesis of the oxidized starch

Sodium periodate (3 g) was dissolved in hydrochloric acid (0.6 mol/L) in a three-necked flask. Cassava starch was added to the solution to form an 8% starch emulsion. The reaction mixture was maintained in a water bath at 35 °C for 2 hours. After the reaction, the resulting product which was washed three times with distilled water, was subsequently centrifuged at 9000 rpm in 10 minutes. The obtained product was dried in an oven at 50 °C for 24 hours to yield the final dried dialdehyde starch as a white powder [15].

2.3. Synthesis of the MMT-oxidized starch

Prior to MMT-oxidized starch preparation, it's necessary to purify the raw MMT. This process was then realized following the procedure in our previous publication [16]. A total of 2 g of MMT was gradually added to 100 mL of distilled water and stirred continuously for 24 hours to achieve a homogeneous suspension. The mixture was then transferred into a column (15 cm in diameter and 1 m in height) and allowed to settle for 6 hours. After settling, the precipitate was collected, and the purified MMT was subsequently centrifuged at 9000 rpm and dried at 100 °C for 4 hours.

Oxidized starch was dissolved in 50 mL of distilled water and stirred at 50 °C for 30 minutes. Separately, 4 g of MMT was dispersed in 250 mL of distilled water and stirred for 24 hours. The oxidized

starch solution was then added to the MMT suspension at a mass ratio of MMT to oxidized starch of 1:0.6, and the mixture was stirred continuously at room temperature for 2 hours. The MMT-oxidized starch composite was separated by centrifugation at 9000 rpm and washed three times with distilled water. The resulting material was dried at 50 °C for 24 hours and sieved to obtain particles smaller than 200 µm. The preparation of MMT with oxidized starch was conducted following the method reported in our previous study [17].

2.4. Adsorption experiments

Batch adsorption experiments were performed using a KS 3000 IC Control shaker (IKA) at a constant agitation rate. For each MMT and oxidized starch ratio, 50 mL of dye solution with a known concentration was used. The conditions evaluated included varying adsorbent dosages (2 to 24 g/L), initial dye concentrations (20 to 200 mg/L), and initial pH values (4 to 10). The mixtures were shaken at 150 rpm and subsequently centrifuged at 9000 rpm for 10 minutes to separate the adsorbent. The supernatant was analyzed using a UV-Vis spectrophotometer (Cary 50 Conc, Varian) to assess the adsorption efficiency. The percentage removal of crystal violet dye was calculated using the following equation:

$$q_e = \frac{(C_o - C_e)}{m} \text{ x } V \quad (1)$$

$$\text{Dye removal (\%)} = \frac{(C_o - C_e)}{m} \text{ x } 100 \quad (2)$$

where q_e (mg/g) is the adsorption capacity at equilibrium; C_o and C_e (mg/L) represent the initial and final concentrations of crystal violet dye, respectively; V (L) is the volume of the dye solution, and m (g) is the mass of the adsorbent.

3. Results and Discussion

3.1. Charaterization of MMT-oxidized starch

The infrared (IR) spectrum (Figure 1a) shows characteristic peaks for MMT and oxidized starch. For MMT, a broad absorption peak at 3300 cm^{-1} indicates the stretching vibration of the -OH functional group while a peak at 1077 cm^{-1}, corresponds to the bending vibration of the Si-O-Si functional group. On the other hand, oxidized starch exhibits a peak at 1728 cm^{-1}, attributed to the carbonyl functional group, and a peak at 2897 cm^{-1}, associated with the asymmetric stretching vibration of the C-H group in the molecule. When MMT is modified with oxidized starch, the resulting MMT-oxidized starch material exhibits additional peaks at 1149 cm^{-1} and 997 cm^{-1}, corresponding to the stretching and asymmetric stretching vibrations of the C-O-H and C-O-C bonds in oxidized starch. Thus, the infrared spectrum results of the synthesized MMT-oxidized starch material indicate that the oxidized starch molecules could intercalate into the tetrahedral and octahedral clay layers of MMT.

The XRD diffraction patterns of the samples MMT, oxidized starch, and MMT-oxidized starch, are presented in Figure 1b. The MMT sample shows a dictinct diffraction peak at $2\theta = 9°$, characteristic of the d_{001} interlayer spacing, indicating a crystalline structure due to regular tetrahedral and octahedral layer alignment. Oxidized starch, produced by $NaIO_4$ oxidation, lacks a regular crystalline structure, as evidenced by the absence of diffraction peaks. The MMT-oxidized starch composite also shows no significant peaks, suggesting that oxidized starch disrupts MMT's crystalline order.

The thermogravimetric analysis (TGA) results (Figure 1c) reveal differing thermal stability profiles among MMT, oxidized starch, and MMT-oxidized starch composite. Oxidized starch begins to lose water at around 250 °C. As the temperature increases to 500 °C, the remaining mass of the starch is about 25 %, and its structure is completely destroyed when the temperature exceeds 800 °C. For MMT and MMT-oxidized starch, when the temperature reaches 450 °C, the degradation process of MMT-

oxidized starch occurs very slowly, with a mass loss of about 5 % compared to a 3 % loss for MMT. Thermal degradation proceeds slowly up to 600 °C, where the mass reduction is approximately 8 % for MMT-oxidized starch. Between 650 °C and 800 °C, thermal degradation reactions occur without significant mass loss due to the stable structure of MMT and MMT-oxidized starch. The analysis results indicate that the synthesized oxidized starch has low thermal stability, with a rapid mass loss between 200 °C and 600 °C because its structure is composed of polysaccharide chains, which have a low decomposition temperature. In contrast, MMT has a very stable structure formed by interconnected tetrahedral and octahedral layers, creating tightly bonded clay mineral layers. Therefore, MMT exhibits high thermal stability, with a mass loss of about 10 % when the temperature increases from 38 °C to 900 °C. The MMT-oxidized starch material, based on the structures of MMT and oxidized starch, also demonstrates high thermal stability due to MMT's highly ordered crystalline network, which results in minimal mass loss during thermal degradation.

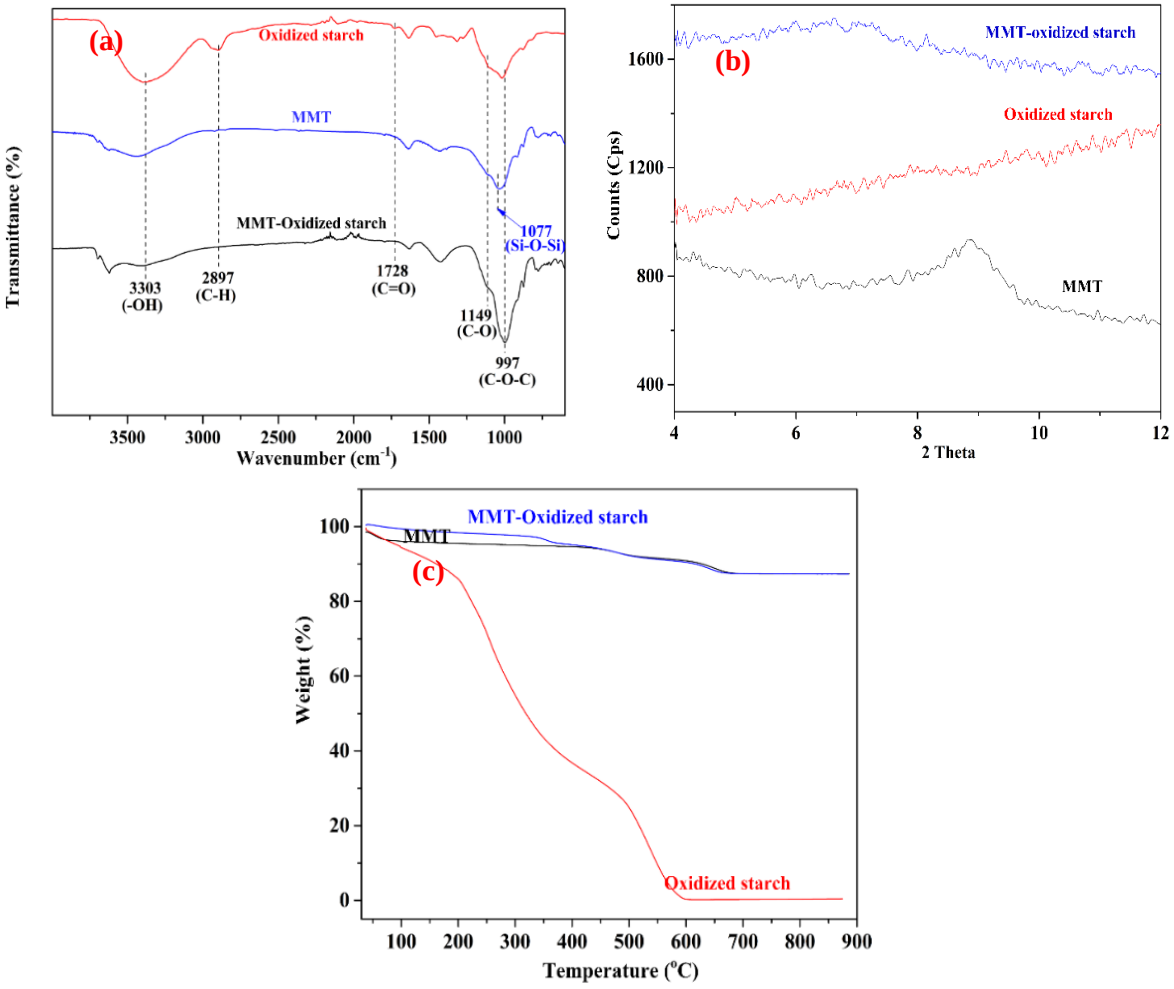

Figure 1. *FTIR spectra (a), XRD diffraction patterns (b), and TGA curves (c) of MMT, oxidized starch and MMT-oxidized starch.*

The morphology of MMT, oxidized starch, and MMT-oxidized starch is presented in Figure. 2. Scanning electron microscopy analysis of the MMT structure reveals a layered structure consisting of thin, uneven flakes that overlap to form multiple layers. In contrast, the oxidized starch features uniformly distributed particles with a clear morphology. After modification, the MMT-starch material exhibits a spherical shape with interspersed particles of varying sizes.

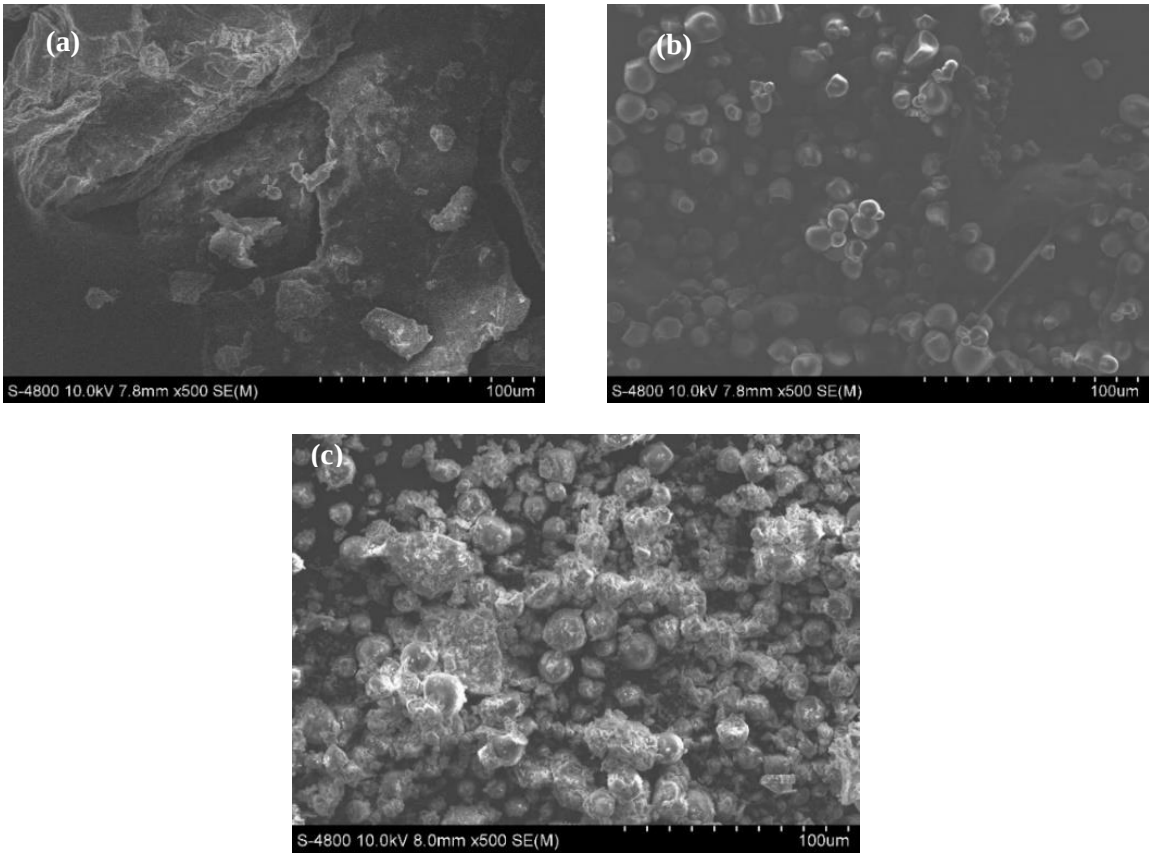

Figure 1. *FTIR spectra (a), XRD diffraction patterns (b), and TGA curves (c) of MMT, oxidized starch and MMT-oxidized starch.*

3.2. Factors Influencing Dye Adsorption

3.2.1. Effect of pH

pH significantly impacts the adsorption efficiency for CV dye on MMT-oxidized starch, with nearly 100% dye removal achieved within a pH range of 4 to 7. At pH levels above 7, the surface of the material becomes negatively charged, leading to decreased interaction with the CV dye. Optimal adsorption occurs at pH 7 (Figure 3a), where ion exchange between the -OH groups on MMT-oxidized starch and the dye enhances adsorption capacity. Beyond this range, surface saturation limits further adsorption.

3.2.2. Effect of Initial Dye Concentration and Contact Time

Adsorption efficiency increases rapidly within the first 30 minutes, reaching equilibrium at 60 minutes (Figure 3b). As the dye concentration increases from 20 mg/L to 200 mg/L, the adsorption capacity rises from 3.22 mg/g to a maximum of 25.4 mg/g. This trend is attributed to the saturation of active sites on the composite surface. The adsorption rate remains stable after 60 minutes due to site saturation, indicating limited capacity for further adsorption at higher concentrations.

3.2.3. Effect of MMT-oxidized starch Dosage

Increasing the adsorbent dose from 2 g/L to 24 g/L enhances adsorption efficiency from 80% to 96% (Figure 3c). However, the adsorption capacity decreases as adsorbent mass increases, from 24.50 mg/g to 2.44 mg/g, due to particle aggregation, which obstructs active sites. This reduction underscores the trade-off between adsorbent mass and adsorption efficiency in batch processes.

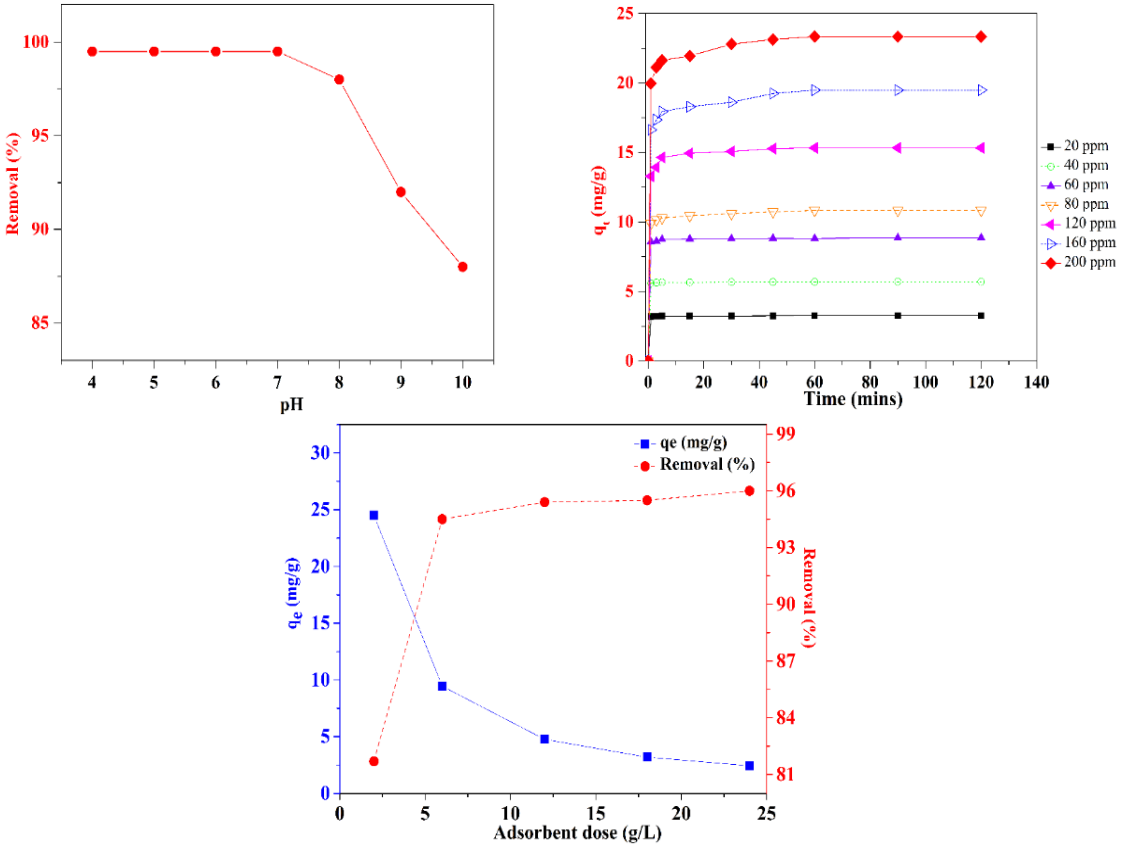

Figure 3. *Effect of (a) pH, (b) initial dye concentration and contact time, and (c) adsorbent dose.*

3.3. Adsorption kinetics

The characteristic of adsorption kinetics on MMT-oxidized starch and CV dye were examined at equilibrium. The experimental data for CV dye on the adsorbent were analyzed using two kinetic models: the pseudo-first order model and the pseudo-second order model, represented by the following equations:

$$\mathrm{Ln}\,(q_e - q_t) = \ln q_e - k_1 t \tag{3}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{4}$$

Where q_t and q_e (mg/g) are the dye adsorption capacity at time t and at equilibrium state. k_1 is the adsorption constant of pseudo-first order model ($gmg^{-1}min^{-1}$) using ln (q_e-q_t) against ln q_e and k_2 is the adsorption constant of the pseudo-second order model ($gmg^{-1}min^{-1}$) using t/q_t against (t).

The adsorption kinetics of CV dye on MMT-oxidized starch were analyzed using pseudo-first-order and pseudo-second-order models (Figures 4a,4b), with adsorption times ranging from 1 minute to 120 minutes and concentrations from 20 mg/L to 200 mg/L. The correlation coefficient of the second-order kinetic model (R^2 from 0.999 to 1) is more linear compared to the first-order kinetic model (R^2: 0.82 to 0.97). Furthermore, when comparing the adsorption capacity at equilibrium calculated by the two kinetic models, the second-order kinetic model is found to be more suitable than the first-order model. Thus, the MMT-oxidized starch material exhibits strong interactions with the CV dye on the adsorbent's surface through chemical adsorption, with the functional groups on the adsorption surface. playing a key role in determining the reaction. This model better represents the adsorption process, indicating strong chemical interactions between the CV dye and functional groups on the MMT-oxidized starch surface.

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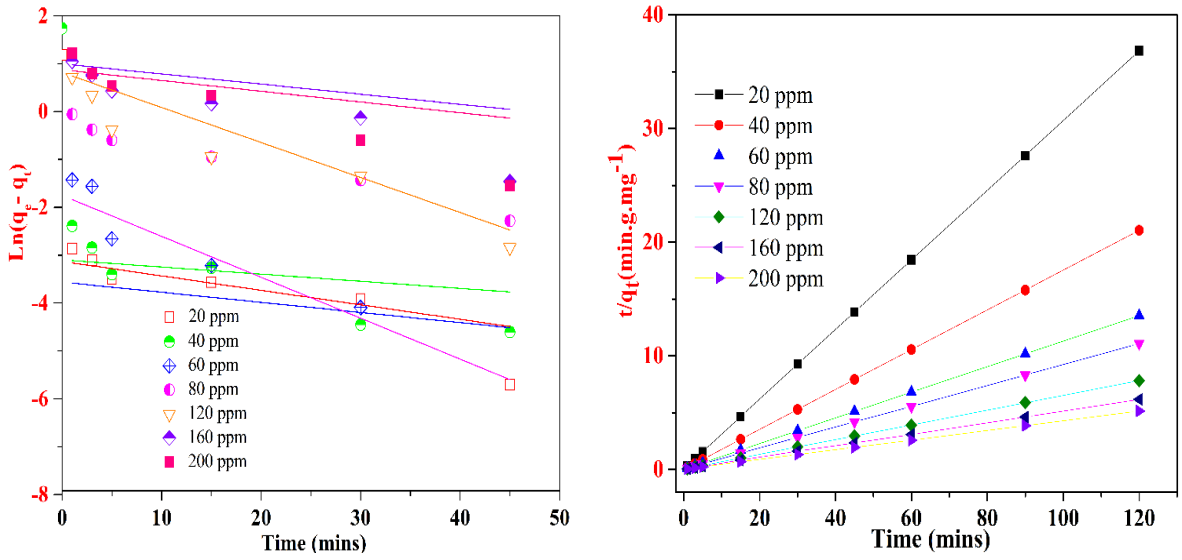

Figure 4. *Pseudo-first-order (a) and Pseudo-second-order models (b) of CV on MMT-oxidized starch adsorbent.*

3.3. Adsorption isotherm

The analysis results are presented in Figure 5, listing the parameters of the two adsorption models along with their corresponding experimental values (Table 1). Furthermore, the correlation coefficient for the Langmuir model is higher (R^2 = 0.998) compared to the Freundlich model (R^2 = 0.984). Therefore, the Langmuir model is more suitable for the adsorption process of CV dye, indicating that the adsorption is characterized as monolayer adsorption

Table 1. *Adsorption parameter values of MMT- oxidized starch in CV dye on Langmuir and Freundlich models.*

Isotherm fitting models	Experimental data	
Langmuir	K_L (L mg^{-1})	0.07
	q_{max} (mg g^{-1})	25.4
	R^2	0.998
Freundlich	K_F (mg g^{-1})	1.73
	n (L mg^{-1})	2.35
	R^2	0.984

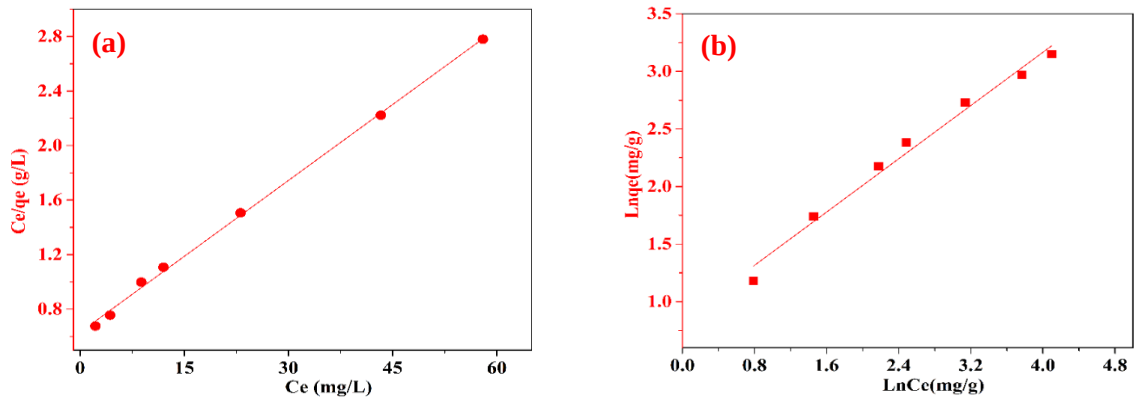

Figure 5. *The adsorption isotherm models of (a)Langmuir, (b)Freundlich on MMT-oxidized starch and CV dye.*

The dominance of the Langmuir isotherm over the Freundlich model in this study suggests monolayer adsorption of crystal violet (CV) dye on the oxidized starch-MMT composite. This outcome can be linked to the structural characteristics of the MMT-oxidized starch, where the availability of uniform active sites may lead to a monolayer adsorption mechanism. Highlighting this can reinforce the composite's potential as an efficient adsorbent for specific pollutants that require controlled, predictable adsorption behavior. Since the Langmuir model suggests uniform adsorption sites and no interactions between adsorbed molecules, it implies that CV dye molecules adhere individually rather than clustering on the adsorbent. This aspect could open up further studies on how the material's surface chemistry can be tuned to optimize interactions with other pollutants.

4. Conclusions

In this study, the oxidized starch-MMT composite was prepared by the thermo-physicomechanical method. The synthesized composite was characterized by developing techniques such as FTIR, XRD, FESEM, TGA were also studied the removal of crystal violet dye from aqueous solutions. The results indicated that the dispersion of the oxidized starch and MMT in this material, enhanced the functional properties in the composite due to the significant interaction between the MMT-oxidized starch and CV dye. Additionally, the results indicated that the Langmuir adsorption model best described the adsorption behavior of the material, with a high adsorption capacity of 25.4 mg/g at pH 7. The adsorption process followed a pseudo-second-order kinetic model, demonstrating the suitability of oxidized starch-MMT as an alternative adsorbent for the removal of crystal violet dye from aqueous solutions.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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