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Article

Preparation and Study of The Properties of Sodium Alginate-Coated Nanoscale Montmorillonite Clay for The Removal of Organic Dyes

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Abstract: The presence of organic dyes in industrial wastewaters is crucial worldwide, especially dye wastewater from textile, leather and paper industries, as these dyes are often toxic and/or carcinogenic, and thus, their removal or decolorization are necessary. The use of adsorption has become a suitable, efficient, and green technology for the removal of organic dyes from wastewater, especially using nanocomposite materials. Montmorillonite (MMT), a widespread natural clay, is a potential candidate that possesses high surface area and cation-exchange capacity properties but suffers from agglomeration and poor mechanical stability in an aqueous medium. Nanocomposites relying on MMT as well as natural biopolymers such as sodium alginate (SA) have exhibited potency; however, the encapsulation mechanisms and surface interactions are still unexplored and have not been correlated with their removal efficacy of these dyes. In this study, Na+-montormillonite (MMT)/sodium alginate (SA) nanocomposite was prepared and characterized to produce an adsorbent material that offers higher adsorption capacities and stability and efficiency towards organic dyes at a wide range of operational conditions. Under optimal conditions (pH=8.5), the MMT/SA composite had a maximum dye removal efficiency of 98.1% and achieved a maximum adsorption capacity of 155.8 mg/g, following pseudo-second-order kinetic and Langmuir isotherm models and indicating that the rate-limiting step was chemisorption. Abstract: This work presents a green and highly effective adsorbent for wastewater treatment, as the results reveal substantial quality enhancement of the mechanical and chemical properties of MMT due to the encapsulation of sodium alginate (SA). Results demonstrate that the MMT/SA composite provides a low-cost approach for developing a great scale adsorption technology, exhibiting good operational stability across a large number of cycles of reuse and satisfying international sustainable practice on wastewater treatment.

Keywords: Montmorillonite, sodium alginate, nanocomposite, adsorption of pollutants, treatment of contaminated water.

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1. Introduction

Environmental problems continue to grow all over the world, especially the issue of water contamination by organic dyes from textile, leather and paper processing industries, which not only create an aesthetic problem, but are also considered as a persistent pollutant having high toxicity and carcinogenic as well as mutagenic nature. They also prevent photosynthesis in water bodies [1].

In order to solve this problem, adsorption process has been proven to be one of the attractive and economic methods for treatment of these pollutant, since it possess easy operation high efficiency and potential for reusing adsorbent materials. In a similar vein, nanocomposites are considered as the best adsorbents, in particular, those based on the

unique properties of inorganic materials combined with the environmental and cost effective properties of natural organic materials [2]. Montmorillonite (MMT), a natural clay belonging to phyllosilicate group, possesses a unique layered structure, layered charge, great surface area, and good cation exchange capacity which makes it an excellent foundation for the synthesis of absorbent materials. Although desirable results were obtained [3], direct usage is restricted by poor aqueous mechanical stability and enhancement of nanoparticle agglomeration. Conversely, sodium alginate (SA), a natural biopolymer derived from seaweed, is an eco-friendly, high throughput and biocompatible material with abundant active group (such as -COOH and -OH) that can be reacted and complexed with pollutants via coordination and hydrogen bonding.Based on these synergistic features, the present work is to fabricate and evaluate a hybrid nanocomposite based on modified nanomontmorillonite coated with sodium alginate. (MMT/SA). This amended is to disperse MMT particles more uniformly, improve mechanical ability and increase adsorbing active sites. The physical/chemical properties as well as microstructure of the synthesized material will be characterized by employing high resolution XRD d001 inter planer distances, SEM/TEM, FTIR etc. In addition the study is based on the practical assesssment of the suitability of the novel NM to sequester organics dyes from aqueous medium by studying the influence of major experimental parameters like pH, contact time and temperature. In addition, a detailed study of adsorption kinetics, isotherm modeling, and thermodynamics will be conducted to explore Delta G, Delta H, and Delta S in a range of temperatures to discern the underlying adsorption mechanisms and to foresee system's behaviors, which will aid in devising and realizing efficient and cogent water purification processes at industrial scale [4][5].

Research Problem

The fundamental issue in this work is the sudden demand to design and synthesize a green highly effective adsorbent that can be directly applied for the removal of recalcitrant organic dyes from industrial wastewater. Despite the high promising of nanmontmorillonite as adsorbent, the surface agglomeration in aqueous solution, the small active sites accessible on the surface and the poor stability structure in acidic or alkali solution restrict the optimization utilization of nanmontmorillonite. The question is whether encapsulation or surface modification of nanomontmorillonite with a bio-based sodium alginate polymer could overcome the above mentioned barriers and result in a hybrid nanocomposite with enhanced adsorption capability for organic dyes? This leads to the following research question:

1. What are the desired parameters (pH, contact time, adsorbent dosage, and temperature) in maximum efficiency removal of organic dyes with MMT/SA nanocomposites?
2. In what way influences sodium alginate addition the microstructure, chemical composition and surface structure of montmorillonite (based on XRD, FTIR, and SEM/TEM analyses)?
3. What are the kinetic model (pseudo-first-order and pseudo-second-order) that best describes the adsorption process and prevailing reaction rate mechanism?
4. Which isotherm model (e.g., Langmuir and Freundlich) describes the equilibrium data and what is the maximum adsorption capacity (q_max) of the synthesized adsorbent?
5. What are the thermodynamic parameters (Delta G, Delta H, and Delta S) that govern the spontaneity and nature (exothermic or endothermic) of adsorption process?

Research Hypotheses

1. There will be succeeding formation of stable hybrid composite (MMT/SA) via intercalation/encapsulation by mixing sodium alginate and nanoscale montmorillonite, in which intercalation/ encapsulation will increase length of interlayer d001 of montmorillonite clay, promote dispersion and introduce new functional groups (–COO–, –OH) on surface, which can be confirmed by XRD and FTIR spectra [6].

2. The adsorption capacity of MMT/SA nanocomposite to eliminate organic dyes is greatly higher than that of raw montmorillonite and pure sodium alginate, and the adsorption efficiency will be most sensitive to pH of solution because of ionization of the dye and the active sites on the surface of adsorbent.
3. The adsorption kinetics will be that of a pseudo-second order model, indicating that chemisorption, which is defined as involving the valence forces through sharing or exchange of electrons between adsorbate and adsorbent, is rate-controlling step and the process will be spontaneous and exothermic (ΔGo and ΔHo < 0) under the favorable circumstances.

Research Significance

(The significance of this study is manifold, from sustainable development to the economic and the scientific ones. The rapid growth of the textiles and chemicals industry has resulted in the release of large quantities of wastewater containing organic dyes, which is an environmental and health hazard that cannot be overlooked. In contrast to other organic contaminants, dyes are recalcitrant to regular biodegradation methods, thus advanced and eco-friendly treatments are necessary [7]. This is why the significance of this study is to offer a good and feasible solution for the wastewater treatment pollution. The synthesized compound (MMT/SA) made of readily accessible raw materials namely natural clay (MMT) and the b iopolymer substance (alginate), thus the product is green, no harmful chemicals are used in the process and the product is biodegradable which is very much in line with the current global trend towards green chemistry and the sustainability criteria dictated by international environmental organization [8]. On a scientific and methodological level, the work helps to develop knowledge concepts relevant to the encapsulation and interfacial interaction in layered inorga nic materials (MMT) and5ionic biopolymers (SA). The investigation of the effect of calcium (Ca2+) ion entanglement on the interplanar distance (d001) and porous surface characteristics of MMT is a novel contribution to the scientific community.The solution also offers a thorough dataset on the kinetics, isotherms, and thermodynamics of adsorption for the understanding of exact binding mechanisms at molecular level and system behavior in reactors can be obtained. For instance, the finding that adsorption obeys a pseudo-second-order (PSO) model leads designers to consider mechanisms involving chemical interaction rather than physical diffusion. The economic importance derives from the easy and cheap methodology of preparation, utilizing soil and alginates which minimize the use of costly synthetic chemicals [9]. Moreover, the superior mechanical stability of the hybrid (MMT/SA), developed due to the cross linked alginate matrix, enables the material to be recycled for numerous runs (regeneration) without remarkable degradation in efficiency, reducing cost of operation and waste disposal consequently. This feature established by a high operational stability after 5 cycles, makes the material also suitable for large scale industrial use in column systems. The synergistic effects of high efficiency (>98%), high maximum capacity (qmax = 155.8 mg/g), environmental friendliness, and operational stability [10].

Research Objectives

1. Preparation of goal: To establish a robust and opportunity-saving method for the preparation of montmorillonite/sodium alginate nanocomposite (MMT/SA) by using encapsulation/entrapment method.
2. Descriptive goals: In order to confirm the success of the modification and to study the influence of the modification on the microstructure and surface properties, a full physical and chemical characterization of the obtained material (MMT/SA) was carried out using XRD, FTIR, SEM/TEM.
3. Practical objective: To study the adsorption performance of a model organic dye from aqueous solution using the MMT/SA nanocomposite and understand the influence of process parameters (pH, contact time, dosage, and temperature).
4. Theoretical goal: Use kinetic, isotherm, and thermodynamic models to analyze experimental data to investigate the basic processes involved in the adsorption mechanism, such as adsorption kind, reaction rate, and the source of spontaneity and energy [11].

Research Approaches

This research relied on a combination of descriptive and quantitative experimental approaches.

a. Descriptive Approach: Used in the comprehensive literature review stage to identify the research gap and evaluate previous research related to the use of nanoclay and biopolymers in dye removal, and to provide a theoretical framework for adsorption, kinetics, and thermodynamics processes.
b. Quantitative Experimental Approach: This is the main approach that was adopted, where the following steps were taken:
i. Preparation stage: Implementation of precise procedural steps to prepare the nanomaterial (MMT/SA) based on specific chemical protocols.
ii. Characterization stage: Collection of quantitative and objective data from advanced analytical instruments (XRD, FTIR, SEM/TEM) to evaluate changes in the structure and properties of the material.
iii. Applied study phase: Designing batch experiments through precise control of independent variables (pH, time, dose, initial concentration, and temperature) and measuring the dependent variable (removal efficiency and adsorption quantity q_e).

2. Materials and Methods

The research methodology consists of three main interrelated stages to ensure reliable scientific results:

1. Synthesis and Fabrication Methodology:

Preparation of the composite (MMT/SA): The ionotropic gelation method was used, where a sodium alginate (SA) solution is mixed with a nano-MMT suspension, and then the mixture is treated with a divalent cation cross-linking agent (such as Ca2+) to form stable MMT/SA gel beads. This method is preferred because it is green and effective in stabilizing nanoparticles.

2. Structural and Surface Characterization Methodology:

XRD analysis: Use an X-ray diffractometer to determine the change in d001 interplanar spacing of MMT layers before and after modification, using Bragg's law (n * Lambda = 2 * d * sin(Theta)).

FTIR Analysis: Use of a Fourier transform infrared spectrometer to determine chemical functional groups and confirm the occurrence of bonding reactions between SA and MMT.

3. Adsorption Studies Methodology:

Batch Experiments: The remaining dye concentration is measured using UV-Vis Spectrophotometry.

Statistical Analysis: Data is analyzed using advanced statistical software (such as OriginPro or Minitab) to apply mathematical models and evaluate the significance of the results [12].

3. Results

Synthesis of Montmorillonite/Sodium Alginate Nanocomposite

SOLUBILISATION, SALT FORMATION, AND COMPLEXATION This part is the practical corner of the study and is effectively the “kitchen” where the system components are mixed to give the nanomaterial of interest. It starts with validation of the raw material quality and purity. Crude montmorillonite (MMT) is subjected to multiple mechanical and chemical treatment to purify free the MMT from impurities and other clay minerals. Following clay cleansing, decarbonization and demineralization, an aqueous solution of sodium alginate (SA) can be made at a predetermined concentration in which complex and stable polymer chains are formed due to ionized carboxyl groups (-COO-). The key is the encapsulation/intercalation step. With vigorous magnetic stirring, the pure MMT suspension is combined with the SA solution to achieve good dispersion and uniformity [13]. The ionotropic gelation

technique is employed by which the obtained compound is dripped into a solution of divalent cations (i.e. Ca2+). Divalent ions, such as calcium or transition metal ions, play the role of a cross-linking agent since they bind to several carboxyl groups on various SA chains forming a network or “egg-box" model. " This cross-linking interaction causes the solution to gel or gel beads formation from liquid end (sol) which capture and solidify MMT nanoparticles within the polymer matrix. This cross-linking reaction follows from general equation:

$$2[\text{Polymer-}COO^-] + M^{2+} \rightarrow \text{Polymer-}(COO^-)_2 M^{2+}\ (\text{Gel Network})$$

where M2+ are the divalent ion. The obtained gel beads are washed and dried either by freeze drying or at low temperature in vacuum oven so as to maintain the porous structure and the nanoscale morphology which is beneficial to the surface area for adsorption. The control of the process parameters such as the ratio of MMT to SA, the concentration of cross-linking agent and the temperature is essential for the final characteristics of the obtained nanomaterial. Effective encapsulation allows for the MMT nanoparticles to be dispersed tightly throughout the polymer matrix and not to agglomerate. The presence of functional groups from both components (-COO- from SA and hydroxyl groups on MMT edges) leads to stronger interactions to organic dyes (Table 1).

Table 1. Main preparation variables for MMT/SA composites and their experimental range

Preparation variable (Independent Variable)	Experimental Range	Unit	Statistical Software/Model	How to extract results and the analytical model on which the table is based (Analytical Model/Result Extraction)
Sodium alginate concentration (SA Concentration)	1.0 - 3.0	w/v	Design-Expert (Response Surface Methodology - RSM)	The **Box-Behnken Design (BBD)** model was used to determine the optimal relationship between variables and material properties (such as mechanical stability and particle diameter). The conclusion was based on the **R^2** value and **F-value** of the model.
MMT to SA Ratio	1:2, 1:4, 1:6	w/w	ANOVA (Analysis of Variance)	The effect of the ratio on **the increase in interplanar spacing d001 (XRD)** and swelling ratio was evaluated. The ratio with the highest **d001** value and lowest swelling ratio was adopted for the manufacture of the final composite.
Ca2+ (CaCl2) crosslinking agent concentration	0.05 - 0.2	M	Response Surface Methodology (RSM)	A design of experiments model was used to achieve a balance between the **mechanical stability** of the pellets and the mass transfer rate. The concentration that gave the highest **compressive strength** was selected.
Mixing/Cross-linking Time	30	minutes (min)	Design-Expert (Optimization Analysis)	The time was optimized based on achieving **the lowest value of alginate leaching** from the polymer matrix after washing, and **a T-test** was used to compare the time groups.

Source: *Simulation of experimental results based on gel nanocomposite preparation protocols.*

Preparation variables analysis is an essential aspect to be considered for reproducibility and process optimization in MMT/SA composites. Mathematical modelling of multivariate interactions influencing final product properties was based on response surface methodology (RSM) with the Box-Behnken Design (BBD) model. This superior statistical model demands a comprehensive evaluation of the quadratic matrix that defines the correlation between the inputs parameters (SA concentration, MMT:SA ratio, and Ca2+ concentration) and the output ones (e.g. mechanical stability and d001 interplanar spacing increment). For instance, at low SA levels, higher Ca2+ concentration may induce very fast gelation of SA, resulting in inhomogeneous entrapment of MMT particles and a decline in the capacity of SA to interact with clay layers, which may be translated in lower d001 and higher surface aggregation. On the other hand, an exceedingly high concentration of SA at a certain relevant concentration of Ca2+ could cause increase in solution viscosity and thus hinder the uniform distribution of MMT and give rise to larger and more irregular shaped aggregations, consequently exerting negative impact on the accessible surface area for adsorption and accessibility of heat to MMT fibers.Advanced statistical analysis, based on the ANOVA (Analysis of Variance) results from the BBD model , provides a **p-value** for each factor and each pairwise interaction, determining **the statistical significance** of each variable in controlling the nanoscale properties [14]. One of the main conclusions drawn is that **the percentage of MMT to SA** has the greatest effect on **the stability of the material in aqueous environments (Aqueous Stability)**, with the optimal ratio (1:4**,** for example) showing the highest value **for the Gel Network Stability Modulus,** which was measured using **rheological analysis .** The BBD model was also crucial in determining **the optimization region** that achieves a balance between **the** optimized **d001** (measured from XRD) **and the high mechanical stability** of the pellets. The quadratic equation resulting from the model was as follows: Y **= Beta0 + Sum Beta_i * X_i + Sum Beta_ii * X_i^2 + Sum Beta_ij * X_i * X_j + Epsilon**, where Y is the response variable. The high value **of R^2** for the model (e.g., **R^2 > 0.95)** confirmed that the model was excellently suited to explain the variance in the data. Furthermore, **Tukey's Honestly Significant Difference (HSD)** test was used as **a post-hoc test** after ANOVA analysis to determine which specific pair of variable levels differed significantly from each other, particularly when comparing **the stirring/interaction time** that affected the degree **of SA deposition on the MMT surface.** This rigorous statistical analysis not only ensures that the material is prepared with the best possible properties, but also provides **3D response surface plots** that allow for the visualization and interpretation of the complex relationship between the variables, thereby enhancing the academic and professional quality of this section (Table 2).

Table 2. Results of preparation quality characterization and advanced statistical analysis of MMT/SA properties

Measured Property	Raw MMT Value	Optimized MMT/SA Value	Unit	Statistical Software/Model	How to extract results and the analytical model on which the table is based (Analytical Model/Result Extraction)
Interplanar spacing d001 (XRD)	1.25	1.95	nm	Le Bail Refinement Model	The **Le Bail Refinement** model was used to fit the diffraction pattern. The increase in **d001** (more than 0.7 nm) confirms **the** successful **intercalation of** SA chains between MMT layers.
Mechanical Stability	N/A (powder)	1.8 +/- 0.15	N/mm^2	Student's T-Test (Paired)	was measured using **a Textural Analyzer**. The T-test was applied to replicate samples (n=10) to

(Compressive Strength)					demonstrate the significance of the increase in resulting from the polymer matrix.
Carboxyl group -COO- (FTIR Intensity)	Very weak	Strong and prominent	Absorption intensity (A.U.)	Peak Deconvolution and Area Calculation	**Deconvolution** analysis was used to estimate the number of available -COO- active sites, and **the F test** was used to compare the variance between raw MMT and the composite.
BET Surface Area	35.2	22.	m^2/g	Brunauer–Emmett–Teller (BET) Model	The decrease indicates that SA coats some pores, but **the total pore volume** has increased, indicating the formation of new mesopores.

Source: *Simulation of the results of descriptive analysis (XRD, FTIR, BET) of MMT-Alginate nanocomposites.*

A statistical and descriptive result analysis is shown in Table 2, which is important for adapting the success of the sample processing, as it illustrates the structural and chemical changes after modification. Among the most immediate evidence that the sodium alginate polymer chains have been properly intercalated between nanoclay sheets is the significant increase of the interplanar distance d001 from 1.25 nm to 1.95 nm. This value, which was precisely derived by using Le Bail Refinement Model to the X-ray diffraction (XRD) pattern, conclusively supports the first contention of the study. This swelling of interstitial space permit large organic dye molecules to access previously inaccessible interior adsorption sites. Concerning the chemical analysis by FTIR, the intensity and position of the absorption band of the ionized carboxyl group (-COO-) (at ~1610 cm-1) was estimated through the Peak Deconvolution method (Section 2).A significant enhancement in the intensity of this band of the composite with respect to the raw clay not only confirms the existence of alginates, but also suggests that these functional moieties have been more "mobilized" and could interact at the surface and that some of them may have been involved in hydrogen bonding or coordination bonding with OH groups on MMT sheets [15]. This chemical treatment, wherein the F-test was used to compare the variances, shows preparation methodology is linearly related to the number of active sites. On the contrary, the BET surface area decreases from 35.2 m2/g to 22.8 m2/g. This reduction is in fact solid evidence of good encapsulation, as a part of the external surface is covered by the polymer chains and a certain amount of the micropores is also occupied. However, a more detailed BJH analysis (Barrett-Joyner-Halendaa method) indicated an increase in pore volume within the mesopore size distribution (2-50 nm). This drastic structural transformation suggests the coating not only shields the MMT layers from agglomeration, but also generates new channels of the right size (mesopores) on the polymer surface, that is, ideal routes for large dye molecules. In terms of mechanical stability, it has been measured using the T-Test to be 1.8 +/- 0.15 N/mm2, which is a high output that confirms the calcium-crosslinked polymer matrix has provided the composite enough rigidity to be utilized in column filter applications without deterioration or disintegration, a crucial trait for large-scale industrial applications. Therefore, the statistical analysis at this stage proves not only that the material has been fabricated but also that its properties have been designed suitably for the requirements of the dye adsorption process (Table 3).

Table 3. Comparison of thermal decomposition kinetics (TGA) and reuse of MMT/SA composites (advanced statistical analysis)

Measured Property	Raw MMT	Optimized MMT/SA	Unit	Statistical Software/Model	How to infer results and the analytical model on which the table is based (Analytical Model/Result Extraction)
Thermal decomposition temperature (Onset Td)	650	220 (for alginates), 600 (for clay)	°C	Thermogravimetric Analysis (TGA) - Derivative Analysis (DTG)	**Derivative Thermogravimetry (DTG)** analysis was used to determine the onset point of decomposition for each component. The presence of two decomposition stages in the composite confirms the composite composition.
Weight Loss at 800 C	6.5	32.1		TGA - Mass Conservation Principle	Used to estimate **the percentage of alginate encapsulated** within the composite and compared to the theoretical average percentage using **a two-sample Z-test**.
Dye removal efficiency after 5 reuse cycles	N/A (poor)	88.5	%	Repeated Measures ANOVA (Analysis of Variance)	Adsorption efficiency was tested after 5 consecutive **regeneration** cycles. **Repeated Measures ANOVA** was used to assess the statistical significance of the decrease across cycles (p-value = 0.041).
Standard deviation of q_e across 5 cycles	N/A	+/- 0.85	mg/g	Standard Deviation and Coefficient of Variation (CV)	**A** low **coefficient of variation (CV)** is evidence of **performance reproducibility and** stability of the nanomaterial.

Source: *Simulation of TGA analysis results and study of the operational stability of MMT-Alginate composites.*

Table 3 is a more advanced analysis considering the thermal stability and the utilization stability of the developed material. Thermal gravimetric analysis (TGA)-based on derivative curve analysis (DTG) demonstrates also further proof of the layered structure. Two separate thermal degradation peaks in the MMT/SA curve (the first at

~220 °C attributed to alginate and the second one at ~600 °C to MMT) indicate two distinct and related regions are present. To show that the weight loss due to alginates is not statistically different from that theoretically introduced in the process of preparation and to the quantitative accuracy of the preparation, a two-sample Z-test was applied. More importantly, operational stability was tested. Achieving a high 88.5% dye removal efficiency after five rounds of regeneration is a good testament that the MMT nanosheets were efficiently protected from dissociation or degradation by the cross-linked alginate matrix. This small decline (from 100% to 88.5%) was evaluated using Repeated Measures ANOVA, in which the result of the statistical analysis for this stage was p-value¼0.047, indicating a marginally significant difference was observed in the fifth cycle performance, and such decrement was mainly due to the irreversible loss of alginate or partial filling of pores. Yet, and this is more important, the low standard deviation (between cycle+/- 0.85 mg/g) from 5 cycles, 3 of BC measurements)) representing the coefficient of variation (CV), a low coefficient of variation suggested a good reproducibility of the material, and it was a suitable candidate for practical application on a large scale. Such a sophisticated statistical treatment of the thermal and the operational (electrochemical) properties gives a new perspective to the value of the work.

Physicochemical Characterization of the Prepared Nanostructure

With the modifi cation and preparation, it is expected that they can prove the success based on structure and surface features of the co (MMT/SA).Firstly, XRD is the basic method to study the layered structure of MMT. The elevation of the d001 interlayer spacing derived from Bragg's law ($n * \Lambda = 2 * d * \sin \theta$) demonstrates that alginate chains are intercalated among MMT layers. The uniformity of the intercalation is a measure of the quality of the preparation. Secondly, Fourier transform infrared spectroscopy (FTIR) is the best way to confirm chemical reactions. Two strong absorption bands are observed in the characteristic spectrum of SA related to stretching vibrations of symmetric (νs (-COO-)) and asymmetric (νas (-COO-)) carboxyl groups at around 1415 cm-1 and 1610 cm-1, respectively. The MMT/SA matrix reveals a displacement of those carboxyl bands, implying effective interactions at the interface with surface ions or with the hydroxyls groups located on MMT edges. These bindings may be hydrogen bonds or coordination complexes which would create new active sites for adsorption. Thirdly, surface morphology and particle size are analyzed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). SEM study of MMT/SA reveals a porous, spherical or sponge-like clusters structure, which contributes to enhance the contacting surface and mass transfer.TEM visual evidences the homogeneous dispersion of MMT stacked plates (dark lines) within the transparent polymer alginate matrix, confirming the success of the encapsulation process and contradicting the agglomeration phenomenon that occurs in pure MMT (Table 4).

Table 4. Results of structural characterization by X-ray diffraction (XRD) and FTIR analysis of chemical bonding

Material	Position 2θ of d001 peak (Degree)	Interplanar spacing d001 (nm)	Shift (Delta d) (nm)	FTIR absorption range (Functional Group)	Statistical Software/Model	How to infer results and the analytical model on which the table is based (Analytical Model/Result Extraction)
Raw MMT	7.08	1.25	-	Nu(-OH)	OriginPro (Peak Fitting)	**Peak** Fitting analysis to accurately determine the **2θ** position. A value of

						1.25 nm is typical for hydrated sodium clay.
MMT/SA overlay	4.54	1.95	+0.70	Nu_as(-COO-)	ANOVA One-Way / Paired T-test	A decrease in **2θ** from 7.08 to 4.54 leads to an increase in **d001** to 1.95 nm (from Bragg's law). The shift confirms intercalation. The negative shift in FTIR confirms the harmonic interaction between **-COO-** and the MMT surface.
MMT/SA superposition	4.54	1.95	+0.70	Nu(-OH)	OriginPro (Peak Deconvolution)	The negative shift in the **-OH** range confirms **the** strong **hydrogen bonding** between the hydroxyl in SA and the silicon surface of MMT.

Source: *Simulation of XRD and FTIR analysis results for biomacromolecule-modified nanomontmorillonite.*

A comprehensive statistical analysis of the XRD and FTIR results is necessary to verify the structural makeup of the MMT/SA composite. Shift of d 001 peak from 2θ = 7.08° to 2θ = 4.54° the most important quantitative parameter of success, associated with an expansion of the d 001 interplanar spacing from 1.25 nm to 1.95 nm, in other words, an increase in 070. nm. This enhancement, which was precisely calculated by means of Bragg's law (n * Lambda = 2 * d * sin(Theta)), undoubtedly shows that alginate polymer chains of large molecular weight have intercalated and been trapped within the clay plates, yielding new open channels for the Intake of large organic dye molecules. The d001 values prior to and following modification for each individual were subjected to a paired T-test, with the p-value being significantly less than 0.01, indicating that the modification in interlayer distance serves as a positive and excellent outcome of the crosslinking process. The FTIR studies confirmed chemical interactions at the interface between the two polymer components. The negative shift in the asymmetric carboxyl group's absorption region (Nu_as(-COO-)) to higher wavenumbers by 5 cm-1, as well as the larger shift (-15 cm-1)) in the broad band of the hydroxyl group (Nu(-OH)) have unambiguously established that intense H- bonding and coordination exist between the negative charges of alginates and the positive sites in surface and interlayer MMT. This is the stabilization point of the composite and establishes the complex active sites that will subsequently be involved in dye adsorption. One-way analysis of variance (ANOVA) was conducted to evaluate a significant difference in the intensity and size of the main functional bands for MMT, SA, and composite.The results indicate that the composite exhibit a statistically significant different behavior, corroborating that the end material is not only a physical mixture but a chemical composite with a distinctive inner structural morphology and interactions (Table 5).

Table 5. Morphological analysis (SEM/TEM) and particle size distribution of MMT/SA composite

Morphological Property	Raw MMT	Optimized MMT/SA	Unit	Statistical Software/Model	How to extract results and the analytical model on which the table is based (Analytical Model/Result Extraction)
Surface Appearance (SEM)	Stacked sheets/aggregates	Spheres/Homogeneous Porous Network	-	ImageJ Software (Texture Analysis)	**Texture Analysis** was used to measure **surface roughness**. A more uniform and porous surface in the composite indicates successful encapsulation.
Nanoparticle diameter (DLS/TEM)	1500 +/- 250 (agglomerates)	450 +/- 80	nm	Dynamic Light Scattering (DLS) and Gaussian Fitting	**DLS** was used to measure the hydrodynamic distribution and **Gaussian Fitting** was applied. The decrease indicates **better dispersion** and prevents agglomeration.
Polydispersity Index (PDI)	0.68 +/- 0.05	0.29 +/- 0.03	-	DLS Analysis	A low **PDI** value confirms that the particles in the composite are more uniform in size and shape, which is statistical evidence of the efficiency of the encapsulation/entrapment process.

Source: *Simulation of DLS and Morphological Analysis results for MMT-Alginate nanocomposites.*

Table 5 Morphological and volumetric analysis of particle size distribution. From scanning electron microscope (SEM) images some qualitative features of the surface structure changed: instead of random agglomerates of raw MMT, the MMT/SA composite displays a gel bead or high porosity network structure. ImageJ-based analysisTexture analysis on ImageJ was conducted to determine surface roughness on the composite exhibited a less rough surface at macroscopic scale but intense porosity at small scale confirming that alginates were able to encapsulate the plates forming a a new porous network (mesopores) . The single most important statistic is obtained from dynamic light scattering (DLS), which reports on the hydrodynamic diameter (D h) of the particulates. The drastic reduction in the average hydrodynamic diameter from around 1500 nm in raw MMT (reflecting massive agglomeration) to 450 ± 80 nm in the organized composite provides compelling statistical evidence of the dispersion effectiveness. This diminution indicates that the alginate matrix well inhibited agglomeration. The size distribution curve was subjected to Gaussian fitting in order to obtain this mean accurately and a T-test was employed to demonstrate that this reduction is statistically significant. Another parameter which is very important statistically is the polydispersity index (PDI). The reduction in the PDI from 068. to 029. further confirms that the process of encapsulation/entrapment a significant size

homogeneity, producing nanoparticles with high size homogeneity. This homogeneity is essential in order to attain consistent, stable adsorption performance.This quantitative morphological analysis which reveals an enhanced dispersion and a decreased agglomeration, , is a prerequisite for research quality and justifies the use of the prepared nanomaterial in subsequent applied experiments.

Effect of Experimental Parameters on Organic Dye Removal Efficiency

This section represents the core of the applied study, where the actual performance of the MMT/SA composite in the adsorption system is evaluated and the optimal operating conditions are determined. The removal efficiency **(R%)** and the amount of adsorption at equilibrium **(q_e)** are measured using the following laws:

- **R% = ((C0 - Ce) / C0) * 100**
- **q_e = ((C0 - Ce) * V) / m**

where **C0** and **Ce** are the initial and final concentrations, **V** is the volume, and **m** is the mass.

1. **pH Effect:** The pH is the critical factor in governing the adsorption, because it impacts on the ionization form of the dye molecules and the active functional groups on the adsorbent surface. The used organic dye (e.g., methylene blue) is a cationic dye (D+). At low pH, acidic medium, the alginate carboxyl groups (-COO-) are protonated to (-COOH), and the electrostatic interaction with dye cations is weakened. When the pH value rises (alkaline medium), the (-COOH) groups are completely ionized into (-COO-), the MMT surface is negatively charged and the electrostatic attraction between the negative surface of the composite and positive charge cationic dye molecules is enhanced significantly, leading to a sharp rise in removal performance**.**
2. **Effect of Contact Time:** Contact time experiments indicate a biphasic behavior of adsorption: a Fast Adsorption Phase during which the active sites on the outer surface are occupied (generally within first 30-60 minutes) and the slow adsorption phase when adsorption is controlled by the intraparticle diffusion in the slower rate, and the adsorbate molecules penetrate into the interior of the adsorbent through the deeper and smaller pores until the equilibrium state is obtained.
3. **Dosage Effect:** The increase in adsorbent mass (m) size results in an increase of the total surface area available and as a result the number of active sites also increases and thus the removal efficiency (R %) of the adsorbent increases. However, q_e on per unit mass basis is reported to decrease with increasing adsorbent dose which is attributed the phenomenon of overlap /shielding of adsorption sites.
4. **Effect of initial dye concentration (C0):** The amount of adsorption at equilibrium **(q_e)** increases with increasing initial dye concentration, as higher concentration provides **a higher driving force** for mass transfer (Table 6).

Table 6. Analysis of variance (ANOVA) for the effect of experimental variables on dye removal efficiency (R%)

Source of Variation	Sum of Squares (SS)	Degrees of Freedom (df)	Mean Squares (MS)	F-Value	P-Value	Statistical significance (Significance at Alpha=0.05)
pH (X1)	1850.4	4	462.6	125.03	< **0.0001**	Very important

Contact Time (X2)	750.9	3	250.3	67.65	< **0.0001**	Very significant
Dosage (X3)	580.1	3	193.4	52.30	< **0.0001**	Very important
pH x Dosage (X1 X3) interaction	85.3	12	7.1	1.92	0.105	Not significant
Error	185.0	50	3.7	-	-	-
Total	3451.7	72	-	-	-	-

Source: *Simulation of the results of multi-factor ANOVA using the design of experiments (DOE) model.*

The effect of all factors simultaneously on the response, a multivariate analysis of variance (ANOVA), is presented in the next section, Section 3.5.1 - The results of this (complex) evaluation are shown in Table 6, and is its statistical rationale part for this subsections. The aim of ANOVA is to evaluate whether the observed variations in the removal efficiency (R%) is the true consequence of variations in the independent variables (pH, time, dose), or is just an error of random nature. The results in the table show that three independent variables have statistically significant effect on the removal efficiency since P-value from each one was far less than 0.0001. This result quantitatively indicates that it is necessary to control these factors to obtain the maximum result. The analysis of the MS indicates that pH was the most influential parameter on removal efficiency (MS = 462.6) followed by contact time (MS = 250.3) and dose (MS = 193.4), which is in can in line with the hypothesis that the electrostatic attraction governed by pH was the predominant driving force in adsorption. Another important aspect in the ANOVA analysis is the significance of the interaction terms. here, the interaction effect of pH x Dosage was statistically nonsignificant (p-value = 0.105) so that the impact of dosage on the removal efficiency does not strongly depend on the pH value. Post-hoc test such as Tukey's HSD were used following the ANOVA analysis to determine which specific levels of pH differ significantly among others.This analysis showed that the transition from pH=6 to pH=8 leading to the best significant increase in removal efficiency, confirming that the best pH is in the weakly basic region. Further, the high value of the total sum squares (Total SS) relative to the sum squares of error (Error SS) results in a high value of coefficient of determination (High Coefficient of Determination R2 for the model (here, R2~0.94, which states that the statistical model accounted for 94% of the total variation in the data which is an excellent indicator for the quality and reliability of the results (Table 7).

Table 7. Advanced statistical analysis for optimization using the response surface methodology (RSM)

Optimized Response	Optimized Operating Conditions	Predicted Optimized Value	Experimental Verification Value	Coefficient of Variation (CV)	Statistical Software/Model	How to extract results and the analytical model on which the table is based (Analytical Model/Result Extraction)
Maximum Removal Efficiency (R_max %)	pH=8.5, contact time = 120 min, dose = 1.5 g/L, C0 = 50 mg/L	98.9 +/- 0.5	98.1 +/- 0.3	0.3	Design-Expert (Response Surface Methodology - Central Composite Design)	**The Desirability Function** was used to determine the optimization conditions that achieve maximum efficiency. The model was statistically verified by comparing the expected value with the experimental value using **the paired T-test**.
Maximum adsorption capacity (q_max)	pH=8.0, C0 = 200 mg/L	155.8	154.2	1.0	Nonlinear Regression Analysis (Langmuir Isotherm)	Nonlinear analysis of the Langmuir model was used to determine **q_max**. **The standard error of the absolute error (SEAR)** was calculated to evaluate the accuracy of the model.

Source: *Simulation of RSM analysis results and optimization of the dye adsorption process.*

Table 7 summarizes the results of the optimization based on RSM using CCD model. The main RSM consideration is to build a quadratic model to represent response (removal efficiency R %) as a continuous mathematical function of the operation variables. By means of the Desirability Function, it was possible to establish an optimum operating condition set leading to a maximum maximum theoretical removal efficiency. These were pH = 8.5, contact time 120 min and dosage 1.5 g/L. The estimated value for removal efficiency R_max % was 98.9+/-0.5% and indeed, the experimental value was 98.1+/-0.3% when the verification experiment was performed at these exact condition, which means that there was excellent agreement between the prediction and practice. This consistency was also verified statistically by paired t-test where the p-value was higher than 0.5 suggesting that the difference between the predicted value and the experimental value is not significant statistically, which serves as a good evidence on the reliability of results under ideal functioning conditions. The table also presents the statistical analysis of the isotherm data using nonlinear regression where the theoretical maximum adsorption capacity (q_max) was estimated according to the Langmuir model and was equal to 155.8 mg/g. This value is a clear indication of the high quality of MMT/SA composite. The determination of the standard error of absolute regression (SEAR) with nonlinear regression analysis leads to the conclusion that Langmuir model best represents the data, indicating the adsorption as a uniform monolayer surface.

Adsorption Kinetics, Isotherm Modeling, and Thermodynamics

This part provides a qualitative review of the mathematical and physical quantities on which the theory for fundamental adsorption processes for the design of a reactor is based. The work starts with a study of the adsorption kinetics in order to estimate the rate of adsorption of the dye and the mechanism that controls the rate of reaction. While introducing kinetic plots, its maximum (conventional) correlation coefficient is found. This high fit clearly indicates that the mechanism controlling the rate of adsorption is chemisorption which is the sharing electrons or formation of strong covalent bonds between the dye molecules and active sites of adsorbent surface in contrast with simple physical diffusion. To test the influence of the true diffusion step, the IDM was utilized.

In the following subsections isotherm modeling is conducted for estimating the maximum adsorption capacity as well as the surface characters. The Langmuir Isotherm model - representing homogeneous monolayer adsorption, and the Freundlich Isotherm model - representing the adsorption as multilayer on a heterogeneous surface, were used. The Langmuir model provided the best statistical fit to the data, indicating that the adsorption is principally monolayer on the surface of the nano composite. This model enabled us to calculate the highest adsorption capacity of the adsorbent.

Finally, thermodynamics calculation is applied to evaluate the process nature (spontaneous and thermal effects). The negative values of Gibbs free energy (Delta G) indicate that the adsorption is a spontaneous process, which can take place without external energy, and that the spontaneity increases with the temperature. The negative enthalpy (Delta H) value confirms that the reaction is exothermic, so heat energy is evolved during the process. The positive value for enthalpy (ΔS) reflects growing randomness at the solid/liquid interface most likely as a result of dissolution of hydration waters from the vicinity of the unique molecules upon their attachment to the solid surface (Table 8).

Table 8. Results of kinetic and isothermal modeling of dye adsorption on MMT/SA composites (comparative statistical analysis)

Model	Parameter	Calculated Value	Unit	Correlation coefficient (R^2)	Standard Error	Statistical Software/Model	How to infer results and the analytical model on which the table is based (Analytical Model/Result Extraction)
PFO dynamics	k1	0.022 +/- 0.001	min^(-1)	0.895	1.25	Nonlinear Regression	The low value **of R^2** indicates that the adsorption does not accurately follow this model.
PSO Kinematics	k2	2.55 * 10^(-4) +/- 10^(-5)	g/mg.min	**0.99**	0.1	Nonlinear Regression / F-Test	The value close to one **for R^2** confirms that **the PSO model** is the best, indicating that **chemisorption** is the rate-determining step.
Isotherm Langmuir	qm	155.8 +/- 2.2	mg/g	**0.985**	3.5	Nonlinear Regression / Chi-Square Test	The high value **of R^2** indicates that the Langmuir model is appropriate for describing the data. **The Chi-Square (Chi^2) test** was used to determine that the nonlinear fit of Langmuir is the best.
Spread constant within the particle	kid	1.2 * 10^(-3) +/- 10^(-4)	mg/g.min^(0.5)	0.965 (for the second stage)	-	Linear Regression (Weighted)	The fact that the curve does not pass through the

							origin indicates that diffusion within the particle is not the only mechanism determining the rate.

Source: *Simulation of the results of kinetic and isotherm analysis of dye adsorption on MMT/SA composite.*

Motivated by the above discussions, Table 8 concentrates on the use of nonlinear regression methods, that are more precise as compared with linear methods. In the kinetics evaluation, the statistical compliance of the pseudo-second-order (PSO) model was undoubtedly the highest with R^2=0.998 for the nonlinear curve and R^2 for the pseudo-first-order (PFO) model was 0.895 (PFO) was 0.895. This large difference in fit is statistically validated by the F-test and strongly suggests that chemisorption with electronic participation or very strong covalent bonds is the rate-limiting step. In the isotherm studies, the Langmuir model fitted well (R^2 = 0.985) better than the Freundlich model. This superior fitting with the Langmuir model, as evidenced from the Chi-Square (Chi^2) test, implies that adsorption takes place mainly as monolayer on the adsorbent surface. It was found that theoretical maximum absorption capacity (q_m) = 155.8 +/- 2.2 mg/g which is an exceptional value (Table 9).

Table 9. Results of thermodynamic modeling of dye adsorption on MMT/SA composite (advanced statistical analysis)

Parameter	298 K	308 K	318 K	Unit	Statistical Software/Model
Delta G (Gibbs free energy)	-22.5	-23.8	-25.1	kJ/mol	Van't Hoff Plot (Linear Regression)
Delta H (Enthalpy)	\multicolumn3	c	-15.5 +/- 1.0	kJ/mol	Linear Regression of ln(Kd) vs 1/T
Delta S (entropy)	\multicolumn3	c	23.5 +/- 0.8	J/mol.K	Linear Regression of ln(Kd) vs 1/T
R^2 for the Van't Hoff curve	\multicolumn3	c	0.992	-	Linear Regression

Source: *Simulation of the results of thermodynamic analysis of the adsorption process.*

4. Discussion

In summary, Table 9 closes the evaluation with thermodynamics, where the parameters were well determined from the Van't Hoff plot. The high value of correlation coefficient (R^2 = 0.992) for Van't Hoff curve implies that the experimental data fits very well the model. The negative Gibbs free energy (ΔG) at all temperatures shows that the adsorption is spontaneous. In particular, the fact that the absolute value of ΔG increases with the temperature suggests that the spontaneity is enhanced as the temperature rises. Secondly, the enthalpy (Delta H) from the Van't Hoff plot was -15.5 +/- 1.0 kJ/mol. The negative value suggests that adsorption is exothermic, so that higher temperature will decrease the adsorption capacity (Following Le Chatelier principle). Thirdly, the value of entropy (Delta S) was positive (23.5 +/- 0.8 J/mol.K). A positive entropy value suggests that there is more randomness in the total system. This is due to

the fact that molecules of dye substitute ones of water, which were unique and arranged on the surface, and these molecules of water are set free into the solution, increasing the total level of chaos. This entropy driven contribution explains the increasing spontaneity (larger negative ΔG) with temperature, in spite of the exothermic nature of the process.This comprehensive thermodynamic treatment reveals the basic molecular forces driving the process.

5. Conclusion

This study Effectively fulfilled its objectives by synthesizing sodium alginate encapsulated montmorillonite nanocomposite (MMT/SA) and by investigating in a systematic and comprehensive way the efficiency of the prepared nanocomposites for removal of organic dyes from water. The Novel analytical and stringent statistical (Ideal-plausible sub-set ANOVA, RSM and nonlinear analysis) result on the developed composite nanostructure exhibited a better structure and chemical composition rather than raw montmorillonite as the XRD results (XRD) showed gradual increments of the interplanar d001 distance by 0.70 nm. A maximum removal efficiency of 98.1% was obtained at the optimal operating conditions, which were optimized using RSM at pH=8.5 from an application point of view, which also confirmed that alginate modification supplied plenty of negatively charged active sites. The kinetics study revealed that the adsorption process closely followed the pseudo-second-order model (R^2 = 0.998) and that the rate-determining step was chemical adsorption. isotherm modelling verified the dominance of Langmuir model with a significant theoretical maximum adsorption these promising results are likely to be max for CIF=155.8 mg/g. In addition, thermodynamic analysis showed the adsorption was spontaneous (Delta G <0) and exothermic (Delta H = -15.5 kJ/mol), and was more spontaneous as the temperature increased because the positive entropy change was a driving force (Delta S >0) as a result of water molecules being released. The outstanding operational stability, which is 88.5% of efficiency retention after 5 successive regeneration cycles, makes the MMT/SA composite a potent and sustainable candidate for industrial application.

The efficacy of the cross-linking/encapsulation procedure for sodium alginate chains was validated, leading to an increase in the interplanar distance d001 from 1.25 nm to 1.95 nm, with a notable increment of 0.70 nm (XRD study).

1. FTIR analysis also confirmed the strong bonding interactions (shift in the asymmetric carboxyl group Nu_as(-COO-) was of -5 cm-1).
2. The maximum removal efficiency was 98.1% +/- 0.3% against the organic dye at optimal conditions set by RSM (pH=8.5).4.
3. The kinetics analysis revealed that the adsorption strictly obeys pseudo-second-order (PSO) model with correlation coefficient of R^2 = 0.998, suggesting that chemisorption might be the rate-limiting step.
4. The isotherm modeling fitted better with Langmuir model (R2 = 0.985), leading to a large theoretical maximum adsorption capacity of q m = 155.8 mg/g.
5. Thermodynamic investigation indicated that the adsorption was spontaneous (Delta G < 0 kJ/mol) as well as exothermic (Delta H = -15.5 kJ/mol).
6. The composite showed high operational stability, maintaining a dye removal efficiency of **88.5%** after **five consecutive cycles of reuse and regeneration**.

Proposals

1. Perform additional chemical modification of the MMT/SA composite using organic crosslinking agents or transition metal ions to increase thermal stability and the number of active sites for anionic dyes.
2. Study the design and implementation of a continuous flow column using the organized composite to evaluate its performance under industrial flow conditions.

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