



Original Article

Fabrication of Composite Material Based on Lignin Extracted from Sugarcane Bagasse and Modified with Chitosan for the Treatment of Methylene Blue in Aqueous Environment

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Abstract: A potential adsorbent material was synthesized based on the interaction between chitosan and lignin, aimed at adsorbing and treating methylene blue (MB) in aqueous environments. The highlight of this study is that the lignin was extracted from sugarcane bagasse, an agricultural by-product, aligning with green and circular economy principles. The FTIR spectroscopy analysis of the material showed the formation of bonds between chitosan and lignin, implying the successful creation of a new material - chitosan/lignin composite material. The effects of factors such as reaction temperatures, contact time, initial concentrations, and chitosan/lignin ratios on the MB adsorption of the material were investigated to evaluate the applicability of the material in dye-contaminated wastewater treatment. Experimental results indicate that the material with a chitosan/lignin ratio of 1.5/3 (mL/g) exhibited the best treatment efficiency at room temperature within 60 min. In this study, isotherm adsorption models for MB on the material were also examined. The data show that the Langmuir model better describes the adsorption process than the Freundlich model. The research findings demonstrate that the chitosan/lignin material has the capability to treat dyes in water under normal conditions, highlighting its potential application in treating textile dyeing wastewater.

Keywords: Methylene blue, chitosan, lignin, sugarcane bagasse, dye treatment.

1. Introduction

Industrial wastewater pollution is a serious environmental challenge, particularly in the textile manufacturing industry, which generates effluents containing large amounts of synthetic

dyes such as methylene blue (MB). Upon entering natural bodies of water, MB becomes a major contaminant due to its high solubility and resistance to degradation, persisting in aquatic environments and being difficult to remove through natural processes [1]. One of the biggest concerns with MB contamination is its impact on aquatic ecosystems, as it absorbs visible light when dissolved in water, reducing sunlight penetration and inhibiting

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photosynthesis in underwater plants and algae. This disruption decreases oxygen production, threatening aquatic life [2]. Additionally, MB can form stable chemical complexes with other organic compounds, further extending its persistence in water. Beyond environmental impacts, the presence of MB in water can lead to oxidative stress and bioaccumulation in aquatic organisms, posing health risks to humans when these organisms are ingested. Exposure to MB with a high concentration and long-term can lead to methemoglobinemia, neurological issues, and oxidative DNA damage [3]. Given these environmental and health concerns, finding an effective, affordable, and sustainable method to remove MB from textile dyeing wastewater is crucial.

Several techniques have been utilized to remove MB from wastewater, including chemical oxidation, filtration, and biodegradation, but these methods often have drawbacks that limit their practicality for large-scale wastewater treatment [4]. Chemical oxidation processes like ozone treatment and photocatalysis effectively break down MB molecules but require high energy inputs and can generate toxic by-products such as chlorinated organic compounds. Membrane filtration techniques, including ultrafiltration, reverse osmosis, and nanofiltration, physically remove MB but suffer from high operational costs, frequent membrane fouling, and significant water loss due to high rejection rates [5]. Coagulation-flocculation using metal salts like aluminum sulfate and ferric chloride can precipitate MB from wastewater but produces large amounts of chemical sludge that require further treatment and disposal [6]. Advanced oxidation processes involving hydroxyl radical-based treatments efficiently degrade MB but rely on hazardous chemicals such as hydrogen peroxide and persulfates [7]. Biological treatments using bacteria, fungi, or algae show potential for MB biodegradation but have slow reaction rates, require highly controlled environmental conditions, and often fail to

break down highly stable dyes in real wastewater settings [8]. Adsorption-based methods using activated carbon, biochar, or clay minerals provide high removal efficiency, but activated carbon is costly to produce and regenerate, while other adsorbents often have limited dye-binding capacity at industrial wastewater concentrations [9]. Given these limitations, efforts are underway to develop more efficient and sustainable MB treatment methods.

On the other hand, chitosan and lignin are emerging as promising alternative adsorption materials due to their sustainability, renewability, low toxicity, and strong affinity for organic pollutants. Chitosan, a naturally occurring biopolymer derived from chitin in shrimp and crab shells, consists of D-glucosamine and N-acetyl-D-glucosamine units that provide strong binding sites for pollutants. Its amine ($-NH_2$) and hydroxyl ($-OH$) groups enable interactions with contaminants through electrostatic attraction, hydrogen bonding, and metal ion chelation. Moreover, in acidic solutions, the protonation of amine groups ($-NH_3^+$) enhances its ability to bind negatively charged pollutants like MB, making it an effective, stable, and environmentally friendly adsorbent [10]. Lignin, a complex polyphenolic macromolecule found in plant cell walls, contains carboxyl ($-COOH$), hydroxyl ($-OH$), and methoxy ($-OCH_3$) groups, which allow it to bind dyes through hydrogen bonding, electrostatic interactions, and π - π stacking [11]. Although naturally hydrophobic, lignin can be modified or combined with more hydrophilic materials to enhance its adsorption efficiency and dispersibility in wastewater [12], making it a strong candidate for sustainable wastewater treatment, especially as part of composite materials.

Recent studies have shown that combining chitosan and lignin into a hybrid adsorbent significantly improves adsorption efficiency by enhancing surface area, functional group availability, and pollutant-binding capacity.

This synergy arises because the porous structure allows more contaminants to be trapped, the positive charge of chitosan interacts with negatively charged pollutants, and the aromatic rings in lignin form π - π interactions with MB molecules [13]. Significantly, this modification leverages the inherent strengths of chitosan and lignin to improve mechanical properties, enhance thermal stability, synergize functional groups of an eco-friendly composite material with better performance for applications like food packaging, biomaterials, and environmental treatment. A key highlight of this study is the utilization of sugarcane bagasse, an abundant agricultural waste product, to extract lignin. Bagasse-derived lignin is low-cost and readily available in sugar-producing countries like Vietnam, aligning with sustainable and circular economy principles [14]. Ultimately, repurposing agricultural waste into bio-adsorbents not only aids industrial waste disposal but also promotes an eco-friendly approach to water treatment, reducing dependence on non-renewable resources.

2. Experimental

2.1. Materials

Sugarcane bagasse was collected after sugarcane was pressed to remove its juice. Chitosan (deacetylation degree $\geq 90\%$) was produced by Shanghai Zhanyun Chemical Co., Ltd (China). Sulfuric acid (95 - 98%), acetic acid (99.5%), sodium hydroxide ($\geq 96\%$), and methylene blue (98.5%) was produced by Xilong Scientific Co., Ltd (China). The chemicals were dissolved or diluted to desired concentrations by distilled water.

2.2. Extraction of Lignin from Sugarcane Bagasse

Lignin was extracted from sugarcane bagasse using a modified procedure derived from previously published works [15, 16]. Briefly, sugarcane bagasse was washed with

clean water and exposed to sunlight until dry, and then crushed to a size of 1 mm. Ground sugarcane bagasse was soaked in a 10% sodium hydroxide solution with the ratio of 1:15 (w/v). The mixture was well stirred for 5 h at 90 °C (Figure 1a-i). Subsequently, the solution part was collected by using a centrifuge process. A 50% sulfuric acid solution was added to the solution with the ratio of 1:10 (v/v) to precipitate down lignin (Figure 1a-ii). Finally, the mixture was centrifuged to obtain lignin (Figure 1a-iii).

2.3. Synthesis of Chitosan/lignin Materials

Figure 1b depicts the synthesis procedure of chitosan/lignin (Cs/Lg) materials, which was modified from those described in previous studies [17,18]. Firstly, a 2% (w/v) chitosan solution was prepared in a 1.5% acetic acid solution. Subsequently, 30 g of lignin extracted from sugarcane bagasse were added to 50 mL of the chitosan solutions with various ratios (Figure 1b-i). For the experiments of the effect evaluation of material ratio, the weights of lignin were varied while the volumes of the chitosan solution were fixed. The mixtures were vigorously stirred for 30 min to obtain Cs/Lg materials (Figure 1b-ii). The Cs/Lg materials would be used to evaluate the methylene blue adsorption capability in further experiments (Figure 1b-iii).

2.4. Effect of Temperature on the MB Adsorption Capability of the Cs/Lg Material

To examine the effect of temperature on MB adsorption capability of the material, three clean 100-mL conical flasks were prepared. For each flask, 3 g of Cs/Lg material and 25 mL of 10 mg/L MB solution were added. The mixtures in the flasks were then shaken for 60 min at various temperatures of 25 °C, 75 °C, and 90 °C. Finally, the samples were centrifuged to remove solid parts and the remaining MB concentrations in solution parts were determined by using a UV-Vis spectrophotometer (Hach DR5000) (Figure 1b-iv).

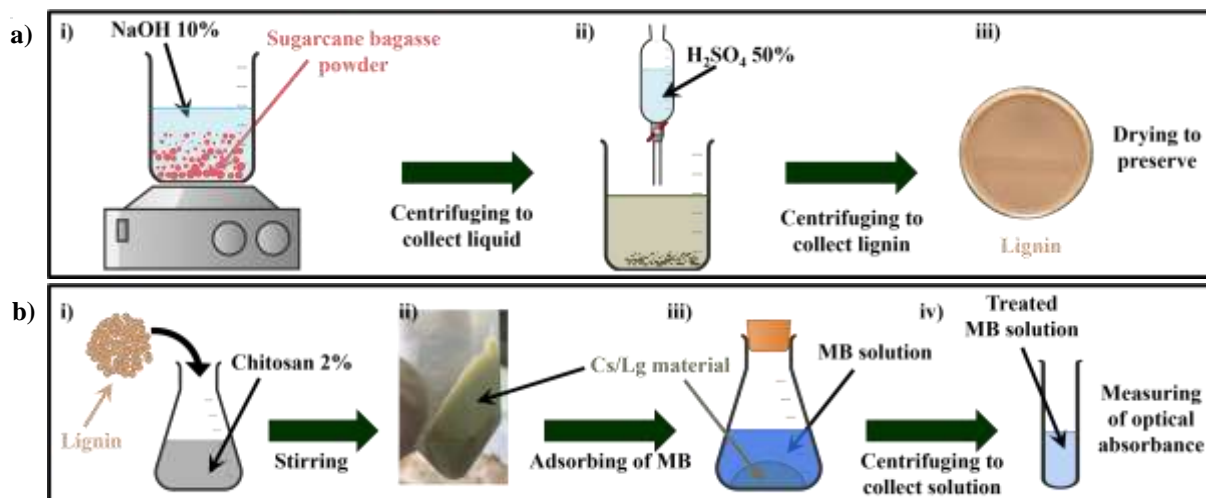


Figure 1. Schematic diagram depicting an experimental procedure. (a) extracting of lignin from sugarcane bagasse. (b) synthesizing of a chitosan/lignin material and treating MB solution by the material.

2.5. Effect of Contact time on the MB Adsorption Capability of the Cs/Lg Material

Five clean 100-mL conical flasks were each prepared with 3 g of Cs/Lg material and 25 mL of 10 mg/L MB solution to assess the effect of contact time on MB adsorption. The mixtures in the flasks were shaken at a temperature of 25 °C for various periods of time, i.e. 15, 30, 60, 90, and 150 minutes. The remaining concentrations of MB in the solutions were measured by using the UV-Vis spectrophotometer after applying a centrifuge process to discard solid parts.

2.6. Effect of Initial MB Concentrations on the MB Adsorption Capability of the Cs/Lg Material

In this experiment, 10 mL of MB solutions with various concentrations from 1.0 mg/L to 10.0 mg/L were shaken with 3 g of Cs/Lg material in separate 100-mL conical flasks at 25 °C for 60 min. The obtained mixtures were centrifuged to collect solution parts, following by spectrophotometry measurements to quantify MB concentrations.

2.7. Effect of Chitosan/lignin Ratios on the MB Treatment Efficiency of the Cs/Lg Material

Five Cs/Lg material samples were prepared by mixing 3g of lignin with different volumes

of the 2% (w/v) chitosan solution (0.2, 0.5, 1.5, 3.0, and 5.0 mL) to evaluate the effect of chitosan/lignin ratio in the Cs/Lg materials on MB adsorption capability. After centrifugation, the Cs/Lg material samples were shaken with 25 mL of 10 mg/L MB solutions in separate 100-mL conical flasks for 60 min at 25 °C. And then, the remaining concentrations of MB in the solutions were determined as described above.

2.8. Determination of MB Concentrations in Solutions and Treatment Efficiency

The concentrations of MB in solutions were determined using a linear curve fitting method. Briefly, a series of MB solutions with identified concentrations in the range of 0.05 - 12.0 mg/L was prepared from a stock MB solution. The optical absorbance (Abs) values of these solutions were measured by using a Hach DR5000 spectrophotometer at 665 nm. The linear regression equation that describes the relationship between Abs and MB concentration (C) was built to calculate the concentration of MB in samples. In this case, the equation was determined to be $\text{Abs} = 0.1857 \times C + 0.0598$ with $R^2 = 0.9975$.

To quantitatively evaluate MB treatment process of Cs/Lg materials, the adsorption capability q_e of the materials for MB was calculated by using equation (1).

$$q_e \text{ (mg/g)} = \frac{(C_0 - C_t) \times V}{m} \quad (1)$$

where C_0 and C_t are the initial and final concentrations of a MB solution; V is the volume of the MB solution; and m is the weight of a Cs/Lg material.

Additionally, the treatment efficiency H of the process was obtained by using equation (2).

$$H \text{ (%) } = \frac{C_0 - C_t}{C_0} \times 100 \quad (2)$$

3. Results and Discussion

3.1. Characteristics of Cs/Lg Materials

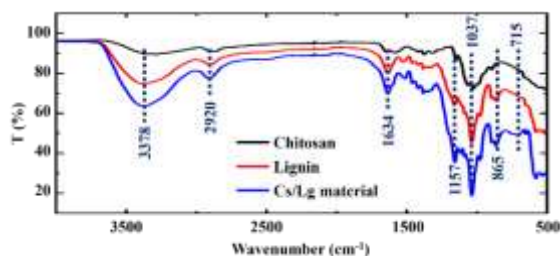


Figure 2. FTIR spectra of chitosan (black), lignin (red), and Cs/Lg material (blue).

Figure 2 shows the spectra of chitosan and the Cs/Lg material synthesized from chitosan and lignin. Compared the spectrum of the Cs/Lg material with that of chitosan and lignin, the strong peak around 3378 cm^{-1} is attributed to the stretching vibration of O-H and N-H bonds in chitosan and lignin molecules. The absorption bands at 2920 cm^{-1} corresponds to C-H stretching bonds which presents in alkyl and aromatic groups. The vibration bands around 1634 cm^{-1} can be assigned to the C=O bond stretching and amine deformation while the peak at 1157 cm^{-1} may be related to C-O-C stretching. The peaks around 1037 and 865 cm^{-1} is the characteristic of the syringyl ring with C-O stretching [19-21]. These results indicate that both characteristic bands in the FTIR spectra of chitosan and lignin are observed in that of the Cs/Lg material. Moreover, a variation band in area 715 cm^{-1} only appears in the spectrum of the Cs/Lg

material, which may represent the vibrations of bonds forming by the cross linking between chitosan and lignin. This evidence implies that our synthesis process could apply to produce a new composite material between chitosan and lignin.

3.2. Effect of Reaction Temperatures on MB Adsorption Capacity of the Material

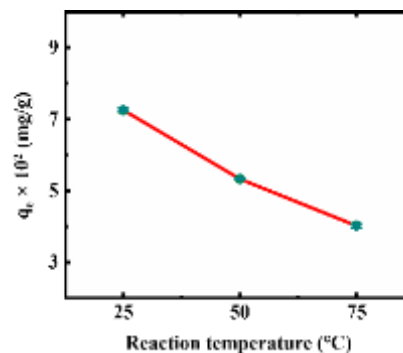


Figure 3. Plotting the variation in MB adsorption capacity of Cs/Lg material with the change of reaction temperatures. Data are expressed as means \pm standard deviation (SD) ($n = 3$).

Figure 3 shows the changes of MB adsorption capacity of Cs/Lg material at different reaction temperatures. The result presents a gradual decrease in adsorption capacity q_e with a reaction temperature increase. This can be explained by the fact that a high temperature may weaken the interactions between the adsorbents and the dye molecules. This trend also implies that the adsorption process of MB onto Cs/Lg material is exothermic, which is consistent with the results of a previous work [22]. Importantly, the findings highlight that this adsorption process could spontaneously occur at room temperature with high efficiency ($H = 90\%$), demonstrating the application capability of the Cs/Lg material in dye-containing wastewater treatment. This characteristic enhances the practicality of adsorption materials in wastewater treatment, as they can be used under normal conditions without the need for additional energy input, reducing operational costs and improving scalability for industrial applications.

3.3. Effect of Reaction times on MB Adsorption Capacity of the Material

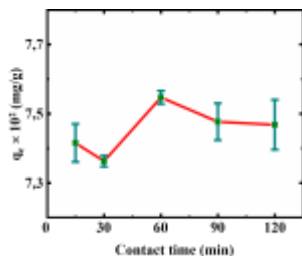


Figure 4. Plotting the variation in MB adsorption capacity of Cs/Lg material with the change of contact times. Data are presented as means \pm SD.

Figure 4 presents the dependences of MB adsorption capacity q_e on the contact time between Cs/Lg material and MB solutions. The experimental results show that the highest q_e value was observed at 60 min. However, this highest q_e value is negligibly different from q_e values obtained at different contact time periods. The data suggests that the adsorption process is rapid under the given conditions, quickly reaching an equilibrium state. This was also reported in several published works, namely that the adsorption processes using lignin-based adsorbents could reach equilibrium states after 30 to 90 min or earlier [17, 18, 21]. The rapid adsorption capability of the Cs/Lg material may help reduce the operational time of a treatment process, therefore, enhancing the practicality of Cs/Lg materials in wastewater treatment.

3.4. Effect of Initial MB Concentrations on the MB Adsorption Capability of the Material

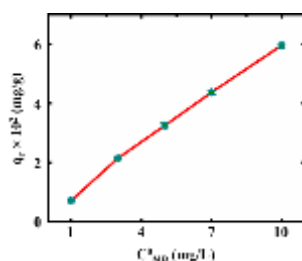


Figure 5. Plotting the variation in MB adsorption capacity of Cs/Lg material with the change of initial MB concentrations. Error bars show SD values.

The evaluation results of the effect of initial MB concentrations on the adsorption capacity of Cs/Lg material are given in Figure 5. The concentrations of MB solutions used in this experiment are in the range of 1 mg/L - 10 mg/L. The data reveals a clear positive correlation between the initial concentrations of MB solutions and the adsorption capacity of the Cs/Lg material, indicating that the material effectively captures dye molecules without reaching saturation within the studied concentration range. This phenomenon is likely due to the porous structure formed by the integration of chitosan molecules with lignin substrate, which enlarges surface area and available adsorption sites. This observation for such low concentration MB solutions was shown in published studies [13, 18]. The high adsorption capacity of the Cs/Lg material is consistent with the characteristics of lignin-based materials which comes from lignin's structure with abundance of aromatic rings, hydrocarbon chains, and hydroxyl groups, reinforcing the material's structural advantages in dye removal [11]. Additionally, the fact that saturation was not reached implies that the material may be suitable for treating wastewater with varying and even higher dye concentrations. These results highlight the strong potential of Cs/Lg as an efficient and scalable adsorbent for dye removing applications.

3.5. Adsorption Isotherm Modelling

Adsorption isotherm modelling analysis is crucial in understanding the adsorption mechanism of MB onto Cs/Lg material. Simple and common adsorption isotherm models are Langmuir model and Freundlich model. The Langmuir model is widely accepted for monolayer adsorption with finite number of uniform adsorption sites while the Freundlich model is applied to describe reversible and non-monolayer adsorption. The models are depicted by the following equations.

$$\text{Langmuir model: } \frac{1}{q_e} = \frac{1}{K_L \times q_m} \times \frac{1}{C_e} + \frac{1}{q_m}$$

Freundlich model: $\ln q_e = \frac{1}{n} \times \ln C_e + \ln K_F$

where q_e (mg/g) is an equilibrium adsorption capacity; C_e (mg/L) is an equilibrium MB concentration; q_m (mg/g) is a maximum adsorption capacity; K_L (L/mg) is a Langmuir constant; K_F and n are Freundlich constants.

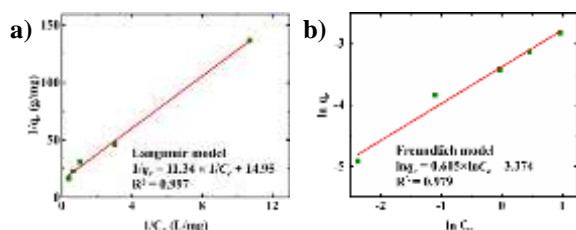


Figure 6. Adsorption isotherm models of MB for Cs/Lg material. a) Langmuir isotherm; b) Freundlich isotherm. Data are given as means \pm SD.

The parameters of the Langmuir and Freundlich models were calculated via the intercept and slope values of the plots of $1/q_e$ versus $1/C_e$ (Figure 6a) and $\ln q_e$ versus $\ln C_e$ (Figure 6b), respectively. The fitting analysis on our data shows that the values of q_m , K_L , K_F and n were 0.0669 mg/g, 1.32 (L/mg), 0.0343 and 1.65, respectively. Additionally, the determination coefficients R^2 were also determined to be 0.997 (Langmuir model) and 0.979 (Freundlich model), indicating that the Langmuir isotherm model is more suitable for the description of adsorption isotherm of MB adsorption on Cs/Lg material than the Freundlich isotherm model. This suggests that MB adsorption occurs as a monolayer on a homogenous surface, rather than multilayer adsorption on heterogeneous sites. The findings reinforce the efficiency and predictability of the Cs/Lg material as an adsorbent, aligning with previous studies and highlighting its practical applicability in dye removal [17, 18]. Furthermore, the K_L is quite small, implying that the affinity between the Cs/Lg material and MB molecules are weak. This suggests that the material can be reconstructed and efficiently reused, making it a promising candidate for sustainable wastewater treatment solutions.

3.6. Effect of Chitosan/lignin Ratios on MB Treatment Efficiency of the Material

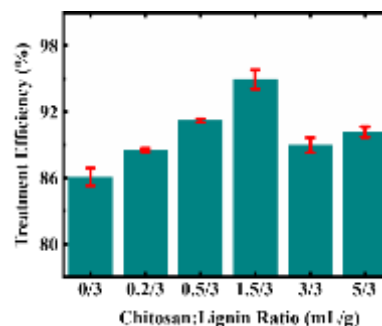


Figure 7. Effect of chitosan contents in Cs/Lg materials on the treatment efficiency of MB in a solution. Error bars indicate SD values.

To demonstrate the crucial role of chitosan in enhancing the MB treatment efficiency of Cs/Lg, different contents of chitosan in Cs/Lg materials were used to test MB adsorption. Figure 7 shows the MB treatment efficiency H of Cs/Lg materials synthesized with various volumes of a 2% (w/v) chitosan solution. The result clearly reveals that the treatment efficiency reached its highest level at the optimal chitosan/lignin ratio of 1.5/3 (mL/g). The efficiency of MB treatment processes rose from 86% for an Lg material without Cs to 95% for a material with a 1.5/3 ratio. The sharp increase in efficiency at this ratio suggests that chitosan could increase the surface area and adsorption sites of the material, significantly improving dye removal. However, exceeding chitosan contents lead to a decline in efficiency, likely due to structural changes in the material or the occupation of active adsorption sites by chitosan resulting in the hinderance of the interaction between MB with the material. Specially, the highest efficiency for MB treatment using the Cs/Lg material is $\sim 95\%$, which outperforms other adsorbents such as barley bran and soursop leaves reported in previous works [23]. This confirms again that the Cs/Lg material exhibits strong potential as an effective and sustainable adsorbent for treating dye-contaminated wastewater. These findings highlight the feasibility of lignin-based

materials for environmental applications, offering a promising alternative to conventional adsorbents.

4. Conclusion

The study successfully synthesized a novel adsorbent material by combining chitosan and lignin extracted from sugarcane bagasse. FTIR analysis confirmed the formation of new bonds between chitosan and lignin, enhancing the material's adsorption capabilities. Experimental investigations demonstrated that the Cs/Lg material with a chitosan/lignin ratio of 1.5/3 (mL/g) achieved the highest MB removal efficiency at room temperature within 60 minutes. Furthermore, adsorption isotherm analysis reveals the goodness-of-fit of the Langmuir model, suggesting monolayer adsorption on a homogenous surface for the MB adsorption of the Cs/Lg material. These findings indicate the material's strong potential for real-world applications in wastewater treatment, particularly in addressing dye contamination from textile industries. The results of our work also offer an eco-friendly and cost-effective alternative to conventional treatment methods. Moreover, this work should open a new direction in the treatment of colorants using agricultural by-products, promoting sustainable solutions for environmental remediation.

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