

Integration of Chitosan and Sugar Cane Bagasse as Adsorbent for Remazol Red Dyes Removal

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Abstract: In this study, the sugarcane bagasse biomass was modified and purpose as a bio-adsorbent to remove Remazol red dye from wastewater. The sugarcane bagasse was calcined at 200°C, followed by the treatment with 1% chitosan solution. The adsorbent was morphologically characterized using FTIR, SEM, XRD analysis to study further the effect of chemical modification techniques on calcined biomass of sugarcane bagasse. Treated calcined sugarcane showed an irregular surface and appearance of pores effects of an acidic chitosan solution. It also clarified the presence of an active site for dye absorption on the adsorbent's surface, which is necessary for the dye adsorption attachment site. The adsorption performance revealed that treated sugarcane bagasse performed better with an optimum adsorbent dosage of 150 mg at an equilibrium time of 60 min. Remazol Red removal favored acidic conditions at pH 4 and decreased as dye concentration increased. With an R² value, the equilibrium data adsorption fitted well with the Langmuir adsorption isotherm.

Keywords: adsorbent; adsorption; chitosan; dyes; sugarcane.

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1. Introduction

Textile effluents are ranked among the most harmful pollutants to the environment because of the high discharge volume of synthetic dyes that are toxic and reactive [1]. Furthermore, these textile effluents are a significant concern for our environment because of their carcinogenic and mutagenic properties, which can cause severe water pollution [2]. In addition, it is difficult to remove or treat, primarily when solely dependent on traditional water treatment methods [3,4].

Various physical or chemical approaches, such as adsorption, coagulation, electrochemical separation, and membrane separation, are already available, each with advantages and disadvantages in the water treatment system practical for specific types of

wastewater or coloring agents. Biological treatment methods have been shown to reduce biochemical oxygen demand (BOD) in dyeing wastewater; however, decolorization rate tends to decrease due to the non-biodegradability of commercial dyes. The use of chemicals for coagulation generates a large volume of sludge-containing chemicals, necessitating proper waste disposal management. Microbubbles have also been extensively researched for use in cleaning and bleaching applications. Physical contact between the microbubbles provides sufficient energy to remove the biofilm from the surface or strip impurities from a fluid [5, 6]. Membrane filtration is quite effective for rapidly treating large effluent volumes containing various dyes, with its only limitation being the high capital costs involved [7].

Adsorption has emerged as a promising dye removal technique. Compared to other available technologies, it is the most preferred technology due to its ease of operation and design to minimize hazardous pollutants in discharged water. It also contributes to its broader applicability in water pollution control [8]. The adsorbent chosen is critical to the efficiency of adsorption operations. Common adsorbents used in wastewater treatment were activated charcoal, zeolites, silica gel, and alumina. However, the majority of these adsorbents are prohibitively expensive, and research efforts to develop an industrially sustainable, cost-effective, and environmentally friendly adsorbent for wastewater treatment have been motivated [9].

Agricultural waste is preferred as an economic adsorbent due to its nature, and lower processing requirements than industrial adsorbents offsets the cost issue [10–12]. These adsorbents are less expensive and easier to find. It also has a strong preference for pigment and is easily accessible at no cost to replace expensive therapy. It also contributes to reducing the industrial waste disposal crisis, which pollutes the soil, water, and air, industrial wastes as adsorbents have been proposed as single-use products to avoid the issue of regeneration. The filled adsorbent could be disposed of quickly by landfill or incorporation. The primed adsorbent can be safely disposed of after treatment by landfill or incineration [13]. Hence, this study aimed to modify the sugarcane bagasse biomass using chitosan to improve the adsorption of Remazol dyes in the textile effluent wastewater.

2. Materials and Methods

2.1. Materials.

The sugarcane bagasse was obtained from a local cafe in Gong Badak, Kuala Terengganu, Malaysia. All the chemicals used are analytical grade and used directly. Remazol Red dye and commercial chitosan powder were purchased from Sigma-Aldrich (M) Sdn Bhd (Petaling Jaya, Selangor). Acetic acid glacial were supplied by Fisher Scientific (New Hampshire, United States). Sodium hydroxide pellets were purchased from Sigma-Aldrich (M) Sdn Bhd (Petaling Jaya, Selangor).

2.2. Preparation of adsorbent.

Sugarcane bagasse was washed using boiled distilled water to remove any impurities and dried for 5 h in an oven at 105°C. After that, it was sieved to extract particles sizes ranging from 100 to 500 µm. The dried sugarcane bagasse was labeled as an untreated sample and placed in an airtight jar. The sugarcane bagasse was treated by a two-stage process: heat treatment and immersion in chitosan solution that acts as a ligand for adhesion of the adsorbate. In the heat treatment, the sugarcane bagasse was heated in a furnace at 200°C for 2 h. Heat

treatment was used to create pores on the surface of the adsorbents. Next, 10 g of chitosan powder was placed in 1 L of the volumetric flask containing 100 ml of 0.1 M acetic acid and filled with distilled water. Then this mixture was heated to 50°C for 30 min to dissolve the chitosan powder. After that, the dried sugarcane bagasse was added to the dissolved chitosan solution and left for a day at room temperature. Next, 1 L of 0.1 M of NaOH was added dropwise to the mixture and left for another 24 h to neutralize the acidic solution. Lastly, the mixture was rinsed with distilled water to remove excess solvent, followed by drying the treated sugarcane bagasse in an oven for 24 h at 60°C.

2.3. Characterization of adsorbents.

Scanning Electron Microscope (SEM) modeled JEOL-JSM-6260L in INOS, UMT was used to determine the surface morphologies for untreated and treated sugarcane bagasse samples. It is necessary to coat the sample with a conductor material such as gold before images are taken. This gold plating will inhibit charging, reduce thermal damage and improve the image quality. The Fourier Transform Infrared (Shimadzu IRTracer-100) equipment was utilized to analyze the chemical characterization of the bio-adsorbent effect of treatment treated in the range of 4000–400 cm^{-1} by KBr pellet technique. The crystallinity pattern of the sugarcane bagasse samples was analyzed using an X-ray diffraction machine (MiniFlex II Benchtop X-ray Diffractometer, Rigaku) available in Physics Laboratory, INOS UMT. The X-ray diffraction analysis was carried out for 2θ values ranging from 10° to 80° utilizing Cu $K\alpha$ radiation at a wavelength of $\lambda = 1.540 \text{ \AA}$.

2.4. Batch adsorption experiment.

Batch adsorption experiments were performed to regulate the optimal conditions for dye adsorption onto adsorbents. These data were also required to generate adsorption isotherm. The batch experiment took place at room temperature and under a constant stirring of 165 rpm. Parameters that affected the adsorption have been determined; the effect of different types of adsorbents, contact time (30-180 min), adsorbent dosage (50-150 mg in 100 ml dye solution), initial concentrations (10-100 mg/L), and initial pH of dye solution (2 - 10). The sample taken for reading was centrifuged for 4 minutes at 29°C and 4000 rpm before being measured using UV-VIS at wavelength 504 nm obtained from the calibration curve. The equilibrium sorption capacity was determined from Eq. (1):

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

where q_e is the amount of dye adsorbed per gram of adsorbent (mg/g), C_i and C_e were the initial and equilibrium dye concentration (mg/l). V and m were the volume of solution and Mass of adsorbent (g), respectively. The removal efficiency of dyes was measured as Eq. (2) below.

$$\% \text{ Removal of dye} = \frac{C_i - C_e}{C_e} \times 100 \quad (2)$$

2.5. Adsorption isotherm.

The adsorption isotherm is a constant temperature that achieves equilibrium between the amount of adsorbate per unit of adsorbent (q_e) with and an equilibrium concentration solution (C_e). The modeling will also represent the monolayer sorption and homogenous distribution of active sites on the surface of the adsorbent. A visual plot of solid-phase

concentration versus liquid phase will show the feasibility of an equilibrium adsorption density [14]. Eq. (3) calculates the adsorption density q_e (mg/g) will be determined.

$$q_e = \frac{(C_i - C_e) \times V}{1000 \times m} \quad (3)$$

Several equations and models are available to describe functions of isotherm, such as Freundlich and the Langmuir equations. The Langmuir model will assume that the adsorption happens in a monolayer with similar and energetically equal active sites. Following is the isotherm equations:

$$q_e = \frac{(q_{max} K C_e)}{1 + K C_e} \quad (4)$$

where both the maximum adsorption capacity (mg/g), q_{max} and Langmuir equilibrium constant (L/mg), K will be obtained from the correlations. This Freundlich model explains a heterogeneous system. The system can be defined in the following equations:

$$q_e = K_f C_e^{(1/n)} \quad (5)$$

Linear regression will be widely employed to find the best-fitting isotherms. The linear that displays the maximum linearity when the equations are applied will validate the experimental results, the coefficient of the isotherm may be derived [15].

3. Results and Discussion

3.1. Characterization of adsorbent.

Scanning Electron Microscopy (SEM) micrograph presented in Figure 1 is the untreated sugarcane bagasse sample in various magnifications. Before the treatment, the surface of the sugarcane bagasse shows basic and compact fiber surface structures, as seen in Figure 1(a) under a magnification power of 50× [16]. Raw sugarcane bagasse magnified to 6000× has no or very few pores on the surface, thus acquiring less surface area for the adsorption process [17].

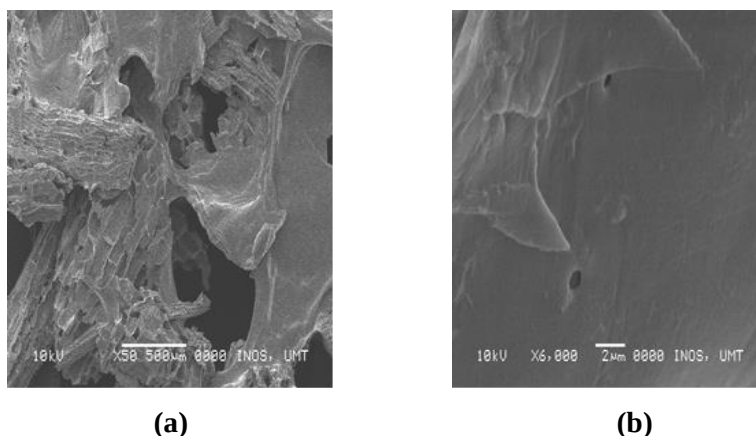


Figure 1. SEM image of untreated sugar cane at (a) 50x magnification; (b) 6000x magnification.

Images obtained by SEM in Figure 2 below revealed the structural surface differences of sugarcane bagasse after treatment with chitosan at different magnification power. Figure 2(a) indicates that the adsorbents have an uneven and rough surface morphology with a high potential for dyes to be trapped and adsorbed due to increased surface area [18]. This morphology also demonstrates that the material has a porous structure, as illustrated in Fig 2(b), demonstrating that sugarcane bagasse is composed of a carbonaceous skeleton [19]. It

was also supported by [17] that a suitable adsorbent should possess the characteristic of a porous structure to increase the surface area. The sugarcane bagasse was heated in the furnace for 2 h at 200°C before being submerged in chitosan solution, resulting in substantial pore growth, as seen in Figure 1, and a greater surface area and dye adsorption capacity to be removed [20].

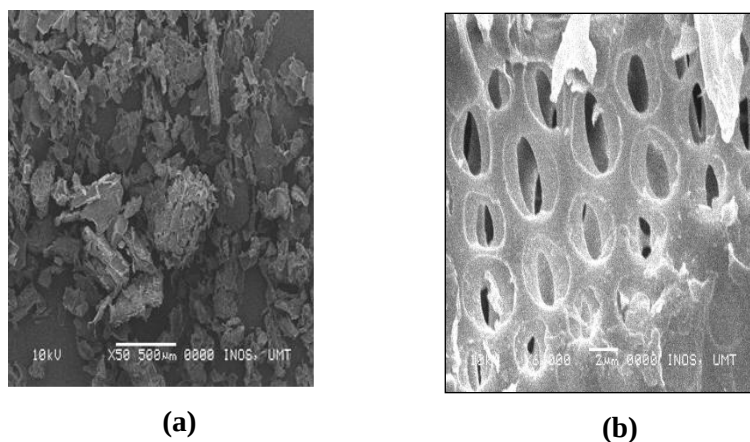


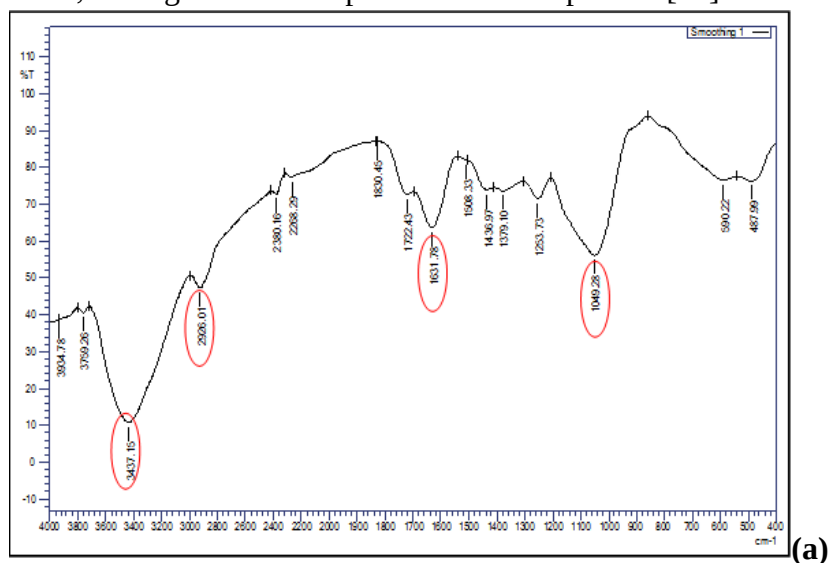
Figure 2. SEM images of treated sugar cane with chitosan at **(a)** 50× magnification; **(b)** 6000× magnification.

The SEM morphology also shows that chitosan was successfully assembled with the sugarcane bagasse when immersed into chitosan solution for 24 hours in the treatment process, as seen in the image. In addition, the coated sugarcane surface with chitosan helped attract anionic dye molecules onto its positively charged surface for dye removal [21].

3.2. Fourier Transform Infrared (FTIR).

Figures 3(a) and (b) show the adsorbent's FTIR spectra before and after the treatment process in the region of 4000–400 cm^{-1} . The FTIR spectrum of untreated sugarcane bagasse presented the specific broadband at 3437.15 cm^{-1} postulates to the presence of hydroxyl group (–OH).

This band's position and asymmetry at lower wavenumbers imply the presence of strong hydrogen bonding. Absorbed water aliphatic primary and secondary alcohols present in cellulose, hemicellulose, and lignin are examples of –OH compounds [22].



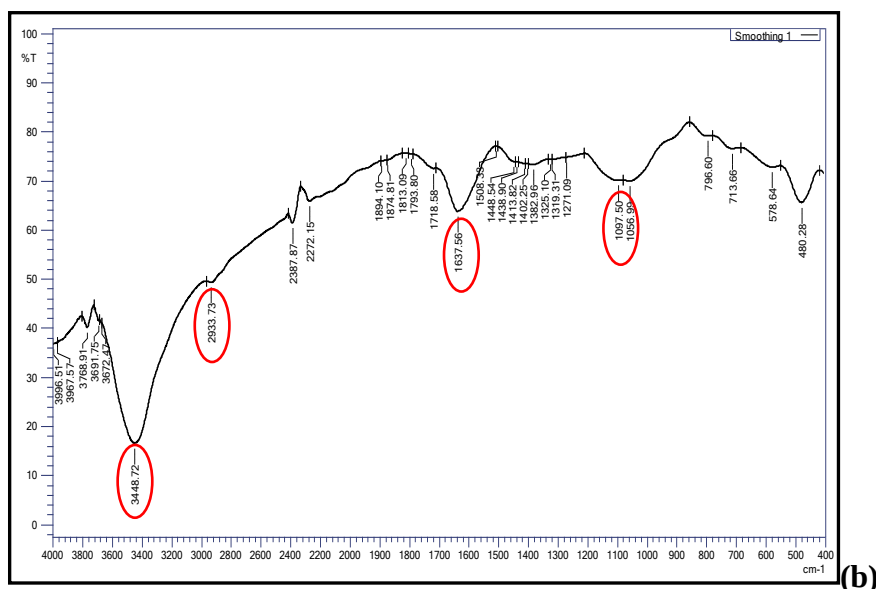


Figure 3. FTIR spectra analysis of (a) untreated sugarcane bagasse; (b) chitosan treated sugarcane.

The shift number increased for -OH compounds may also indicate hydrogen bonds forming between both components and lead to the interface's good adhesion. C–H stretching vibrations should be related to the band at 2926.01 cm^{-1} . The stretching vibrations of C–C in an aromatic skeletal mode of lignin and CO of the alpha-keto carbonyl matched the peak at 1631.78 cm^{-1} . The aromatic C H bending vibrations of the syringe and guaiacyl units in lignin and the stretching vibrations of the C–O–C bridge in hemicellulose and cellulose were ascribed to the peak at 1049.28 cm^{-1} . The signal at 475 cm^{-1} in the carbon samples indicates the presence of silica [23].

Figure 3 (b) shows that the adsorbent contains all the primary adsorption peaks with additional peaks at 1637.56 cm^{-1} assigned as a vibration of -NH_2 of amide groups, confirming that chitosan is present on the surface of sugarcane bagasse [24]. The amide group's frequencies have shifted to 1637.53 cm^{-1} , suggesting that the acetic acid residue is bonded to the amine group in the chitosan chain. The electrostatic interaction between these groups and the negatively charged sites might explain the amide group shift.

The spectra of treated sugarcane bagasse also show the band between $3500\text{--}3300\text{ cm}^{-1}$ contributed to the NH_2 stretching of the primary amine, and a broad spectrum was attributed to NH_3 [25]. A peak in the range of 1400 cm^{-1} ascribed to the aromatic C=C in-plane stretching vibration. A weak absorption band at 796.60 cm^{-1} was also assigned to aromatic C–H and C–C out of plane vibrations, suggesting the emergence of polynuclear hydrocarbon structure.

3.3. X-ray diffraction (XRD).

The pattern of raw sugarcane bagasse and treated sugarcane bagasse with chitosan were reported similarly, as shown in Figure 4. The diffractogram of untreated sugarcane presented a typical peak of cellulose at $2\theta = 15.5^\circ$, and $2\theta = 21.4^\circ$ and the pattern depicted an amorphous character which is postulate to no crystallinity of sugarcane bagasse [26]. This amorphous composite indicates a good adsorbent characteristic by having more active sites accessible for the adsorbate.

Two diffraction peaks exhibited a weak diffraction peak centered at diffraction angle $2\theta = 16.7^\circ$ and sharp diffraction peaks at $2\theta = 22.0^\circ$ for sugarcane bagasse treated with chitosan

in Figure 4 [27]. As demonstrated in Figure 4, the strong diffraction peak of treated sugarcane bagasse suggests a high degree of crystallinity morphology of chitosan after treatment [28].

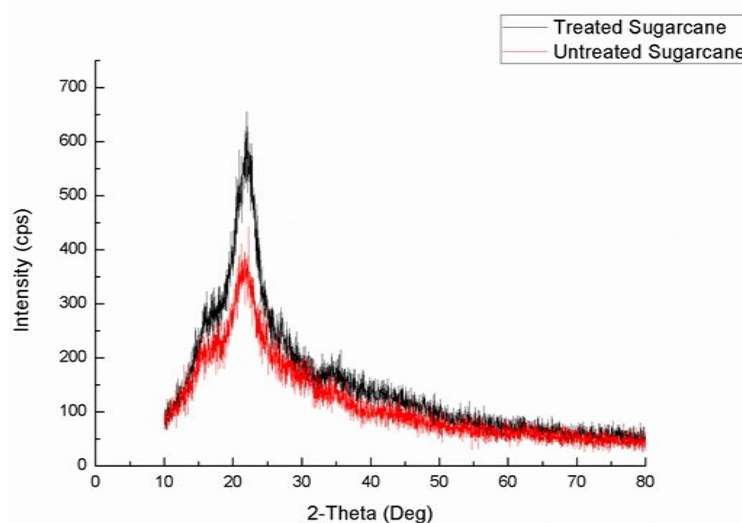


Figure 4. XRD pattern for both untreated and treated sugarcane bagasse.

3.4. Effect of types of adsorbent and contact time on Remazol Red dyes removal.

Figure 5 shows the graph of removal efficiency based on types of adsorbent used for Remazol Red dye. This observation shows that an untreated sugarcane bagasse sample alone cannot perform as a promising adsorbent. The highest removal obtained from untreated sugarcane was only 34.7%, with a contact time of 180 min due to the surface of the sugarcane bagasse before the treatment having smooth and minimal pores, leading to less surface area for adsorption to take place [17].

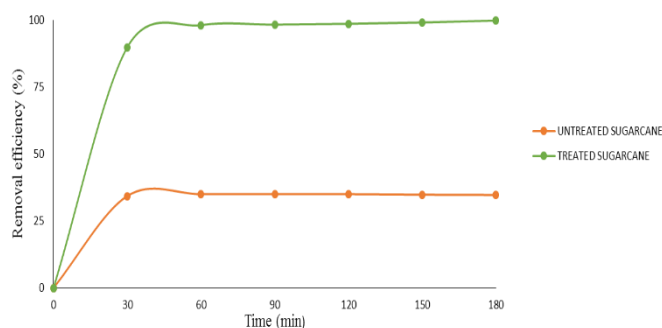


Figure 5. The removal efficiency of Remazol Red using different types of adsorbents.

However, the treated sugarcane with chitosan displayed a very high removal efficiency of 99.8 % after 180 min due to amino and hydroxyl groups in chitosan, which served as additional active sites for the adsorption process [29]. As shown in SEM images, the heat applied during the treatment has caused extensive pore development, resulting in a higher uptake capacity of dyes to be removed [20].

Both samples undergo rapid adsorption in the first 30 min, referring to the abundant availability of active sites on the surfaces in their initial stage. The adsorption rate gradually increased, leading to equilibrium at 60 min of adsorption. The reaching of equilibrium adsorption might be attributed to a decrease in the accessible active adsorption sites on the adsorbent with time, resulting in the reduced mass transfer of the adsorbate molecules from the bulk liquid to the adsorbent's external surface [30]. The experiment results demonstrate that the sugarcane bagasse treated with chitosan had the maximum removal effectiveness, with an equilibrium time of 60 min.

3.5. Effect of dosage on Remazol Red dyes removal.

The dosages were manipulated in the range of 50 mg to 150 mg of treated sugarcane bagasse as adsorbent, as shown in Figure 6. The percentage removal of Remazol Red increased with the increase in dosage of adsorbent.

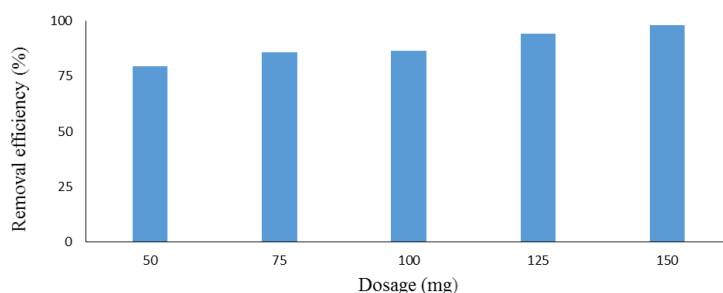


Figure 6. The efficiency of Remazol Red using different adsorbent dosages.

The lowest dosage of adsorbent would give the lowest removal efficiency of 79.4%, shown by 50 mg of treated sugarcane with chitosan. The results exhibited an increase in trend when 75 mg, 100 mg, 125 mg of adsorbents in 100 ml of 10 mg/L dye solution. The results achieved more than 80 % removal percentage, 86%, 86.6%, and 94.4 %. The adsorbent dosage of 150 mg displayed the highest removal percentage of 98.1% at 60 minutes because of the increasing availability of active sites resulting from the increased dosage and conglomeration of the adsorbent [31]. Thus, an increase in adsorbent dosage increased the number of active sites available to the solute for adsorption, which resulted in an increasing rate of adsorption. The adsorbent dosage of 150 mg of treated sugarcane bagasse was chosen for further experiments.

3.6. Effect of initial concentration on Remazol Red dyes removal.

The effects of initial Remazol Red dye concentration on percentage removal utilizing treated sugarcane bagasse were investigated in Figure 7. The percentage removal of the dye reduces as the initial dye concentration increases due to a decrease in rapid solute adsorption. It also lacks the accessible active sites necessary for Remazol Red's high initial concentration [32]. The results reveal that when the initial concentration increased from 10 mg/L to 100 mg/L for 150 mg adsorbent for 60 min, the percentage of dye removal reduced from 98.1% to 61.5%

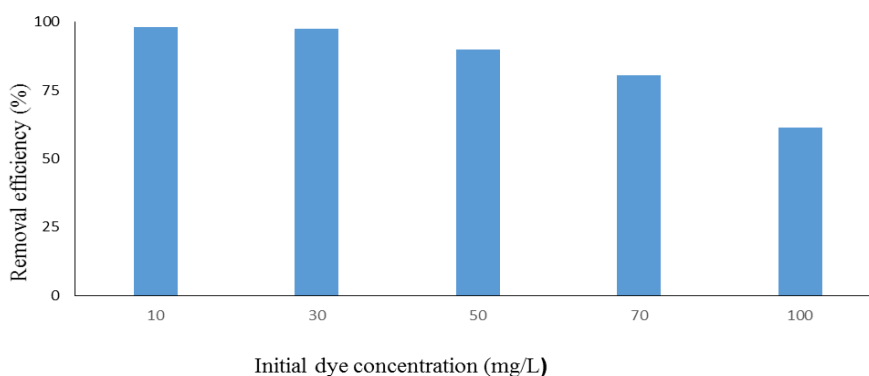


Figure 7. The efficiency of Remazol Red using different initial concentrations.

Increased initial concentration decreased the removal efficiency of dyes and required a more extended time to reach equilibrium. Therefore, an initial concentration of 50 mg/L of treated sugarcane bagasse was used for subsequent studies.

3.7. Effect of pH on Remazol Red dyes removal.

A change in pH value might affect adsorbent adsorption capacity by altering the surface charges of the adsorbent. It may also affect the active sites available for adsorption [20]. Using 0.01 M HCl and 0.01 M NaOH, the pH values were changed from 2 to 10. As shown in Figure 8, the optimal pH was obtained at pH 6, with a maximum removal percentage of 98.2 %. When the pH of the solution was reduced, the proportion of anionic dye removed decreased. Chitosan comprises neutral ($-\text{NH}_2$) or cationic ($-\text{NH}_3^+$) amino groups. These groups were protonated in acidity and kept neutral in an aqueous environment, absorbing negative counter-ions. In this situation, we have an ionic exchange mechanism involving electrostatic interactions between the chitosan molecules on the sugarcane surface and the Remazol red dye molecules [33].

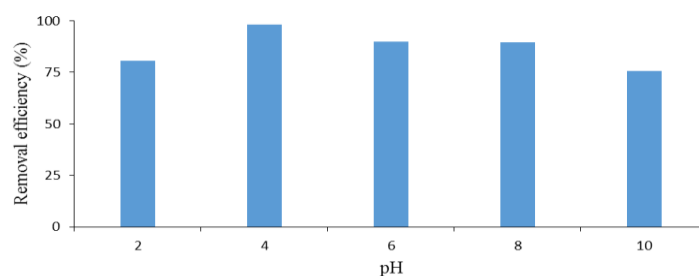


Figure 8. The efficiency of Remazol Red using different pH.

The results demonstrate that anionic dyes adsorb more significantly in pH 4. At pH 2 (acid), the anions in the solution compete with the anionic dye, resulting in a reduction in adsorption. The polymer's amino groups are deprotonated in an alkaline pH solution [34]. In summary, at a low pH, more protons are available to protonate the chitosan amine groups to create ($-\text{NH}_3^+$) groups on the composite surface, enhancing the electrostatic attraction between negatively charged anion dyes and positively charged adsorption sites, causing dye adsorption to increase.

3.8. Adsorption Isotherm.

The Langmuir and Freundlich isotherms were plotted, as illustrated in Figures 9 and 10. The values of isotherms constants and correlation coefficients were determined for both isotherm models and were calculated and summarised in Table 1.

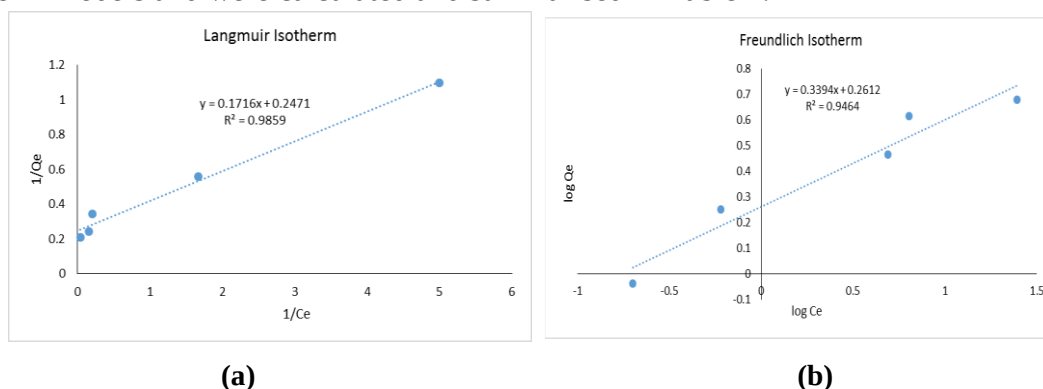


Figure 9. (a) The Langmuir isotherms; (b) the Freundlich isotherms.

The value of isotherms constant and correlation coefficients was calculated for both isotherm models, as stated in Table 1, using the data obtained from Figure 9.

Table 1. Langmuir and Freundlich isotherms parameters for Remazol Red adsorption onto treated sugarcane bagasse with chitosan.

Langmuir				Freundlich		
Q _{max} (mg/g)	K _L (1/mg)	R _L	R ²	K _f (1/mg)	1/n	R ²
4.04	1.44	0.07	0.985	1.825	0.3394	0.946

According to the correlation coefficient of treated sugarcane bagasse with chitosan, the Langmuir model complemented the data better than the Freundlich model. The maximum adsorption predicted by the Langmuir isotherm model of adsorbents was 4.04 mg/g. The R_L value was determined to be 0.07, confirming that the Langmuir Isotherm was favorable for Remazol Red adsorption onto the adsorbents utilized in this investigation. Confirmation of the experimental findings with the Langmuir isotherm model suggests that the surfaces of the adsorbents are homogenous.

4. Conclusions

The present study has proven that the sugarcane bagasse had better performance evaluation when treated with chitosan. It was found that the highest uptake of dyes was using 150 mg of treated sugarcane bagasse with chitosan, which displayed 99.8% removal efficiency after 180 minutes. The adsorbed quantity of dyes decreased when the concentrations of dye were increased. It was also studied that removal of Remazol red favored acidic conditions (pH 4) due to protonated of amine groups in chitosan to formed (-NH₃⁺) groups on the composite surface which increased the electrostatic attraction between the negatively charged anion dyes and positively charged adsorption sites which helped in the uptake of dyes. The adsorption studies also showed that the Langmuir isotherm was favorable for the adsorption of Remazol Red onto the adsorbents used in this study. Confirmation of the experimental data with the Langmuir isotherm model indicates the adsorbents' surfaces' homogeneous nature.

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Conflicts of Interest

The authors declare no conflict of interest.

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