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Original Research Article

Investigation of Characterisation for Biosorption of Congo Red from Textile Wastewater Using *Spathodea Campanulata* Leaves: FTIR, SEM, and XRD Analysis

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ABSTRACT

In the present study, the bio sorbent was investigated for its surface functional groups using Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), and X-ray diffraction (XRD). Before and after bio sorption studies, The FTIR spectra for the study's bio sorbent were acquired, and it was discovered that the sites on the bio sorbent that accommodate the exchangeable dye could function as active sites for the exchange of dye. The significant numbers of heterogeneous pore layers in the bio sorbent that are clearly visible in the SEM microscopic image indicate that there is a good chance for the adsorption of the dye. However, the surface of the dye-loaded bio sorbent demonstrates unmistakably that the bio sorbent's surface was covered in dye. The XRD analyses verified the bio sorbent's amorphous and crystalline character.

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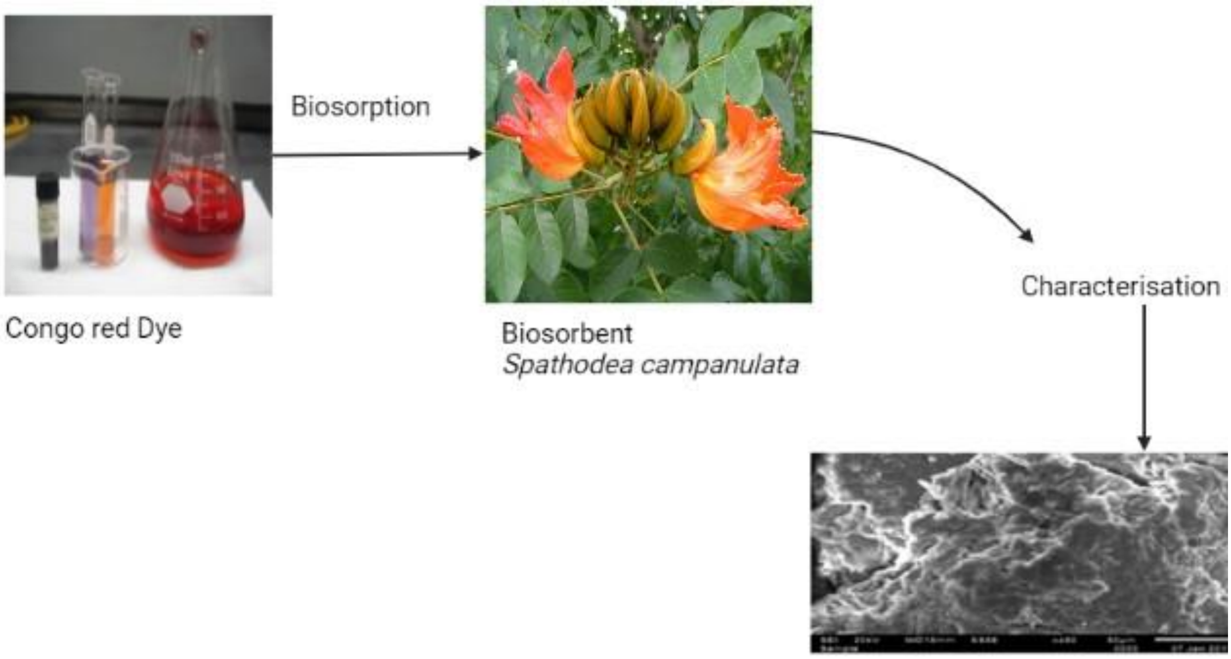
GRAPHICAL ABSTRACT



Introduction

Dyes are used in industries such as rubber, paper and pulp dyeing, and dye intermediates pharmaceutical, tanning, food technology, hair dyeing, plastic, and cosmetic, *etc.* [1]. The textile industry consumes more than 107 kg of dyes per year of which 90% are from fabrics [2]. In India the dye industry produces about 60000 tons of dyes which is about 6.6% of the total global production [3]. According to the central pollution control board (CPCB), India, there are approximately a million known dye and dye intermediates out of which 5000 are produced commercial, based on their use, dyes are classified into 15 groups. They are acid dyes, azo dyes, basic dyes, direct dyes, food dyes, metal Complex, mordant dyes, whitening agent, pigment dyes, reactive dyes, solvent dyes, sulphur dyes, and VAT dyes. According to their chemical structure that can be classified into cationic (basic), anionic (direct, acid, and reactive dyes) and nonionic dyes (disperse dyes). Many researchers found that colorant may cause problems in aquatic ecosystem in several ways as follows:

1. Dyes can have acute chronic effect on exposed organisms, depending on the dye concentration and on the exposure time.
2. Coloration of surface waters which captures the attention of both public and the authorities.
3. Dyes present in the water affect the sunlight entering the water and have a drastic effect on the growth of bacteria and disturb the biological activity.
4. Dyes have complex molecules structure which cannot be removed by municipal wastewater treatment operations.
5. Dyes in wastewater undergo chemical and biological changes that consume dissolved oxygen from the stream and destroy aquatic life.

The process of biosorption is which molecules of a gas or liquid build up on the surface of another solid material (either live or dead biomass) [4-5]. The biosorbent chosen for the present study is available plenty in nature. Therefore, *Spathodea Campanulata* leaves was chosen as biosorbent for the removal of Congo red dye using batch techniques. The functional groups on the surface of the biosorbent that contribute to the biosorption are characterized using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM), and X-Ray Diffraction (XRD) analysis.

Experimental

Preparation of biosorbents

The collected *Spathodea campanulata* leaves were washed with deionized water several times to remove dirt particles. The washing process was continued till the wash water contains no dirt. The washed leaves were then completely dried in sunlight for 20 days. The dried leaves were then cut into small pieces and powdered using domestic mixie. In the present study, the powdered materials in the range of 53-152 mm particle size were directly used as biosorbents without any pretreatment.

Preparation of dye solutions

Stock solutions of crystal violet and congo red concentration 1000 mg/L were prepared by dissolving 1 g of 100% crystal violet and 1 g of 100% congo red in 1000 ml of distilled water. The solution was prepared using standard flasks. The range of concentration of the prepared dye solutions varied between 20 and 200 mg/l was prepared using the stock solution of individual dye.

Surface Characterization of Biosorbent

Scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) were employed for surface investigation of *spathodea campanulata* leaves. The surface morphology of *spathodea campanulata* leaves before and after biosorption was examined using a scanning electron microscope.

Results and Discussion

Fourier transform infrared spectroscope (FTIR)

The functional groups contained in the biosorbent are revealed by Fourier transform infrared spectroscopy (FTIR) in terms of their structure and chemical makeup. FTIR spectra with wave numbers between 400 and 4000 cm^{-1} were used to examine the functional groups present in the leaves of *Spathodea Campanulata* both before and after congo red biosorption, as displayed in Figures 1 and 2 [6].

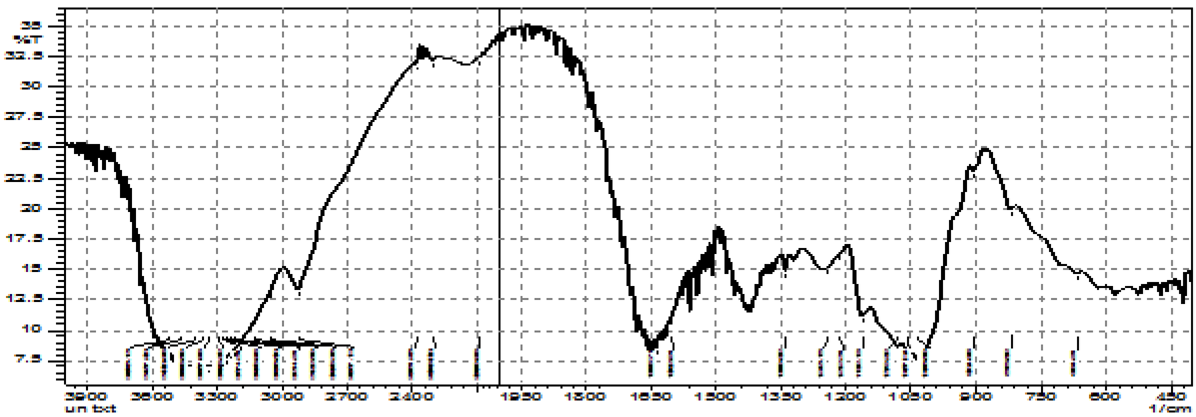


Figure 1: Untreated *spathodea campanulata* leaves as biosorbent

According to Figure 2, it is evident that different functional groups could be responsible for the biosorption of Congo red to the biosorbent. The broad bands at 818.82 cm^{-1} and 904.65 cm^{-1} indicate the presence of C–Cl stretching and C–C stretching bands, respectively. The band at 939.37 cm^{-1} is characteristic of C–S stretching bond. The shifts of the FTIR peaks are presented in Table 1. Fourier transform infrared spectrum analysis reveals the presence of large number of functional groups on the surface of the biosorbent from *Spathodea campanulata* leaves.

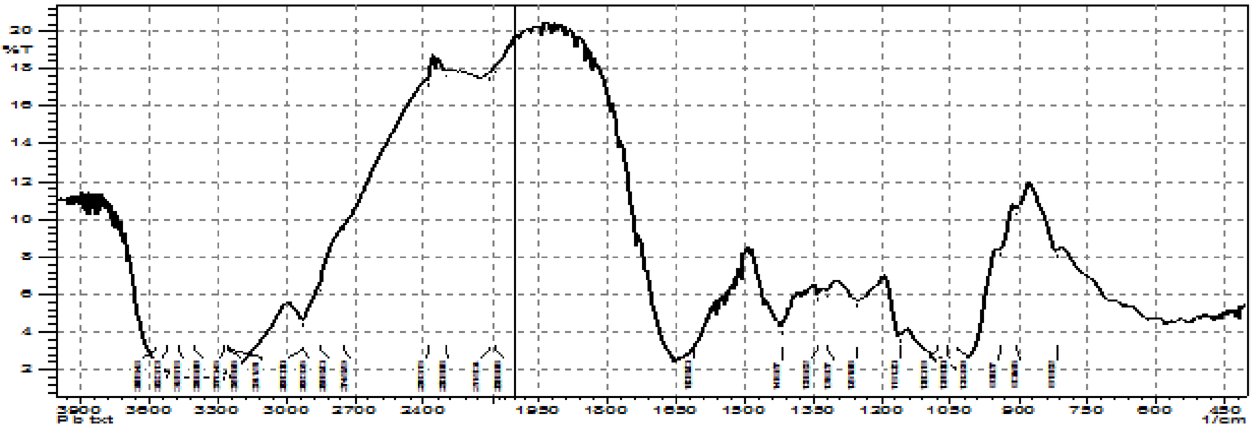


Figure 2: Treated congo red dye

Table 1: Comparison of the biosorption capacity of the adsorbents/biosorbents for the removal of Congo red from textile waste water

Sl No.	Biosorbent	Dye Uptake (mg/g)	Reference
01	Hazelnut shell-activated carbon 750 ^{0}C	8.82	11
02	Coir pith carbon	5.87	12
03	Apricot stones-activated carbon 750^{0}C	4.11	13
04	Fir wood based activated carbon	1.21	14
05	Corncob based activated carbon	0.84	15
06	Calcined raw kaolin	7.59	16
07	Glass wool	2.24	17
08	*Caulerpa racemosa var. cylindracea*	5.23	18
09	Living biomass	1.17	19
10	Cashew nut shell	5.31	20
11	Spathodea campanulata leaves	11.73	Present study

Scanning electron microscope (SEM)

Figures 3 and 4 illustrate the morphological evaluation of the biosorbent from the leaves of *Spathodea campanulata* before and after biosorption of congo red using SEM and microscopic pictures. The crude biosorbent, or before biosorption of dye, has a highly porous and heterogeneous shape, as depicted in Figure 3. This illustration demonstrates the rough surface of the biosorbent. Figure 4 displays SEM microscopic pictures of the biosorbent following the biosorption of the dye congo red [7-8].

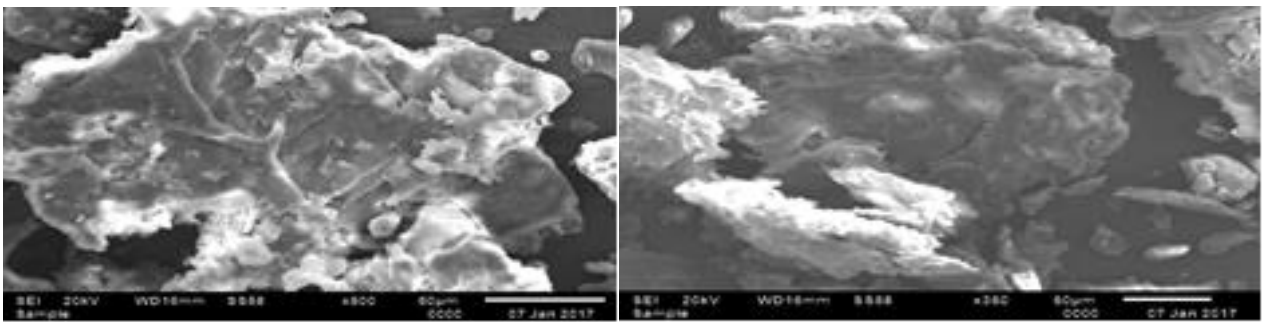


Figure 3: SEM micrographs of *Spathodea campanulata* leaves biosorbent before biosorption

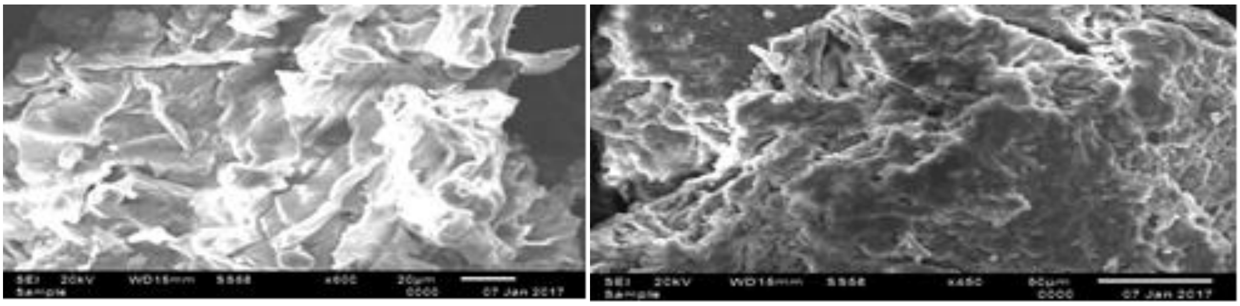


Figure 4: SEM micrographs of *Spathodea campanulata* leaves biosorbent after biosorption with congo red

X- ray diffraction

The X-ray diffraction diffractograms of the biosorbent from *Spathodea campanulata* leaves before and after biosorption of congo red dye are illustrated as plots in Figures 5 and 6. The intensities of diffracted X-ray were recorded as a function of 2ϴ using a copper target (Cu-Kα radiation with wave length λ= 1.5492 A0) at a scanning speed of 20/min [9-10].

X-ray diffractogram of *Spathodea campanulata* treated with the congo red is shown in Figure 6. Accordingly, it can be seen that the XRD pattern has amorphous and crystalline features. The peaks at 2θ values of 0.5577, 0.5538, 0.4788, 0.4165, and 0.4605 show the presence of $Rb_{12}Si_{17}$, $C_{448}Al_{638}N_{56}Na_{560}O_{5866}Si_{2002}Sr_{11}$, $Ca_{13}Cd_{76}$, $Co_{2.37}Zn_{2.63}$ and $BaCd_4Pt_2$(ICDD files). Their corresponding d-values are 4.0539, 4.0750, 4.1247, 4.4991, and 4.1392, respectively.

These variations in biosorption capacity could be due to the differences in the properties of the biosorbents such as active surface area, porosity, functional groups, etc. The data also indicated that the biosorbent has shown high affinity towards removal of congo red. This could be due to the variations in the chemical properties and sizes of dye molecules. The abundant availability, cost effectiveness, good biosorption capacity and rapid biosorption rates suggested that the *spathodea campanulata* leaves can be efficient and effective biosorbents for the removal of congo red dye from textile waste water.

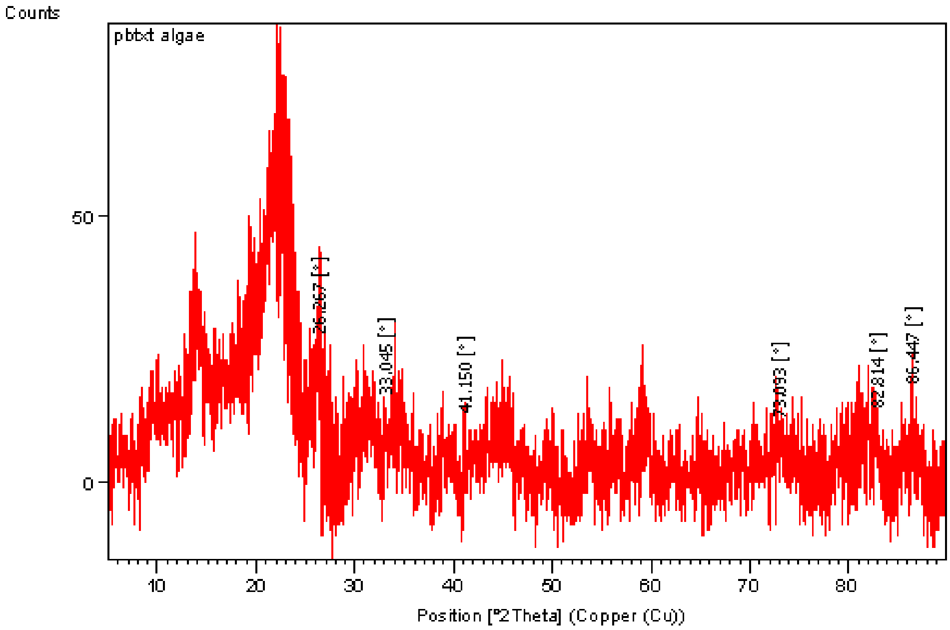


Figure 5: XRD pattern of Congo red treated *Spathodea campanulata*

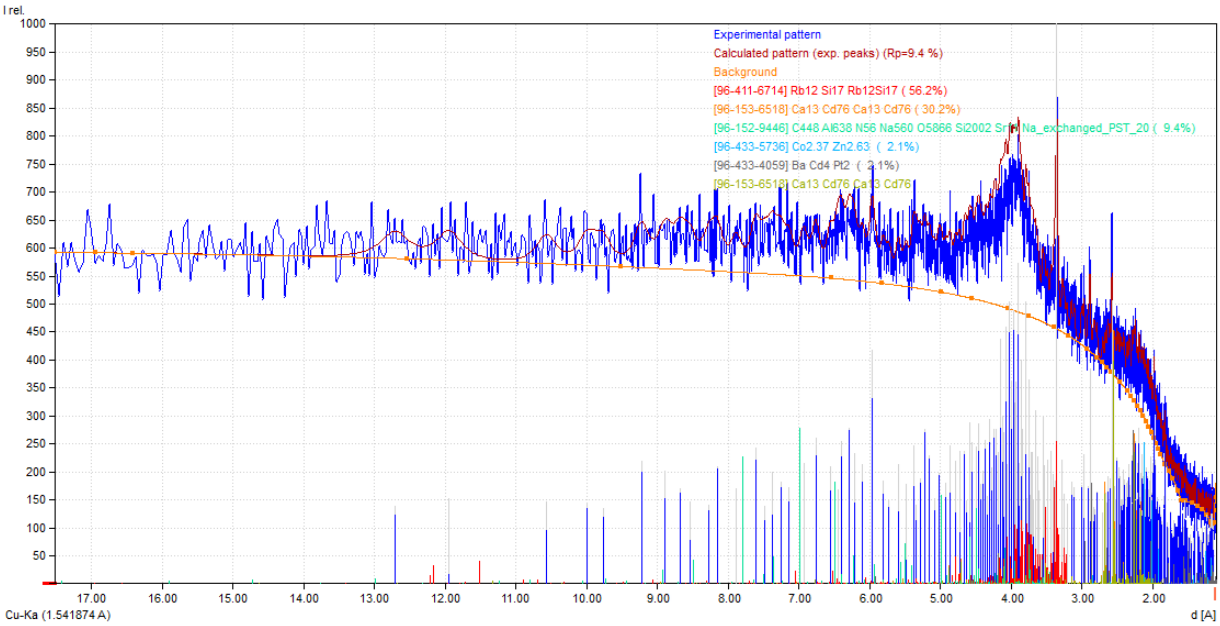


Figure 6: XRD spectrum of Congo red treated *Spathodea Campanulata* with matching compounds

Conclusion

The presence of several functional groups on the surface of the biosorbent was confirmed by FTIR analysis. This investigation verified that the biosorption of dyes involves the presence of the hydroxyl, carboxyl, amino, sulfone, and phenol groups. The existence of several functional groups of cellulose was revealed by XRD analyses. The binding of dyes to the surface of the biosorbent was confirmed by SEM examination. According to the findings, spathodea campanulata is an efficient, affordable alternative to other biosorbents for removing congo red dye from industrial effluent.

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Authors' Contributions

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work

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