Scientific Research Publishing

Advances in Chemical Engineering and Science, 2023, 13, 189-202
https://www.scirp.org/journal/aces
ISSN Online: 2160-0406
ISSN Print: 2160-0392

Adsorption of Anionic and Cationic Dyes from Textile Effluents by Activated Carbon Prepared from Sawdust and Fish Scale

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How to cite this paper: Jahan, R.A., Hassan, M.M., Rana, A.A. and Karim, M.M. (2023) Adsorption of Anionic and Cationic Dyes from Textile Effluents by Activated Carbon Prepared from Sawdust and Fish Scale. *Advances in Chemical Engineering and Science*, **13**, 189-202.
https://doi.org/10.4236/aces.2023.133014

Received: April 13, 2023
Accepted: June 9, 2023
Published: June 12, 2023

Abstract

In Bangladesh, there are thousands of textile-dying industries spread across the country's many regions, the majority of which involve knitting and dying. The dyeing industry uses an enormous quantity of water, as well as colors and chemicals. After the dying process has been completed, they also release a significant amount of wastewater. Cotton, wool, and polyester fiber are typically dyed with textile dyes such as reactive, acid, and disperse dyes. These dyes are utilized most frequently in the respective sectors. The dyes' colorants are extremely poisonous and dangerous to all forms of life, including aquatic life and living things. The present work has been intended to investigate whether or not it is practicable to remove commonly used textile dyes simultaneously from an aqueous dye solution using an adsorption technique that makes use of a variety of different adsorbents. This study focuses on the removal of color from two distinct types of dyes—Methylene Blue and Reactive Blue-250 which are cationic and anionic in nature respectively, using two different types of activated carbon adsorbents prepared from sawdust and fish scale. Dye removal capacity was tested as a function of contact time, the dosage of the adsorbent, pH during the treatment process, temperature and initial concentration of dye. The applicability of the Langmuir and Freundlich adsorption isotherms in describing experimental data was investigated. The micro and mesoporous activated carbon prepared from sawdust and fish scale identified by Scanning Electron Microscopy (SEM) images indicated that such adsorbents with a large surface area have more dye adsorption potential whereas the variation in dye adsorption occurs due to variation in surface area. From the overall experimental data, maximum removal of 95.39% and 87.92% was found for Methylene Blue and Reactive Blue-250 respectively by sawdust, and 90.64% removal of Methylene Blue by using fish scale.

Keywords

Textile Wastewater, Ionic Dyes, Removal, Activated Carbon, Adsorbent

1. Introduction

The textile industry is one of the major sectors that have a significant contribution to the economic development of Bangladesh. The vast majority of these textile enterprises are knit dyeing, and there are approximately 2000 knit dyeing industries in Bangladesh. The dyeing industries utilize a significant quantity of textile dyes and chemicals in order to dye various types of fabrics made of cotton, wool, and polyester in common cases by reactive, acid, and disperse dyes respectively. The dyes used in textiles are extremely hazardous to our natural environment, causing damage not only to plants and aquatic systems but also to human lives [1]. According to the estimation provided by the World Bank, 16 to 22 percent of total pollution of water in Bangladesh is caused by the dyeing and treatment of textiles. As a consequence, this causes a negative impact on the environment.

In order to facilitate the dyeing processes properly, the dye added with some auxiliary chemicals must be dissolved within an adequate amount of aqueous solution. The chemical structure of a pigment, also known as a dye, is made up of a variety of chemical groups, chromophoric groups, and auxochromes [2]. Because of the presence of these chemical groups, the dyes are responsible for a number of diseases including cancer, skin corrosion, kidney damage, and other long-term injuries [3].

There have been a lot of conventional processes developed for removing the dyes from textile effluents such as coagulation, flocculation, biodegradation, and others [4] [5]; however, these are all resulted with excessive sludge production, decreased efficiency, and an inability to effectively combat certain dyes. Other techniques such as using activated carbon, biomass, ion exchange, membrane separation, oxidation, and so on are also available [6] [7] [8] [9], but they have a number of drawbacks such as inability to treat large volumes of effluents, non-economic, ineffectiveness for disperse dyes, a high cost of energy and chemicals that prevent them from being practical applications.

Considering the efficiency, ease of development and relatively low cost, adsorption especially by the carbon-based materials, has become an increasingly popular method for the removal of textile dyes in recent years [10]. Nowadays, numerous efforts have been made to utilize wastes as raw materials in producing activated carbon [11]. Because of the chemical and structural characteristics of activated carbon adsorbents, it is possible for them to adsorb dye particle from an aqueous solution and therefore to function as an appropriate material for the dye removal [12]. One of the most significant characteristics of activated carbon is to be the high degree of micro porosity with a low volume. This increases the surface area that is available for chemical reactions. Based on the results of gas adsorption, only one gram of activated carbon possesses a surface area that is greater than 3000 m^2 (32,000 sq ft) [13]. The capacity of these adsorbents to perform their intended function is contingent upon a number of variables, including the dosage of the adsorbents, pH of the solution, temperature, and the type of dye that needs to be removed.

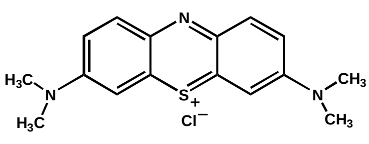
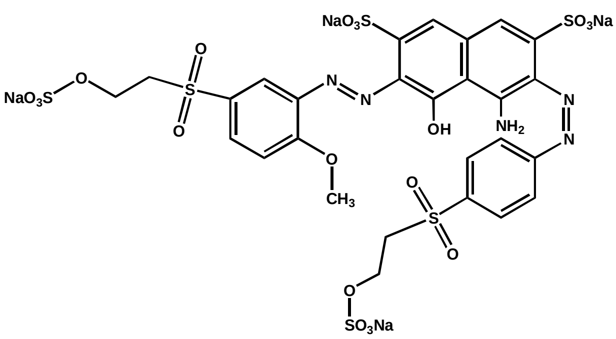
Numerous studies have been reported on activated carbons from natural and agricultural wastes including rice husk, fish scale, jute stick, bagasse, sawdust, coconut shell etc. [4] [14] [15] [16]. Since Bangladesh is mostly an agriculture-based country, a wide variety of agricultural and natural wastes are readily available here. In the current study, sawdust and fish scale were used as precursor raw materials which is then treated physically and chemically to produce activated carbon on a laboratory scale using activating agents to improve the adsorption capacity of the prepared adsorbent.

2. Materials and Methods

2.1. Selected Dyes

Methylene Blue and Reactive Blue-250 were used for the adsorption study since these two dyes are frequently used in the textile industries of Bangladesh. The textile wastewater containing the dyes was collected from Nice Fabrics Ltd., which is situated at Mawna, Sreepur, Gazipur and for standard sample preparation, the primary standard Methylene Blue and Reactive Blue-250 were also collected from the same source. The chemical and structural properties of the selected dyes are shown in **Table 1**.

Table 1. Chemical and structural properties of Methylene Blue & Reactive Blue-250.

Name of the dye	Methylene Blue	Reactive Blue-250
Formula	$C_{16}H_{18}ClN_3S$	$C_{27}H_{23}N_5Na_4O_{20}S_6$
Molecular weight	319.85 g/mol	1021.9 g/mol
Chemical structure		
Structural category	Thiazine dye	Azo dye
Dye type	Cationic	Anionic

2.2. Chemicals

All the reagents and chemicals used in this work were of pure analytical grade, including phosphoric acid (H_3PO_4), hydrochloric acid (HCl), potassium hydroxide (KOH) and were used without further treatment.

2.3. Preparation of Adsorbents from Sawdust and Fish Scale

Sawdust was collected from a sawmill, located at Khilgaon in Dhaka and fish scale was collected from a local market, named Anandabazar, located near the University of Dhaka. The raw materials were washed properly with distilled water to remove dirt and impurities, dried and the materials were prepared to discrete sizes. After being washed, the sawdust was kept in an open environment for sun drying. The clean and dry sawdust was then pre-carbonized at 350˚C for 1 hr in the absence of air. The chemical activation of the prepared powder was carried out by mixing with 1 M H_3PO_4 and kept for around 24 h followed by washing with hot distilled water to neutralize the excess H_3PO_4. The sample was finally activated at a temperature of 600˚C for 2 hours in a muffle furnace. This activated sample was again washed with distilled water and heated at 110˚C in an oven overnight. The oven-dried sample was then ready for the treatment of textile wastewater.

The raw fish scale was washed with hot distilled water to take out the impurities and water-soluble substances and dried naturally. The pre-carbonization of the clean and dry fish scale was carried out at 350˚C in an inert atmosphere. Then the pre-carbonized material was mixed with 1 M KOH at a weight ratio of 1:1, in order to ensure chemical activation keeping the mixture for 24 hours. The obtained product was then washed with 0.5 M HCl to neutralize the excess KOH followed by washing with distilled water for several times till the obtention of neutral pH. The activated sample was then finally carbonized at temperature 900˚C for 60 minutes in a muffle furnace. After being carbonized, the final product was obtained by washing and drying at 120˚C.

2.4. Instruments and Software

Shimadzu UV-visible spectrometer (Model: UV-1800, Shimadzu, Japan), with 1 cm matched quartz cells was used for spectrometric measurements. UV-visible spectra of the samples treated with adsorbents were accumulated at wavelength ranging from 200 to 800 nm against a solvent blank. The spectral data were obtained by using software compatible to the instrument. Scanning Electron Microscopy (SEM) images were taken by the instrument (Model: JSM-6490LA, JEOL, Japan) at an accelerated electron voltage of 15 kV at different magnifications in micrometer scale. Structural as well as material identification of the produced activated carbon adsorbents have been performed using X-ray Powder Diffractometer (XRD) (Model: Ultima IV, Rigaku Corporation, Japan) and Fourier Transform Infrared spectrophotometer (FT-IR) (Model: IR Prestige-21, Shimadzu, Japan) respectively.

3. Results and Discussion

SEM micrography

Figure 1(a) and Figure 1(b) display the SEM images of the adsorbents prepared from sawdust and fish scale respectively. According to the figures, the adsorbent, prepared from

sawdust, has more porous and uniform surface morphology than the adsorbent prepared from fish scale indicating that the optimum conditions of activation temperature and chemical treatment of sawdust were found to be more efficacious to produce well-developed pores on the surface of the prepared activated carbon, which seems to be the important factor that resulted in the higher adsorption capacity of methylene blue and reactive blue-250. It has also been appeared that the size of the porous carbon particles of the both adsorbents were in the range of micrometer scale.

XRD patterns

Characteristic X-ray diffraction pattern of the prepared adsorbent from sawdust is shown in Figure 2. Two broad peaks appeared at approximately $2\theta = 25^\circ$ and 44° indicating the emergence of amorphous structure of activated carbon, which is an important property for porous adsorbents [17]. It has been reported that peaks around $2\theta = 44^\circ$ are due to the formation of pores by the decomposition of carbon towards the graphitic structures [18]. The relatively well-organized aromatic carbon which is more stable than amorphous-like carbon is produced in such cases.

FT-IR spctroscopic analysis

Activated carbon produced after the thermal and chemical treatment of fish scale and sawdust were analyzed by FT-IR to identify the chemical bonds present on the carbon surface and structural changes between these two types of adsorbents that came through different activation and carbonization processes. FT-IR spectra of the adsorbents prepared from fish scale and sawdust have been shown in Figure 3(a) and Figure 3(b) respectively.

The major characteristic absorption peaks observed for activated carbon adsorbent from the fish scale were assigned at 3495 cm^{-1} (O-H stretching bond of

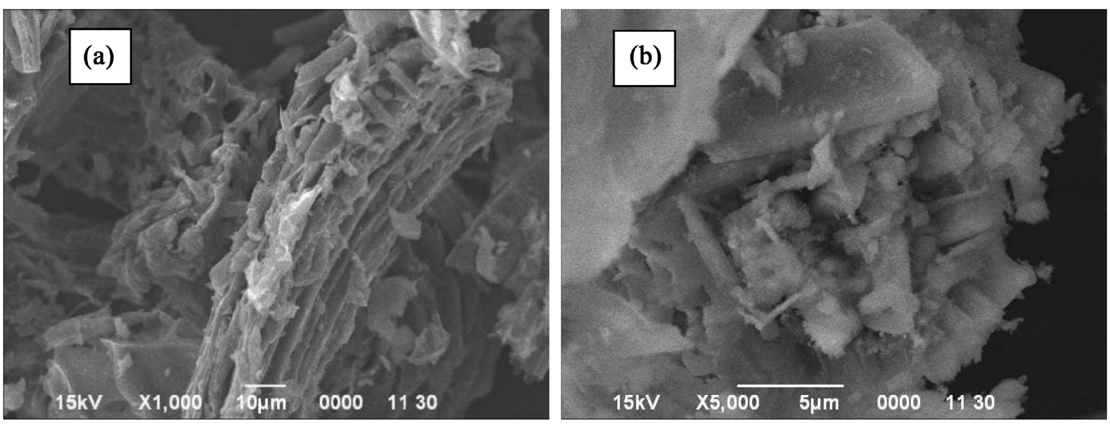


Figure 1. SEM images of the adsorbents prepared from sawdust (a) and fish scale (b).

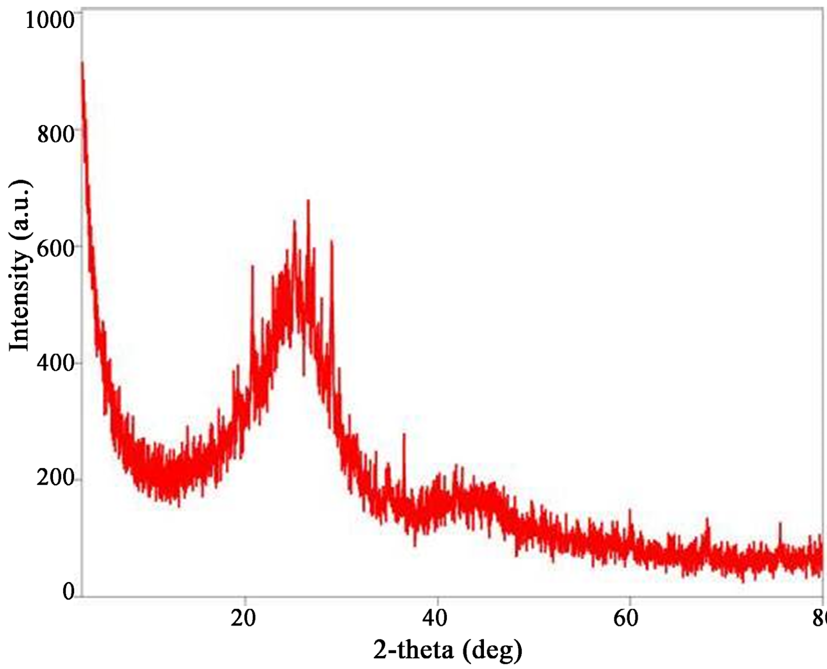


Figure 2. XRD pattern of the adsorbent prepared from sawdust.

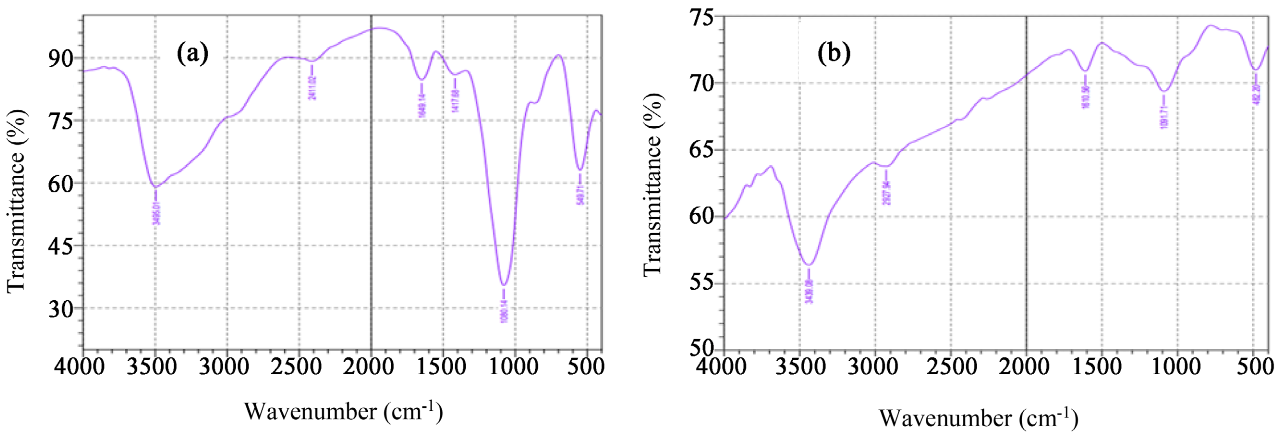


Figure 3. FT-IR spectra of the adsorbents prepared from fish scale (a) and sawdust (b).

free alcohol group), 2411 cm^{-1} (O=C=O stretching bond of carbon dioxide), 1649 cm^{-1} (C-H bending bond of aromatic compound), 1417 cm^{-1} (O-H bending bond of alcohol), 1080 cm^{-1} (C-O stretching bond of primary alcohol) and 549 cm^{-1} (C-Cl stretching bond). Similarly, in the FT-IR spectrum (**Figure 3**) of the adsorbent from sawdust, the obtained characteristic peaks were assigned at 3439 cm^{-1} (O-H stretching bond of intermolecular bonded alcohol group), 2927 cm^{-1} (O-H stretching bond of carboxylic acid), 1610 cm^{-1} (C=C stretching bond of conjugated alkene) and 1091 cm^{-1} (C-N stretching bond of amine) [19] [20].

Standard calibration curve by UV-Vis Spectroscopy

To estimate the concentration of the selected dyes, present in the collected textile effluents, UV-Vis spectroscopic analysis of the samples was carried out based on the standard calibration plot of light absorbance vs. standard solutions of 5, 10, 15, and 20 ppm of pure dye concentration (Methylene Blue) at its maximum absorbance 664 nm illustrated in **Figure 4**. Similarly, the calibration curve for Reactive Blue-250 was prepared spectrophotometrically using standard solutions at 614 nm which is the maximum absorbance of the corresponding dye. From the abovementioned standard curves, the value of the initial concentrations of Methylene Blue and Reactive Blue-250 in the textile wastewater were measured which were found to be 17.83 and 21.65 ppm respectively.

Treatment of the dye-containing textile wastewater

The textile wastewater containing Methylene Blue and Reactive Blue-250 was treated with the addition of the prepared adsorbents to calculate the removal percentage of the dyes from the effluent by varying the dosage and contact time with the adsorbents along with the change in initial dye concentration during treatment. The concentration of the two dyes in the textile wastewater was determined separately before and after the treatment with adsorbent by using the UV-Visible spectrophotometric technique and the dye removal percentage (%) was calculated by the following formula:

$$\text{Dye removal\%} = \frac{C_0 - C_e}{C_0} \times 100\% \tag{1}$$

where, C_0 is the initial concentration of dye and C_e is the final concentration of dye after treatment.

Effect of dosage on the removal of two dyes by the adsorbent prepared from sawdust

Effect of dosage of the adsorbent prepared from sawdust for the removal of cationic and anionic dyes Methylene Blue and Reactive Blue-250 respectively was examined by adding different dosages of adsorbent to the effluent wastewater

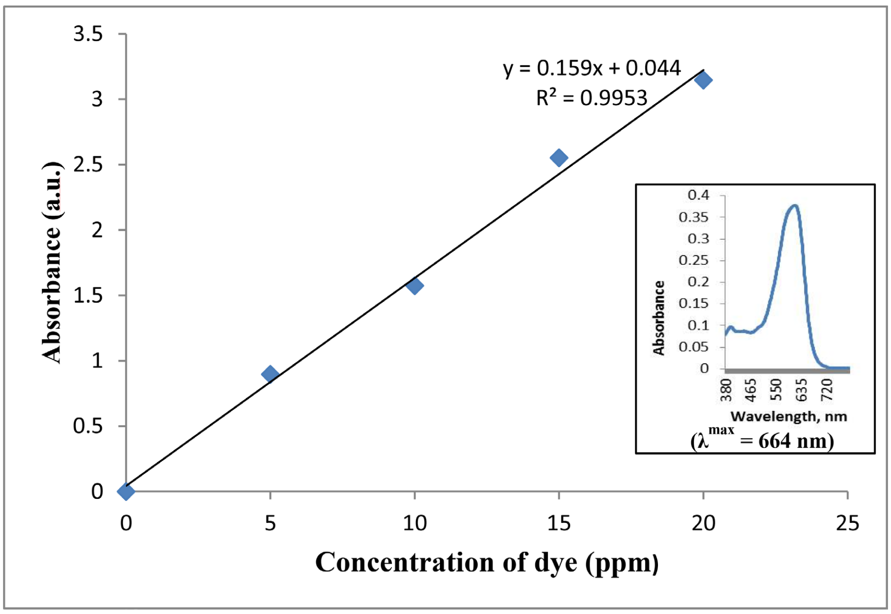


Figure 4. Standard calibration curve for methylene blue; inset shows the maximum absorbance of methylene blue at 664 nm.

at the reaction conditions listed in Table 2. After treatment with the adsorbent dosages (0.01, 0.03, 0.05, 0.10, and 0.15 g), the removal percentage of Reactive Blue-250 & Methylene Blue was determined by measuring the concentration of the resulting solution from UV-Vis absorption spectra and illustrated in Figure 5. From the plot of the amount of adsorbent dosage vs. % removal of dyes, the optimum dosage was determined 0.05 g for removing Reactive Blue-250 with the removal percentage of 84.78% and 0.03 g for the removal of Methylene blue with the removal percentage of 94.25%.

Effect of contact time on the removal of Methylene Blue by adsorbents from sawdust & fish scale

Removal percentage of Methylene Blue from the textile wastewater by adding fixed amount of adsorbents prepared from sawdust and fish scale to the effluent solution was measured UV-Vis spectrophotometrically at different contact time starting from 15 to 120 minutes while keeping other reaction conditions same as listed in Table 2. For both the adsorbents, the concentration of Methylene Blue in the wastewater was measured from the UV-Vis absorption spectra to calculate the percent removal of the dye and plotted against the contact time as shown in Figure 6. At various contact times for dye removal treatment with shaking, the impact of the period of shaking observed as the optimum dye removing percentage was found to be 93.08% at 30 minutes for the adsorbent from sawdust and 89.18% at 60 minutes for the adsorbent from fish scale.

Table 2. Conditions of treatment for removal of Reactive Blue-250 and Methylene Blue from textile effluent by adsorbent from sawdust.

Dye	Initial conc. (ppm)	pH	Sample amount (mL)	Treatment time (mins)	Agitation speed (rpm)
Reactive Blue-250	21.65	5.8	30	60	200
Methylene Blue	17.83	8.3	30	30	200

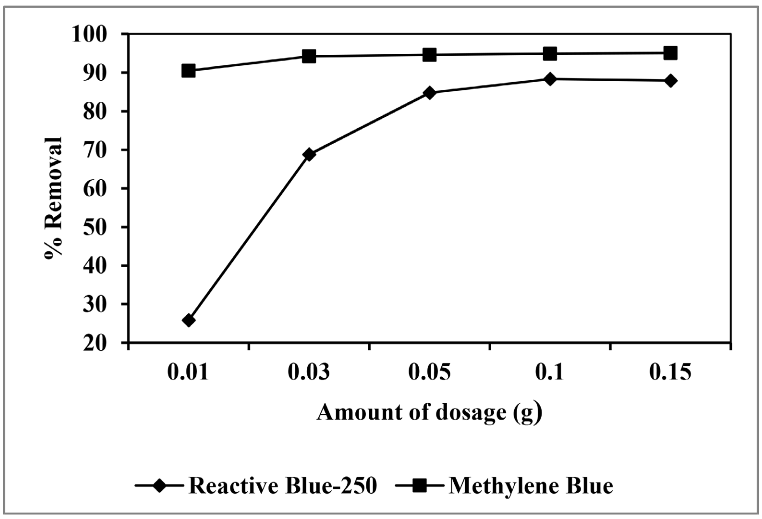


Figure 5. Effect of dosages of adsorbents from sawdust on % removal of two dyes.

Effect of initial dye concentration of the solution during treatment

The effect of initial dye concentrations on removal percentage of Methylene Blue and Reactive Blue-250 with the addition of adsorbent prepared from sawdust were observed by using different concentrations of the dyes. The experiment was carried out at room temperature using sample volume 30 ml, adsorbent dosages of 0.05 g at pH around 7 and a rotation of 200 rpm on the shaker with contact time of 60 minutes. The initial concentrations taken for Reactive Blue-250 and Methylene Blue were 21.65 and 17.83 ppm respectively whereas the other concentrations 30, 40 and 50 ppm of the dyes were prepared in an aqueous solution by adding a known amount of the dyes and the optimum dye concentration for adsorption was identified. The plot of initial dye concentration vs. % removal is illustrated in Figure 7.

For different initial dye concentrations, at equilibrium, the adsorption capacity of the adsorbent was obtained from following equation:

$$q_e = (C_0 - C_e) \times V/w \tag{2}$$

where q_e is the equilibrium dye adsorption capacity (in mg/g), C_0 and C_e are the

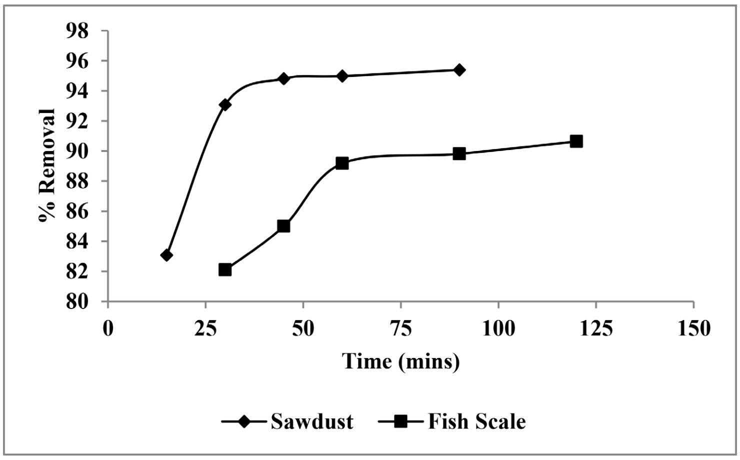


Figure 6. Effect of contact time of adsorbents on % removal of methylene blue.

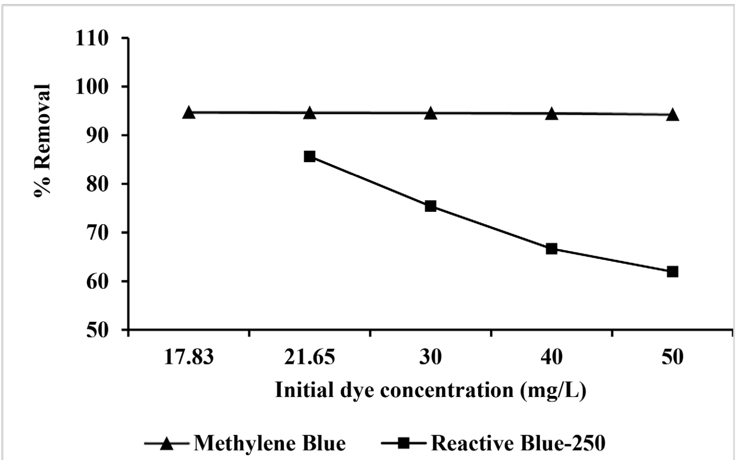


Figure 7. Effect of initial dye concentration on % removal of the dyes.

initial and equilibrium concentrations (in mg/L) of the dye respectively, V is the volume of the sample (in L) and w is the weight of the adsorbent (in g).

For Reactive Blue-250, the maximum q_e value was found 18.59 mg/g at the initial dye concentration 50 ppm. However, the removal percentage was reduced from 85.64% at initial concentration 21.65 ppm to 61.97% at 50 ppm. In the case of Methylene Blue, maximum q_e value was found 47.16 mg/g at the initial dye concentration 50 ppm. There is no substantial effect of initial concentration of Methylene Blue was observed as the removal percentage reduced from 94.81% at initial concentration 17.831 ppm to 94.33% at 50 ppm.

Adsorption isotherm

The molecular distribution of adsorbate on the surface of the adsorbent is determined by examining the experimental results with various isotherm models mostly to the Langmuir and Freundlich isotherms since these two equilibrium adsorption isotherms are applied for predicting the adsorption capacity and nature of the adsorbent in particular, information regarding the extent of homogeneity and heterogeneity of the adsorbent surface. In order to optimize the adsorption process of a system to remove dyes, the establishment of the appropriate correlations of the data for each system at equilibrium is required.

Langmuir adsorption model

In this regard, for the adsorption system of Methylene Blue and Reactive Blue-250 onto the adsorbent prepared from sawdust, the Langmuir model for adsorption has been tested since the best fit was achieved using this isotherm, which is valid for monolayer adsorption onto a homogeneous surface containing a finite number of identical sites [21]. The model assumes the uniformity regarding the energies of adsorption onto the surface. Langmuir adsorption parameters can be derived by transforming the Langmuir equation into linear form [22] as follows:

$$\frac{C_e}{q_e} = \frac{C_e}{q_{\max}} + \frac{1}{q_{\max} K_L} \quad (3)$$

where, C_e is the equilibrium concentration of dye (in mg/L), q_e is the amount of dye adsorbed per unit mass of adsorbent (in mg/g) at equilibrium, $q_{\max}$ and K_L represent the maximum adsorption capacity (in mg/g) and energy of adsorption (in g/L) respectively known as the Langmuir constants.

The value of Langmuir parameter was obtained by the linear correlation of the values of C_e/q_e versus C_e at room temperature as given in **Figure 8**, in which the equilibrium adsorption isotherm for Methylene Blue (**Figure 8(a)**) and Reactive Blue-250 (**Figure 8(b)**) onto the adsorbent surface prepared from sawdust has been shown.

The Langmuir adsorption isotherm parameters K_L and $q_{\max}$ were calculated from the slope and intercept of the plot of C_e/q_e versus C_e were presented in **Table 3** along with the correlation coefficient R^2. Further, the essential features of the Langmuir adsorption isotherm denoted by equilibrium parameter R_L, which is a dimensionless constant commonly known as separation factor. The equation

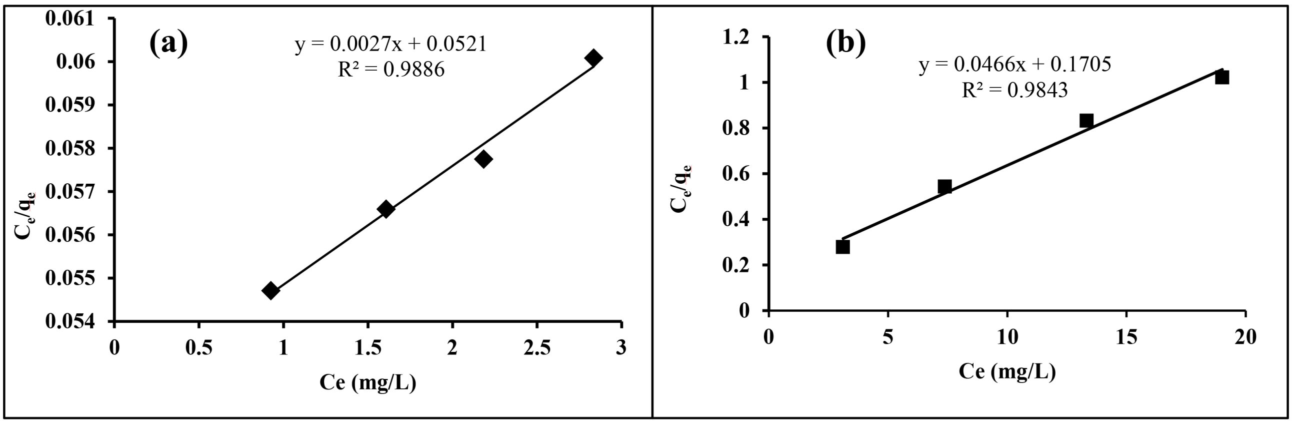


Figure 8. Langmuir adsorption isotherm for the removal of (a) Methylene Blue and (b) Reactive Blue-250 onto the adsorbent prepared from sawdust.

Table 3. The adsorption isotherms parameters for Langmuir and Freundlich model.

Adsorption parameters	Methylene Blue	Reactive Blue-250
Langmuir isotherm		
K_L	0.0027	00466
q_{max}	0.0521	0.1705
R_L	0.8810	0.3003
R^2	0.9886	0.9843
Freundlich isotherm		
K_f	1.2613	0.9028
$1/n$	0.9207	0.2766
R^2	0.9996	0.9849

of R_L is shown below:

$$R_L = \frac{1}{1+K_L C_i} \tag{4}$$

where, C_i is the highest initial dye concentration used in the adsorption experiments. R_L is the qualitative information regarding the favorability of the adsorption process; R_L greater than 1 implies unfavorable adsorption whereas R_L between 0 & 1 indicates a favorable adsorption process [23]. In the present study, for the adsorption of Methylene Blue & Reactive Blue-250 onto the adsorbent prepared from sawdust, the RL values (Table 3) indicate that for the treatment of both the cationic and anionic dyes, adsorbent, prepared from sawdust poses a favorable adsorption process for efficient dye removing.

Freundlich Adsorption model

At equilibrium, the amount of adsorbed dye can be predicted by using the Freundlich isotherm model which is commonly used to demonstrate the characteristic adsorption for the heterogeneous surface of adsorbent has also been tested for the adsorption system described in the present work. The linearized form of equation is shown below:

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \quad (5)$$

Here, the constant K_f approximately measures the adsorption capacity while $1/n$ is an indicator of the strength of adsorption or in other words, the effectiveness of adsorption, q_e represents the amount of dye adsorbed per unit mass of adsorbent (in mg/g) and C_e is the equilibrium concentration of dye (in mg/L).

From the plot of $\log C_e$ vs. $\log q_e$ as shown in **Figure 9**, both the values of $1/n$ and K_f can be calculated from the slope and intercept respectively. All the adsorption parameters of Freundlich model are compiled and shown in **Table 3**. The value of n indicates the ease of adsorption on the adsorbent surface [24]; with the decrease of n, the adsorption becomes more difficult to take place. Comparison of the n values obtained for the two dye systems investigated using the adsorbent from sawdust indicates the better adsorption of Reactive Blue-250 dye than Methylene Blue onto the adsorbent surface according to the Freundlich adsorption isotherm.

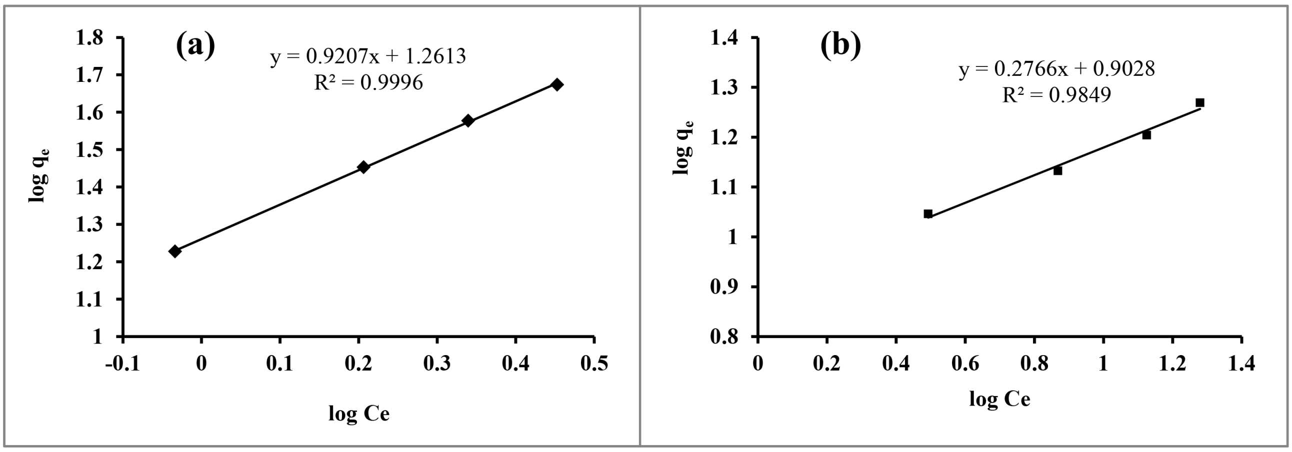


Figure 9. Freundlich adsorption isotherm for the removal of (a) Methylene Blue and (b) Reactive Blue-250 onto the adsorbent prepared from sawdust.

Both the adsorption isotherm models' parameters and significant linear relationships as indicated by the R^2 values which are found to be close to unity, signify the applicability of these two adsorption isotherms and the monolayer coverage on the adsorbent surface.

4. Conclusion

Cationic dye Methylene Blue and anionic dye Reactive Blue-250 from the textile effluents were effectively removed simultaneously at room temperature by using low cost and natural source derived adsorbents from sawdust and fish scale. The removal of dye is influenced by the initial concentration of dye, the amount of adsorbent used and the contact time to the adsorbents and the optimum removal condition has been determined as well. It has been found that adsorbent from sawdust has a more effective dye-removing capacity than that of the adsorbent from the fish scale for both cationic and anionic dyes. The Adsorption behavior has been described by a monolayer Langmuir type isotherm. It was also found that the adsorption of Reactive Blue-250 onto the sawdust adsorbent is more facilitated compared to Methylene Blue according to the Freundlich isotherm model. The present study concludes that the prepared activated carbon from natural and agricultural wastes as low-cost adsorbents is conveniently applicable for the removal of dyes particularly, for the removal of Methylene Blue and Reactive Blue-250 from solution and wastewater of industrial effluents.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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