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Characterization of *Ficus carica* fiber by Scanning Electron Microscope in Adsorption Isotherms Studies of Dye Removal from Aqueous Solution

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ABSTRACT : The biosorbent extracted from bast of *Ficus carica* plant has significant potential for removal of toxic Methylene blue dye from aqueous solution by adsorption. The unbalance forces on the surface of the biosorbent (natural fiber) are responsible to attract the adsorbate (dye-molecules) from the waste water for purification purposes. In the present scenario natural Ficus carica fiber has been characterized by scanning electron microscope (SEM) before and after adsorption of Methylene blue (MB) dye. The scanning electron microscope is the basic tools to characterize the adsorbents at finer scale which is beyond the preview of other techniques. Equilibrium behavior of natural fiber was investigated by performing batch adsorption experiments. Adsorption isotherm models, Langmuir and Freundlich were used to reproduce the equilibrium data. The Langmuir equation was found to have the correlation coefficient value in good agreement. Also, it was found that Ficus carica fiber (FCF) has an elevated adsorption capacity for MB dye (57.61 mg/g) and separation factor ($R_L < 1$) accounts for feasibility of adsorption system.

Keywords: *Ficus carica* fiber, Methylene blue, scanning electron microscope, Langmuir isotherm, Freundlich Isotherm.

I. INTRODUCTION

The release of dyes into industrial wastewater causes serious environmental problems, because their chemical structure gives them a persistent and recalcitrant nature. The discharge into natural waterways may have an inhibitory effect on photosynthesis affecting aquatic ecosystem [1]. Besides, dye molecules are broken down during the anaerobic processes occurring in the sediment, generating toxic amines that may cause a risk to the environment as well as threaten human health through the food chain. Therefore, it is very important to determine an effective way to remediate dyes contaminations [2]. Various treatment and disposal methods exist include chemical precipitation, electrode deposition, reverse osmosis, adsorption, etc [3]. The adsorption is one of the efficient methods to remove dyes from wastewater.

Adsorption, a surface phenomenon require characterization of adsorbent by scanning electron microscope to determine the topography and morphology of the adsorbate surface. Electron microscopy is a specialized branch of science, which uses the electron microscope to magnify the pore structure, pores arrangement, nature of surface etc. Recently, many natural biological macromolecules and microbial biomass such as chitosan [4,5], sawdust and rice husk [6] modified cellulose [7], peanut hull [8], cotton [9] etc, have been used as adsorbents. However, the adsorption capacity of these adsorbents is not very large; to improve adsorption performance, new absorbents are still under development. Ficus carica Bast fibre, an agricultural biopolymer, is more attractive for industrial use because of its renewability, biodegradability, and low unit cost. This can be satisfactorily utilized as a dye scavenger in adsorbing MB dye, because it has inherently adsorbate interaction capacity. Hence, the aim of this paper was to study the adsorption behavior of Methylene blue dye onto natural fibre and characterization of biosorbent by scanning electron microscope. The Langmuir and Freundlich equations were used to fit the equilibrium isotherm. These results will be useful for further applications in basic dyes removal from wastewater.

II. MATERIALS AND METHODS

Ficus carica bast was collected and washed with water to remove the cementing and gummy materials. The material was waterlogged for ten to fifteen days and trodden till the bast fiber alienated. The fiber so obtained was scoured in 1% sodium hydroxide solution at 60°C for 30 min and washed with deionized water thoroughly several times and air dried for seven days. The material was slashed into particle size of 1 mm to 1.5 mm and stored in desiccators to promote research when desired. All chemicals used in this research were of analytical reagent grade. Deionized water was used for all dilutions.

In adsorption experiment 0.1g of dried fiber was immersed in 100ml dye solution having concentrations between 10-110mg/l. Different initial concentration of dye solution was prepared by proper dilution from stock solution (1000 mg/l). These samples were placed into Gerhardt Thermoshake machine at 303K with 100 rpm. After shaking for 24 hrs, the equilibrium concentrations of the solution samples were measured using a UV-Visible spectrophotometer at 665 nm. The surface and cross section of FCF were observed with a scanning electron microscope (JSM-6100) after coating the material with gold. Batch adsorption studies were performed at different pH (2, 4, 6, 8, and10), adsorbent amount (0.1, 0.2, 0.3, 0.4 and 0.5 mg/ 100ml), and contact time (30, 60, 90, 120, 150, 180, 210, 240, 270 and 300 min) to obtain the equilibrium data. All experiments were performed in triplicate and the results average was reported. The amount of dye adsorbed onto the fiber, Q_e (mg/g), was calculated by a mass balance relationship [1].

$$Q_e = (C_0 - C_e)\ V/M$$

Where C_0 and C_e are the initial and equilibrium liquid-phase concentrations of dye solutions respectively (mg/l), V the volume of the solution (L), and M the weight of the dry fiber used (*g*).

RESULTS AND DISCUSSION

SEM Analysis : The scanning electron microscope provides the surface information. The surface structure of the biosorbent was observed with scanning electron microscope at different magnifications. The microscopic images clearly depicted that surface topography/morphology and internal architecture of both unloaded and MB loaded fiber were different. The biosorbent had irregular and porous structure as shown in Fig. 1. (*a*) and (*b*); however, remarkable dye adsorption by FCF was indicated in and Fig. 2. (*c*) and (*d*) It was observed porosities had increased surface area and adsorption capacity.

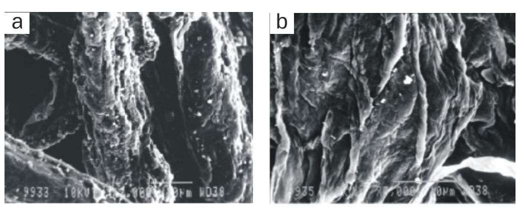

Fig. 1. SEM images (*a*) and (*b*) Unloaded *Ficus carica.* fiber

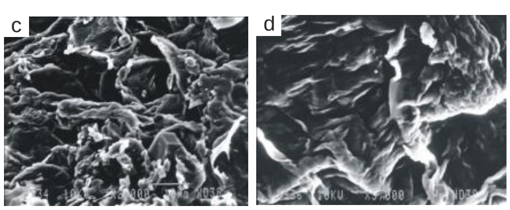

Fig. 2. SEM images (*c*) and (*d*) Methylene blue loaded *Ficus.*

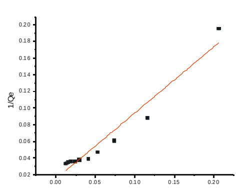

Fig. 3. Langmuir isotherm for the adsorption of MB dye.

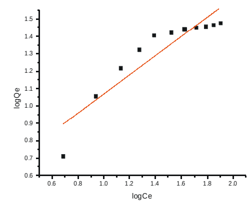

Fig. 4. Freundlich isotherm for the adsorption of MB dye onto *Ficus carica* fiber FCF.

Adsorption isotherm models : To determine mechanistic parameters associated with dye adsorption, the results obtained were analyzed by Langmuir and Freundlich models [10]. The Langmuir adsorption isotherm is applied to equilibrium adsorption assuming monolayer adsorption onto a surface with a finite number of identical sites.

The Langmuir equation may be written as

$$1/Q_e = (1/Q_m) + (1/K_L Q_m)\ (1/C_e) \qquad ...(2)$$

Where, K_L is Langmuir constant (l/mg) related to the affinity of binding sites and the free energy of adsorption; Q_e is Dye concentration at equilibrium onto fiber (mg/g); C_e is Dye Concentration at equilibrium in solution (mg/l); Q_m is Dye Concentration, when monolayer forms onto fibers and the saturation is attained (mg/g). A plot of $1/Q_e$ vs. $1/C_e$ gives a linear relationship, from which K_L and Q_m can be determined from the slope and intercept of the plot respectively (Table 1).

Table 1. Langmuir and Freundlich isotherm constants for the adsorption of MB dye onto FCF.

Langmuir Parameters				Freundlich Parameters			
Q_m (mg/g)	K_L	R^2	R_L	K_F(mg/g)	1/n	n	R^2
57.61	0.021	0.948	0.347	3.390	0.562	1.781	0.838

The essential features of the Langmuir Isotherm can be expressed in terms of a dimensionless constant separation factor (R_L), which is defined by the following relationship:

$$R_L = 1/\ (1 + K_L\ C_0) \qquad ...(3)$$

Where C_0 (mg/l) is the initial concentration of adsorbate.

The Freundlich equation, which is also often used for heterogeneous surface energy systems, may be written as

$$\ln \quad Q_e = \ln K_F + (1/n) \ln C_e \qquad ...(4)$$

The intercept K_F and slope (1/*n*) obtained from the plot of log Q_e versus log C_e is roughly a measure of the adsorption capacity and the adsorption intensity respectively (Table 1). The magnitude of the term (1/n) gives an indication of the favorability of the adsorbent/adsorbate systems [11]. It is generally stated that the values of n in the range of 1 to 10 represent high-quality adsorption.

CONCLUSION

In this study, batch adsorption experiments for the removal of Methylene blue from aqueous solution by using Ficus carica bast fibre have been carried out. For the maximum adsorption, the optimal operating parameters, pH of solution, adsorbent dosage, contact time and temperature were selected as 7.8, 0.1g, 170 min and 303K, respectively. Scanning Electron Microscopic pictures showed that a remarkable amount of Methylene blue dye was absorbed by Ficus carica bast fibre. Equilibrium data fitted well in the Langmuir isotherm equation, confirming the monolayer adsorption capacity (57.61mg/g) of Methylene blue onto Ficus carica bast fiber. The dimensionless separation factor R_L (0.347) showed that natural fibers can be used efficiently and economically for removal of Methylene blue from aqueous solutions for water purification purpose.

Thus the natural bast fiber can be used as an effective adsorbent. The dye adsorption on natural fiber was more dependent on the surface chemistry, the nature of components and the porous structure of the fiber.

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