



## Jute stick powder as a potential biomass for the removal of congo red and rhodamine B from their aqueous solution

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### ABSTRACT

Jute stick powder (JSP) has been found to be a promising material for adsorptive removal of congo red (C.I. 22120) and rhodamine B (C.I. 45170) from aqueous solutions. Physico-chemical parameters like dye concentration, solution pH, temperature and contact time have been varied to study the adsorption phenomenon. Favorable adsorption occurs at around pH 7.0 whereas temperature has no significant effect on adsorption of both the dyes. The maximum adsorption capacity has been calculated to be 35.7 and 87.7 mg/g of the biomass for congo red and rhodamine B, respectively. The adsorption process is in conformity with Freundlich and Langmuir isotherms for rhodamine B whereas congo red adsorption fits well to Langmuir isotherm only. In both the cases, adsorption occurs very fast initially and attains equilibrium within 60 min. Kinetic results suggest the intra-particle diffusion of dyes as rate limiting step.

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### 1. Introduction

Effluents of textile, paper and pulp, paint, printing and cosmetic industries must be treated to bring down the concentration of dyes present in it to permissible limit before discharging into water bodies as required under environmental regulation act [1–3]. Most of the dyes are toxic, carcinogenic and can cause allergic dermatitis, skin irritation, mutation, etc. [4–6]. Beside these they impart intense color to wastewater streams causing reduced photosynthesis of aquatic plants due to inhibition of the sunlight penetration [3,7]. Thus endanger aquatic life. It is difficult to treat dye containing wastewater by conventional methods as most of the dyes have complex aromatic structure of synthetic origin and thus hardly amenable to biodegradation [8–11]. Available methodologies in this regard such as flocculation combined with coagulation [12], reverse osmosis [13], membrane separation [14], oxidation or ozonation [15,16], etc., are either expensive or inadequate in removing dye from wastewater [17].

Recently removal of dye through adsorption on activated carbon has been found to be very effective [18] but high cost limits its application [19] in many countries including India. This promotes search for adsorbents mainly from biological origin such as peat [20–22],

fly ash [23], orange peel [17], coir pith [24], banana pith [25], biogas residual slurry [26], hardwood sawdust [27], maize stalk [28], rice husk [29], peanut hull [30], bagasse pith [31], jute processing wastes [32], *Azadirachta indica* leaf powder [2], etc., for this purpose. Still depending upon the availability and cost there is always a necessary to search for new effective adsorbent. The present investigation deals with the removal of congo red (Fig. 1a) and rhodamine B (Fig. 1b) as model anionic and basic dye, respectively, using jute stick powder (JSP), an agricultural waste. Physico-chemical parameters including probable binding mechanism involved in the adsorption process has also been reported.

### 2. Materials and methods

#### 2.1. Chemicals

Congo red (C.I. 22120) and rhodamine B (C.I. 45170) used in this study were purchased from BDH, England. All other chemicals and reagents were procured from Merck, Germany.

#### 2.2. Preparation of adsorbent

Jute stick was collected from the local market; cut into small pieces; washed with water to remove any adhering substances and dried at 80 °C. This was powdered by grinding in a mechanical blender and sieved. The 80–100-mesh size fractions were used for further study. The specific surface area of the powdered biomass was determined by BET surface area analyzer

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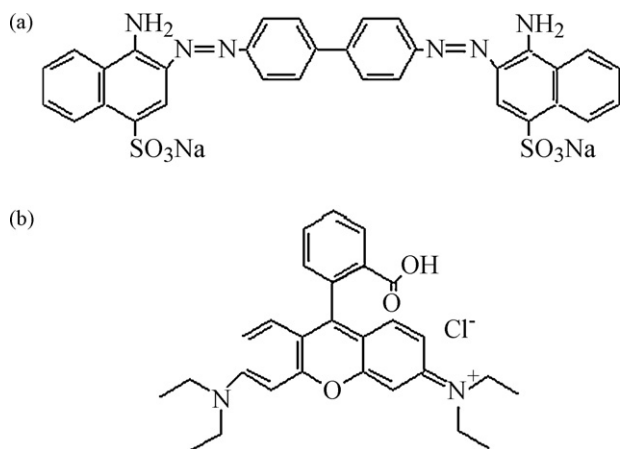


Fig. 1. Chemical structures of (a) congo red and (b) rhodamine B.

(Quantachrome Instruments, Autosorb-1-C) following  $N_2$  sorption method [33].

### 2.3. Preparation of dye solution and estimation

Stock solutions (1000 mg/l) of congo red and rhodamine B were prepared in deionized and double distilled water and diluted to get the desired concentration of the dyes. Calibration curves for the dyes were prepared by measuring the absorbance of different concentrations of the dyes at their optimum  $\lambda_{max}$ , 497 nm (congo red) and 554 nm (rhodamine B), respectively, using UV–vis spectrophotometer (Varian CARY 50). The dye concentrations in the experimental samples were evaluated from the calibration curves. Amount of dye uptake,  $q$  (mg/g), was calculated using the following equation:

$$q = \frac{(C_i - C_f)V}{1000W}$$

where  $C_i$  (mg/l) is the initial dye concentration,  $C_f$  (mg/l) is the dye concentration after adsorption,  $W$  (g) is the amount of biosorbent and  $V$  (ml) is the volume of the solution.

### 2.4. Equilibrium studies

Adsorption experiments were conducted with 50 ml dye solution (50 mg/l, pH ~6.0) and 0.5 g JSP taken in 250-ml Erlenmeyer flask. The mixture was agitated (120 rpm) at 30 °C for 24 h unless otherwise stated. At the end of incubation, JSP was separated by filtration through glass wool and the concentration of the dye in the solution was determined as described above.

### 2.5. Effect of pH and temperature

The influence of hydrogen ion concentration on the adsorption process was studied over a pH range of 2.0–10.0. Prior to this, the biomass was conditioned in phosphate buffer of desired pH for 2 h with agitation, separated by filtration and washed with deionized water for use in this experiment.

The experiment was repeated by varying the incubation temperature from 10 to 50 °C and at pH 7.0 and 6.0, being optimum, respectively, for congo red and rhodamine B.

### 2.6. Adsorption kinetics

Kinetics of adsorption of dye by JSP was studied at regular intervals of time up to 180 min. The experiment was conducted at

optimum pH values of the dyes. Each data point was obtained from individual flask and therefore no correction was necessary due to withdrawal of sampling volume.

### 2.7. Scanning electron microscopy

Pristine and dye adsorbed JSP were coated with platinum [34] for scanning electron microscopy and micrographs were recorded on FESEM (JEOL JSM-6700 F) instrument equipped with energy dispersive X-ray analysis.

### 2.8. FTIR analysis

Fourier transform infrared spectroscopy (FTIR) spectra of pristine JSP biomass were obtained by NICOLET Magma 750 FTIR spectrometer. Pressed pellets were prepared by grinding the powder specimens with IR grade KBr in an agate mortar.

## 3. Results and discussion

### 3.1. Characterization of the adsorbent

The specific surface area of JSP as determined by a BET surface area analyzer was 32.6 m<sup>2</sup>/g. The nature of the functional groups present on the surface binding-sites of the adsorbent is very important to understand and interpret the adsorption phenomenon. FTIR, a superb tool for identifying types of chemical bonds in a molecule, was recorded with pristine JSP biomass (Fig. 2). The region below 1500 cm<sup>-1</sup> is the 'fingerprint region' and the absorption can hardly be clearly assigned to any particular vibration system.

The region between 1800 and 3500 cm<sup>-1</sup> presents two major peaks centered around 3406 cm<sup>-1</sup> (the H-bonded –OH group) and 2900.7 cm<sup>-1</sup> (the C–H stretching of the –CH<sub>2</sub>– groups) corresponding the pristine JSP. According to the literature [35–38], the peak wavenumber of the carboxyl acid groups in the organic compound is approximately 1740 cm<sup>-1</sup>. This peak number is more or less identical to 1737.7 cm<sup>-1</sup> in this study. The two sharp bands in the region 1600.8 and 1506.3 cm<sup>-1</sup> are most probably due to stretching modes of benzene ring [38] of lignin present in JSP.

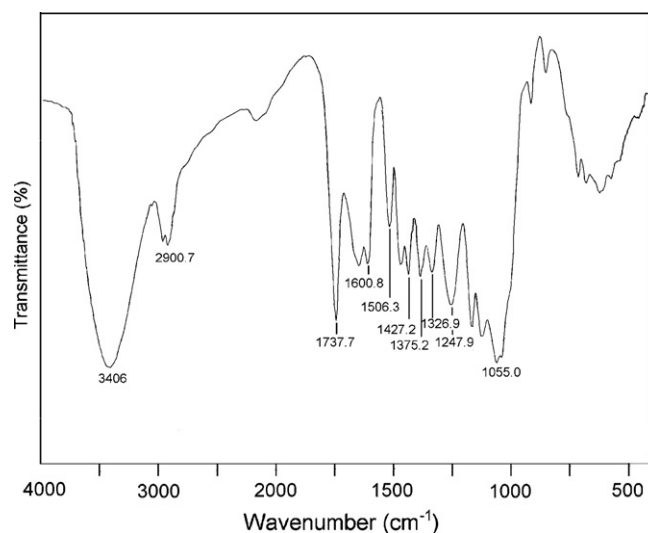


Fig. 2. FTIR spectra of jute stick powder.

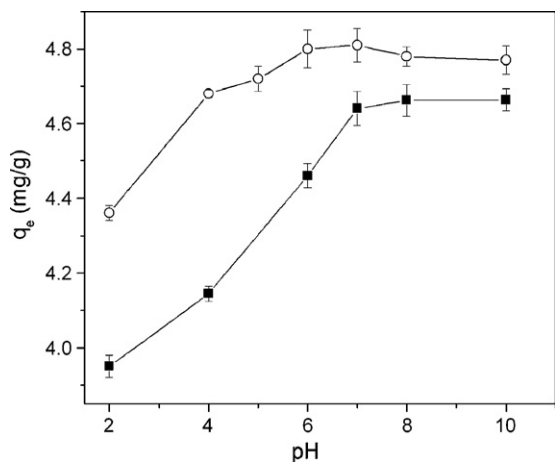


Fig. 3. Effect of pH on congo red (○) and rhodamine B (■) adsorption by JSP biomass.

### 3.2. Effect of pH and temperature

Adsorption of a dye biomass may be dependent [3,39] or independent [40] of solution pH as Annadurai et al. [39] obtained maximum adsorption of rhodamine B on orange peel at pH 7.0, whereas for banana pith [25] no significant effect of pH was noted by Namasivayam et al. [17]. Only limited influence of pH on the adsorption of congo red by neem leaf powder was reported by Bhattacharyya and Sharma [2]. In the present investigation a small increase in adsorption of the dyes by JSP was noted with increase in pH up to 7.0 (Fig. 3). Solution pH influences chemical nature of the surface of the adsorbent [41] and at lower pH, predominating adsorbed species is the molecular form of the adsorbate, while ionized form is preferentially adsorbed. It may be interpreted from present adsorption process that the surface of JSP had more preference for the dissociated species of the dye at neutral pH. JSP is a heterogeneous polymer of cellulose, hemicellulose and lignin [42] and thus contains various functional groups, such as carboxyl and hydroxyl, etc. The interaction of the dye molecules with these functional groups may follow extremely complicated patterns. There may be weak electrostatic interaction between the dye molecules and electron deficient sites on the surface of the JSP particles. The extent of adsorption of congo red and rhodamine B on JSP remained nearly constant in the pH range of 7.0–10.0 and therefore, the adsorption of the dye on JSP might be attributed to weak electrostatic interaction between the dye molecules and the solid surface.

As various textile dye effluents are discharged at relatively high temperature (50–60 °C), so temperature will be an important design parameter affecting the biosorption process. Only a little change in the adsorption of dye by biosorbents has been reported from different laboratories. In the present investigation only a small increase in adsorption of congo red and rhodamine B by JSP with increase in temperature from 10 to 50 °C is noted (data not shown).

### 3.3. Adsorption isotherm

Adsorption isotherms are prerequisites to understand the nature of the interaction between adsorbate and the adsorbent used for the removal of organic pollutants [1]. In the present study, the adsorption of both rhodamine B and congo red on JSP (Fig. 4) shows that the adsorption of the dyes increases with increase in dye concentration and tends to attain saturation at higher concentrations. The experimental data have also been analyzed by two well known adsorption

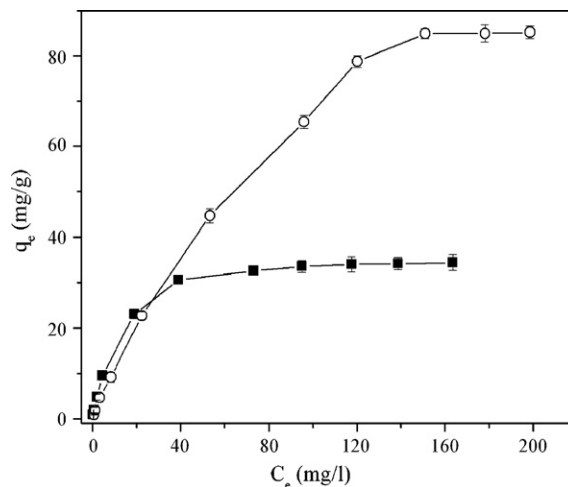


Fig. 4. Equilibrium adsorption of congo red (■) and rhodamine B (○) adsorption. Data represent an average of four independent experiment.  $\pm$ S.D. shown by error bar.

isotherm models, e.g., Langmuir (Eq. (1)) and Freundlich (Eq. (2)):

$$\frac{C_e}{q_e} = \frac{1}{K_L} + \frac{a_L C_e}{K_L} \quad (1)$$

where  $C_e$  and  $q_e$  are the equilibrium dye concentration in the solution (mg/l) and on JSP (mg/g), respectively,  $a_L$  and  $K_L$  are the Langmuir constants. Fig. 5 shows that adsorption of both the dyes on JSP fits well to the Langmuir isotherm model with a correlation coefficient of 0.999 (congo red) and 0.998 (rhodamine B), respectively, supporting monolayer coverage of the adsorbate on the surface of adsorbent. The theoretical monolayer saturation capacities of JSP have been calculated to be 35.7 mg/g (0.051 mmol/g) and 87.7 mg/g (0.183 mmol/g) for congo red and rhodamine B, respectively. This value is higher to that of other biosorbents such as GAC [43], coir pith [3], PAC [43], banana pill and orange pill [39]. However, adsorption capacity of neem leaf powder [2] and waste coir pith [44], respectively, towards congo red and rhodamine B is higher to a small extent.

Langmuir equation can also be used to obtain,  $R_L$ , the dimensionless equilibrium parameter or the separation factor from the expression:

$$R_L = \frac{1}{1 + a_L C_0}$$

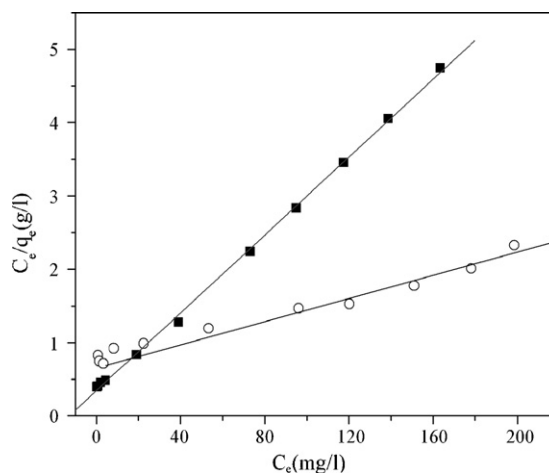


Fig. 5. Langmuir plots for the adsorption of congo red (○) and rhodamine B (■).

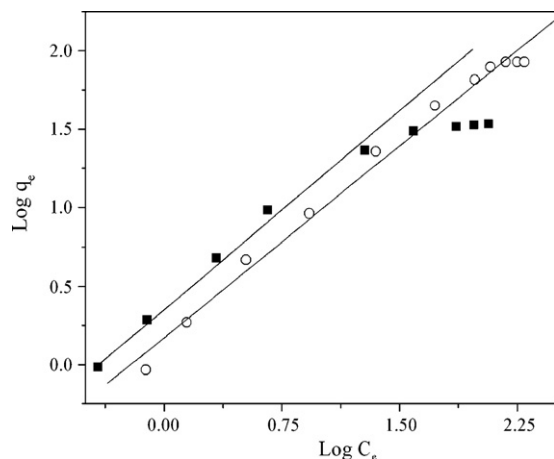


Fig. 6. Freundlich plots for the adsorption of congo red (■) and rhodamine B (○).

where  $C_0$  is the initial concentration of the adsorbate and  $a_L$  is the Langmuir coefficient as described above. The value of  $R_L$  has been determined to be 0.049 and 0.235 for congo red and rhodamine B, respectively, for an initial dye concentration of 250 mg/l indicating that the adsorption process is favourable.

The data were also analyzed by the linearized form of Freundlich isotherm model

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (2)$$

where  $C_e$  and  $q_e$  are the equilibrium concentration of dyes in the solution (mg/l) and on JSP (mg/g), respectively.  $K_F$  is the Freundlich constant and  $1/n$  is the heterogeneity factor. Fig. 6 shows that Freundlich model also fits for rhodamine B (correlation coefficient 0.997) but fails to describe the congo red (correlation coefficient 0.879) adsorption process.

### 3.4. Kinetic study

The rate as well as mechanism of adsorption process can be elucidated on the basis of kinetic study. Dye adsorption on solid surface may be explained by two distinct mechanisms; (i) an initial rapid binding of dye molecules on the adsorbent surface followed by (ii) relatively slow intra-particle diffusion. Progress of the adsorption of dyes by JSP with agitation time (Fig. 7) shows that the rate of

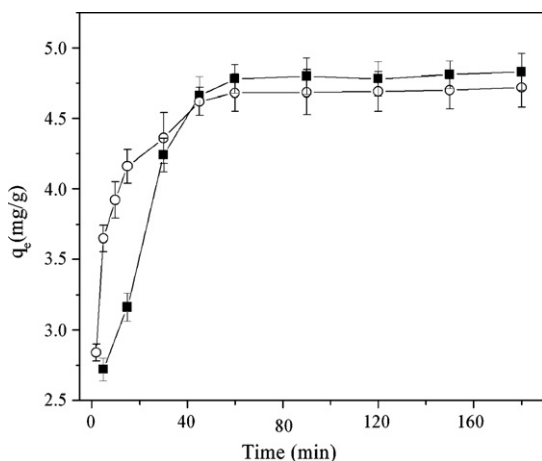


Fig. 7. Effect of contact time on congo red (■) and rhodamine B (○) adsorption by JSP biomass. Data represent an average of four independent experiment.  $\pm$ S.D. shown by error bar.

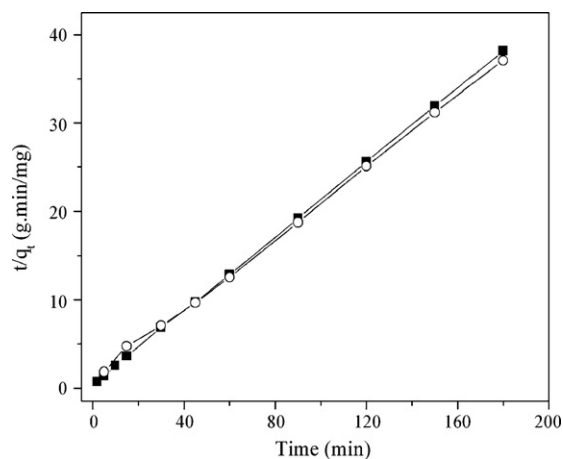


Fig. 8. Second-order plot of adsorption of congo red (○) and rhodamine B (■).

adsorption of the dye molecules was initially very fast and then gradually slows down to reach equilibrium within 1 h. To determine the rate controlling and mass transfer mechanism, experimental kinetic data were correlated to linear form of the pseudo first- (Eq. (3)) and second-order (Eq. (4)) rate models.

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

where  $q_e$  is the equilibrium concentration,  $t$  is the time,  $k_1$  and  $k_2$  are the first- and second-order rate constants, respectively;  $q_t$  is the uptake per gram of JSP biomass at time  $t$ . Experimental data show high degree of nonlinearity (figure not shown) and poor correlation coefficients (0.88 for congo red and 0.89 for rhodamine B) for pseudo first-order model. On the other hand, linearity (Fig. 8) with high correlation coefficients (0.999 for both the dyes) indicates that the present sorption process follows second-order rate model. Transportation of the dyes from the solution phase into the pores of the adsorbent may also be considered as the rate controlling stage in batch experiments under rapid stirring condition. The rate parameters for intra-particle diffusion [45] for both of the dyes were determined using the following equation (Eq. (5)):

$$q_t = K_d t^{0.5} \quad (5)$$

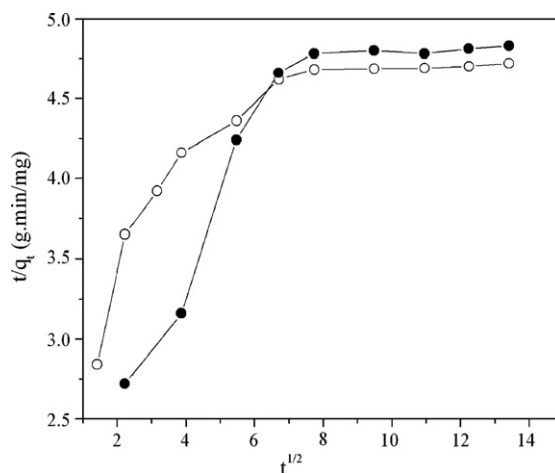


Fig. 9. Test of intra-particle diffusion model for adsorption of congo red (○) and rhodamine B (●).



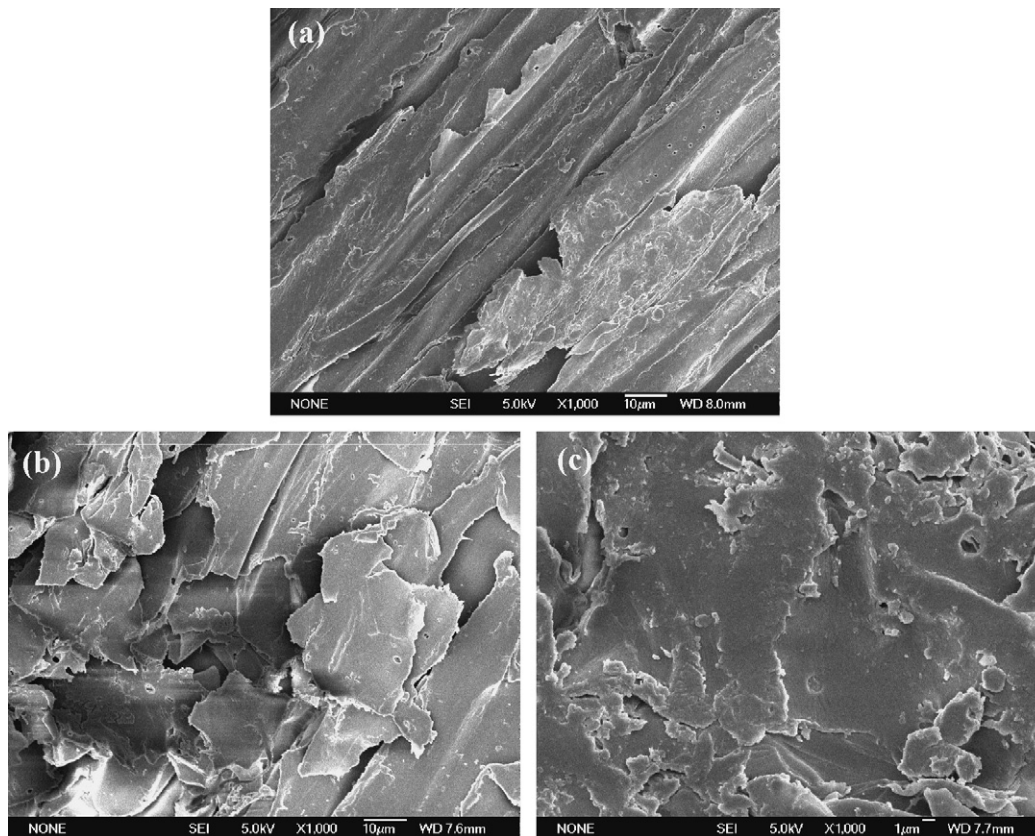


Fig. 10. Scanning electron micrographs of (a) JSP biomass, (b) congo red and (c) rhodamine B adsorbed JSP biomass.

where  $q_t$  (mg/g) and  $K_d$  represent the amount of dye adsorbed per unit mass of JSP at time 't' and the intra-particle diffusion rate constant, respectively.

Biphasic nature of the curves (Fig. 9) indicates that the process occurs in two steps. Initial steep portion may be attributed to the transport of dye molecules from bulk solution to the surface of the adsorbent and the linear portion to the binding of the dye molecules on the active sites of the adsorbent.

### 3.5. Scanning electron microscopy

The surface morphology of an adsorbent can be extensively characterized using scanning electron microscopy [46]. The SEM image of the pristine JSP (Fig. 10a) is found to be smooth and layered structure over a large area corresponding to dye–surface interaction. Significant changes in the surface morphology are noted, which became uneven and irregular after adsorption of congo red (Fig. 10b) and rhodamine B (Fig. 10c). Relatively thick layer of rhodamine B on JSP surface also supports the greater adsorption capacity (0.183 mmol/g) of the biomass than that of congo red adsorption (0.051 mmol/g).

## 4. Conclusions

JSP, an agricultural waste, is efficient in removing both congo red and rhodamine B from their aqueous solutions at an optimum pH ~7.0. Equilibrium isotherm indicate that the adsorption process follows both Langmuir and Freundlich isotherm models reasonably well for rhodamine B only the former one for congo red. The maximum adsorption capacities of the biomass are found to be 35.7 and 87.7 mg/g of the biomass for congo red and rhodamine B, respectively. The adsorption process is very fast initially and the

intra-particle diffusion to reach equilibrium within 1 h following pseudo-second-order rate kinetics.

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