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RESEARCH ARTICLE

ADSORPTION STUDIES OF RHODAMINE B FROM AQUEOUS SOLUTION USING LOW COST MATERIAL: EQUILIBRIUM AND KINETICS STUDIES

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ABSTRACT

Rhodamine B (RhB), one of the toxic dyes which are extensively used for dyestuffs, textile, paper and plastics industries. RhB does not easily biodegrades in aqueous medium and show harmful effect on aquatic as well as human life. In the present work adsorption studies of RhB onto cardboard (CB) was examined in aqueous solution at 28°C with the effect of pH, dye concentrations and contact time. Highest 68% uptake efficiency recorded was for 10 mg/L solution concentration onto 25mg of cardboard. Langmuir and Freundlich adsorption isotherm models were applied to describe the equilibrium isotherms. The kinetic data were fitted to pseudo-second-order kinetic model and intraparticle diffusion kinetic was also investigated.

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INTRODUCTION

The development of many branches of industries and use of modern technologies, that are accompanied by tougher for environmental protection and search for possibly cheap and most effective adsorption materials. The industrial wastewater usually contains a variety of organic compound and toxic substances which are harmful to fish and other aquatic life. Color is the first contaminant to be recognized in wastewater (Banat *et al.*, 1996), Synthetic dyes are extensively used in paper, textile, food, and pharmaceutical industries. About 40,000–50,000 tons of dyes are continuously entering the water systems due to improper processing and dying methods from industries (Filipkowska *et al.*, 2002).

A wide range of methods have been developed for the removal of synthetic dyes from waters and wastewaters to decrease their impact in environment. Among these physico-chemical methods like adsorption (Gupta *et al.*, 2004; Mittal *et al.*, 2005), electrochemical coagulation (Yang and Mc, 2005), activated carbon adsorption (Tan *et al.*, 2008) and oxidation or ozonation (Malik and Saha, 2003), membrane separation (Sachdeva and Kumar, 2009), sonochemical degradation (Abbasi and Razzaghi, 2008). are popular now days. Many industrial wastes and agricultural by-products have been used as adsorbents for the removal of organic compounds, dyes, color, surfactants, metals, etc. from wastewater (Gupta *et al.*, 1990; Nadeem, 2013; Bousher, 1997). Various naturally

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available materials such as bagasse pith (Al-Dury et al., 1990), cotton waste, hair, bark and rice husk (Bishnoi, 2004), tea waste ash (Balasubramnaian and Muralisankar, 1987), algae (Nadeem, 2010), rice bran (Gupta et al., 2010), maize leaves (Nadeem, 2013) maize leaves carbon (Nadeem, 2013), zeolite (Nadeem 2013) and other agricultural residues (Nigam, 2000) have been investigated widely as adsorbents. Rhodamine B (RhB), is one of the water soluble xanthenes class dyes (Ilayaraja et al., 2013), a basic red cationic dye. It is often used in textile, cotton, wool, silk and food industries. It is potentially harmful to humans as it can cause eye burn, irritation to skin (Jain et al., 2013), gastrointestinal and the respiratory tracts. For these reasons, the treatment of dve effluents is essential prior to their discharge into the receiving water bodies (Arivoli et al., 2009). Molecular structure of RhB dye is shown in Fig. 1. In the present work a waste material, cardboard was applied as an adsorbent for the removal of crystal Rhodamine B (RhB), from aqueous solutions. The main objective of the research is to investigate the adsorption efficiency of cardboard (CB) for Rhodamine B. Cardboard is a low cost, easily available and biodegradable.

MATERIALS AND METHODS

Adsorbent

Cardboard (CB) can be obtained from anywhere as a waste. Firstly it was washed using tap water and finally with double distilled water to remove the suspended impurities, dust and soil and then dried in oven at 80°C for 12 hours to dry and grinded by mixer and stored in air tightened bottle. All the

chemicals in the study were of analytical-grade. The main constituent of cardboard is cellulose (Fig. 2), is a very important biopolymer. Cellulose is inexhaustible and renewable raw material (Ciolacu, 2011).

Fig. 1. Molecular structure of Rhodamine B

Fig. 2. Molecular structure of Cellulose: a major constituent of cardboard

Dye Solution Preparation

The dye Rhodamine B [C.I C745170, formula weight = 479.02, λ max-543nm supplied by Thomas Baker Chemical Limited Mumbai, India was used as such without further purification. An accurately weighed quantity of dye was dissolved in double distilled water to prepare 250 mg/L stock solutions. RhB is an amphoteric dye, although usually are basic as it has an overall positive charge.

Instruments

UV–Vis absorption measurements were recorded by using a spectrophotometer (Systronics 2201), X-ray diffraction was recorded using Cu K α radiation (n = 1Å) on a Philips X' Pert-PRO PMRD system, FT-IR spectra were recorded using a Perkin-Elmer FT-IR.

Batch Adsorption Experiments

Batch mode adsorption studies were carried out to study the effect of initial pH, agitation time, and initial dye concentration (10 to 100 mg/L) on the adsorption process for 25 mg adsorbent dose and 25 ml of dye solution. Proper concentrations of the adsorbate were prepared from the stock solution through proper dilution. The required concentration for the adsorption experiments were prepared by serial dilution. The pH of the dye solution was adjusted by using 0.01M, 0.1M, and 1M NaOH and 0.01M, 0.1M, and 1M HCl solutions with pH Meter (Systronics 335, pH meter with a combined pH electrode). The batch adsorption experiments were performed

on an orbital shaker. The samples were withdrawn from the shaker at predetermined time intervals and solutions were separated from the adsorbent by centrifugation. To determine the residual dye concentration, the absorbance of the supernatant solution was measured with double beam spectrophotometer. Experiments were carried out twice and the concentrations were calculated by the average values. All the adsorption experiments were carried out at room temperature (28 °C). The adsorption efficiency R(%) and amount of adsorption in batch experiments, q_e (mg/g) were calculated as follows show in Equation (1) and (2) respectively:

$$R(\%) = \frac{C_{i} - C_{f}}{C_{i}} \times 100$$
 (1)

$$q_{\rm e} = \frac{(C_{\rm i} - C_{\rm e})V}{m} \tag{2}$$

Where, C_i is the initial concentration (mg/L), C_e is the equilibrium concentration (mg/L) V is the volume of solution (mL), m is the mass of adsorbent (g), q_e is the amount adsorbed (mg/g)

pH Stability Study Aqueous Rhodamine B Dye Solution

The aqueous Rhodamine B dye solution was found to be stable over pH range 4–10 (Fig. 3) λ_{max} remains constant at 556nm (The emission spectra depict that there is a broad band with a maximum of intensity that corresponds to S1 \rightarrow S0 transition), but absorbance different. At PH 1, pH 2 and pH 3 the λ max is shifted from lower to higher at 560nm. This absorption behavior of RhB dye with pH was performed by taking 3 mg/L of dye concentration and the pH was maintained from 1 to 10.

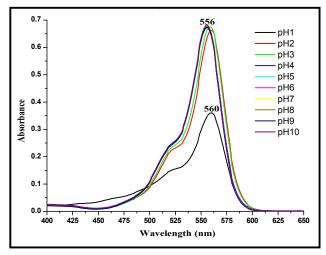


Fig. 3. Absorption spectra of Rhodamine B as afunction of pH

RESULTS AND DISCUSSION

Adsorption Equilibrium Studies of RhB dye on CB

Employing the batch method, the adsorption behavior of the dye on cardboard was investigated as a function of; pH of the aqueous dye solution, contact time for batch adsorption and concentration of the dye solution. In the adsorption process pH of the dye solution plays an important role, particularly on

adsorption efficiency. The adsorption amount of dye on CB surface was found less in acidic media as compare to basic media but remained almost constant in basic conditions. The adsorption capacities of RhB onto cardboard is higher at pH 3 (Fig. 4) and due to this reason all experiments were done at pH 3. As a function of contact time, the uptake of RhB dye by cardboard was rapid at room temperature, and reaches 46% of dye adsorption within 15 minutes as shown in Fig. 5. It was observed that on increase of the initial dye concentration there is a decreased uptake of RhB. Maximum adsorption efficiency of RhB was observed at lower concentration of dye and on increasing dye concentration, adsorption efficiency decreases gradually (Fig. 6). The decrease in the dye uptake with increasing the concentration of dye might be due to the lack of available adsorption sites (Shukla *et al.*, 2002).

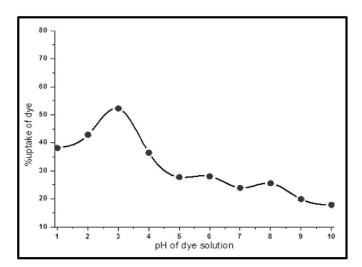


Fig. 4. The effect of pH on adsorption efficiency of dye on CB

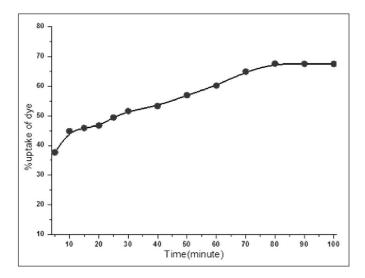


Fig. 5. The effect of contact time on adsorption efficiency of dye

Adsorption Isotherms

Since, the adsorption isotherm is important to describe how adsorbate will interact with adsorbents and so is critical for design purpose, therefore, data using an equation is essential adsorption operation (Hashem *et al.*, 2007). Modelling of

equilibrium data is fundamental for the industrial application of adsorption since it gives information for comparison among different adsorbent under different operational conditions, designing and optimizing operation procedure (Benguella and Benaissa, 2002). The result of batch equilibrium was used to characterize the equilibrium between the amount of adsorbate that accumulated on the adsorbate and the concentration of dissolve adsorbate. The experimental isotherm data set obtained was fitted using adsorption models including the Langmuir and Freundlich isotherm (Langmuir, 1918; Freundlich, 1907).

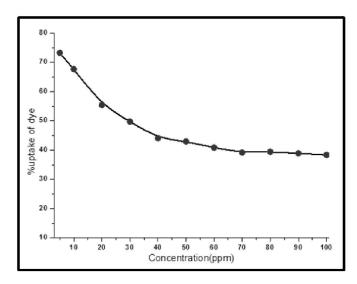


Fig. 6. The effect of concentration on adsorption efficiency of dye

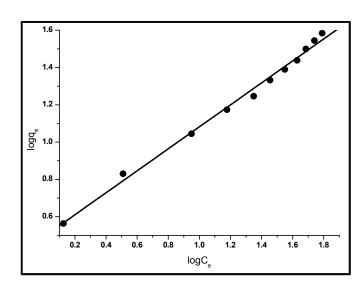


Fig. 7. Linearized Freundlich isotherm plot for RhB on CB

Freundlich Isotherm

The isotherm constants of Freundlich were calculated using normal linearization method as given in Equation (3).

$$\log q_{\rm e} = \log K_{\rm f} + \frac{1}{n} \log C_{\rm e} \tag{3}$$

Where, K_f = Freundlich isotherm constant (mg/g), n = adsorption intensity, C_e = the equilibrium concentration of adsorbate (mg/L), q_e = the amount of dye adsorbed per g of the adsorbent at equilibrium (mg/g).

The plot of $\log q_e$ versus $\log C_e$ was linear shown in Fig.7, with a slope equal to 1/n and an intercept equal to $\log K_f$. The constant K_f is an approximate indicator of adsorption capacity, while 1/n is a function of the strength of adsorption in the adsorption process (Voudrias *et al.*, 2002).

Langmuir Isotherm

The Langmuir isotherm equation may be expressed in a linearized form as shown in Equation (4):

$$\frac{C_{\rm e}}{q_{\rm e}} = \frac{1}{q_{\rm max}K_{\rm L}} + \frac{C_{\rm e}}{q_{\rm max}} \tag{4}$$

Where q_{max} is the monolayer capacity of the adsorbent (mg/g) and K_L is the Langmuir adsorption constant (dm³/mg). The plot of C_e/q_e versus C_e was linear shown in Fig. 8, with a slope equal to $1/q_{max}$ and an intercept equal to $1/(q_{max}K_L)$. The Freundlich and Langmuir parameters are given in Table 1.

Table 1. Freundlich and Langmuir isotherm constants for RhB adsorption on CB

| Langmuir Parameters | | Freundlich Parameters | |
|---|--------|-----------------------|--------|
| Q _{max} , (mg g ⁻¹⁾ | 50.251 | $K_f(mg g^{-1})$ | 3.1103 |
| $K_L (dm^3mg^{-1})$ | 0.3336 | 1/n | 0.5891 |
| R^{2} | 0.858 | \mathbb{R}^2 | 0.993 |

Table 2. Pseudo first order, Pseudo second order and model and Intraparticle diffusion kinetic parameter for dye adsorption

| Pseudo-first-order model | | | |
|---|--------|--|--|
| K ₁ | 0.031 | | |
| • | 3.734 | | |
| $rac{q_e}{R^2}$ | 0.889 | | |
| Pseudo-second-order model | | | |
| K_2 | 0.013 | | |
| qe | 7.342 | | |
| $\begin{array}{c} q_e \\ R^2 \end{array}$ | 0.989 | | |
| Intraparticle diffusion model | | | |
| K_d | 0.3942 | | |
| C | 3.0005 | | |
| \mathbb{R}^2 | 0.9796 | | |

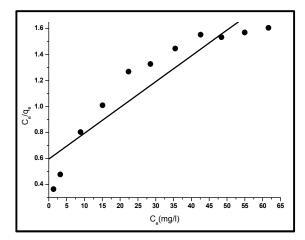


Fig. 8. Linearized Langmuir isotherm plot for RhB on CB

The essential characteristics of the Langmuir equation can be expressed in terms of a dimensionless constant which is called equilibrium parameter defined as Equation (5):

$$R_{\rm L} = \frac{1}{1 + K_{\rm L} C_{\rm i}} \tag{5}$$

Where K_L is the Langmuir constant, which is used to determine the enthalpy of adsorption, and C_i is the highest initial dye concentration employed. The value of R_L indicates whether the type of isotherm observed is unfavorable (R_L >1), linear (R_L =1) or favorable (R_L <1) (Koswojo *et al.*, 2010). The R_L values plot are shown in Fig. 9. R_L values were in the range 0< R_L <1, indicating that the adsorption process was favorable.

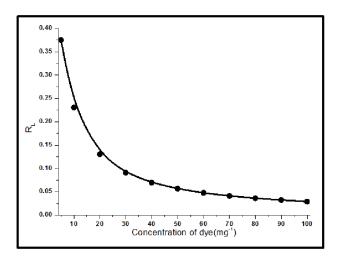


Fig.9. R_L vs initial concentration of dye

Kinetic Studies

Kinetic models have been proposed to determine the mechanism of the adsorption process which provides useful data to improve the efficiency of the adsorption and feasibility of process scale-up (Eftekhari *et al.*, 2010). The rate constants were calculated by using pseudo-first-order and pseudo-second-order kinetic models and the rate controlling step was determined by intra-particle diffusion model. The pseudo-first-order model was presented by Lagergren (1898). The Lagergren's first-order reaction model is expressed in linear form as Equation (6):

$$\log(q_{e} - q_{t}) = \log q_{e} - \frac{K_{1}}{2.303}t \tag{6}$$

The adsorption data was also analysed in terms of pseudo-second-order mechanism, described by Y.S.Ho and McKay (1999), the linear form of the Equation (7):

$$\frac{t}{q_{t}} = \frac{1}{K_{2}q_{e}^{2}} + \frac{1}{q_{e}}t \tag{7}$$

Where, K_2 is the rate constant of pseudo-second-order adsorption (g/mg min), $K_2q_e^2$ is the initial rate of adsorption (mg/g /min). The plot of log (q_e-q_t) versus t would be linear

with a slope of $-K_1/2.303$ and an intercept of log q_e shown in Fig.10. The plot of t/q_t against t of equation (7) and shown in Fig.11.

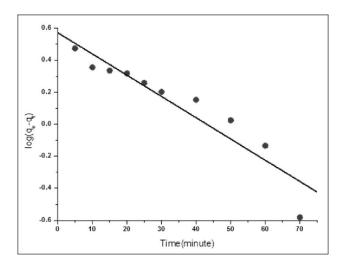


Fig. 10. Pseudo-first-order kinetic plot for dye adsorption on CB

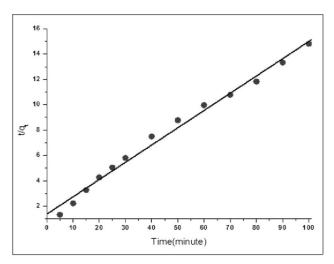


Fig. 11. Pseudo-second-order kinetic plot for dye adsorption on CR

In adsorption systems where there is the possibility of intraparticle diffusion being the rate-limiting step, the intraparticle diffusion approach described by Weber and Morris (1963), is used. The linear form of the equation (8) is as follows:

$$q_{\rm t} = K_{\rm d} t^{1/2} + C \tag{8}$$

The plot of q_t against $t^{1/2}$ of equation (8) should give a linear relationship with a slope of K_d and an intercept of C is shown in Fig. 12. The data demonstrate good compliance with pseudosecond-order rate law rather than the pseudo-first-order rate law. This shows that the pseudo-second-order kinetic model shows better explanation of the kinetic adsorption data obtained in the present study. This was probably true in the present case CB negatively in nature which attracts the positively charged dye charged. This would allow electrostatic interaction between the positively RhB and the CB surface.

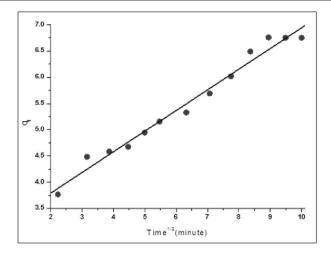


Fig. 12. Intraparticle Diffusion kinetic plot for dye adsorption on CR

XRD Analysis

The physical status of cardboard and dve adsorbed cardboard has been compared with the help of XRD. Before recording XRD, cardboard fibers were washed with double distilled water and dried in the oven at 100 °C, for 24 h. XRD studies had demonstrated that the cellulose fibers are constituted by crystalline and amorphous regions (Inpanya et al., 2012). XRD pattern of cardboard shows a hump at $2\theta = 11.63$ and a peak of low intensity at 2θ =22.6, indicating that cardboard has amorphorous in nature. After the adsorption of crystal violet dye on the cardboard the hump of shift towards higher 20 =14.6 value with lower intensity hump (Fig. 13). The peak intensity as well as the number of XRD peaks increases. Suggesting that crystal violet dye get adsorbed on the active sites of cardboard by ion-ion interaction, ion- dipole interaction, dipole- dipole interaction or through hydrogen bonding. The increase in the intensity of the cellulose after dye adsorption indicates that the plane at that particular 20 values increases. That may due to the dye that gets adsorbed on the CB.

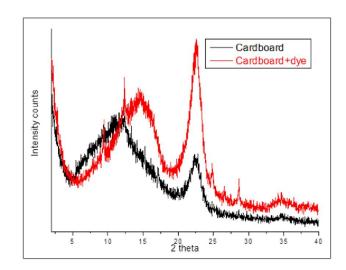


Fig. 13. XRD patterns of cardboard and dye adsorbed cardboard

FT-IR Analysis

FT-IR spectra of cardboard, RhB and RhB adsorbed cardboard was recorded in the wave number range of 4000-400 cm⁻¹ shown in Fig. 14. The spectrum of cardboard's adsorption peak at 1647 cm⁻¹ assigned H₂O and 1059 cm⁻¹ assigned to C-O (Garside, and Wyeth, 2003) and strong broad band at 3300-3500 cm⁻¹ for OH stretching, C-H stretching in methyl and methylene groups at 2900-3000 cm-1, 1730 cm-1 is assigned carbonyls and in ester groups (Jorgea et al., 2013). Appearance of the band near 1600 cm⁻¹ is a relative pure ring stretching mode strongly associated with the aromatic C-O-CH₃ stretching mode. The FT-IR of pure RhB show a characteristic peaks at 3422 cm⁻¹ assigned to O-H, 1589 cm⁻¹ for C=C and 1475 cm⁻¹ assigned to C-N, 1453cm⁻¹ and 2900 cm⁻¹ assigned ascribed to the C-H stretching modes that might be attributed to aromatic rings (Owen and Thomas, 1989). Two spectrum of cardboard at 1705 cm⁻¹ and 1376cm⁻¹ are disappear due to interaction of RhB dye.

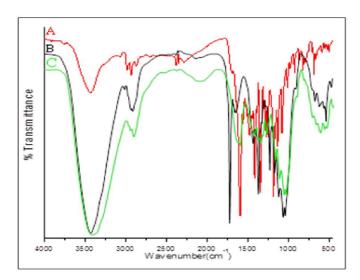


Fig.14. FT-IR spectra of : (A) RhB dye, (B) Cardboard and (C)
Cardboard + RhB

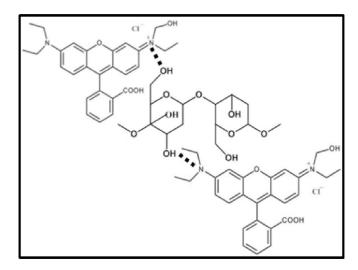


Fig.15. Absorptive interaction mechanism of RhB dye with cellulose which in the main constituent of cardboard

Proposed Mechanism for Adsorption of RhB on CB

Dyes are organic compounds that can be used to impart bright, permanent colors to fabrics. The affinity of a dye for a fabric depends on the chemical structure of the dye and fabric molecules and on the interactions between them. Chemical bonding thus plays an important role in how and why dyes work. RhB dye have positive charge on nitrogen atom which attract the electron pair containing oxygen atom through electrostatic interaction and second process hydrogen bonding between hydrogen atom of hydroxyl of adsorbent with nitrogen atom, which have unshared pair of electrons of RB dye (see Fig. 15). The formation of dye fiber hydrogen bonds is controlled by the presence of hydrogen bonding functional groups in the fiber and the dye and also by the macromolecular fiber structure (Simona *et al.* 2000).

Conclusions

The present study indicates that cardboard is a good adsorbent and it can be used for the removal of RhB dye from wastewater. Cellulose is the major constituent of cardboard which contains electron pair containing oxygen atom which attract the positive charge containing nitrogen atom of RhB dye by electrostatic interaction and in second process hydrogen bonding between hydrogen atom of hydroxyl of the cardboard with nitrogen atom, which have unshared pair of electrons of RhB dye. Maximum 68% of dye uptake on 25 mg of cardboard for and 10 mg/L of 25 mL dye solution was observed. The adsorption data for RhB dye investigated in this work fitted well to Langmuir adsorption isotherm equation and pseudo-second-order kinetic model.

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