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Dynamic and Equilibrium Studies on Adsorption of Direct Dye (Direct Yellow 28) by Low Cost Activated Carbon Derived from *Jatropha curcas* Stem Waste

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Abstract

Activated carbon materials were prepared from abundantly available Jatropha curcas stem waste. The carbon samples were prepared by subjecting the carbon material to various chemical processes. The adsorption capacity of selected derived carbon sample using Jatropha curcas stem waste prepared by dolomite process (CaCO₃) was much greater than the capacities of the other carbons prepared by various processes. The adsorption of Direct Dye (Direct Yellow 28) on this dolomite treated activated carbon was investigated to assess the possible use of this adsorbent for the processing of textile dyeing effluent wastewater. The effect of major variables governing the efficiency of process such as initial dye concentration, agitation time and temperature on the adsorption capacity has been studied. The percentage removal of dye is observed to decrease with the increase in initial dye concentration. With increase in temperature, the adsorption of dye also decreases, indicating exothermic nature of the reaction. Adsorption isothermal data could be interpreted by the Langmuir and Freundlich equations. Kinetic data have been studied using elovich, pseudo-first order and pseudo-second order equations for understanding the reaction mechanism. The results have established good potentiality for the activated carbons material to be used for the adsorption of Direct Yellow 28 from wastewater.

Keywords: Activated Carbon; Adsorption; Direct Yellow 28; Jatropha curcas.

1. INTRODUCTION

The textile dying units release large quantities of dyes in their effluents (Nabi Bindhendi Gh *et al.* 2007). Many industries such as paper, rubber, plastics, paint, printing and leather discharge colored effluent indiscriminately, which cause pollution in receiving water bodies. Textile effluents are known toxicants, which inflict acute disorder in aquatic organisms' uptake of textile effluents through food chain in aquatic organisms may cause various p^H ysiological disorders like hypertension, sporadic fever, renal damage, cramps etc., (Karthikeyan *et al.* 2001). Hence the treatment of

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wastewater containing dye is a challenging problem (Karthikeyan et al. 2006). Activated carbon has been extensively used for the purpose of water purification. In particular, it has been commonly used for the removal of organic dyes from textile waste water. Discharge of organic pollutants like dyeing industry wastewater into water bodies contaminates the environment. The adsorptive properties of activated carbon for removal of pollutants are well documented (Macias et al. 1993). Adsorption of hazardous soluble chemicals from wastewater in to surface of a solid adsorbent has provided a new dimension to wastewater technology (Benfield et al. 1982). One of the major

challenges associated with adsorption by activated carbon is its cost effectiveness. This has led many workers to search for cheaper substitutes. Crab shells (Ann et al. 2001), peanut hull pellets (Brown et al. 2000), corn starch (Kweon et al. 2001), soyabean hull and sugar beat fibre (Jambulingam et al. 2005), rice husk (Low and Lee, 1997), fly ash (Nagarnaik et al. 1976), Balsamodendron caudatum wood waste (Sivakumar et al. 2012), Ipomoea carnea stem (Karthikeyan et al. 2007; 2012), turmeric waste (Karthikeyan et al. 2008), coconut shells and husk (Banerjee et al. 1976), pomegranate peel (Jambulingam et al. 2005), maize cob (Bousher et al. 1997) and sawdust (Marshall and Johns, 1996) are some new adsorbents, which have been tried with varying success. Moreover, the affinity of carbon surface towards solute molecules must be enhanced in order to increase the extent of solute adsorption. Comparison of size information available for commercial activated carbon pores and solute molecules in natural waters suggest that some fractions of solute will be able to access finer carbon pores (Namasiyayam and Yamuna, 1999). In the present work, we have prepared Nanoporous activated carbon from Jatropha curcas stem waste which is used as an adsorbent for Direct Yellow 28 removal and the adsorption capacity of chosen adsorbent was regulated by many influencing factors such as temperature, pH variations and initial dye concentrations. The kinetic and equilibrium adsorption data obtained were correlated to characterize the prepared carbon sample for the adsorption of Direct Yellow 28.

2. MATERIALS & METHODS

2.1 Adsorbent

The study of *Jatropha curcas* based carbon material is used as adsorbent which is expected to be economical, environmentally safe and it has practical importance. The raw stems of *Jatropha curcas* were collected from in and around Namakkal district, Tamil Nadu, India. They were cut into small pieces and dried in sunlight until the moisture was evaporated. The dried materials were used for the preparation of activated carbons using physical and chemical activation methods. *Jatropha curcas* stem waste material was treated with dolomite for a period of 24 hrs. Then the material was placed in the muffle furnace carbonized at 400 °C. The dried materials were powdered and activated in a muffle furnace kept at 800 °C for a period

of 60 minutes. After activation, the carbon obtained was washed sufficiently with plenty of water, dried and sieved then to desired particle size. The N_2 adsorption-desorption isotherms of activated carbons were measured at 77 K using a gas sorption analyzer (NOVA 1000, Quanta Chrome corporation) in order to determine the surface areas and the total pore volumes. The surface areas were calculated using the BET equation.

2.1.1 Characterization of adsorbent

Physico-chemical characterizations of the adsorbent were presented in table 1.

Table 1. Characteristics of the dolomite treated activated carbon prepared from *Jatropha curcas* stem waste (JSAC)

Parameter	JSAC
Bulk density (g/mL)	0.857
Ash content (%)	10.26
рН	6.10
Moisture content (%)	17.10
Surface area (m ² /g)	845.23
Iodine number(m g/g)	810.33
^{pH} pzc	4.0
Porosity (%)	0.624

2.2 Adsorbate

The textile dye, Direct Yellow 28, was purchased from Sigma-Aldrich (Germany) and all the chemicals used were obtained as research grade chemicals and were used without purification. A stock solution of Direct Yellow 28 was prepared by dissolving 1 g of dye in 50 mL of double distilled water and used for further studies by diluting as concentrations required.

Table 2. Properties of Direct Yellow 28

Parameter	Values
Name	Direct Yellow 28
C. I Number	19555
Chemical Class	Mono Azo
Molecular Formula	$C_{28}H_{18}N_4Na_2O_6S_4$
Formula Weight	636.00
$\lambda_{ ext{max}}$	390 nm

2.3 Batch adsorption studies

To study the effect of important parameters like adsorbent dose, p^H , contact time, initial

concentration on the removal of Direct Yellow 28, batch experiments were conducted at 32 °C. For the determination of optimum adsorbent dose. 50 mL dye solution was treated with adsorbent dose range 20 to 60 mg/L, till equilibrium was attained. The effect of pH on dye removal was studied over a pH range 3-9 pH was adjusted by the addition of dilute aqueous solution of HCl or NaOH (0.10 M). For each experiment run, 50 mL of Direct Yellow 28 solution of known concentration, pH and a known amount of the adsorbent dose were taken in a 250 mL conical flash. This mixture was agitated in a temperature controlled orbital shaker at a constant speed of 150 rpm at 32 °C. Samples withdrawn at appropriate time interval were filtered then analyzed spectrophotometrically. In order to investigate the kinetics of adsorption of dye onto adsorbent, various kinetic models, pseudo-first order, pseudo-second order and elovich diffusion were used. Langmuir, Freundlich have been used to describe the equilibrium nature of adsorption of dye in the present study. The concentrations of dye in solutions were determined before and after adsorption using Elico UVvisible spectrophotometer. The amount of dye adsorbed and adsorption efficiency were calculated.

2.4 Determination of Direct Yellow 28

Direct Yellow 28 was estimated spectrophotometrically. The amount of Direct Yellow 28 adsorbed in mg/L at time, t was computed by using the following equation.

$$qt = \frac{CO - Ct}{ms} \times V \tag{1}$$

where, C_0 and C_t are the Direct Yellow 28 concentration in mg/L initially and at given time t, respectively, V is the volume of the Direct Yellow 28 solution in ml and m_s is the weight of the activated carbon.

The percentage of removed Direct Yellow 28 in solution was calculated using equation

% of Removal =
$$\frac{\text{Co-Ct}}{\text{Co}} \times 100$$
 (2)

The initial concentration of Direct Yellow 28, p^H and temperature were investigated by varying any one parameters and keeping the other parameters constant.

2.5 Adsorption dynamics

The study of adsorption dynamics describes the solute uptake rate and evidently this rate controls the residence time of adsorbate uptake at the solid-solution interface. The kinetics of Direct Yellow 28 adsorption on the activated carbon was analysed using pseudo first order (Lagergren, 1898), pseudo second order (Ho and Mckay, 2000) kinetic models and elovich equation (Chien and Clayton, 1980). The conformity between experimental data and the model predicted values was expressed by the correlation coefficients. A relatively high R² value indicates that the model successfully describes the kinetics of Direct Yellow 28 adsorption.

2.5.1 The pseudo first - order equation

The pseudo first - order equation (Lagergren, 1898) is generally expressed as follows.

$$\frac{dqt}{dt} = k1(qe - qt) \tag{3}$$

where, q_e and q_t are the adsorption capacity at equilibrium and at time t, respectively (mg g⁻¹), k_1 is the rate constant of pseudo first-order adsorption (l min⁻¹). After integration and applying boundary conditions t=0 to t=t and t=0 to t=t to the integration form of equation (3) becomes.

$$log(qe - qt) = \frac{log(qe) - k1}{2.303} \times t$$
 (4)

The value of log (q_e-q_t) were linearly correlated with t. The plot of log (q_e-q_t) Vs t should give a linear relationship from which k_1 and q_e can be determined from the slope and intercept of the plot, respectively.

2.5.2 Pseudo second order kinetic model

To describe dye adsorption, the modified pseudo second order kinetic equation is expressed as (Ho and Mckay, 2000).

$$\frac{dqt}{dt} = k2(qe - qt)2 \tag{5}$$

$$\frac{1}{\text{ge-gt}} = \frac{1}{\text{ge}} + \text{k1} \tag{6}$$

Where, k_2 is the pseudo second order rate constant. A plot of t/q_t vs t (fig. 3) enable calculation of

the rate constant k_2 , which in turn is used to calculate the initial sorption rate h as follows:

$$h = k_2 q_e^2 \tag{7}$$

The $q_{\rm e}$ value calculated from the pseudo second order model system is in good agreement with the experimental $q_{\rm e}$ values.

2.5.3 Elovich model

The Elovich or Roginsky-Zeldovich equation is generally expressed as follows:

$$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t) \tag{8}$$

where,

 q_t = The amount of dye adsorbed in mg/g, at time t.

 α = The initial dye sorption rate, mg/g. min.

 β = The adsorptions constant, g/mg during the experiment.

To simplify the Elovich equation, Chien and Clayton assumed $\alpha\beta>>1$ and on applying the boundary conditions $q_t=0$ at t=0 and $q_t=q_t$ at t=t, the above equation becomes,

$$q_{t} = \frac{1}{\beta} \ln(\alpha \beta) + \frac{1}{\beta} \ln t$$
 (9)

Thus the constants α and β can be obtained from the slope and intercept of linear plot of q_t vs ln t (fig. 4). The above equation will be used to test the applicability of the elovich equation to the kinetics of chosen adsorbent adsorbate system.

2.6 Isotherm studies

2.6.1 Langmuir isotherm

The Langmuir adsorption isotherm is based on the assumption that an adsorbent adsorbs the dye at specific homogeneous sites and once that site is occupied, no further adsorption take place at that site. Theoretically, the adsorbent has a finite capacity to absorb the adsorbate and hence there is a limit beyond which no

further adsorptions can occur. The monolayer capacity can be represented by the expression:

$$q_e = Q_0 k_L C_0 / 1 + k_L C_e$$
 (10)

The linear form of the above equation is represented as:

$$C_e / q_e = 1/Q_0 k_L + C_e / Q_0$$
 (11)

Where, k_L is related to the free energy of adsorption (L/mg) and q_0 is the maximum adsorption capacity. The value of Q_0 and k_L were calculated from the slop and intercept of the linear plot (fig. 5). The equilibrium concentration and hence the amount of dye adsorbed were calculated by varying the dye concentration between 20 mg/L and 60 mg/L with a fixed adsorbent dose and all other experiment condition kept constant.

The isotherm constants for the Langmuir isotherm under study were calculated from the linear form of the model and the correlation coefficients are given in table 4. The essential characteristics of Langmuir isotherm can be expressed in terms of a dimensionless constant called the equilibrium parameter.

$$R_{L}=1/1+K_{L} \times C_{0}$$
 (12)

The value of $R_{\rm L}$ indicates the type of isotherm to be favourable (0< $R_{\rm L}$ <1), linear ($R_{\rm L}$ =1), unfavourable ($R_{\rm L}$ >1) or irreversible $R_{\rm L}$ =0, where $K_{\rm L}$ is the Langmuir constant and C_0 is the highest initial dye concentration (mg/L).

2.6.2 Freundlich Isotherm

Freundlich isotherm is an empirical equation employed to describe heterogeneous system. The Freundlich Equation is commonly given by

$$q_e = k_f c_e^{1/n} \tag{13}$$

Where, q_e is the amount of solute adsorbed per unit weight adsorbed (mg/g), C_e the equilibrium concentration of solute in the bulk solution (mg/L), kf and n are Freundlich constant indicated the relative adsorption capacity and sorption intensity of the adsorption (mg/g). A linear form of the Freundlich

expression can be obtained by taking logarithms of then on linear form and can be written as

$$\log q_e = (1/n) \log C_e + \log k_f$$
 (14)

The Freundlich constant kf and n can be calculated from the slop and intercept of the linear plot with log $q_e v_s \log C_e$ (fig. 6).

3. RESULT & DISCUSSION

3.1 Surface characters of activated carbon

The morphology of the surface of the prepared Nanoporous activated carbon sample was examined using Scanning Electron Micrographs.

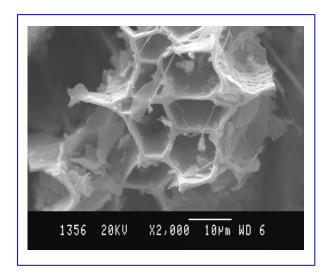


Fig. 1: SEM image of Jatropha curcas stem waste

These micrographs provide positive reception of the porosity of adsorbents and consequently a qualitative evaluation of their ability to adsorb the dye molecules in solution. *Jatropha curcas* activated carbon (ICAC) has many pores, small cavities and rough areas with micro pores which were clearly found on the surface. This shows that dolomite treatment created well developed pores with uniform distribution on the surface of the precursor, therefore leading to the activated carbon with large surface area and porous structure. The morphological study by SEM of the above adsorbent shown in the fig. 1 revealed that it is highly porous in nature (Yoshida *et al.* 1993).

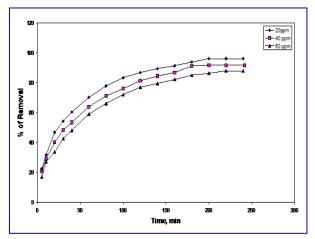


Fig. 2: Influence of Agitation time on Direct Yellow 28
Adsorption

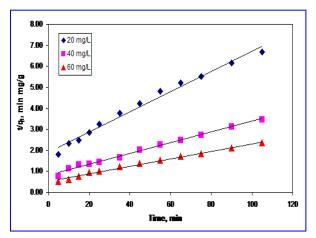


Fig. 3: Influence of initial dye concentration on pseudo second order plot for direct yellow 28 adsorption

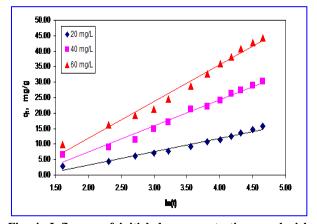


Fig. 4: Influence of initial dye concentration on elovich plot for direct yellow 28 adsorption

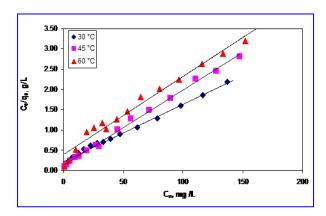


Fig. 5: Influence of temperature on langmuir plot for direct yellow 28 adsorption

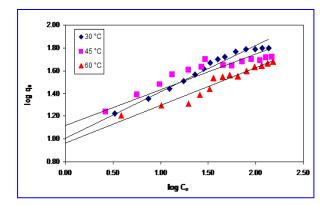


Fig. 6: Influence of temperature on freundlich plot for direct yellow 28 adsorption

3.2 Effect of pH

The p^H value of the solution being an important controlling parameter in adsorption is mainly influenced by two factors:

- (i) Distribution of the dye ionized species in the solution p^H case and
- (ii) Overall charge of the adsorbent.

Therefore the interaction between dye molecule and the adsorbent is basically a combined result of charges on the dye molecules and the surface of the adsorbent. The adsorption behavior of the dye on the adsorbent was studied over a wide p^H range of 3-9. It is observed that the p^H significantly affects the extent of adsorption of dye over the adsorbent and a reduction in the amount adsorbed dye with increasing p^H was observed. The maximum uptake of the Direct Yellow 28 is observed at pH 6.0 as well as a large decrease in adsorption capacity was observed as the p^H is increased above 6.5.

3.3 Effect of Agitation time and Initial dye Concentration

The uptake of Direct Yellow 28 from water by activated *Jatropha curcas* stem waste carbon increases when the agitation time was varied from 10 to 150 minutes and attains equilibrium at 30 °C and at p^H 6.5, when the initial concentration of the Direct dye solution used was 20 mg/L and the adsorbent dosage of 50 mg. The increase in adsorption of direct dye with increase in agitation time may be attributed to the increased intra particle diffusion occurring at long shaking time fig. 2.

3.4 Effect of Temperature on kinetic rate constant and rate parameters

For this adsorption process, adsorption experiments were carried out with fixed initial dye concentration (20 mg/L) and $p^{\rm H}$ 6.5 at different temperatures viz. 30 °C, 45 °C and 60 °C.

Table 3. The adsorpt	tion kinetic model rate constants for activated carbon JSAC at different temperatures

ı, é		Pseudo First Order	Pseudo Second Order			Elovich Model			
Adsorbent	Initial Temp.	K ₁ Min ⁻¹	\mathbb{R}^2	K ₂ g mg ⁻¹ min ⁻¹	h g mg ⁻¹ min ⁻¹	\mathbb{R}^2	βg mg ⁻¹ min ⁻¹	α g ⁻¹ mg ⁻ ¹ min ⁻¹	\mathbf{R}^2
	30 °C	0.0272	0.9601	0.0044	2.2614	0.9810	0.2114	0.0506	0.9625
JSAC	45 °C	0.0276	0.9719	0.0060	2.8261	0.9906	0.2270	0.8676	0.9585
	60 °C	0.0299	0.0979	0.0080	3.3409	0.9958	0.2451	0.7550	0.9568

The analysis of the data in table 2 reveals that the influence of temperature on the dye adsorption has little effect on the pseudo first order and elovich (fig. 4) rate constants. The table 2 also reveals that the influence of the temperature of dye on pseudo second order (fig. 3) rate constant is appreciable. It is obvious that the adsorption of dye onto the adsorbent is best described by pseudo second order rate equation with regression coefficient value which is greater than 0.99.

3.5 Isothermal modeling

The Langmuir adsorption isotherm fig. 5 and the values of high correlation coefficients (R^2) , obtained in this study indicates the applicability of Langmuir adsorption isotherm.

Table 4. Parameters of Langmuir and Freundlich adsorption isotherms for the adsorption of Direct Yellow 28 onto JSAC

Temp.	Langmuir Isotherm			ındlich therm	
	$\mathbf{K}_{\mathbf{L}}$	Q_0	1/n	$\mathbf{k_f}$	
30 °C	4.50	70.89	0.0023	10.1135	
45 °C	7.31	67.58	0.0014	13.2282	
60 °C	7.58	54.02	0.0015	9.2449	

The values of adsorption intensity (n) reveal that the applicability of Freundlich adsorption Fig. 6 is not good when compared to Langmuir adsorption isotherm. The values of 1/n and k_f are given in table 3. The values of 1/n which lie between 0 and 1 clearly describe the surface heterogeneity. The study of temperature effects on the freundlich parameters reveals decreasing trend in the adsorption capacity (Q_0) with increase in temperature. These data are useful for practical design purposes.

4. CONCLUSION

In the present study, adsorption of Direct Yellow 28 on activated *Jatropha curcas* stem waste carbon has been investigated. The data obtained through this work support that the *Jatropha curcas* carbon is an effective low cost adsorbent for the removal of Direct Yellow 28 from aqueous solution. The adsorption of Direct Yellow 28 is dependent on the initial concentration and agitation time. Equilibrium of Direct Yellow 28 adsorption reaches at 150 min. The values of correlation co-efficient (R²) obtained in this study

indicate the applicability of Langmuir adsorption isotherm. The study of temperature effects on the Freundlich parameters reveals that decreasing trend in the adsorption capacity with increase in temperature. These data are useful for practical design purposes.

The Elovich and pseudo-first order equations provided a best fit description for the sorption of Direct Yellow 28 onto *Jatropha curcas* stem waste carbon, but the pseudo-second order equation had better correlation coefficient value than elovich equation. Therefore, pseudo-second order was considered the most appropriate due to high correlation coefficient when compared to elovich equation and pseudo first order equation. The adsorption of Direct Yellow 28 onto activated *Jatropha curcas* stem waste carbon gave better results.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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