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Adsorption of Basic Textile Dye from Aqueous Solution By Prepared Activated Carbon

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ABSTRACT

Activated carbon was prepared from Jackfruit waste and it was used to remove basic dyes like Basic Fuchsin (BF) from aqueous solution. The prepared carbon was named as JC_{HNO_3} . Batch adsorption experiments were carried out to study the impact of variables such as pH of the solution, initial dye concentration, particle size, adsorbent dose and contact time on adsorption process. The experimental analyses reveal that the solution pH has tremendous effect on the sorption process. The JC_{HNO_3} was characterised by BET, SEM and FTIR analyses. In order to describe the adsorption equilibrium Freundlich, Langmuir, Tempkin and Dubinin- Radushkevish (D-R) Isotherms were used. The kinetics of adsorption was studied using pseudo-first order, pseudo-second order, Intraparticle diffusion, pore distribution and Elvoich models. The BET surface area of prepared carbon JC_{HNO_3} was found to be $987m^2g^{-1}$.

Keywords: Adsorption, Basic Fuchsin, Isotherms, Kinetics.

INTRODUCTION

Environmental problems arise because of the dismissal of industrial waste water which contains dyes. The reason for such a problem was due to the chemical structure of the dyes which gives them consistency and makes them non-cooperative in nature. Due to the discharge of dyes, other major problems are also caused for instance the released dye in water streams embody the danger of eco toxicity and also the threat of bioaccumulation. As a consequence, there was even greater hazard that was faced by the humans because of the transport of these pollutants through the food chain as it may affect their health.

Hence, this type of discharged dyes from industries especially textile waste waters has been one of the major challenges for researchers in the last two decades. It was known fact that textile dyes possess complex aromatic (odour) molecular structures. The behaviour of these molecular structures was inert and

do not biodegradable [1], so the textile dyes should be treated properly otherwise, these effluents may be discharge to near-by water bodies and caused for water pollution. There are various types of conventional waste water treatments such as coagulation, flocculation, filtration, oxidation (or) reduction and complex formations. All these are expensive textile treatment process [2]. Other than these, there is another important and useful technique to get pure water that is adsorption onto activated carbon [3-8]. But since the commercial activated carbons are costly a lot of interest was shown in finding alternate sources like Pine cone [9], Coaca shell [10], Pomegranate peel [11] Coconut shell [12], Rice husk [13] by researchers so as to enable the preparation of low cost activated carbons.

The Asia Pacific Association of Agricultural Research Institution (APAARI) made a survey and discovered and stated that the cultivation of land for Jack fruit in India was more than one lakh hectors [14]. The waste generated from this fruit leads to pollution, if it was not properly disposed. Thus present study focuses on the use of Jack Fruit Pichiparai-1 to prepare activated carbon and highlights on its efficiency on adsorption of Basic Fuchsin dye from aqueous solution.

MATERIALS AND METHODS

Preparation of activated carbon: As mentioned above, Jack Fruit Pichiparai-1 was selected for the preparation of activated carbon. The rind and pulp waste was cleaned with hot distilled water to remove dirt. Later it was dehydrated at 105° C until constant weight was secured. The withered waste was then cut into tiny pieces and mixed with K₂CO₃ solution at impregnation ratio. The impregnation ratio was given by the weight of K₂CO₃ solution / weight of water in grams. It was then dehydrated in an oven overnight at 105° C. The material was carbonised in uniform nitrogen flow at 600°C. The heating was provided at the rate of 10° C min⁻¹ from room temperature. The carbon that was obtained was cooled to room temperature and washed with hot distilled water to remove remaining chemical and filtered. The same process was repeated until the filtrate shown neutral pH. The activated carbon was then sieved to 150, 90, 75, and 45 mesh and subjected to liquid phase oxidation with 0.1N in HNO₃ for 3 hours individually. In present sorption process activated carbon having a size of 45mesh was used and named as JC_{HNO₃}

Preparation of Adsorbate: In the preparation of adsorbate, all the reagents were used belong in analytical grade. In this study experiment for the preparation especially the cationic textile dye, BF, was used supplied by SIGMA ALDRICH. It was taken directly to prepare stock solution without any further purification. The preparation of stock solution (1000 mg 1^{-1}) of selected dye was prepared by dissolving 1g dye into one litre double distilled water. The structure of the BF was shown in figure.1.





Figure 1. Molecular structure of Basic Fuchsin

Determination of zero point charge (pHzpc): The zero point charge is the pH at which external surface charge is zero [15]. The manner of preparation is 50 cm³ of 0.01.M NaCl solution was placed in a closed Erlenmeyer flask. Then the pH is adjusted to a value between 2 and 12 by adding 0.1 N Hcl or NaOH solution. Then 0.15 g of carbon sample is added and the final pH measured after 48 hours under agitation at room temperature. The *pHzpc* is the point where the curve pH (final) *vs* pH (initial) crosses the line pH (final) = pH (initial) takes as the *pHzpc* of the given carbon [16].

BET surface area: The characteristics of carbons with regard to pore structure were determined by nitrogen adsorption at -196° C by a Quanta chrome instrument (Nova 2200). The carbon was degassed at 150° C in a vacuum condition for 24 h before being measured by gas adsorption. The measured isotherms range from approximately 0.03 to 0.995. The BET surface area was determined by means of the standard BET equation applied in the relative pressure range from 0.06 to 0.3.

SEM analysis: In SEM analysis to study the texture of the surface of the prepared activated carbon by Scanning Electron Microscope SEM Hitachi- S520 (OXFORD LINK-ISIS), before and after adsorption of dye.

FTIR Analysis: To identify the surface functional groups, the surface characterization of JC_{HNO_3} before and after dye adsorption was performed with the help of Fourier Transform Infrared Spectroscopy (FTIR), was recorded by Thermo Nicolet Nexus 670 spectrometer in the wave number range 400 – 4000 cm⁻¹.

Batch adsorption studies: All the experiments were carried out in 250 mL conical flasks with 100 ml test solution at room temperature ($25\pm2^{\circ}$ C). 1 g of the adsorbent is added to the text solution and makes it to a neutral pH. The flasks were shaken in horizontal shaker at 120 rpm to study the equilibration time (10-100 minutes) to ensure maximum adsorption of dye and to know the kinetics of adsorption process. At the end, the samples were filtered using Whatman no. 42 filter paper, and these filtrates were analyzed for residual dye concentration at wave length of 544 nm.

The test solution of dye of 50 mg 1⁻¹ concentration was prepared from fresh stock solution. The same process of filtration is done. The first part of the filtrate was discharged to avoid the effects of adsorption onto the filter paper and the remaining filtrate was analyzed for residual dye concentration using UV-Visible spectrophotometer. Control experiments were executed in duplicate to rectify any adsorption of dye in containers.

$$\% removal = \frac{(C_i - C_e)}{C_i} X100 \dots \dots \dots \dots \dots \dots (1)$$

Where C_i = initial concentration of dye solution in mg L⁻¹, C_e = equilibrium concentration of dye solution in mg L⁻¹, m = mass of the adsorbent in grams (g), V = Volume of test solution in liters (L).

Effect of adsorbent dosage: Studies were made on the removal of percentage of BF by adsorption on to JC_{HNO_3} in the range 0.1 to 1.4 g with 50 mg 1⁻¹ initial concentration of dye and agitation time of 60 minutes at pH 7 and at temperature (25±2°C). The results are presented on percentage removal of dye versus adsorbent dosage was shown in figure.2.



Figure 2. Effect of adsorbent dose on adsorption of BF on JC_{HNO_2}

Effect of contact time: The adsorption experiments had been conducted to the extent of removal of dye at an optimum initial concentration of dye of 50 mg L⁻¹ with optimum dose (1g) of adsorbent at pH 7 and at room temperature $(25\pm2^{\circ}C)$ by varying the agitation time from 5 to 60 min. The time profile of adsorption of dye on to adsorbent was represented in figure. 3.



Figure 3. Effect of contact time on adsorption of BF on JC_{HNO_3}

Effect of initial concentration of dye: Keeping the control parameter at these optimum conditions, the effect of initial concentration of dye and the removal of dye in terms of percent removal and the amount of dye adsorbed on prepared JC_{HNO_3} has been studied. The effect of adsorption was shown in figure.4.



Figure 4. Effect of initial concentration of dye solution on adsorption of BF

Effect of particle size: Studies and experiments were conducted to evaluate the influence of the particle size of the adsorbent on the adsorption of BF, this was shown in figure.5.



Figure 5. Effect of particle size on adsorption of BF

Effect of pH: Studies were also made on the effect of pH on adsorption by varying the pH from 3 to 12 under constant process parameters i.e. the initial concentration of the standard dye solution which is 50 mg 1^{-1} . The results were shown in figure.6.



Figure 6. Effect of pH on adsorption of BF

Adsorption isotherms: The final data received from the experiments in the present work was tested with Freundlich [16] Langmuir [17] Tempkin [18] and Dubinin-Radushkevich (D-R) [19] isotherms given in table 1.

Freundlich isotherm										
Equation	Graph	R ² logk _F			1/n	k _F	n	q _{e(expt.)}	q _{e(cal.)}	χ2
$q_e = k_F C_e^{1/n}$ $log q_e = 1/n \ log C_e$ $+ log k_F$	logq _e vs logC _e					$(mg^{1-1/n} l^{1/n} g^{-1})$	(g l ⁻¹)	(mg g ⁻¹)	(mg g ⁻¹))
y=0.3099x+0.4257		0.9611	0.4257		0.3099	2.6650	3.2268	4.7240	3.6504	0.2440
Langmuir isotherm										
Equation	Graph	\mathbf{R}^2	a_L/k_L		1/k _L	$\mathbf{q}_{\mathbf{m}}$	R _L	q _{e(expt.)}	q _{e(cal.)}	χ2
$\begin{array}{l} q_e & = \\ k_L C_{e'} (1 + a_L C_e) \\ C_{e'} q_e &= a_L / k_L \ C_e \ + \\ 1 / k_L \end{array}$	C_{e}/q_{e} vs Ce					(mg g ⁻¹)		(mg g ⁻¹)	(mg g ⁻¹))
y=0.1277x+0.2956		0.9891	0.1277	,	0.2956	7.8309	0.3165	4.7240	4.2589	0.0458
Temkin equation										
Equation	Graph	\mathbf{R}^2		В	Bln(A)	В	Α	q _{e(expt.)}	q _{e(cal.)}	χ2
$\begin{array}{l} q_e = RT/bln(Ac_e) \\ q_e = Bln(C_e) + \\ Bln(A) \\ where \ RT/b = B \end{array}$	q_e vs lnC_e						(1 g ⁻¹)	(mg g ⁻¹)	(mg g ⁻¹)	
y=1.1036x+3.2804		0.9583		1.1036	3.2804	1.1036	19.5398	4.7240	4.4008	0.0221
D-R equation										
Equation	Graph	R ²		lnq _m	В	q _m	Е	q _{e(expt.)}	q _{e(cal.)}	χ2
$q_e = q_m e^{-\beta \varepsilon^2}$ $\ln q_e = -\beta \varepsilon^2 + \ln q_m$ where ε = $RT(1+1/C_e)$	$lnq_e vs$ ε^2				(mol ² J ⁻²)	(mg g ⁻¹)	(kJ mol ⁻ ¹)	(mg g ⁻¹)	(mg g ⁻¹)	
y=-0.0021x- 9.4595		0.9757		-9.459	0.0021	22.510	15.43	4.7240	5.2471	0.0579

Table 1: The adsorption Isotherms of BF on JC_{HN}

Chi-square analysis: The chi-square analysis was carried out to recognize the suitable isotherm for sorption of dye on to carbon adsorbents. The mathematical expression was

Where $q_{e(expt.)}$ and $q_e(cal.)$ are the experimental sorption capacity of dye (mg g⁻¹) at equilibrium time and the corresponding value that is obtained from the sorption model. If data from the model are similar to the experimental data, χ^2 will be a small number, while if they differ; χ^2 will be a bigger number [20].

Adsorption kinetics: Five Kinetic models were used to study the adsorption kinetics. These are pseudo-first-order [21], pseudo-second – order [22], Weber and Morris intraparticle diffusion [23], Bangham's pore diffusion [24] and Elovich models [25]. The equations and results relevant to this were shown in table 2.

Pseudo-first-order								
Equation	Graph	\mathbf{R}^2	k ₁ /2.303	log(q _e)	k ₁	q _{e(cal.)}	q _{e(expt.)}	SSE
$dq_t/dt = k_l(q_e-q_l)$ $log(q_e-q_l) = -$ $k_lt/2.303 + logq_e$	$log(q_e - q_t)$ vs t					mg g ⁻¹	mg g ⁻¹	
y=-0.0321x+0.3193		0.9759	0.0321	0.3193	0.07393	2.0859	4.7240	0.31186
Pseudo-second-order								
Equation	Graph	R ²	1/q _e	$1/(k_2 q_e^2)$	k ₂	$q_{e(cal.)}$	q _{e(expt.)}	SSE
$\frac{l/(q_e - q_t)}{t/q_t} = \frac{k_2 t + 1/q_e}{(1/q_e)t} + \frac{l/(k_2 q_e^2)}{t/(k_2 q_e^2)}$	t/q_t vs t					mg g ⁻¹	mg g ⁻¹	
y=0.1989x+0.6657		0.9991	0.1989	0.6657	0.05943	5.0277	4.7240	0.00413
Intraparticle diffusion								
Equation	Graph	R ²	k _{ip}	С	k _{ip}	q _{e(cal.)}	q _{e(expt.)}	SSE
$q_t = k_{ip}t^{1/2} + C$	$q_t vs t^{1/2}$					mg g ⁻¹	mg g ⁻¹	
y=0.3381x+2.6514		0.9799	0.3381	2.6514	0.3381	4.6516	4.7240	0.00023
Pore diffusion								
Equation	Graph	\mathbf{R}^2	Α	$log(k_0/(2.303V))$	ko	q _{e(cal.)}	q _{e(expt.)}	SSE
$loglog(C_i/(C_i-q_im)) = alogt + log(k_0/(2.303V))$	loglog(Ci/(Ci- qtm)) vs logt				$\underset{l}{\overset{ml}{}}_{j} (g l)^{-1}$	mg g ⁻¹	mg g ⁻¹	
Y=0.1525-1.6207		0.9701	0.1525	-1.6207	5.5156	4.6127	4.7240	0.00055
Elovich equation								
Equation	Graph	R ²	1/β	$1/\beta \ln(\alpha\beta t)$	a	q _{e(cal.)}	q _{e(expt.)}	SSE
$q_t = \frac{1}{\beta} \ln(1 + \alpha \beta t)$ $q_t = \frac{1}{\beta} \ln(t) + \frac{1}{\beta}$ $\ln(\alpha \beta t)$	$q_t vs ln(t)$					mg g ⁻¹	mg g ⁻¹	
Y=0.5994X+2.4030		0.9725	0.5994	2.4030	0.8249	4.2688	4.7240	0.00928

Table 2: The adsorption	n kinetics of BF on JC _{HNO3}
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Fitness of the kinetic models: The model that was best suited to among kinetic models was assessed by the squared sum of errors (SSE) values. It was assumed that the model which gives the lowest SSE values is the best model for the particular system [26,27].

RESULTS AND DISCUSSION

The BET surface area of JC_{HNO_3} was found to be $987m^2$ g⁻¹. The study of effect of adsorbent dose discovered that the optimum amount of JC_{HNO_3} was fixed for further analysis for the removal of dye. At 1 g the percentage removal was found to be 94.98 %. From figure.2 it was clear that the rate of removal of dye was found to be increased rapidly with the increase of carbon dose and showed down in later. This could be supported by figure. 3, it was clear that the percentage removal was more by increasing the contact time [28]. But after 40 min, it was gradually approached a constant value, denote the attainment of equilibrium. Therefore 40 min was fixed as optimum contact time. Decreased removal rate towards the end indicative the possible monolayer adsorption on surface of adsorbent.

The percentage removal of dye was decreased from 99.4% to 65.83%, with the increase the dye concentration which was observed in Figure.4 the reason for this, at low concentration of adsorbate the ratio of surface active sites to the total dye is high. It clearly reveals that at low concentration dye molecule contains more number of available active sites. 94.48% of dye was removed at 50 mg L⁻¹ and it was fixed as optimum concentration.

From figure.5 we can conclude that the percentage removal of dye was decreased with increasing particle size of prepared carbon. The reason for this is, large sized particles normally have less surface area. Therefore the particle having the size below 45μ were used for adsorption process.

PZC value of JC_{HNO_3} was found to be 4. its reveals that if pH>*PZC*, acidic functionalities will dissociates, releasing protons into the medium and leaving negatively charged surface on carbon [29]

The percent removal of dye below its *PZC* value (*PZC*=4) is very low that is from 20 -50% which is observed in Figure.6.At pH level the percentage removal of dye attains a value 96%, and almost the same value (95%) is observed at a pH which is equal to 7.This value is fixed as optimum pH. The increased percent removal of dye solution is due to the fact that since the adsorbent surface got negative charge after its *PZC* value and there by increased the electro static force between cationic dye and surface of adsorbent. Figure. 7(a) shows Freundlich plot between logq_e and logc_e. The values of Kf and 1/n were obtained from the slope and intercept of the linear plot and were tabulated in Table.1.The Freundlich correlated coefficient value was found to be 0.9611. This value appears to be more than χ^2 value (0.244). The high χ^2 reveals that this model was not suitable to explain the sorption process [30].

The Langmuir graph of C_e/q_e as function of C_e was depicted in Figure.7(b). From the plot monolayer capacity (q_m) and Langmuir constant (a_L) values are evaluated and is presented in table.1

In order to explain the adsorption phenomena with its high R^2 value (0.9891) and low $\chi 2$ value (0.0458) Langmuir model is far better than Freundlich isotherm. The value of dimension less constant separation factor (R_L) was 0.3165 and it is presented in table.1. The value lies in the range between 0-1 clearly gives as an idea that the adsorption of dosage if favorable [31]. The adsorption capacity q_m was found to be 7.8309 mg g⁻¹.

Figure.7(c) shows the Tempkin plot of lnC_e vs q_e . The slope of the line gives the value of 'B' and intercept values were also given in table.1. The R² value for this isotherm was found to be low (0.9583).

Tempkin isotherm is well suited for dye sorption process because of low χ^2 (0.0221) value. This suggests that the heat of adsorption of all the dye molecules in the layer decreases linearly with coverage due to adsorbent – adsorbate interactions. The adsorption might characterized by uniform distribution of binding energies [16].

Dubinin-Radushkevich plot of ε^2 vs q_e was shown in figure 7(d). The values of slope β , q_m and mean free energy E were given in Table.1. The high value R² (0.9757) and low χ^2 (0.0579) reveals that sorption process is suitable and also confirmed by D-R plot. These values are also proves that those is some degree of heterogeneous surface factor. The value of E (15.43) is within the values of ion exchange, and also indicates adsorption mechanism may be Chemisorption [32].



The different graphs varied kinetic models figure.8 (a) represents Pseudo first order kinetic model plot. This has low R^2 (0.9759) and high SSE value (0.31186). Not only that log (q_e (expt) [i.e., (log (9.64) =0.984)] is not equal to the intercept value (0.3193) of the plot .This indicates that the sorption kinetics does not follow pseudo-first order kinetics.

Figure.8 (b) represents Pseudo second order kinetic model plot. The plot of T vs T/q_t , the slop and intercept values along with R^2 and SSE are given inTable.2. It has high R^2 value (0.9991) and low SSE value (0.00413). Hence the sorption process can be well described by pseudo -second order model confirming to other well established models. It predicts the behaviour over the whole range of studies and it is in agreement with the chemisorptions mechanism being the rate controlling step [33].

Figure 8 (c) shows intra particle diffusion plots of $t^{1/2}$ vs q_t and corresponding results were tabulating in table.2. It is evident from the plot that there are two separate stages; first linear portion (stage I) and second curved path followed by a plateau (stage II). In stage I attributed to the immediate utilization of most readily available adsorbing sites on adsorbent surface. In stage –II, very slow diffusion of adsorbate from

surface site into the inner pores was observed. So, dye removal mechanism assumed to involve the following 4 steps.

1 .Migration of dye from bulk of the solution to the surface of the adsorbent (Bulk diffusion).

- 2 .Diffusion of dye through the boundary layer to the surface of the adsorbent (Film diffusion).
- 3. Transport of the dye from the surface to the interior pores of the adsorbent (Intra particle diffusion).

4. Adsorption of dye at an active site on the surface of the material (Chemisorption via ion exchange as indicated by the fitting of sorption process to Langmuir and Dubinnin Isotherms).

The intercept of line in figure.8(c) did not pass through origin. It indicates some degree of boundary layer control in sorption process. So, intra particle diffusion is not only the rate controlling step. Figure.8 (d) represents Bangham's plot of log (t) vs $\log(\log(C_i/(C_i-q_t m)))$ and the corresponding values were tabulated in table.2. It also has moderate R^2 (0.9701) but very low MSSE Value (0.00055). Therefore the rate determining step of adsorption dye on to adsorbent might be pore diffusion. Figure 8(e) represents Elovich plot of *ln* (*t*) vs q_t and corresponding data were given in Table.2. It also has moderate R^2 value (0.9725) but very low SSE Value (0.000928). Adsorption phenomenon also fitted to Elvoich plot kinetic pattern , which is confirmed to this equation might be taken as evidence that the rate determining step is diffusion in nature [34]. whenever the rate of desorption can be neglected we could use this equation [35].





Figure 8. Adsorption Kinetics of BF on JC_{HNO_2}

Figure. 9 shows SEM image of JC_{HNO_3} with more number of pores, these pores were well closed by dye molecules which were confirmed by FTIR, because of the appearance of new functional groups of dye molecules on dye loaded carbon.



The FTIR spectra of JC_{HNO_3} and JC_{HNO_3-BF} were shown in figure.10. Bands around 3400-3500 cm⁻¹ in JC_{HNO_3} are due to –OH stretches in hydroxyl, carboxylic and phenolic groups. Similarly, in FTIR of JC_{HNO_3} -BF, the intensity of band at 3445 cm⁻¹ was decreased and new bands with specific intensities between 885-811 cm⁻¹ were observed. These bands were characteristic of out of plane deformational vibration of two adjacent hydrogen atoms in 1, 4-disubstituted benzene rings of BF dye molecule [34]. A distinct peak was observed at 1578 cm-1. This is may be due to poly aromatic C=C stretching vibrations in sp2-hybridized carbons [37, 38]. In case of dye loaded carbons the intensity of bands at 1585, 1163-1150 cm⁻¹ was increased. It represents the increased aromatic nature due to adsorption of dye molecules.



Figure.10 FTIR spectrum of JC_{HNO3} and $JC_{HNO3-BF}$

APPLICATIONS

The textile effluent samples have been collected from Dharmavaram in Ananthapur District for the efficiency of prepared carbon adsorbent in decolorizing these textile effluents was studied. The experiments reveal that the prepared carbon successfully removes the color from the effluents. It was observed that besides the removal of color, it was concurrent reduction of COD and BOD of the effluents until the permissible limit.

CONCLUSIONS

On experimentation, the prepared JC_{HNO_3} has successfully removes Basic Fuchsin dye from its 50 mg L⁻¹ aqueous solution. It is found that the adsorption process attains equilibrium time within 40 min and required adsorbent dose is 1 g. The kinetics of the process can be best explained by pseudo second order pattern and indicates that the dye was chemisorbed. Nevertheless pore diffusion is one of the rate controlling steps during sorption process. The prepared activated carbon successfully removed the color of the effluents until the acceptable limit.

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