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Adsorption Study With Different Eco-Friendly Activated Carbons For The Removal Of Basic Dye From Aqueous Solution

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Abstract

A carbonaceous adsorbent prepared from plant material like Acacia Arabic Bark Carbon (AABC) was found to show good porosity, appreciable surface area and consequently adsorbs dyes to an appreciable extent. Effect of various experimental parameters have been investigated using batch adsorption technique at room temperature (30±1°C) and the adsorption of Rhodamine B (RB) on carbonaceous adsorbent confirms to Langmuir equation, is a first-order process and pore diffusion controlled. The efficiency of carbonaceous adsorbent was evaluated by comparing the results with those obtained on a Commercial Activated Carbon (CAC). These results indicate that AABC could be employed as low-cost adsorbent for the removal of cationic dye from industrial wastewater. This investigation suggested that natural adsorbents are from plant materials can be utilized as adsorbent, because of their appreciable extent removal of dyes. FT-IR spectra of the adsorbents were recorded to explore the number and position of functional groups available for the binding of dye onto adsorbents. SEMs of the native and exhausted (CAC and AABC) were recorded to explore the morphology of the adsorbent.

Key Word

Rhodamine B, Commercial Activated Carbon, Acacia Arabic Bark Carbon, Freundlich and Langmuir isotherms, Kinetics of adsorption, low cost adsorbents.

INTRODUCTION

Our environment is composed of atmosphere, hydrosphere, lithosphere, and biosphere, of which hydrosphere plays a vital role. The quality of ground water is very much important, as it is depleting and being contaminated in many parts of the world. Now-a-days the increase in population increases the need for textile, leather, food processing, dyeing, cosmetics, paper and dye manufacturing products. This increased production lead to the release of toxic and hazardous wastes into the environment in solids, liquids or gaseous form [1]. Environmental harm and visual pollution are mostly caused by the release of colored effluent from sectors like textiles, leather, paper, and plastics [2]. In addition to their endurance in water bodies, the organic dyes are distinguished by their intricate structures and synthetic origins poses a serious risk to human health and aquatic life because of their mutagenic, carcinogenic, and toxic consequences [3]. Many industrial processes use different synthetic chemical dyes for various purposes. Some frequent users of these chemicals include paper and pulp manufacturing, dyeing of cloth, leather treatment, printing etc. Most of the used solutions containing such dyes are discharged as effluents [4]. It affects the aesthetic nature and they produced land and water pollution. Thus, pollution caused by industrial wastewater has become a common problem for many countries. From environmental points of view; the discharge of dyes into natural water bodies represents a serious problem because of their persistence and non-biodegradable characteristics [5].

The Rhodamine B (RB) dye family was used in many industrial sectors and was one of the earliest and most popular synthetic dyes. Rhodamine B is one of the basic dyes of the xanthene class that is most eco-friendly, with chemical structure and with a high water solubility [6]. Rhodamine dyes are generally toxic, and are soluble in water, methanol, and ethanol. RB is used for dyeing of various products including cotton, silk and paper, and finds a variety of applications in the analytical and photo-chemical fields [7].

The application of the adsorption technique has assumed remarkable importance in the treatment of contaminated waters and effluents, especially if the adsorbent employed presents low cost and does not require any treatment before

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its utilization [8]. Adsorption process using activated carbons are widely used to remove pollutants from wastewaters. Therefore, the development of suitable adsorption techniques for the removal of dyes from aqueous medium becomes an important approach [9]. Many adsorbents based on low cost and agricultural by-products have been used for dye adsorption from wastewater, which included banana pith [10], sawdust [11], babul seed [12], fly ash [13], ocimum sanctum carbon [14], moringa oliefera bark [15], orange peels [16] and jute sticks [17] have also been reported as efficient adsorbent for removing colour.

In the present study removal of RB on AABC was studied by batch adsorption model. The effect of process parameters such as pH, adsorbent dose, contact time and initial concentration were investigated. The kinetics of RB adsorption on CAC and AABC was analyzed by fitting various kinetic models. Experimental equilibrium data were fitted to the Freundlich and Langmuir equations to determine the best fit isotherm equation. The data thus obtained may be helpful to design adsorption treatment systems and establishing a continuous treatment plant for water and wastewater.

2.0 MATERIALS AND METHOD

2.1 ADSORBATE

Exactly 50 mL of dye solutions of various initial concentrations were prepared in standard measuring flask by diluting the stock solution of the dye (1000 ppm). The optical density (OD) of the dye solution was measured spectrophotometrically by using UV- Visible spectrophotometer at the wavelength of maximum absorption (λ_{max}) 544 respectively for the RB dye [18].

2.2 PREPARATION AND ACTIVATION OF ADSORBENT

Commercial Activated Carbon (CAC) was procured from E Merck (India). The raw material *Acacia Arabic Bark* (AAB) were collected locally, washed, dried, carbonized at 300-400°C and thermally activated with sodium bicarbonate at 700-900°C to produce CO₂ inert atmosphere at this temperature, which avoids ash formation. The obtained carbon was ground well to a fine powder and sieved (Jeyant Sieve, India) into discrete particle size (90, 125, 150, 180, 210 and 250 micron) and stored. Required particle size especially (90 micron) was activated by acid digestion (4N HNO₃; 2hr at 80°C' and in an air oven (at 120°C for 2 hr) [19]. The resulting carbon washed with distilled water until a constant pH of the slurry reached.

2.3 ADSORPTION EXPERIMENTS

An accurately weighed quantity of the dye (RB) was dissolved in double distilled water to prepare the stock solution (1000 mg/l). The percentage purity of the dye was taken into consideration while preparing the stock solutions. Adsorption experiments were carried out at room temperature (30 \pm 1°C) under batch mode [20, 21]. The values of percentage removal of dye and amount adsorbed (q in mg g⁻¹) were calculated using the following relationships:

Percentage removal = $100 (C_i - C_e) / C_i$ (1) Amount adsorbed (q) = $(C_i - C_e) / m$ (2) Where C_i and C_e are the initial and equilibrium (final) concentration of dye (in mgL⁻¹) respectively, m is the mass of adsorbent, in gL⁻¹.

3.0 RESULTS AND DISCUSSION

3.1 Effect of initial concentration

Effect of initial concentration on the extent of removal of RB (in terms of percentage removal and amount adsorbed) on CAC and AABC were studied. The initial concentration provides an important driving force to overcome all mass transfer resistance of dye anions between the aqueous and solid phases [22]. The relevant data are given in Table 1. The percentage removal is found to decrease exponentially, while the amount of RB adsorbed increases exponentially with the increase in initial concentration of RB (Figure 1). This indicates a decrease in adsorption, which is attributed due to the lack of available active sites required for the high initial concentration of RB.

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Table 1. Effect of process parameters on the extent of removal of RB by CAC and AABC at 30 ±1°C.

| Variation | Adsorbents | Initial concentration (ppm) | Contact time (min) | Dose of ACs (g/l) | Initial pH | Particle size (µ) | Percentage Removal (%) | Amount Adsorbed (mg/L) |
|-----------------------------------|-------------|--------------------------------------|--------------------------|------------------------------|---------------|----------------------|------------------------------|--------------------------------------|
| Initial Concentration (ppm) | CAC AABC | 20 - 200 (100*) 10 - 100 (50*) | 30 30 | 2 2 | 7.5 7.5 | 90 90 | 99.0 - 85.2 96.0 - 82.0 | 4.95 - 42.6 2.4 - 20.50 |
| Contact time (min) | CAC AABC | 100 50 | 5-50 5.50 | 2 2 | 7.5 7.5 | 90 90 | 88.2 - 99.4 90.2 - 99.6 | 22.05- 24.85 11.28 - 12.45 |
| Dose of ACs (g/l) | CAC AABC | 100 50 | 30 30 | 1.5 - 2.4 1.5 - 2.4 | 7.5 7.5 | 90 90 | 84.2 - 98.4 90.0 - 99.4 | 28.07 - 93.60 15.0 - 23.35 |
| Initial pH | CAC AABC | 100 50 | 30 30 | 2 2 | 2-11 2-11 | 90 90 | 85.4 - 98.0 89.2 - 98.0 | 21.35 - 24.50 11.15 - 12.25 |
| Particle size (μ) | CAC AABC | 100 50 | 30 30 | 2 2 | 7.5 7.5 | 45-250 45-250 | 99.2 - 84.4 99.6 - 83.6 | 24.8 - 21.10 12.45 - 10.45 |

^{*} Optimum Initial Concentration

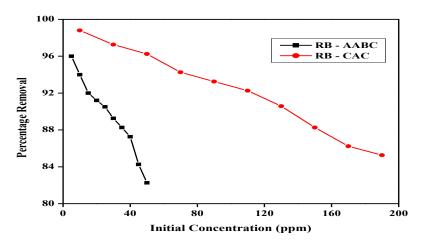


Figure -1 Effect of Initial concentration for the removal of RB onto CAC and AABC

3.1.1 Adsorption Isotherms

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In order to determine the adsorption potential, the study of adsorption isotherm is essential in selecting an adsorbent for the removal of dyes [23]. The adsorption data were analyzed with the help of Freundlich and Langmuir isotherms.

Freundlich isotherms:
$$\log q_e = \log k + (1/n) \log C_e$$
 (3)
Langmuir isotherms: $(C_e/q) = (1/Q_ob) + (C_e/Q_o)$ (4)

Where, k and 1/n are the measures of adsorption capacity and intensity of adsorption, respectively. q is the amount dye adsorbed per unit mass of adsorbent (in mgg^{-1}) and C_e is the equilibrium concentration of dye (in mgL^{-1} or ppm); Q_o and b are Langmuir constants, which are the measures of monolayer adsorption capacity (in mgg^{-1}) and surface energy (in gL^{-1}), respectively. The values of Freundlich and Langmuir constants obtained from the linear correlation between the values of (i) $log q_e$ and $log C_e$ (ii) (C_e/q_e) and C_e . They are found to be linear (Figure 2A, 2B). The applicability of Langmuir isotherm indicates the formation of mono-layer and also nature of adsorption process. The values of adsorption isotherm constants along with the correlation co-efficients are presented in Table 2. In order to compare the validity of each model a normalized standard deviation, $\Delta q(\%)$ is calculated using the following equation.

Where the superscripts, exp. and cal. are the experimental and calculated values of q_t viz., the amount adsorbed at different time t and n is the number of measurements. The Δq (%) values are also given in Table 2. Based on the low values of Δq (%), it is concluded that the adsorption of RB can best be described by the Langmuir adsorption isotherm. This indicates the applicability of Langmuir isotherm and the mono-layer coverage on adsorbent surface [24]. The mono-layer adsorption capacities (Q_0) of the adsorbents are found to be of the order:

Adsorption capacity of AABC is better and nearer to CAC. Further, the essential characteristics of the Langmuir isotherm can be described by a separation factor, R_L , which is defined by the following equation [25].

$$R_L = [1/(1 + bC_i)]$$
 (6)

Where, R_L is the separation factor, C_i and b are the initial concentration of dye (in mg L^1 or in ppm) and Langmuir constant (in g L^1). The value of R_L , indicates the shape of the isotherm and nature of the adsorption process as given below;

| <u>R_L value</u> | Nature of adsorption process |
|----------------------------|------------------------------|
| $R_L > 1$ | Unfavourable |
| $R_L = 1$ | Linear |
| $0 \le R_L \le 1$ | Favourable |
| $R_L = 0$ | Irreversible |

In the present study, the value of R_L (0.123 and 0.139) indicates that the adsorption process is favourable for this low cost adsorbent.

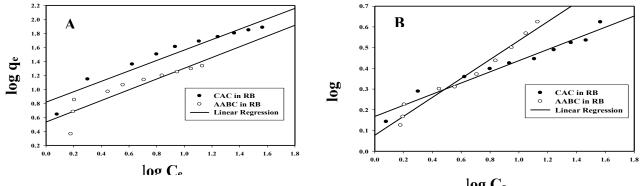


Figure - 2 (A) Freundlich and (B) Langmuir adsorption isotherms for the removal of RB onto CAC and

AABC

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Table 2. Freundlich and Langmuir parameters of adsorption isotherms for the removal of RB by CAC and AABC at 30 ±1°C

| S.No | Parameters | Adsorbents | | | | |
|-------|---------------------------------|------------|-------|--|--|--|
| 0.140 | 1 arameters | CAC | AABC | | | |
| | Freundlich isotherm | | | | | |
| | Slope(1/n) | 0.872 | 0.584 | | | |
| 1 | Intercept (log k) | 53.85 | 5.257 | | | |
| | Correlation coefficient (r) | 0.958 | 0.968 | | | |
| | $\Delta q(\%)$ | 0.017 | 0.008 | | | |
| | Langmuir isotherm | | | | | |
| | Slope(1/Q _o) | 0.039 | 0.178 | | | |
| | Intercept (1/Q _o b)) | 0.185 | 0.598 | | | |
| 2 | Correlation coefficient (r) | 0.937 | 0.987 | | | |
| L | $Q_o (mg/g)$ | 35.874 | 22.78 | | | |
| | b (g/L) | 0.625 | 0.458 | | | |
| | R_{L} | 0.123 | 0.139 | | | |
| | $\Delta q(\%)$ | 0.006 | 0.004 | | | |

3.2 Effect of contact time

The effect of contact time on the amount of RB adsorbed is observed at the optimum initial concentration of RB. The relevant data are given in Table 1. The extent of removal of RB by these adsorbents is found to increase exponentially and reach a maximum value with increase in contact time (Figure 3).

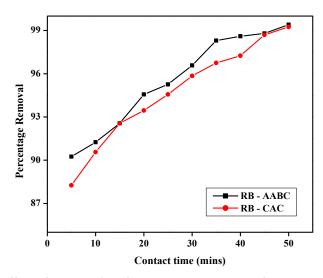


Figure - 3 Effect of contact time for the extent removal of RB onto CAC and AABC

The relative increase in the extent removal of RB after 35min., of contact time is not significant and hence it is fixed as the optimum contact time. The contact time experiments are useful to investigate the influence of system parameters on the rate and extent of adsorption [26].

3.2.1 Kinetics of adsorption

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The kinetics and dynamics of adsorption of RB these two adsorbents have been studied by applying the various first order kinetic equations, proposed by Natarajan-Khalaf-as cited by Kannan and Vanangamudi [27], Lagergren as cited by Trivedi [28] and Bhattacharya- Venkobachar [29].

: $Log(C_i/C_t) = (k/2.303)t$ (7) Natarajan and Khalaf equation

: Log $(q_e - q_t) = \log q_e - (k/2.303) t$ (8) Lagergren equation

: Log [1 - U(T)] = -(k/2.303) t (9) Bhattacharya and Venkobachar equation

Where k is the first order rate consant (min. $^{-1}$) for adsorption of RB. C_i and C_t are the concentration of dye (in mg L¹ or ppm), at time zero and at time t respectively; q_e and q_t are the amount of dye adsorbed per unit mass of the adsorbent (in mg g⁻¹) and at time t respectively,

Where U(T) = $[(C_i - C_t)/(C_i - C_e)]$ (10) Where Ce is an equilibrium concentration of RB (mg L¹). The values of (i) $\log (C_i/C_t)$, (ii) $\log (q_e - q_t)$ and (iii) $\log [1-U(T)]$ are linearly correlated with time (t). The values of first order rate constant (k), Δq (%) and correlation co-efficient (r-value) are given in Table 3. All the linear correlations are found to be statistically significant, indicating the applicability of these kinetic equations and the first order nature of the adsorption process of RB on these adsorbents. The rate of adsorption (k-value) for CAC and AABC are obtained from Lagergren equation and it is found to be almost equal with the value obtained from Bhattacharya and Venkobachar equation. Hence any one of these kinetic equations could be used in future to calculate k-value for the adsorption of dyes. Lagergren plots are shown in Figure 4 (B), based on the high r-values, which are close to unity and low Δq (%) values, it is concluded that the Lagergren equation is applicable to the kinetics of adsorption of RB.

3.2.2 Intra particle diffusion model

The adsorbate species (dye RB) are most probably transported from the bulk of the solution into the solid phase through intra particle diffusion / transport process, which is often the rate limiting step in many adsorption processes, especially in a rapidly stirred batch reactor. The possibility of intra particle diffusion process was explored by using the Weber and Morris intra particle diffusion model [30].

$$q_t = k_p t^{1/2} + C$$
(11)

 $q_t = k_p t^{1/2} + C$ (11) Where q_t is the amount of dye adsorbed (in mg g^{-1}) at time t; C is the intercept and k_p is the intra- particle diffusion rate constant (in mg g^1 min $\frac{1}{2}$). The values of q_t are found to be linearly correlated with values of $t^{1/2}$ (Figure -5A). The k_p values were calculated by using correlation analysis (Table 3). The r-values are found to be close to unity, this reveals the presence of intra particle diffusion process. Based on the calculated values of k_n (mg g⁻¹ min.^{-1/2}) for AABC, indicates that the AABC has more porous like CAC. The large values of intercept (Table 3) give an idea about the boundary layer thickness i.e. larger the intercept; greater is the boundary layer effect.

The intercept (C) value of CAC is more and AABC is less, indicating the boundary layer effect is maximum in CAC and minimum in AABC. The correlation of the values of log (% removal) and log (time) also resulted in linear relationship (Figure 5-B). This indicates that the process of intra particle diffusion is also taking place in these adsorption systems (Table 3). The divergence in the values of slope from 0.427-0.457 indicates the presence of intra particle diffusion process as one of the rate limiting steps, besides many other processes controlling the rate of adsorption, all of which may be operating simultaneously.

Table 3. Kinetics and dynamics of adsorption of RB by adsorption on CAC and AABC at 30 ±1°C.

| | Parameters | CAC | AABC |
|------|--|-------|-------|
| S.No | | | |
| | Natarajan & Khalaf equation. | | |
| 01 | Correlation Coefficient (r) | 0.995 | 0.945 |
| | 10 ² k (min ⁻¹) | 9.102 | 0.545 |

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| | | | | 1 | | |
|----------|---------------------------|---------|---|--------|---------|------|
| | | | Δq (%) | 7.987 | 35.24 | |
| | | | Lagergren equation. | | | |
| | | 02 | 0.999 | 0.998 | | |
| | | UZ | 10 ² k (min ⁻¹) | 17.101 | 6.985 | |
| | | | Δ_{q} (%) | 2.242 | 10.987 | |
| | | | Bhattacharya and Venkobachar equation. | | | |
| | | 03 | Correlation Coefficient (r) | 0.994 | 0.978 | |
| | | 0.5 | $10^2 \text{k (min}^{-1})$ | 16.479 | 9.047 | |
| | | | Δq (%) | 7.015 | 18.01 | |
| | | | Intra Particle diffusion Model. | | | |
| | | | Correlation Coefficient (r) | 0.972 | 0.969 | |
| | | 04 | $k_{p} (\text{mg g}^{-1} \text{ min}^{-1/2})$ | 73.42 | 24.02 | |
| | | | Intercept (C) | 75.28 | 18.24 | |
| | | | Δq (%) | 1.302 | 0.018 | |
| | | 05 | Log (% removal) Vs log (time) | 2 000 | 2.050 | |
| | | | Correlation Coefficient (r) | 0.998 | 0.978 | |
| | | | Slope (m) | 0.427 | 0.457 | |
| | | | Δq(%) | 17.45 | 35.14 | |
| 100 COCt | 4 - 2 - 0 - 8 - 0 - 6 - 0 | A Conta | act time (min) 2.8 C CAC in RB AABC in RB Linear Regression Linear Regression | | 0 25 30 | sion |
| | | | 2.5 10 10 15 10 15 10 15 10 15 10 15 10 15 10 15 10 10 10 10 10 10 10 10 10 10 | 35 | | |

Figure - 4 (A) Natarajan & Khalaf, (B) Lagergren and (C) Bhattachary & Venkobachar Plots

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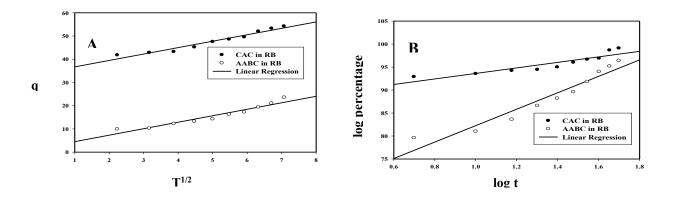


Figure - 5 (A) Intra-particle diffusion model (B) log Intra-particle diffusion Plot

3.3 Effect of dose

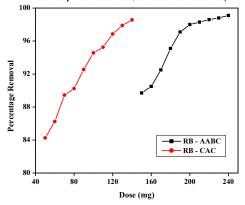
The effect of dose of adsorbent on the amount of RB adsorbed has been studied (Table 1). In this parameter while increasing the addition of the dosage of the adsorbents is enhancing the availability of surface active sites, which indicates the proper conglomeration of the adsorbent. The value of percentage removal of RB (Table 1) increase exponentially with increase in dose of adsorbent (Figure 6-A). This suggests that the adsorbed species (dye) may either block the access to the internal pores or causes particles to aggregate and there by resulting in the availability of active sites for adsorption [31].

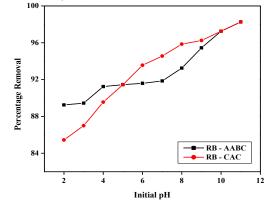
3.4 Effect of initial pH

The effect of initial pH of the dye solution on the amount of dye (RB) adsorbed has been studied by varying the initial pH, under constant conditions of other process parameters (Table 1). The optimum pH value is fixed as 7.5. The results are shown in (Figure 6-B). The increase in initial pH almost linearly increases the amount of RB adsorbed. The change in initial pH values of dye solution significantly affect the adsorption characteristics of basic dye indicating that removal of RB (basic dye) is enhanced by basic solution [32].

3.5 Effect of particle size

The amount of RB adsorbed increases linearly with the decrease in particle size of the adsorbent. This is due to the increase in available surface area with the decrease in particle size [33]. The effect of particle size of AABC and CAC with percentage removal is shown in (Figure 6-C). There exists a linear relationship between the amount adsorbed and particle size, as evidenced by the r-values close to unity.





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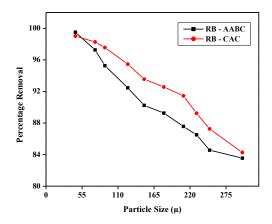


Figure - 6 (A) Effect of dose (B) Effect of initial p^H for the removal of RB onto CAC and AABC (C) Effect of
Particle size for the removal of RB onto CAC and AABC

4.0 Characterization Studies

4.1 Dye Solution (Adsorbate):

The adsorbate Rhodamine B dye (Chemical formula = $C_{28}H_{31}N_2O_3Cl$; C.I = 45170.1; MW = 442.56; FW = 536.10; class = basic dye; pH = 7.5 and λ_{max} = 544 nm; IUPAC Name is N-[9-(ortho-carboxyphenyl)-6-(diethylamino)-3H-xanthen-3-ylidene] diethyl ammonium chloride), which is a highly water soluble, supplied by BDH (India). It is found as a reddish violet powder, used as a colorant in textiles and food stuffs, and is also a well-known water tracer fluorescent [34].

$$(C_2H_5)_2N \\ \Theta \\ CI \\ HOOC$$

$$Rhodamine-B$$

Figure - 7 Structure of Rhodamine B dye

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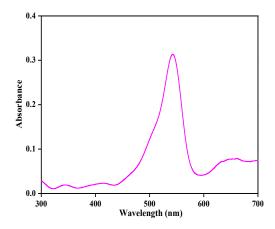


Figure - 8 UV-Visible spectrum of Rhodamine B

4.2 Physico-chemical Studies: Table - 4 Physico-chemical Studies properties of adsorbents

| S.No | Characteristic parameter | *CAC | AABC |
|------|--|--------|--------|
| 01 | Bulk density (gm ⁻²) | 0.9640 | 0.9458 |
| 02 | Moisture content (%) | 10.23 | 2.856 |
| 03 | Loss on ignition (%) | 95.0 | 58.14 |
| 04 | Residue after ignition (%) | 38.12 | 26.47 |
| 05 | pН | 7.5 | 7.5 |
| 06 | Electrical conductivity \times 10 ⁴ (ohm ⁻¹ cm ⁻¹) | 4.62 | 3.58 |
| 07 | Zero point charge (pH _{zpc}) | 7.75 | 5.78 |
| 08 | Surface area (m ² g ¹) | 875 | 775 |
| 09 | Decolourising power (mg g ⁻¹) | 12.57 | 10.45 |
| 10 | Phenol number \times 10 ³ (mg L ⁻¹) | 8.35 | 7.48 |

^{*}Data given by E. Merck, India are close to the experimental observations.

CAC has the high moisture content indicating that it is highly porous in nature. The low moisture content of low cost adsorbents is due to the method of preparation and activation. The range of bulk density (gm⁻²) and moisture content (%) of activated carbon is 0.9458 and 2.856 (AABC).

The moisture content does not affect the adsorption capacity / power directly, but it dilutes the adsorbent dose and therefore necessitates the use of additional quantity of adsorbent for the effluent treatment process [35]. The loss on ignition (%) and Residue after ignition (%) were observed to be in the range of 95.0 (CAC) – 58.14 (AABC) and 38.12 (CAC) – 26.47 (AABC) respectively. The prepared carbons are found to abstract protons from water and the resultant aqueous slurry or solution is found to be alkaline (basic) as evidenced from the pH value of CAC and AABC are 7.5 - 7.5 respectively. The electrical conductivity of CAC and AABC is $4.62 - 3.58 \times 10^4$ ohm⁻¹ cm⁻¹ respectively. The pH_{2pc} zero point charge of CAC is 7.75, while the range of pH_{2pc} for IPACs is 5.47 - 5.78. The pH_{2pc} values are noted to be in the acidic range. The surface area (mgg⁻¹) of CAC and activated carbon are 875 and 775 (AABC) respectively. The order of surface area is found to be

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AABC<CAC

The surface area is found to be linearly related to the range decolourising power (mgg⁻¹) of CAC and ACs are 12.57 and 10.45 (AABC) and phenol number (mgL⁻¹) of CAC and ACs are 8.35 and 7.48 (AABC respectively. The high value of phenol number makes all the low cost activated carbons as very good adsorbent for the removal of cationic dyes [37].

4.3 FT-IR spectra

The FT-IR Spectra of low cost carbons before adsorption and after adsorption of RB dye is shown in figure 9 . The characteristic group frequencies ($\bar{\upsilon}$, in cm⁻¹) observed for various surface functional groups. The surface functional groups are almost common as revealed by some common peaks, which appeared at 3400 – 3550, 2850 – 2950, 1635 – 1822, 1500 – 1600 and 1240 – 1504 cm⁻¹, depending upon the nature and type of raw materials. The characteristic peaks are slightly varying in their position for different IPACs. A peak ranges 2850 – 2950 cm⁻¹ in all the ACs confirm the C – H stretching. The peaks around 1800 cm⁻¹ in all the ACs confirm the presence of carbonyl group. This was further evidenced by another peak around 1631 – 1745 cm⁻¹, which reveals the keto nature. The peak around 3400 – 3558 cm⁻¹ confirm the presence of –OH group. The functional group may be –COOH or –OH. The nature of –CH group is revealed by another peak around 2804 – 2950 cm⁻¹. The IR peaks around 1200 – 1500 cm⁻¹ may be due to the stretching vibrations of lactone (γ and / or δ) =C–O, –CH= and –CH₂– functional groups . In dye loaded carbons, some of the peaks are shifted to 5-30 cm⁻¹ and some of them are disappeared indicating the involvement of the surface functional groups in binding the dye. The IR spectra indicates the presence of surface functional groups like>C=0, -CH, -COOH, -OH, etc., [38] and their involvement in the adsorption process.

Table -5 Characteristic frequency (in cm⁻¹) in FT-IR spectrum of ACs before and after adsorption of dyes.

| ne 3 Cha | uaciei | istic ii | equen | cy (III | CIII / II | 1 1 1 111 | . specu | uiii oi | ACS I | Jetore | anu ai | ter aust | ոքատո | or uy | <i>-</i> 50. |
|---------------|----------------|----------------|-----------------|---------------------------------|-----------------|-------------------|------------------------|----------|---------|----------|----------|----------|-------------------------------|-----------------------------------|-----------------------------|
| Materi als | O H str. | C- H Str | C= O str. | C H ₂ be nd | CO O str. | SO ₃ H | CH ₃ Def | o | N= H | C= N | N= N | =C= C | CH ₃ In plan | C· C H ₃ str. | plan e OH ben d |
| CAC | 34 30 | 28 90 | , | 29 48 | , | , | 139 0 | , | , | 13 80 | , | , | 107 8 | 98 0 | 632 |
| CAC - RB | 34 10 | 28 72 | 163 8 | , | , | | 1 | , | , | 13 82 | 1 | 124 3 | 107 3 | 1 | 640 |
| AABC | 34 49 | 29 32 | 162 8 | , | 152 8 | | 1 | 10 95 | , | , | 1 | , | 109 2 | 10 22 | 644 |
| AABC - RB | 34 60 | 29 24 | 172 0 | | , | 125 6 | 136 5 | , | 72 2 | 13 75 | 155 9 | - | 110 8 | 10 57 | 630 |

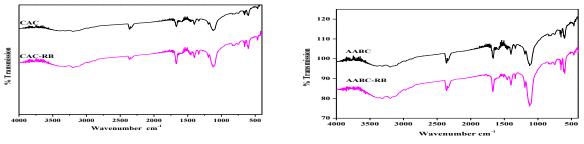


Figure - 9 FTIR spectrum of before and after adsorption of RB on CAC and AABC

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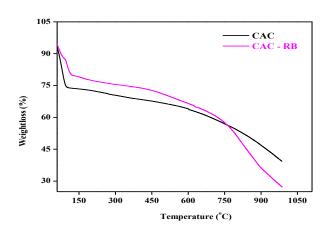
4..4 Thermo gravimetric studies

TGA studies have been made only with CAC and all ACs before and after adsorption of dyes. It has been found that the samples undergo two stages of thermal degradation in the temperature ranges of 110 -150°C and 650 -750°C, respectively. The peak temperature (T in °C) and weight loss (Δm , in %) for the two stages of degradation of the AC and AC - dye samples are listed in the below table.

Table - 6 Weight loss (∆m in %) for the two stages of degradation of AC and AC-Dye samples

| CAC/ACs - | Weight loss (%) | | | | | | | | |
|-----------|-----------------|----------|-------|--|--|--|--|--|--|
| Dye | I Stage | II Stage | Total | | | | | | |
| CAC | 14.67 | 16.64 | 31.31 | | | | | | |
| CAC -RB | 12.86 | 17.66 | 30.52 | | | | | | |
| AABC | 15.17 | 11.97 | 27.14 | | | | | | |
| AABC -RB | 13.97 | 12.48 | 26.45 | | | | | | |

About 60% of the total weight losses were observed in activated carbons with dye samples. It has been reported by E Merck, India that the loss on ignition (LOI) of CAC sample is 95% and hence the residue left after ignition of CAC at 600°C is approximately only 5%. The dye loading on CAC and ACs leads to increase in thermal stability of these ACs [39].



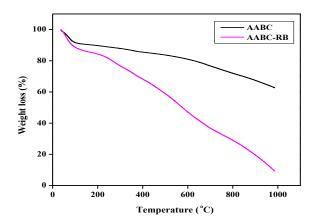


Figure -10 TGA spectrum of before and after adsorption of RB on CAC and AABC

SEM Studies

Scanning electron microscope (SEM) studies provide useful information regarding the textural morphological characteristics of the surface of the adsorbents. The SEM photo of all the IPACs before and after adsorption of RB was recorded. The SEM images of pure CAC and AABC (Figure.11-A&C) show the porosity of the structure. After dye adsorption, a significant change is observed in the surface morphology of these adsorbents (Figure. 11B & D) photographs clearly reveal the surface texture and porosity of the samples. SEM photographs also show that the particles can be roughly approximate as spheres or globules, if the roughness factor is included to account for their regularities. At higher magnification (*viz.*, 1 x 3000), the globules are distinct visible and they appear to be quite uniform with internal pores (porous structures) or holes of the adsorbents. SEM photographs also depict the porosity nature of the carbonaceous materials and the presence of grains in it. The adsorb dye molecules are either engulfed or surrounded by the porous adsorbent particle [40]. The colour of ACs was found to be black and blackish-gray and

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when view under powerful microscope, the presence of the spherical particles was also know the results of the SEM studies indicate that the dye molecules are adsorbed on surface of the adsorbents. Hence, it is concluded that all the prepared low cost carbons are employed as adsorbents for the removal of dyes from water.

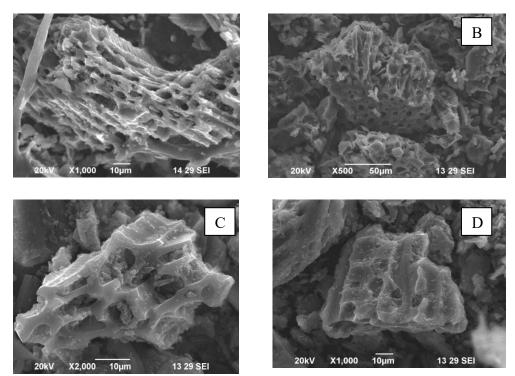


Figure - 11 SEM images of before (A, C) and after (B, D) adsorption of RB on CAC and AABC.

CONCLUSION

The conclusions derived from the present investigation are the percentage of removal of RB increased with decrease in initial concentration of dye, particle size of AABC and increases in contact time, dose of adsorbent and initial pH of the dye solution. Adsorption data obeyed Freundlich and Langmuir adsorption isotherms and first order kinetic equations. The intra-particle diffusion is one of the rate determining steps, and prepared AABC could be employed as adsorbent for the removal of dye / colour in general and Rhodamine B in particular.

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