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Adsorptive removal of acid orange 7 from industrial effluents using activated carbon and conducting polymer composite – A comparative study

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The feasibility of removing Acid Orange7 (AO7) from industrial effluents using two different adsorbents namely Activated Carbon (CAC) and Polypyrrole Polymer Composite (PPC) prepared from Casuarina wood has been analysed. Batch mode adsorption studies are performed in order to investigate the adsorption capacities of these adsorbents by varying initial dye concentration, agitation time, temperature and *p*H. Results reveal that adsorption capacity decreases from 83.4 to 60.5% for CAC and from 95.1 to 72.6% for PPC at 30°C with an increase in the initial concentration from 25 to 100 mg/L. A comparative study on the adsorptive capacities of CAC and PPC have been carried out and it was inferred that PPC had better adsorption capacity than CAC at an initial concentration of 50 mg/L at 30, 35 and 40°C. The calculated q_e values agree very well with experimental values. The regression coefficient values above 0.98 confirm that adsorption follows second order kinetics. The increase in Langmuir adsorption capacity (Q_0) from 7.8036 to 9.5082 mg/g for CAC and 8.1873 to 11.0087 mg/g on increasing the temperature from 30 to 40°C accounts for the endothermic nature of the process. The present study confirms the potentiality of an abundant low cost solid waste material and its availability for the removal of acidic dyes from industrial effluents

Keywords: Acid Orange, Activated carbon, Adsorbent, Polymer composite, Isotherm.

Textile industries ranks first in the usage of dyes when compared to other industries like food, paper, cosmetics and carpet industries[1]. Decolourization of textile effluents using conventional technologies is not effective due to their limitations[2]. Many physical and chemical methods such as adsorption, coagulation, precipitation and filtration have been used to remove harmful dyes from coloured waste water. Adsorption is the most effective and economical method for removal of dyes. Many researchers have proved several low cost materials such as pear millet husk carbon[3], *Aspergillus niger*[4], rice husk, banana pith, cotton waste, kaoline[5], coir pith[6], guava seeds[7], and neem saw dust[8], clay[9] and mango seed kernel[10] as suitable adsorbents for the removal of dyes.

Polymer composites or green composites are viable alternative for the existing waste water treatment technologies. One efficient way of increasing the adsorption capacity of saw dust is by the polymerization of monomer on the surface of saw dust. In recent years, conducting electro active polymers such as Polypyrrole has received great attention due to their electrical conductivity and electro activity[11-13].

In this study, Poly pyrrole saw dust composite was chemically prepared by polymerizing pyrrole on saw dust surface. Activated carbon was prepared by carbonization of Casuarina followed by chemical impregnation with H_3PO_4. A comparative study of the conducting polymer and activated carbon for the adsorption of acid orange 7 from textile wastewater was conducted by batch mode adsorption studies.

Experimental Section

Preparation of activated carbon

The seeds and branches of Casuarina wood were collected and cut into small pieces (2 cm), washed with distilled water and dried in sunlight for 10 days. The dried material was soaked in a boiling solution of 40% H_3PO_4 for 1 h and kept at room temperature for 24 h. After 24 h, the wood material was separated, air dried and carbonized in muffle furnace at 400°C. The carbonized material was then powdered and activated in a muffle furnace at 800°C for a period of 10 min. After activation, the material was repeatedly washed with plenty of distilled water. The characteristics of CAC and PPC are analyzed as per the standard procedures[14,15] and are given in Table 1.

Preparation of poly pyrrole composite

Saw dust prepared from Casuarina wood was used for the preparation of PPC. The saw dust was first washed with distilled water in order to remove the impurities and finally dried at 60°C for 2 h. In order to prepare polymer coated saw dust, 5.0 g of saw dust was immersed in 50 mL of 0.20 M freshly distilled pyrrole solution for 12 h before polymerization. 50 mL of 0.5 M $FeCl_3$ as the oxidant solution was added into the mixture gradually and the reaction was allowed to continue for 4 h at room temperature[11]. The polymer coated saw dust was filtered, washed with distilled water, dried in an oven at 55-60°C and sieved before use[16].

Preparation of dye solution

Acid Orange 7 (M.wt: 350.33, Mol.Formula: $C_{16}H_{11}N_2NaO_4S$, λ max: 482 nm) used in this study is of commercial quality and used without further purification. Different initial concentrations were prepared by diluting the stock solution using double distilled water as solvent. Dye concentration was analyzed by measuring the absorbance values with UV-VIS spectrophotometer (Model: JASCO V-570). The *p*H measurements were made using *p*H meter (Model LI 610 ELICO). The *p*H adjustments of the solution were made by 0.1M HCl or 0.1M NaOH. The chemicals were of analar grade and all the adsorption experiments were carried out at room temperature (27±2°C).

Characterization studies

Physico-chemical characteristics of PPC and CAC were studied as per the standard testing methods[17,18]. Scanning electron microscopy (SEM) was used to visualize the morphological features and surface characteristics of CAC and PPC as shown in Fig. 1.

Batch mode adsorption experiments

The batch technique was selected because of its simplicity. The experiments were carried out in a mechanical shaker (KHAN shaker - KEMI make) working at a speed of 150 rpm. Dye solutions (50 mL) of desired concentrations and initial *p*H values were used. Blank samples were run under similar experimental conditions without using adsorbents. After shaking, the adsorbents were separated by centrifugation and the supernatant solutions were estimated by measuring absorbance at maximum wavelengths using UV-visible spectrophotometer (Model: JASCO V-570) at the desired wave length. The effect of each parameter like adsorbent dose, adsorbent particle size, different dye concentrations and agitation time was studied by fixing the values of other parameters[19]. Amount of dye adsorbed by the CAC and PPC were calculated using the following equation:

$$qe = \frac{(C_o - C_e)}{w} v \qquad \ldots. (1)$$

where q_e(mg/g) is the amount of dye adsorbed at equilibrium onto CAC and PPC; C_o and C_e(mg/L), the initial and equilibrium liquid-phase concentrations of dye; V(L), the initial volume of dye solution; and W(g), the weight of CAC and PPC .The adsorbents CAC and PPC of 2g were added to each flask and then the flasks were sealed to prevent any change in volume during the experiments. It was agitated for predetermined time intervals at room temperature in the mechanical shaker.

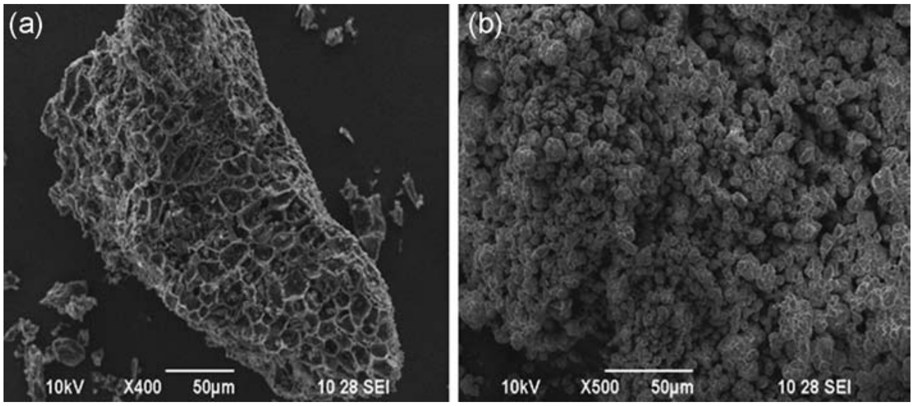

Fig. 1 — SEM image of (a) CAC and (b) PPC

Results and Discussion

Analysis of adsorbent characteristics

The physico-chemical characteristics of CAC and PPC prepared from Casuarina are summarized in Table 1.

Analysis of adsorption parameters

Effect of initial dye concentration

Initial dye concentration of AO7 ranging from 25 mg/L to 100 mg/L was prepared and adsorption experiments were conducted using 2g of CAC and PPC. When the concentration of dye increases, the limited capacity of the adsorbent checks any further adsorption of dye. Hence, the overall removal percentage decreases[20] since the adsorbent has limited number of active sites which becomes saturated at certain concentration. The equilibrium controls the maximum adsorption and decreases the final removal percentage.

The amount of AO7 dye removal by CAC is less than PPC. This might be due to the heterogeneity obtained by the presence of functional groups present on the surface of the polymer composites[21]. From the literature[16] it was suggested that the dye removal was high due to the ion exchange mechanism between the oppositely charged functionalities originating from monomer and the ionic dye molecules.

Effect of contact time

The effect of contact time was studied by agitating 2 g of CAC and PPC separately with 50 mg/L of AO7 solution. The adsorption efficiency is found to increase with increase in time. CAC shows 53.5% removal of AO7 whereas PPC shows 77.8% removal of AO7 within 50 min. Both the adsorbents attain equilibrium at 90 min and hence it is selected as the equilibrium time for further studies. In the process of adsorption, initially, the dye molecules have to first encounter the boundary layer effect and then it has to diffuse from boundary layer film onto adsorbent surface. Finally, it has to diffuse into the porous structure of the adsorbent. This phenomenon will take relatively longer contact time[22].

Table 1 — Physico-chemical characteristics of CAC and PPC.

S.No	Properties	CAC	PPC
1	*p*H	6.87	7.90
2	Moisture content, %	16.30	6.25
3	Conductivity, µS/cm	0.12	5.73
4	Volatile matter, %	5.7	14.6
5	Methylene blue Number, mg/g	525	43
6	Iodine Number, mg/g	1186.9	96

Effect of pH

Adsorption process for the treatment of dye containing waste water is *p*H dependent. The effect of *p*H on the dye removal efficiency of CAC and PPC was studied at varied *p*H range of 2-10. The maximum percentage removal of AO7 occurs at acidic *p*H 2.8 and adsorption decreases with increase in *p*H. It is known that, the ionic dyes, upon dissolution, releases colored dye anions/cations into solution. The adsorption of these charged dye groups onto the adsorbent surface is primarily influenced by the surface charge on the adsorbent, which, in turn, is influenced by the *p*H[23] of the solution˙ Similar results have been reported for adsorption of Eosin Y using conducting electro polymers[24].

Effect of temperature

The effect of temperature on dye adsorption was studied at 30, 35 and 40°C. The results indicated that the amount of dye adsorbed at equilibrium increases with increasing temperature. This is due to the mobility of the dye molecules with increase in temperature[25]. The percentage removal of AO7 increased from 38.5 to 90.7% for CAC and from 42.6 to 98.4% for PPC at 30°C indicating that the adsorption is an endothermic process.

Adsorption kinetics

In order to investigate the mechanism of adsorption, various kinetic studies like pseudo first order, pseudo second order and intra-particle diffusion models were analyzed.

Pseudo first order kinetic model

The pseudo first order model of Lagergren[26] is based on the assumption that the rate of change of adsorbed solute with time is proportional to the difference in equilibrium adsorption capacity and the adsorbed amount. The pseudo-first order equation[27] is expressed as follows:

$$\frac{dq}{dt} = k_1(q_e - q_t) \qquad \ldots. (2)$$

The integrated form of equation is expressed as follows:

$$\log\,(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \qquad \ldots. (3)$$

where q_e (mg/g) and q_t (mg/g) are the adsorption capacity per unit weight of adsorbent at equilibrium and at time t (min) respectively. k_1 is the pseudo-first order rate constant. Linear plot of log $(q_e - q_t)$ *versus t* gives the value of rate constant k_1. The values of first order rate constant k_1 and q_e were calculated from the intercepts and slopes of the plot of log ($q_e - q_t$) *versus* t and the results are summarized in Tables 2(a) and 2(b) for CAC and PPC respectively. The correlation coefficients are low for both CAC and PPC and it is found that the pseudo-first order equation does not fit well with whole range of adsorption process, as it is applicable for the initial stages of adsorption processes[28] and after that it starts deviating from the theory. This infers that the adsorption of AO7 onto CAC and PPC does not follow first order mechanism.

Pseudo second order kinetic model

The pseudo second order model[29] is based

on the assumption that the rate–limiting step involves chemisorption. The dye adsorption described by a modified second order equation is expressed as follows:

$$\frac{t}{q_t}=\frac{1}{k_2qe^2}+\frac{1}{q_e} \qquad \text{.... (4)}$$

where k_2 is the pseudo second order rate constant (g/mg/min). The value of k_2 was found to decrease with increase in dye concentration due to decrease in available vacant sites for adsorption. The values of second order rate constant k_2 and q_e (Tables 2 (a) and (b)) were calculated from the intercepts and slopes of the plot of t/q_t *versus* t as shown in Figs. 2 (a) and 2(b) for CAC and PPC respectively. Based on the values of the correlation co-efficient which is greater than 0.98, the second order kinetic model is found to be more suitable to describe the adsorption process than Pseudo-first order model.

Intra-particle diffusion study

In a rapidly stirred batch reactor, the transport of adsorbent species takes place from the bulk of the solution into solid phase through intra particle diffusion process[30]. The expression for the intra-particle diffusion model is given by the following equation:

$$q_{t=k_{dif}}t^{1/2}+C \qquad \text{.... (5)}$$

where C_e (mg/g) is the intercept; and K_{dif} is the intra-particle diffusion rate constant (mg/g/min). The values of q_t are found linearly correlating with values of $t^{1/2}$ for both the adsorbents and the rate constant K_{diff} was directly evaluated from the slope of the regression line. The values of intercept C provide

Table 2(a) — Kinetic model values for adsorption of AO7 onto CAC

Conc mg/L	First order kinetics			Second order kinetics		
	k_1×10^{-2} /min	$q_{\text{e mg/g}}$	r^2	k_2× 10^{-3} g/mg/min	$q_{\text{e mg/g}}$	r^2
25	4.83	0.4764	0.9659	4.3	7.5463	0.9905
50	2.66	0.8657	0.9809	4.4	7.0264	0.9949
75	1.38	1.6736	0.9885	5.5	5.9186	0.9959
100	7.80	2.9436	0.9269	12.1	4.4487	0.9989

Table 2(b) — Kinetic model values for adsorption of AO7 onto PPC

Conc mg/L	First order kinetics			Second order kinetics		
	k_1×10$^{-2/\text{min}}$	$q_{\text{emg/g}}$	r^2	k_2× 10^{-3} g/mg /min	$q_{\text{e mg/g}}$	r^2
25	3.22	0.7153	0.9749	4.8	7.4752	0.9915
50	3.27	0.7035	0.9806	4.9	7.0834	0.9939
75	1.54	1.4961	0.9857	6.3	5.9826	0.9959
100	7.60	3.0188	0.9167	13.9	4.4159	0.9912

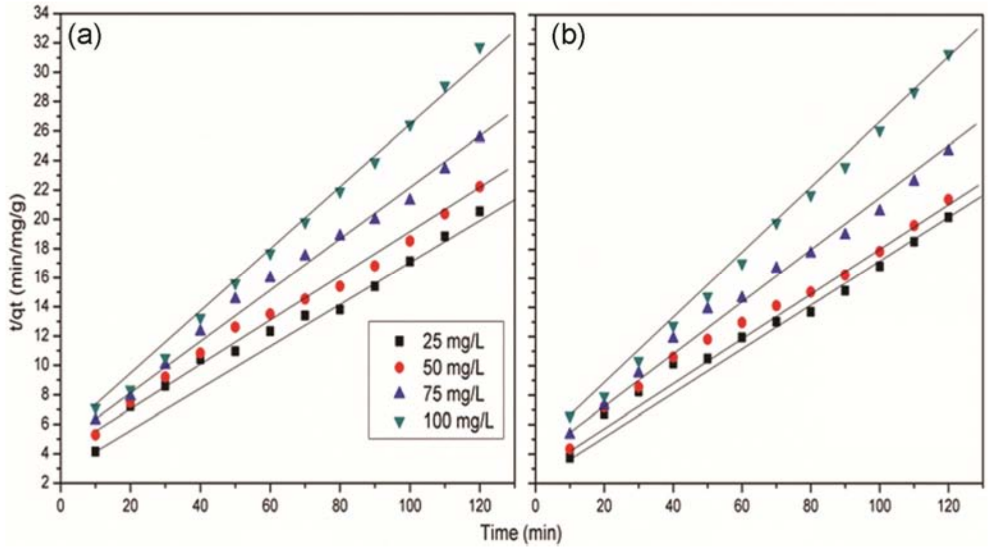

Fig. 2 — Pseudo Second-order plot for adsorption of AO7 onto (a) CAC and (b) PPC

information about the thickness of the boundary layer. The resistance to the external mass transfer increases as the intercept increases. The results are shown in Table 3.

If the intra-particle diffusion is involved in the adsorption process, then the plot of q_t *versus* square root of time would result in a linear relationship and the intra-particle diffusion would be the controlling step if this line passes through the origin. When the plots do not pass through the origin, it indicates some degree of boundary layer control and it further shows that the intra-particle diffusion is not the only rate controlling step, but also other processes may control the rate of adsorption. Such plots may present multi-linearity, indicating that two or more steps take place.

It is evident from Figs 3(a) and 3(b) that initially the diffusion of adsorbate takes place through the solution to the external surface of adsorbent. Gradually the intra- particle diffusion takes place and at the final equilibrium stage intra-particle diffusion starts to slow down due to extremely low adsorbate concentrations in the solution. Figure 3(a) and 3(b) also indicates that the plot of q_t versus $t^{1/2}$ does not pass through the origin. The correlation coefficient values from Table 3. indicate that pore diffusion[31] plays a major role for the adsorption of AO7 onto CAC and PPC.

Adsorption isotherm studies

Adsorption isotherm indicates the relationship between the adsorbate in the liquid phase and the adsorbate on the surface of the adsorbent at equilibrium constant temperatures[32]. The applicability of the isotherm equation is compared by judging the correlation coefficient (r^2). Langmuir[33], Freundlich[34] and Dubnin–Radushkevich (D–R) isotherm models were used to describe the adsorption of AO7 onto CAC and PPC.

Langmuir isotherm

Langmuir isotherm is used to determine the maximum capacity of the adsorbent.

The Langmuir equation can be written as follows:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b_L} + \frac{C_e}{Q_0} \qquad \text{.... (6)}$$

where Ce is the equilibrium concentration (mg/L), q_e is the amount of dye adsorbed at equilibrium (mg/g) and Q_0 (mg/g) and b_L (L/mg) are Langmuir constants related to adsorption capacity and energy of adsorption respectively. The Langmuir isotherm is based on the assumption of structurally homogeneous adsorbent and monolayer coverage with no interaction between the sorbate molecules. Once a dye molecule occupies a site, no further adsorption can take place at that site[35]. The values of Q_0 and b_L for CAC and PPC are calculated from the slopes and intercepts of the linear plots of C_e / q_e and the results are summarized in Table 4.

The values of adsorption efficiency Q_0 and adsorption energy b_L increases with increasing the

Table 3 — Intra particle diffusion values for the adsorption of AO7 onto CAC and PPC

Conc mg/L	CAC			PPC		
	k_{diff} mg/g/min	C	r^2	k_{diff} mg/g/min	C	r^2
25	1.0101	0.7991	0.9772	0.9732	1.0821	0.9862
50	0.9553	0.6097	0.9836	0.9329	0.8948	0.9865
75	0.7849	0.6776	0.9875	0.7735	0.9261	0.9962
100	0.5496	1.0957	0.9811	0.5256	1.2668	0.9923

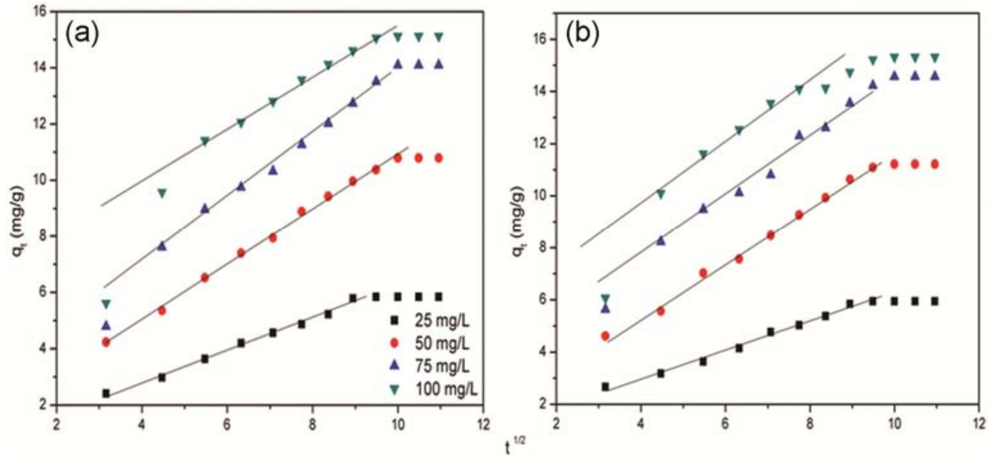

Fig. 3 — Intra particle diffusion plot for the adsorption of AO7 onto (a) CAC and (b) PPC

temperature suggests that the maximum adsorption corresponds to a saturated monolayer of dye molecules on all the adsorbents. Further it confirms the endothermic nature of processes involved in the system[36].

The essential characteristics of Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter R_L[37] which could be defined by the following equation:

$$R_L = \frac{1}{(1+bC_0)} \quad \text{.... (7)}$$

where C_0 is the highest initial solute concentration. R_L value indicates the type of adsorption isotherm either to be unfavorable ($R_L > 1$), favorable ($R_L = 1$) or irreversible ($R_L = 0$). Langmuir model is more appropriate to explain the nature of adsorption of AO7 with correlation coefficient of 0.9834 to 0.9909.

Freundlich isotherm

The Freundlich equation is an empirical relationship describing the sorption of solutes from a liquid to a solid surface. Linear form of Freundlich equation is expressed as follows:

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \quad \text{.... (8)}$$

where K_f is a constant for the system, related to the bonding energy. K_f can be defined as the distribution coefficient and represents the quantity of dye adsorbed onto adsorbent for unit equilibrium concentration. $1/n$ indicates the adsorption intensity of dye onto the adsorbent or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. A value for 1/n below 1 indicates a normal Freundlich isotherm while 1/n above 1 is indicative of cooperative adsorption.

The data obtained by plotting log q_e *Vs* log C_e from linear Freundlich isotherm for the adsorption of AO7 onto CAC and PPC is presented in Table 4(a) and 4(b) respectively. The correlation coefficient shows that the Freundlich model is comparable to Langmuir model. The value of 1/n lesser than 1 (Tables 4(a) and 4(b)) indicate that the adsorption of AO7 onto CAC and PPC is favourable.

Dubnin-Radushkevich (D-R) isotherm

The isotherm proposed by Dubnin and Radushkevich is useful in estimating the mean free energy and the energy of activation. From the energy of activation it can be predicted whether an adsorption is physisorption or chemisorption. The D-R model equation Eq. (9) is shown below:

$$q_e = q_D . e^{-B\varepsilon^2} \quad \text{.... (9)}$$

where, B is a constant related to the adsorption energy; q_D, the theoretical saturation capacity; ε , the Polanyi potential, which is calculated from the following equation:

$$\varepsilon = RT \ln\left(1 + {}^{1}\!/_{C_e}\right) \quad \text{.... (10)}$$

The plot of ln q_e versus ε^2 gives the slope B [$mol^2 KJ^{-2}$)] and the intercept which yields the adsorption capacity, Q_D (mg/g). The mean free energy of adsorption (E), defined as the free energy change, when one mole of ion is transferred from infinity in solution to the surface of the solid, was calculated from the B value using the following relationship:

$$E = 1/(2B)^{1/2} \quad \text{.... (11)}$$

The calculated value of D-R parameters is given in Table 5. If the energy of activation is less than 8

Table 4(a) — Isotherm parameters of AO7 onto CAC

Temp, °C	Isotherm models					
	Langmuir			Freundlich		
	Q_0 mg/g	b_L L/mg	r^2	K_f mg/g	r^2	1/n
30	7.8036	0.4517	0.9834	3.0510	0.6771	0.4032
35	8.9819	0.1103	0.9894	3.2639	0.7759	0.3863
40	9.5082	0.0932	0.9909	3.5473	0.8691	0.3781

Table 4(b) — Isotherm parameters of AO7 onto PPC

Temp, °C	Isotherm models					
	Langmuir			Freundlich		
	Q_0 mg/g	b_L L/mg	r^2	K_f mg/g	r^2	1/n
30	8.1873	3.3519	0.9863	3.50436	0.7679	0.3772
35	9.5445	0.0979	0.9916	3.79672	0.8790	0.3646
40	11.0087	0.0687	0.9958	4.45055	0.9031	0.3425

Table 5 — D–R isotherm values for the adsorption of AO7 onto CAC and PPC

Temp, °C	CAC				PPC			
	Q_D mg/g	B×10^{-4} mol^2 KJ^{-2}	E KJ/mol	r^2	Q_D, mg/g	B ×10^{-4} mol^2 KJ^{-2}	E KJ/mol	r^2
30	10.5034	9.9	22.48	0.9868	10.5974	8.0	25.00	0.9896
35	10.6168	8.9	23.69	0.9862	10.5908	7.5	25.82	0.9866
40	10.4283	7.3	26.17	0.9825	10.4449	5.8	29.36	0.9835

KJ/mol, the adsorption is physisorption and if it is greater than 16 KJ/mol, the adsorption is chemisorption in nature[38]. The values of E, calculated using Eq. (12) are 22.48 to 26.17 KJ/mol for CAC and 25.0 to 29.36 KJ/mol for PPC, respectively. This indicates that the adsorption of AO7 onto CAC and PPC is chemisorption in nature.

Conclusion

(i) The present study reveals that CAC and PPC prepared from the seeds and wood of casuarina are used effectively as adsorbents for the removal of AO7 from aqueous solution.

(ii) The amount of dye adsorbed is found to vary with initial *p*H, temperature and contact time. Maximum adsorption occurs at *p*H 2-3 for AO7.

(iii) Kinetic studies predict that the adsorption of AO7 onto CAC and PPC follows pseudo second-order kinetics.

(iv) The adsorption isotherms like Freundlich, Langmuir and Dubnin-Radushkevich were analyzed for the adsorption of AO7 onto CAC and PPC. D-R isotherm predicts that the adsorption is chemisorption in nature. The equilibrium data fits very well into the Langmuir isotherm model which indicates monolayer adsorption. Freundlich isotherm shows poor fit into the isothermal data.

(v) The adsorption capacity of CAC and PPC increased with rise in temperature indicating endothermic nature of adsorption.

(vi) Intra-particle diffusion model predicts that pore diffusion plays a major role for the adsorption of AO7 onto CAC and PPC.

On comparing the results, it is obvious that, for the adsorption of AO7, PPC is an efficient, economic and alternative biomaterial than CAC.

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