

Removal of disperse red 9 from textile wastewater using acid activated carbon and polypyrrole nano composite – A comparative study

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Abstract

The present study analyses the feasibility of removing Disperse red 9 (DR9) from industrial effluents using two different adsorbents namely Phosphoric acid impregnated Activated Carbon (PAC) and Polypyrrole Polymer Composite (PPC) prepared from Casuarina wood. Batch mode adsorption studies were performed in order to investigate the adsorption capacities of these adsorbents by varying initial dye concentration, agitation time, temperature and pH. A comparative study on the adsorptive capacities of PAC and PPC were carried out and it was inferred that PPC had better adsorption capacity than PAC at an initial concentration of 50 mg/L at 30⁰C, 35⁰C & 40⁰C. 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 7.48 to 9.41 mg/g for PAC and 8.65 to 11.73 mg/g for PPC on increasing the temperature from 30⁰ to 40⁰C accounts for the endothermic nature of the process.

Keywords: Activated carbon, Adsorbent, phosphoric acid, Polypyrrole composite, Disperse Red, regression coefficient, kinetics, Langmuir.

1. Introduction

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], coffee husks [7], 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 (PPC) was chemically prepared by polymerizing pyrrole on saw dust surface. Activated carbon (PAC) 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 DR 9 from textile wastewater was conducted by batch mode adsorption studies. This study also evaluates the potential of the polymer composite prepared from *Casuarina* saw dust. The study also includes an evaluation of the effects of various operational parameters such as initial dye concentration, contact time, pH and temperature on the dye removal process. The adsorption kinetic models, equilibrium isotherm models related to adsorption processes were also performed and reported.

2. Materials and Methods

2.1 Preparation of Activated Carbon (PAC)

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 hour and kept at room temperature for 24 hours. After 24 hours, 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 minutes. After activation, the material was repeatedly washed with plenty of distilled water. The characteristics of PAC and PPC are analyzed as per the standard procedures [14] and are given in Table 1.

2.2 Preparation of Poly pyrrole composite (PPC)

Saw dust prepared from *casuarina* was used for the preparation of polymer composite. The saw dust was first washed with distilled water in order to remove the impurities and finally dried at 333 K for 2 hours. In order to prepare polymer coated saw dust, 5.0 g of saw dust was immersed in 50 ml of 0.20M freshly distilled pyrrole solution for 12 hours before polymerization. 50 ml of 0.5 M FeCl₃ as the oxidant solution was added into the mixture gradually and the reaction was allowed to continue for 4 h at room temperature [15]. The polymer coated saw dust was filtered, washed with distilled water, dried in an oven at 55-60° C and sieved before use [16]. The characteristics of PPC were studied as per the standard procedures [17] and the surface morphology was analyzed by Scanning Electron Microscope images.

2.3 Preparation of dye solution

Disperse Red 9 (M.Wt: 237.25, Mol.Formula: C₁₅H₁₁NO₂, λ max: 503 nm) used in this study is of commercial quality and used without further purification. A stock solution of 1000 mg/L of DR 9 dye was prepared by dissolving appropriate amount of dye in one litre of distilled water. The experimental solutions were obtained by diluting the dye stock solutions in accurate proportions to different initial concentrations. The concentration of the dye was determined using Elico make uv-vis spectrophotometer at wavelength 503 nm. The structure of DR 9 dye is shown in fig.1.

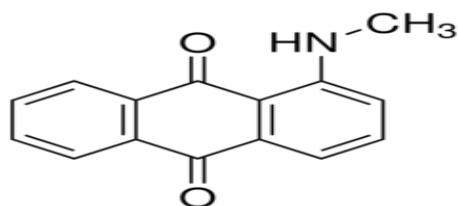


Fig.1. Structure of Disperse Red 9

2.4 Characterization Studies

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

2.5 Batch mode adsorption studies

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 150rpm. Adsorption experiments were conducted at room temperature by agitating 0.20 gm of adsorbent with 50 ml of dye solutions of 25 to 100 mg/L concentrations in 250 ml stoppered flask. 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. Blank samples were run under similar experimental conditions without using adsorbent. After shaking, the adsorbent was separated by centrifugation and the supernatant solutions was analyzed by measuring absorbance at maximum wavelengths using UV-Visible spectrophotometer (Model: JASCO V) at the wave length of 503 nm.

The mixture was withdrawn at specified intervals, centrifuged using electrical centrifuge (universal make) at 5000 rpm for 20 minutes and the unadsorbed supernatant liquid was analyzed for the residual dye concentration using Elico make uv-vis spectrophotometer at wavelength 503 nm. The effect of solution pH on the equilibrium adsorption of dyes (50 mg/L) was investigated using dilute HCl and NaOH solutions between pH 2 to 10. The effect of temperature on equilibrium adsorption was studied at 303, 308 and 313 K. 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]. To correct any adsorption of dye in container walls, control experiments were carried out in duplicate. There was no adsorption by the container walls. The amount of dye adsorbed by PAC and PPC were calculated using the following equation:

$$q_e = \frac{(c_0 - c_e)}{w} v \quad \dots\dots \quad (1)$$

Where q_e (mg/g) is the amount of dye adsorbed at equilibrium onto PPY; C_0 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 PAC and PPC.

3. Results and discussion

3.1 Characterization Studies

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

Table 1. Physico-chemical characteristics of PAC and PPC.

S.No	Properties	PAC	PPC
1	pH	6.87	7.90
2	Moisture content, %	16.30	6.25
3	Conductivity, $\mu\text{S}/\text{cm}$	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

Physico-chemical characteristics of PAC and PPC were studied as per the standard testing methods. In order to characterize the surface structure and morphology of PAC and PPC, SEM analysis was carried out using Scanning Electron Microscope as shown in fig.2.

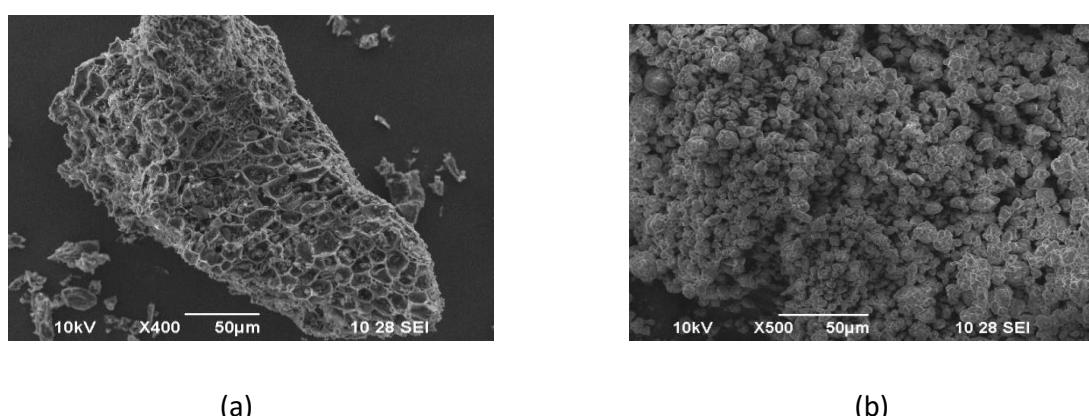


Fig.2. SEM image of (a) PAC and (b) PPC

3.2 Effect of initial dye concentration and agitation time

The rate of adsorption is a function of the initial dye concentration and contact time which is an important factor for effective adsorption. Initial dye concentration of DR 9 ranging from 10 mgL^{-1} to 120 mgL^{-1} was prepared and adsorption experiments were conducted using 2g

of PAC and PPC. The effect of time on the removal of DR9 by PAC and PPC is shown in fig.3 and this reveals that as the concentration of PAC and PPC increases the percentage removal decreases. As expected when the concentration of dye increases, the limited capacity of the adsorbent checks any further adsorption of dye. Hence, the overall percentage removal decreases [20].

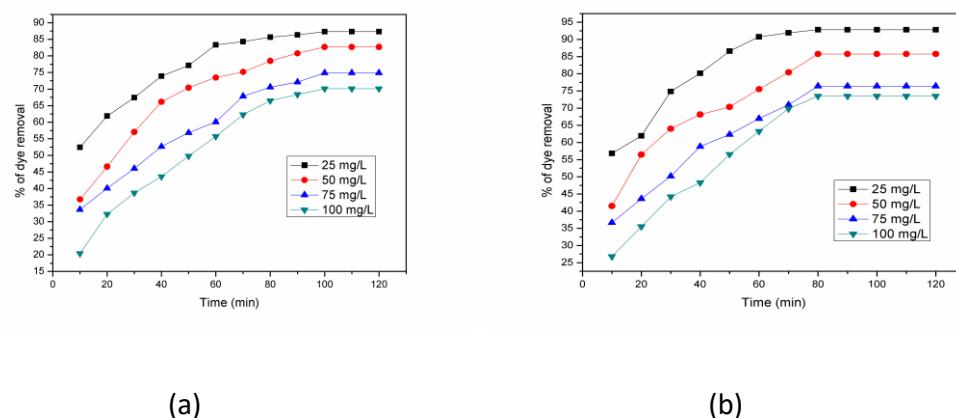


Fig.3 Effect of initial dye concentration on the removal of DR9 by a) PAC b) PPC

3.3 Effect of pH

The pH of the dye solution has been recognized as one of the most important factors influencing the adsorption process. Adsorption process for the treatment of dye containing waste water is *pH* dependent. To determine the effect of *pH* on the removal of dye, its adsorption was studied at varied *pH* range of 2 – 10. Figure 4 shows that the maximum percentage removal of DR 9 occurs at acidic *pH* 2 and adsorption decreases with increase in *pH*. In acidic medium, the surface of the adsorbent is positively charged due to higher concentration of H⁺ ions, so the electrostatic attraction between the adsorbents and dye is enhanced. But in alkaline conditions, electrostatic repulsion occurs resulting in decreased adsorption.

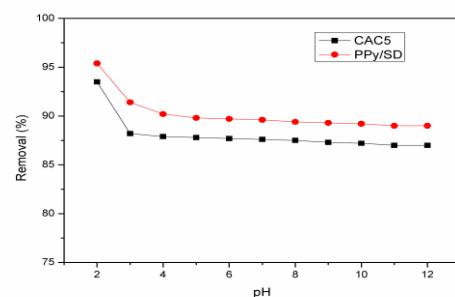


Fig.4 Effect of pH on the percentage removal of DR9 by PAC and PPC

3.4 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 as shown in fig.5. This is due to the mobility of the dye molecules with increase in temperature (Khashayar Badii et al., 2010). It has been observed that the percentage removal of DR 9 increases with increasing temperature indicating that the adsorption is endothermic in nature [21]. These adsorption data were further analyzed with adsorption isotherm models to find out the suitable model.

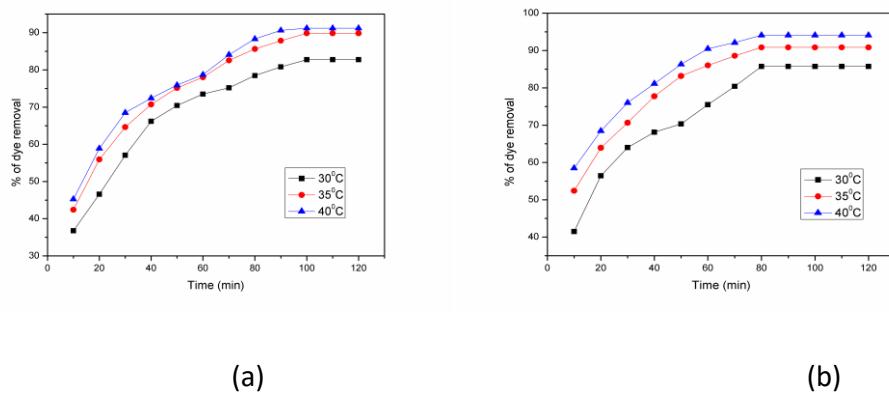


Fig.5 Effect of temperature on the percentage removal of DR9 by a) PAC b) PPC

3.5 Equilibrium Adsorption Isotherm

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. The applicability of the isotherm equation is compared by judging the correlation coefficient (r^2).

3.5.1 Langmuir isotherm

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

The Langmuir equation can be written as follows:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b_L} + \frac{C_e}{Q_0} \quad \dots \dots (2)$$

Where C_e 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 [22]. The Langmuir plot of " C_e/q_e " vs " C_e " for the adsorption of DR9 onto PAC and PPC at different temperatures (30° , 35° and 40°C) are shown in fig.6. The values of Q_0 and b_L for PAC 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 2.

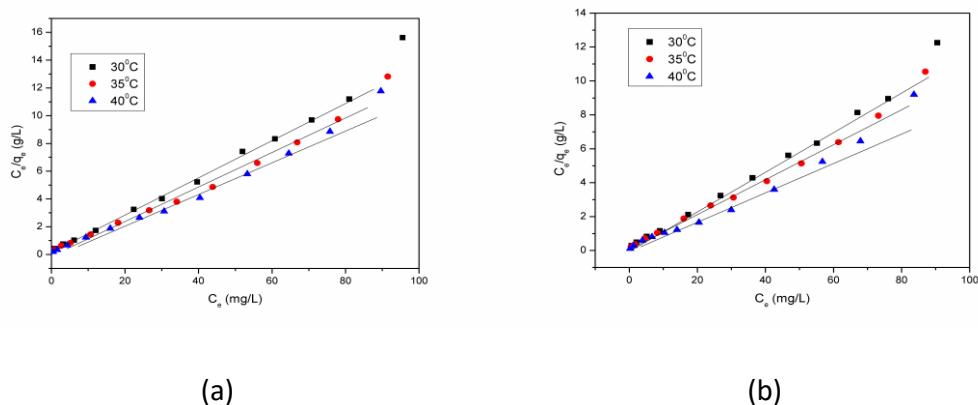


Fig.6 Langmuir isotherm plot for the adsorption of DR9 onto a) PAC b) PPC

The values of adsorption efficiency Q_0 and adsorption energy b_L increases with increasing the 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 [23].

The essential characteristics of Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter R_L [25] which could be defined by the following equation:

$$R_L = \frac{1}{(1+bc_0)} \quad \dots \quad (3)$$

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 ($R_L = 0$).

Table 2. Results of isotherm plots for adsorption of DR9 onto PAC and PPC

Parameters	Temperature (°C)					
	PAC			PPC		
	30	35	40	30	35	40
Langmuir Isotherm model						
Q ₀ (mg/g)	7.48	8.32	9.41	8.65	10.16	11.73
b _L (L/mg)	0.356	0.426	0.452	0.385	0.405	0.507
r ²	0.9988	0.9977	0.9971	0.9991	0.9992	0.9942
Freundlich Isotherm model						
n	3.12	3.46	3.18	3.14	3.31	3.82
k _f (mg ^{1-1/n} L ^{1/n} g ⁻¹)	2.75	2.91	3.54	3.23	3.45	4.04
r ²	0.8434	0.7395	0.907	0.8497	0.9017	0.9184
Dubinin - Raduskevich Isotherm model						
q _D (mg/g)	8.05	9.28	9.46	8.98	9.62	11.82
E (KJ/mol)	25.65	27.52	29.36	26.92	27.92	29.54
r ²	0.9718	0.9958	0.9777	0.9932	0.9808	0.9811
Kx10 ⁻⁴ (mol ² /J ²)	7.6	6.6	5.8	6.9	6.7	5.4

3.5.2 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:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad \dots \quad (4)$$

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. The adsorption intensity of dye (n) onto the adsorbent or surface heterogeneity, becomes more heterogeneous as its value gets closer to zero. The data obtained by plotting log q_e Vs log C_e from linear Freundlich isotherm for the adsorption of DR 9 onto PAC and PPC is presented in Table 2 respectively. The correlation coefficient shows that the Freundlich model is not comparable to Langmuir model.

3.5.3 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:

$$q_e = q_D \cdot e^{-B\varepsilon^2} \quad \dots \quad (5)$$

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:

$$\varepsilon = RT \ln(1 + 1/C_e) \quad \dots \quad (6)$$

The plot of $\ln q_e$ versus ε^2 gives the slope B [mol²KJ⁻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:

$$E = 1/(2B)^{1/2} \quad \dots \quad (7)$$

The calculated value of D-R parameters is given in Table 2. If the energy of activation is less than 8 kJ mol⁻¹, the adsorption is physisorption and if it is greater than 16 kJ mol⁻¹, the adsorption is chemisorption in nature. The values of E, calculated using Eq. (12) are 25.65 to 29.36 KJ mol⁻¹ for PAC and 26.92 to 29.54 KJ mol⁻¹ for PPC. This indicates that the adsorption of DR 9 onto PAC and PPC is chemisorption in nature.

3.6 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.

3.6.1 Pseudo first order kinetic model

The pseudo first order model of Lagergren [24] 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 [25] is expressed as follows:

$$\frac{dq}{dt} = k_1(q_e - q_t) \quad \dots \quad (8)$$

The integrated form of equation is expressed as follows:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad \dots \quad (9)$$

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 table 3 for PAC and PPC respectively. The correlation coefficients are low for both PAC 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 [26] and after that it starts deviating from the theory. This infers that the adsorption of DR 9 onto PAC and PPC does not follow first order mechanism.

3.6.2 Pseudo second order kinetic model

The pseudo second order model [27] 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:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \quad \dots \quad (10)$$

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 (Table 3) were calculated from the intercepts and slopes of the plot of t/q_t versus t as shown in fig.7 for PAC 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.

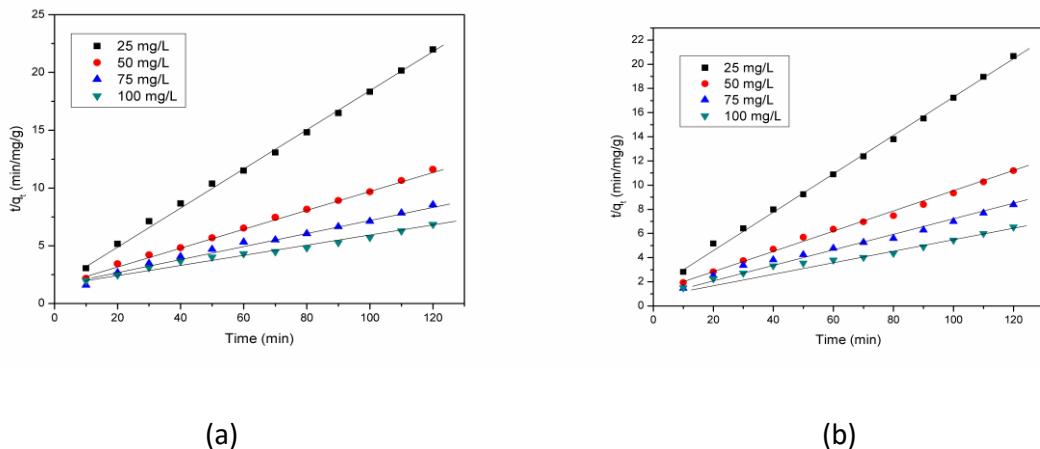


Fig.7 Pseudo second order plot for the adsorption of DR9 by a) PAC b) PPC

3.6.3 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 [28]. The expression for the intra-particle diffusion model is given by the following equation:

$$q_t = k_{dif} t^{1/2} + C \quad \dots (11)$$

Where C (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 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.

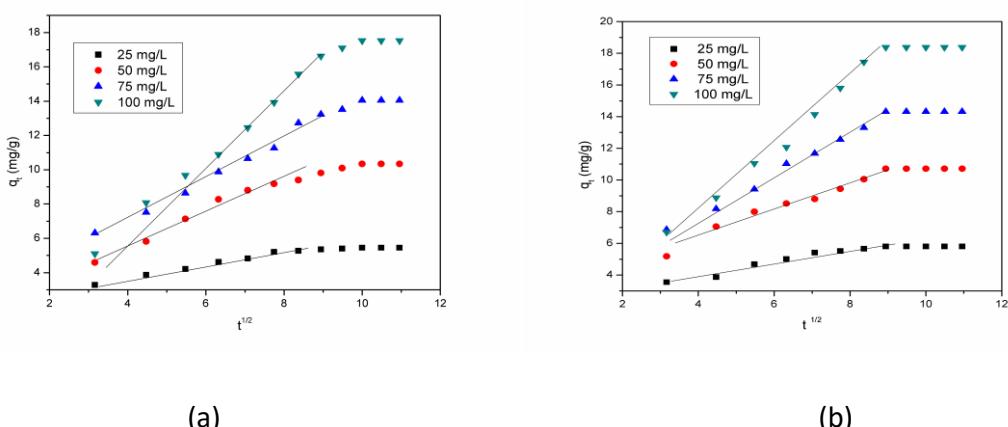


Fig.8 Intraparticle diffusion plot for the adsorption of DR9 onto a) PAC b) PPC

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 fig.8 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. Fig.7 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 plays a major role for the adsorption of DR 9 onto PAC and PPC.

Table 3. Calculated kinetic parameters for the adsorption of DR9 onto PAC and PPC

Parameters	Temperature (°C)					
	PAC			PPC		
	30	35	40	30	35	40
q_e exp.(mg g ⁻¹)	10.34	11.23	11.4	10.72	11.36	11.76
Pseudo first order kinetic model						
$k_1 \times 10^{-2}$ (min ⁻¹)	36.82	29.52	34.4	27.19	45.94	47.86
q_e cal (mg g ⁻¹)	0.6255	0.7801	0.6694	0.8471	0.5013	0.4812
r^2	0.9262	0.9258	0.9313	0.9145	0.9312	0.9225
Pseudo second order kinetic model						
$k_2 \times 10^{-4}$ (g mg ⁻¹ min)	4.26	4.41	4.83	5.01	7.66	8.75
q_e cal (mg g ⁻¹)	12.14	12.99	13.02	12.3	12.52	12.79
r^2	0.9991	0.9987	0.9985	0.9973	0.9993	0.9994
h	0.6295	0.7453	0.8189	0.7575	1.2018	1.4326
Intra particle diffusion model						
k_{dif} (mg g ⁻¹ min ^{1/2})	1.4902	1.3165	1.2535	1.3915	1.2233	1.1431
r^2	0.9677	0.9754	0.9742	0.9676	0.9755	0.9856
C	2.9349	3.6804	4.1505	3.8161	5.4585	6.2552

4. Conclusion

- (i) The present study reveals that PAC and PPC prepared from the seeds and wood of casuarina were used effectively as adsorbents for the removal of DR 9 from

- aqueous solution.
- (ii) The amount of dye adsorbed is found to vary with initial pH, temperature and contact time. Maximum adsorption occurs at pH 2 for DR 9.
 - (iii) Kinetic studies predict that the adsorption of DR 9 onto PAC and PPC follows pseudo second-order kinetics.
 - (iv) The adsorption isotherms like Freundlich, Langmuir and Dubinin – Radushkevich were analyzed for the adsorption of DR 9 onto PAC 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 PAC 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 DR 9 onto PAC and PPC.

On comparing the results, it is obvious that, PPC is an efficient, economic and alternative biomaterial than PAC for the adsorption of DR 9 from aqueous solutions.

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