

Removal of a dye from simulated wastewater by adsorption using treated parthenium biomass

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Abstract

In the present study adsorption of rhodamine-B from aqueous solution on formaldehyde treated parthenium biomass (WC) and phosphoric acid treated parthenium carbon (PWC) was studied. Aqueous solutions of various concentrations (50–500 mg/l) were shaken with certain amount of adsorbent to determine the adsorption capacity of rhodamine-B on WC and PWC. The effectiveness of formaldehyde treated parthenium biomass (WC) and phosphoric acid treated parthenium carbon (PWC) in adsorbing rhodamine-B from aqueous solution has been studied as a function of agitation time, adsorbent dose, initial dye concentration and pH. The adsorption capacities of the studied adsorbents were in the order PWC > WC. Initial pH had negligible effect on the adsorption capacity. Maximum dye was sequestered from the solution within 60 min after the start of every experiment. After that, the concentration of rhodamine-B in the liquid-phase remained constant. The adsorption of rhodamine-B onto PWC and WC followed second-order kinetic model. Adsorption data were modeled using both Langmuir and Freundlich classical adsorption isotherms. The adsorption capacity Q_0 was 59.17 mg/g at initial pH 7.0 for the particle size 0.3–1.0 mm for phosphoric acid treated parthenium carbon (PWC). The FT-IR spectra of the adsorbents were recorded to explore number and position of functional groups available for the binding of dye cation onto studied adsorbents. SEMs of the adsorbents were recorded to explore the morphology of the studied adsorbents.

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1. Introduction

Dyes are widely used by textile industries to color their products. One of the major problems concerning textile industries is the treatment and disposal of dye laden wastewater [1]. When these colored effluents enter rivers or any other surface water system, they upset biological activity. Ground water systems are also affected by these pollutants because of leaching from the soil [2]. This wastewater contains a variety of organic compounds and toxic substances, which are harmful to fish and other aquatic organisms [1]. Rhodamine-B dye is used for the dyeing of cotton, silk, paper, bamboo, weed, straw and leather [<http://www.pdindustries.com/rhodamine>]. Though rhodamine-B is not strongly hazardous, it can cause some harmful effects. Acute

ingestion of rhodamine-B may irritate the gastrointestinal tract and cause toxic effects. It also causes skin and eye irritation. On inhalation, it may irritate respiratory tract [http://www.vetmed.iastate.edu/departments/bm/SOP/QR/rhodamine_b.doc]. Rhodamine-B has been tested in mice and rats by subcutaneous injection and in inadequate studies, by oral administration. It was carcinogenic in rats when injected subcutaneously, producing local sarcomas. The intravenous LD₅₀ in rats is 89.5 mg/kg and 128 mg/kg intraperitoneal mouse LDLO (it is the lowest lethal dose of a chemical determined by testing on animals) [www.osha-slc.gov/dts/sltc/methods]. Therefore, the removal of such dye from process effluent becomes environmentally important.

Among several chemical and physical methods, the adsorption onto activated carbon has been found to be superior to other techniques for the removal of dyes from aqueous solution in terms of methodology, operational conditions and efficiency. Commercially available activated carbons are usually derived from wood or coal, and therefore, are considered expensive

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[3]. This has led to the scientists to develop cheaper but efficient substitutes. Researchers have studied the production of low cost activated carbon based on agricultural wastes, agricultural byproducts and biomaterials such as apricot [4], bottom Ash and de-Oiled Soya [5], hen feathers [6], ground nut shell [7], coconut tree [8], soy meal hull [9], jute fiber [10], *Posidonia oceanica* (L.) fibres [11], *Parthenium hysterophorus* [12,13], red mud [14], etc.

P. hysterophorus L. is a common weed [15] and may cause several health problems. It contains sesquiterpenes lactones (parthenon, hymenin, ambrosin, etc.), which may induce allergic dermatitis. However the relative composition of these lactones varies in different species of parthenium. Accumulation of fumaric acid in the stem and leaves, *p*-anisic, *p*-hydroxybenzoic and vanillic acid in the roots and caffeic acid in all plant parts, except the pollen has been observed. The phenolic acid is said to be responsible for allelopathic impact of parthenium on certain crops. When human beings frequently come in contact of this weed, it may cause allergy, dermatitis, eczema, black spots and blisters around eyes, burning, rings and blisters over skin, redness of skin, asthma, hay fever, etc. in man and livestock [16]. Parthenium is available whole year in abundant quantities at zero prices.

The focus of our research is (i) to evaluate the adsorption potential of formaldehyde treated parthenium weed (WC) and phosphoric acid treated parthenium weed (PWC) for the removal of rhodamine-B dye from aqueous solution under different experimental conditions; (ii) ever increasing unwanted weed would be put to some use; (iii) easy availability and no cost of Parthenium will make it a low cost alternative to activated carbon; (iv) to understand the adsorption, the equilibrium and kinetic data of the adsorption were then studied.

2. Experimental

2.1. Adsorbents

2.1.1. Preparation of formaldehyde treated parthenium carbon (WC)

Fully grown plants of parthenium were collected in and around Hisar City (India), cut in to small pieces of 2–3 cm and dried in sunlight until all the moisture evaporated. The dried mass was ground to a fine powder in a steel blender. The ground powder was treated with 1% formaldehyde in the ratio of 1:5 (parthenium: formaldehyde, w/v) at 50 °C for 4 h to polymerize and immobilize the color and water-soluble substances of parthenium. The sample was filtered out, washed with distilled water several times to remove free formaldehyde and then oven dried at 80 °C for 24 h. The resulting material was again ground followed by sieving in the size range of 0.3–1 mm. The various physico-chemical characteristics of WC are given in Table 1. The material was placed in an airtight container for further use.

2.1.2. Preparation of phosphoric acid treated parthenium carbon (PWC)

The dried biomass of parthenium (as given in Section 2.1.1) was impregnated with H₃PO₄ (50%) in a ratio of 2:1 (acid vol-

Table 1
Physico-chemical characteristics of adsorbents

Parameter	WC	PWC
Color	Brown	Black
Moisture content (%)	0.07	0.26
pH	7.2	2.5
Conductivity (mS/cm)	0.106	0.205
Particle density	0.34	0.54
Bulk density (g/ml)	0.25	0.35
Porosity (%)	27.0	35.1
C (%)	46.6	47.4
H (%)	6.3	3.8
N (%)	2.6	3.2
O (%) by difference	44.53	45.6

ume:weight of parthenium) and allowed to get carbonized at 150 °C in hot air oven for 24 h. The carbon so obtained was washed with distilled water to remove all the free acid until the neutral pH was obtained and dried in hot air oven at 105 °C. Finally it was ground and sieved in the size range of 0.3–1 mm. The various physico-chemical characteristics of PWC are given in Table 1.

2.2. Dye solution preparation

The basic dye, rhodamine-B (Table 2) was used as supplied by S.D. Fine Chemical, Mumbai, India. An accurately weighed quantity of the dye i.e., 1250 mg was dissolved in double distilled water to prepare the stock solution (1000 mg/l). Experimental solutions of desired concentration were obtained by successive dilution.

2.3. Batch mode adsorption studies

Batch adsorption studies were carried out by agitating the 0.2 g adsorbent with 50 ml of dye solution of desired concentration and pH at room temperature (36 ± 2 °C) in a rotary shaker at 180 rpm. After agitation, samples were withdrawn from the shaker and dye solutions were separated from the adsorbent by centrifugation. The residual dye concentration was estimated in supernatant spectrophotometrically by monitoring the absorbance before and after the treatment at wavelength of 552 nm with an UV-vis spectrophotometer (Model SL-177).

Table 2
Physico-chemical characteristics of the rhodamine-B

Parameter	Value
Chemical composition	C ₂₈ H ₃₁ ClN ₂ O ₃
Molecular weight	479.02
C.I.	45170
Dye content (%)	80
Nature	Basic dye
Synonyms	Rhodamine 610, basic violet 10, brilliant pink B
Color	Bright reddish violet powder
Soluble in solvents	Ether, alcohol and water
Melting point	165 °C
pH	7.03
λ _{max}	552 nm

Effect of agitation time on percent removal was studied using dye concentration 50–250 mg/l and adsorbent doses (4.0 g to 20 g/l) for both the adsorbents. Effect of pH was studied by adjusting the pH of dye solution from 3 to 10 with 0.1M dilute HCl and NaOH solutions. All experiments have been carried out in duplicate for all the adsorbents. The removal percentage, defined as the ratio of difference in initial dye concentration before and after adsorption ($C_i - C_e$) to the initial dye concentration in the aqueous solution (C_i), was calculated using the following equation:

$$\% \text{Removal} = \frac{C_i - C_e}{C_i} \times 100 \quad (1)$$

The amount of adsorption at equilibrium, q_e (mg/g) was calculated by the following equation:

$$\text{amount adsorbed } (q_e) = \frac{C_0 - C_e}{W} V \quad (2)$$

where C_0 and C_e (mg/l) are the liquid-phase concentrations of dye at initial and equilibrium, respectively. V is the volume of solution (l) and W is the mass of adsorbent (in g) used.

FT-IR spectra of the native and exhausted adsorbents were recorded with the help of (Perkin-Elmer, PE-RXI) to study the functional groups and binding sites. For the FT-IR study, 1 mg of finely ground biomass was encapsulated in 20 mg of KBr in order to prepare translucent sample disks. The surface morphology of the adsorbents was analyzed by scanning electron

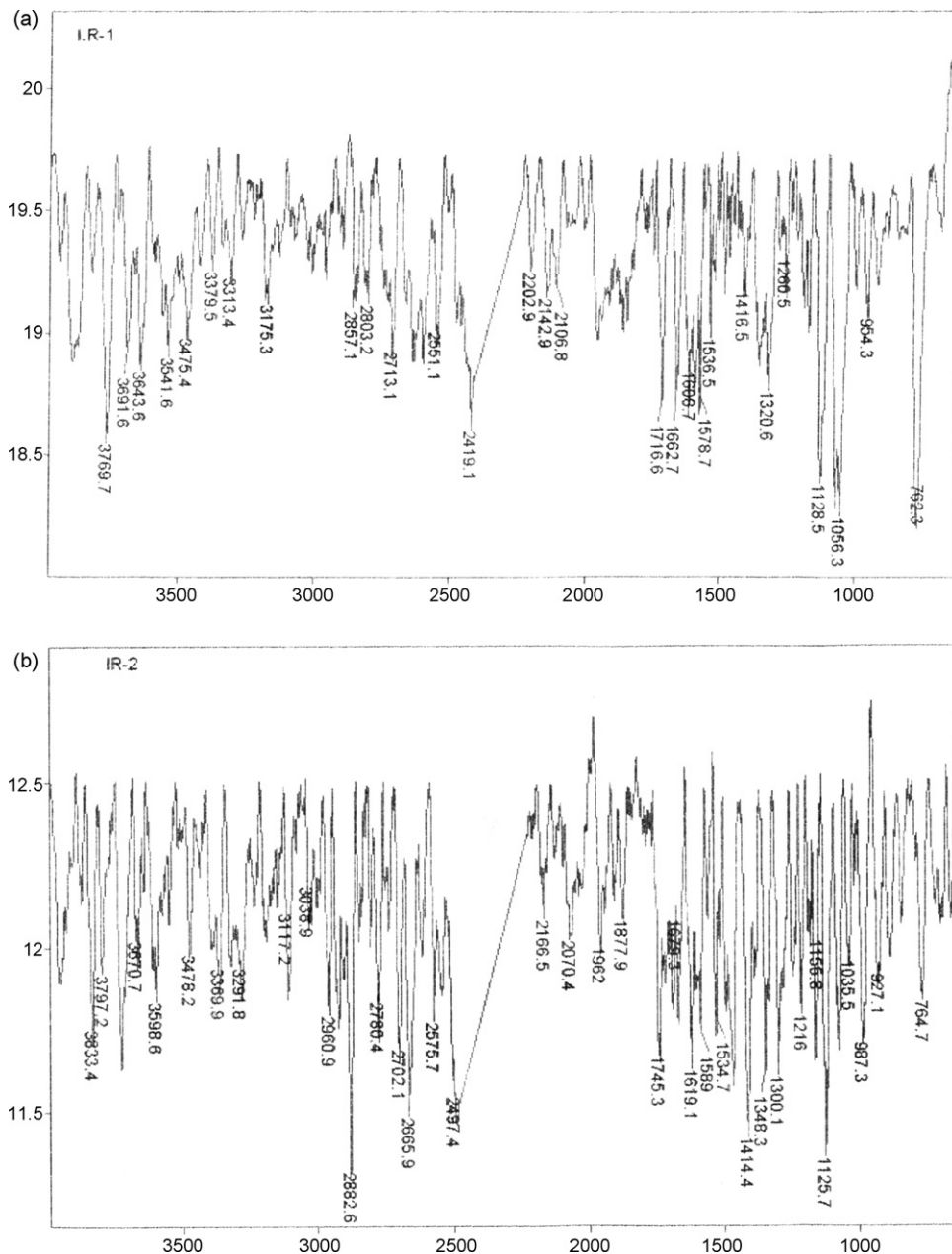


Fig. 1. (a) FT-IR spectra of native PWC and (b) FT-IR spectra of rhodamine-B loaded PWC.

microscopy using LEO 435 VP SEM at 15 kV with background subtraction.

3. Results and discussion

The complete spectrum of the dye solution was recorded before and after shaking it with adsorbent. This indicated that there were no daughter products of the dye and there was no change in λ_{\max} and also supported the facts that dye removal from the solution in this study was through the mechanism of adsorption [12].

3.1. Infrared spectroscopy analysis

FT-IR spectra of native and rhodamine-B loaded PWC are given in Fig. 1a and b. As shown in figure, the spectra display number of absorption peaks, indicating the complex nature of examined adsorbents. The spectra of these adsorbents were measured within the range of 500–4000 cm^{-1} wave number. For PWC, FT-IR spectroscopic analysis indicated broad bands at

3475.4 cm^{-1} representing –OH group. The band observed at 2857.1 cm^{-1} can be assigned to the C–H stretching and those at 1716.6 cm^{-1} to C=O stretching. The peaks around 1662.7 cm^{-1} corresponds to the C=C stretching and the absorption peak at 1416.5 cm^{-1} is assigned to the symmetric bending of the CH_3 . The C–O band absorption peak is observed at 1200.5 cm^{-1} (Fig. 1a). Although slight changes in the absorption peak frequencies can be observed in Fig. 1b, where PWC is loaded with rhodamine-B dye. It seems that these functional groups participate in dye binding. Such kind of changes in FT-IR spectra of native and rhodamine-B loaded WC are also observed (not shown). These changes in FT-IR spectra also confirm the binding of dye cation with functional groups present in the adsorbent. The fundamental peaks of the adsorbents before and after use are given in Table 3.

The SEM images of native WC and PWC (Fig. 2a and c) show the porosity and surface structure. After dye adsorption, a significant change is observed in structure of these adsorbents (Fig. 2b and d). The adsorbents appear to have a rough surface and pores containing a new shiny and bulky particle.

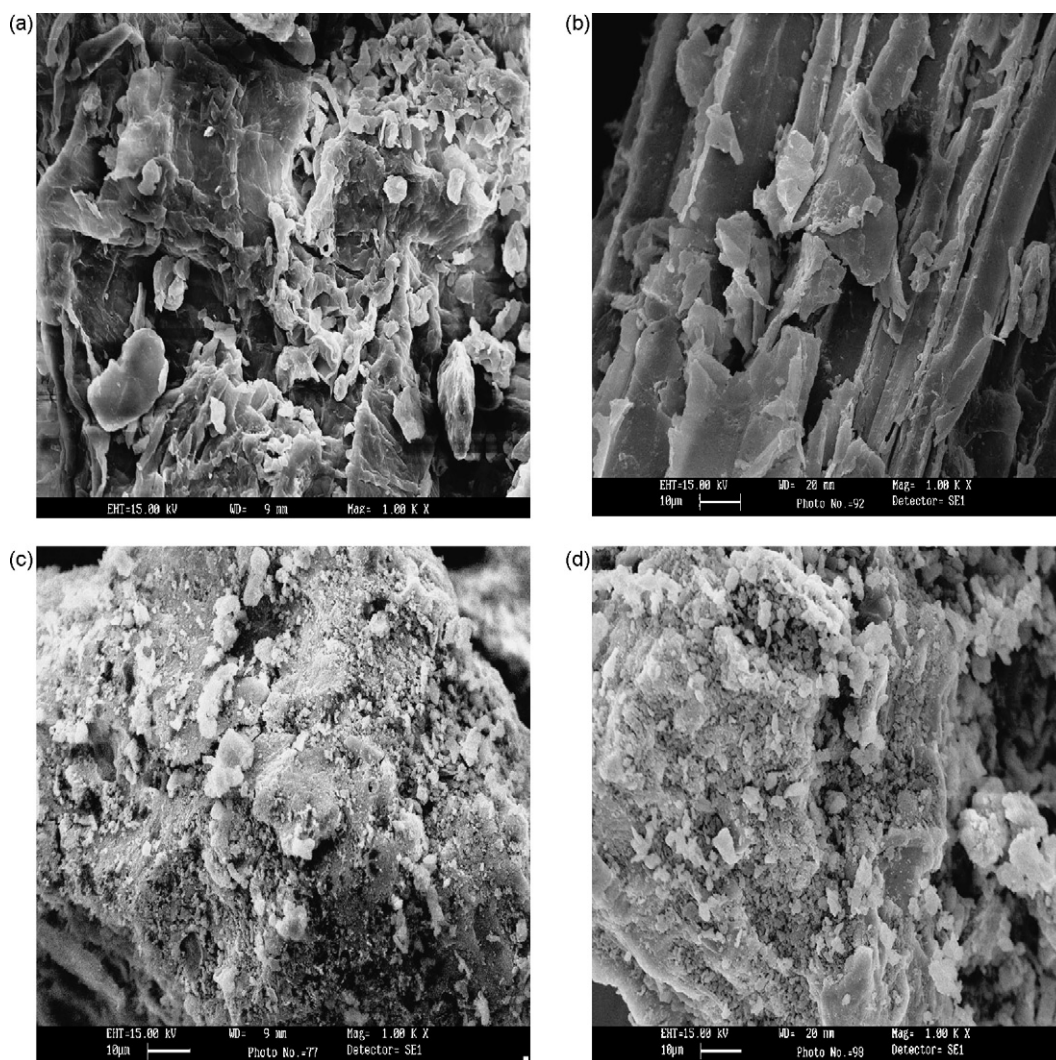


Fig. 2. Scanning electron micrographs (SEM) of (a) native WC and (b) WC after dye adsorption. Scanning electron micrographs (SEM) of (c) native PWC and (d) PWC after dye adsorption.

Table 3
Some fundamental frequencies of the studied adsorbents (before and after use)

Adsorbent	Band position (cm ⁻¹)				
	O–H	C–H	C=O	C–O	Bending vibrations
PWC native	3475.4	2857.1	1716.6	1200.5	987.7, 927.1, 764.7
PWC-RB	3478.2	2960.9	1745.3	1216.0	954.3, 762.3
WC native	3356.2	2923.1	1733.9	1247.2	898.3, 559.5
WC-RB	3400	2924.7	1739.4	1240	897.1, 770.6

3.2. Effect of pH

The pH of the solution is an important process controlling parameter in the dye adsorption process. Its role on the dye adsorption was studied at 50 mg/l dye concentration with 4.0 g/l adsorbent dose at $36 \pm 2^\circ\text{C}$ temperature for 120 min contact time. The initial pH of solution considered for this investigation was from 3 to 10. The dye adsorption by PWC was 95.6–98.3% in the studied pH range followed by WC (62.7–75.2%) (Fig. 3). PWC had more adsorption capacity than WC at all the studied pH values. This can be attributed to different structure and nature of these two adsorbents. The percentage removal of rhodamine-B was not significantly altered when the pH was increased from 3 to 10. Different trends for the adsorption of rhodamine-B on different adsorbents have been reported. The trend was similar to the adsorption of rhodamine-B by biogas waste slurry [17] and waste banana pith [18]. Hence neutral pH was selected for the further adsorption experiments.

3.3. Effect of different adsorbents surface change

To check the percentage removal of adsorbents (PWC and WC) experiments were carried out at different initial dye concentrations (50–500 mg/l) while maintaining the temperature ($36 \pm 2^\circ\text{C}$), pH (7) and contact time (120 min) constant. Fig. 4 indicates that the percentage adsorption by the studied adsorbents were in the order PWC > WC. At 50 mg/l dye concentration, PWC had 1.36 times more adsorption than WC. This may be due to difference in the presence of the function groups on the studied adsorbents (Table 3) or may also be due to difference in the surface characteristics (Table 1) of the adsorbents.

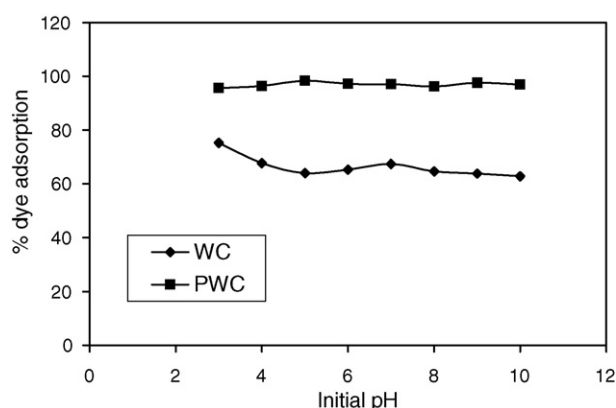


Fig. 3. Effect of pH on rhodamine-B adsorption (dye concentration = 50 mg/l, adsorbent dose = 0.2 g/50 ml, contact time = 2 h).

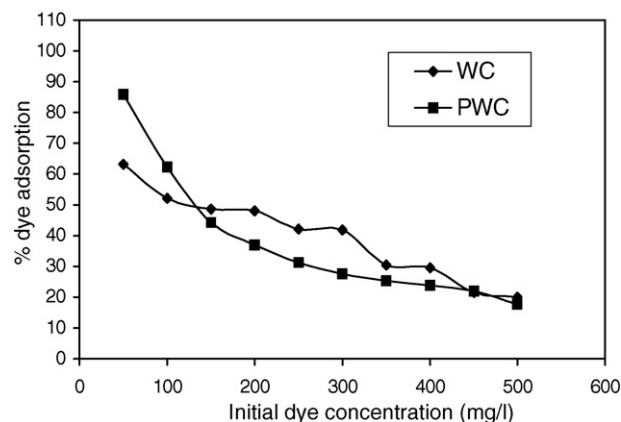


Fig. 4. Adsorption efficiency of rhodamine-B for different adsorbents (pH 7.0, adsorbent dose = 0.2 g/50 ml).

3.4. Effect of contact time and initial dye concentration

Effect of contact time and dye concentration on the removal of rhodamine-B from aqueous solution is presented in (Figs. 5 and 6) by PWC and WC, respectively. The experiments were carried out at 180 rpm, 4.0 g/l mass of adsorbent, ($36 \pm 2^\circ\text{C}$) temperature, (7.0) pH and initial dye concentrations of rhodamine-B (50, 100, 150, 200 and 250 mg/l) for different time intervals up to 120 min. Percent dye removal of PWC and WC decreased with increase in dye concentration. Though the percentage removal decreased with increase in concentration but the uptake of dye (mg/g) increased with increase in dye concentration and remained nearly constant after the equilibrium time. Adsorption capacity at equilibrium for rhodamine-B increased from (12.39, 24.74, 37.0, 49.24 and 61.46) mg/g and (7.35, 12.98, 17.82, 21.45 and 25.27) mg/g for PWC and WC, respectively, as the concentration of rhodamine-B increased from (50, 100, 150, 200 and 250) mg/l. The equilibrium was established

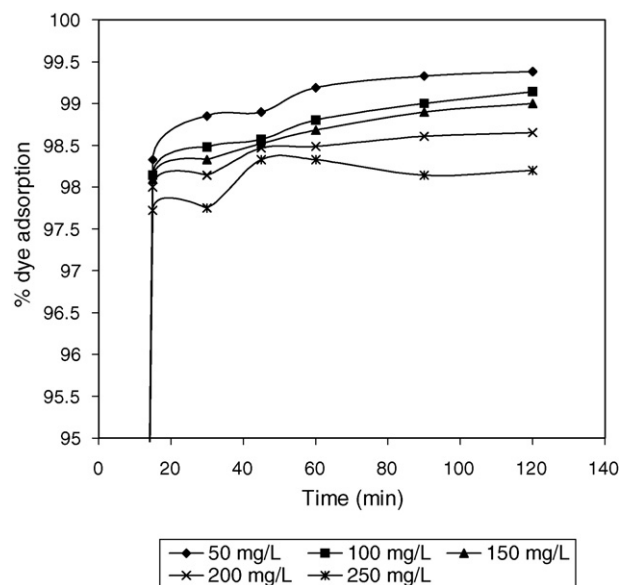


Fig. 5. Effect of initial dye concentration on the adsorption of rhodamine-B on PWC (adsorbent dose = 0.2 g/50 ml, pH 7.0).

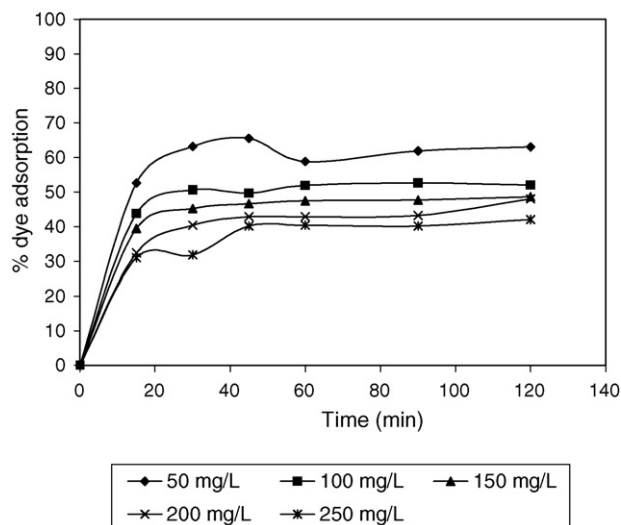


Fig. 6. Effect of initial dye concentration on the adsorption of rhodamine-B on WC (adsorbent dose = 0.2 g/50 ml, pH 7.0).

within 60 min for both the adsorbents at all studied concentrations. It is established that removal of dye depends upon the concentration of dye. The removal curves are single, smooth and continuous leading to saturation.

3.5. Effect of adsorbent dose on dye removal

In order to examine the effect of adsorbent dose on the removal efficiency of rhodamine-B, adsorption experiments were set up with varying amount of adsorbent dose (4.0, 8.0, 12.0, 16.0 and 20.0 mg/g) for PWC and WC while keeping the initial dye concentration of 50 mg/l, temperature ($36 \pm 2^\circ\text{C}$) and pH 7.0 constant at different contact time up to 120 min. The percent adsorption was increased and equilibrium time was decreased with adsorbent dosages (Figs. 7 and 8). The percent adsorption increased from 95.2 to 98.0, as the PWC dose was increased from 4.0 g to 20 g/l at the equilibrium time. Similarly for WC, adsorption increased from 66.4 to 90.2%, as the WC dose was increased from 4.0 g to 20 g/l. Maximum dye was sequestered from the solution within 60 min after the start of every experiment. A larger mass of adsorbent could adsorb larger amount of dye due to availability of more adsorption sites and more surface area of adsorbent. But for each adsorbent, the amount of dye adsorbed per unit weight of adsorbent is different. For PWC, adsorption capacity was decreased from 11.91 to 2.45 mg/g as the adsorbent dosage was increased from 4.0 g to 20 g/l in the solution. This may be due to overlapping of adsorption sites as a result of overcrowding of adsorbent particles [19,20]. For WC, the adsorption capacity was decreased from 8.3 to 2.25 mg/g as the dosage was increased.

3.6. Adsorption isotherm

Adsorption data for a wide range of adsorbate concentration and adsorbent doses can be analyzed by Langmuir and Freundlich isotherm models. The Langmuir model is based on the assumption that maximum adsorption corresponds to sat-

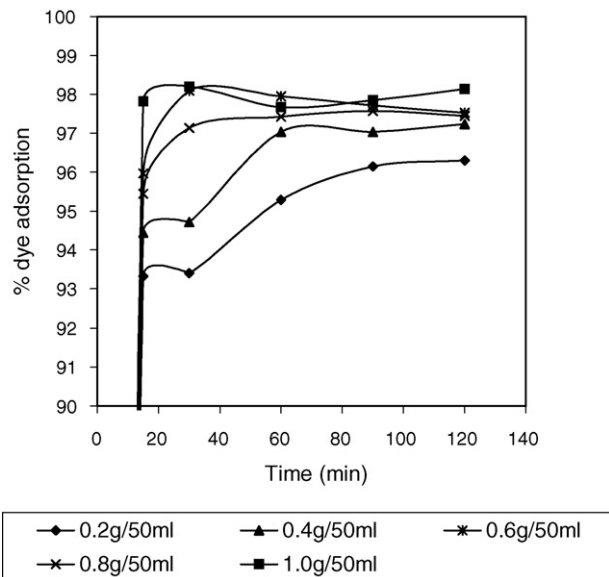


Fig. 7. Effect of adsorbent dose on the adsorption of rhodamine-B on PWC (dye concentration = 50 mg/l, sample = 50 ml, pH 7.0).

urated monolayer of dye molecules on the adsorbent surface, that the energy of adsorption is constant and there is no transmigration of adsorbate in the plane of surface [21]. Langmuir isotherm was obtained by agitating the fixed dose of adsorbate and the different concentrations of dye solution for a constant time. The Langmuir constant Q_0 (related to adsorption capacity) and b (related to energy of adsorption) were calculated from the following equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \quad (3)$$

where C_e is the concentration of dye solution (mg/l) at equilibrium and q_e is the amount of dye adsorbed (mg/g) at equilibrium

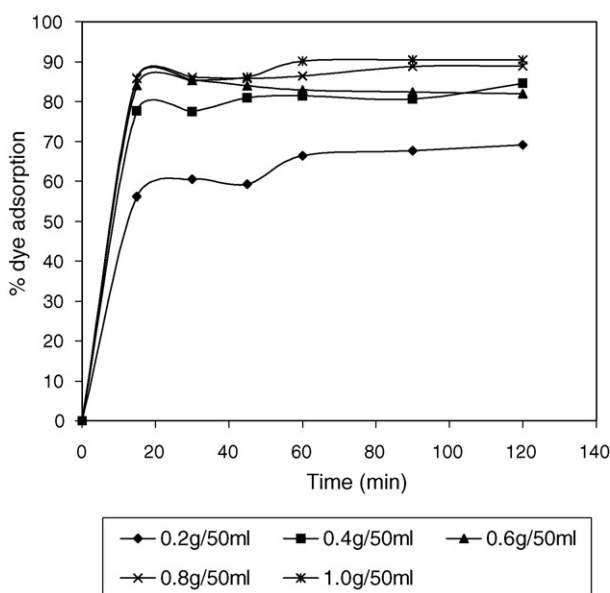


Fig. 8. Effect of adsorbent dose on the adsorption of rhodamine-B on WC (dye concentration = 50 mg/l, sample = 50 ml, pH 7.0).

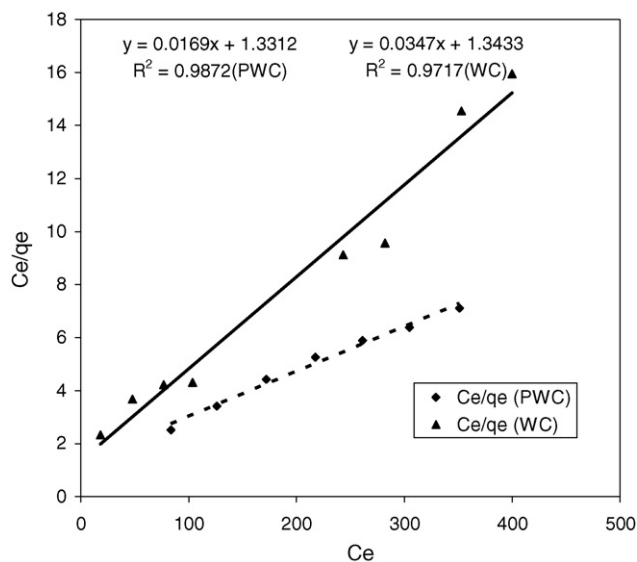


Fig. 9. Langmuir plot of C_e/q_e vs. C_e for dye adsorption.

time. Thus, a linear plot of C_e/q_e versus C_e shows that adsorption follows Langmuir isotherm (Fig. 9). The values of Q_0 and b were calculated from the slope and intercept of the plot are presented in Table 4. The essential characteristics of Langmuir isotherm can be expressed by dimensionless constant called equilibrium parameter R_L [22] which is defined by:

$$R_L = \frac{1}{1 + bC_0} \quad (4)$$

where b is the Langmuir constant ($l\text{ mg}^{-1}$) and C_0 is the highest dye concentration (mg/l). R_L values indicate the type of isotherm. R_L values between 0 and 1 indicate favorable adsorption. The R_L values were found to be between 0 and 1 for the rhodamine-B dye for all the studied concentrations which indicates the favorable adsorption.

Table 4
Values of Langmuir and Freundlich parameters obtained for the studied systems

Adsorbent	Rhodamine-B dye					
	Langmuir parameters			Freundlich parameters		
	Q_0 (mg/g)	b (l/mg)	R^2	K_f (mg/g)	n	R^2
PWC	59.17	0.013	0.9872	14.27	4.93	0.9748
WC	28.82	0.026	0.9717	1.95	1.97	0.9259

Table 5
Kinetic parameters for the removal of rhodamine-B by various adsorbents

Adsorbent	First-order			Second-order		
	k_{ad} (min^{-1})	q_e (mg g^{-1})	R^2	k_2 ($\text{g mg}^{-1} \text{min}^{-1}$)	q_e (mg g^{-1})	R^2
PWC	0.049	1.456	0.849	0.284	24.81	1
WC	0.051	1.346	0.9	0.046	13.23	0.999

Freundlich isotherm was also applied for the adsorption of rhodamine-B dye [23]:

$$\log_{10} \left(\frac{x}{m} \right) = \log_{10}(K_f) + \left(\frac{1}{n} \right) \log_{10}(C_e) \quad (5)$$

where x is the amount of dye adsorbed (mg), m the weight of adsorbent used (g) and C_e is the equilibrium concentration of dye in solution (mg/l). K_f and n are the constants incorporating all factors affecting the adsorption process (adsorption capacity and intensity). Linear plots of $\log_{10}(x/m)$ versus $\log_{10} C_e$ shows that adsorption also follows Freundlich isotherm well. In general, as the K_f value increases, quantity of dye adsorbed onto the surface of carbon also increases. The slope $1/n$ ranging between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero [24]. Values of K_f and n were calculated from the intercept and slope of the plot are given in Table 4. Determination coefficient, R^2 of both, Langmuir and Freundlich equation, is >0.92 , indicating the good agreement data for both the adsorbents.

3.7. Adsorption kinetic study

The removal of dye by adsorption on PWC and WC follows first-order rate expression. The rate constant is determined by the following Lagergren rate equation [17]:

$$\log(q_e - q_t) = \log q_e - \frac{k_{ad}}{2.303} t \quad (6)$$

Where q_t is the amount of adsorbate adsorbed at time t (mg/g), q_e the adsorption capacity in equilibrium (mg/g), k_{ad} the pseudo-first-order rate constant (min^{-1}), and t is the contact time (min). Linear plot of $\log(q_e - q_t)$ versus t were obtained for different adsorbents. The values of adsorption rate constant (k_{ad}) calculated from the slope of linear plots are presented in Table 5. Adsorption rate constant for congo red (50 mg/l) on biogas waste slurry [17], waste banana pith [18] and orange peel [2] were reported to be 0.028 , 0.106 , and 0.0406 min^{-1} , respectively.

Kinetic data were further treated with the pseudo-second-order kinetic model [25], which is expressed as

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (7)$$

where k_2 is the pseudo-second-order rate constant (g/mg/min). For second-order kinetic model, the plot of t/q_t versus t shows a linear relationship. q_e and k_2 can be determined from the slope and intercept of plot, respectively, and values are given in Table 5. Also, this procedure is more likely to predict the behavior over the wide range of adsorption. The correlation coefficients for the pseudo-second-order kinetic model are closer to unity indicating applicability of this kinetic equation and second-order nature of the adsorption process of RB on PWC and WC well.

4. Conclusion

Adsorbents prepared from parthenium weed by the chemical treatment, i.e., phosphoric acid treated parthenium carbon (PWC) and formaldehyde treated parthenium biomass (WC) were used for the removal of rhodamine-B over a wide range of concentration. The results indicate that WC has lower adsorption efficiency (63.16%) for rhodamine-B than PWC (85.95%) under studied experimental conditions. Rhodamine-B removal was highly dependent on initial dye concentration, adsorbent dose and contact time. Higher dye adsorption by PWC and WC was possible provided initial dye concentration was low in solution. Initial pH had negligible effect on the adsorption for this dye. The equilibrium data fitted well in the Langmuir model showing monolayer coverage of dye molecules at the outer surface of adsorbents. The kinetics of rhodamine-B adsorption onto parthenium carbon followed the pseudo-second-order model. Parthenium weed is easily available and has zero cost. These results indicate that PWC and WC could be used as a low cost material for dye removal from dilute aqueous solution.

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