



Research Article

Adsorptive removal of Remazol Brilliant Blue R dye from its aqueous solution by activated charcoal of *Thuja orientalis* leaves: an eco-friendly approach

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Received: 30 August 2019 / Accepted: 17 January 2020 / Published online: 24 January 2020
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Abstract

Anthraquinone dyes are one of the major water pollutants due to their stability and persistence in the aquatic system. The present study describes the use of activated charcoal prepared from *Thuja orientalis* leaves (ACTOL) as an adsorbent to remove reactive anthraquinone dye Remazol Brilliant Blue R (RBBR) from its aqueous solution. In this work, leaves of the plant were carbonated to get fine charcoal which was activated by acid treatment and analysed by BET, SEM and FTIR techniques. Batch experiments were conducted to study the effect of pH, contact time, concentration and amount of adsorbent on the removal of RBBR in the concentration range 0.1–6.65 m mol L⁻¹. The highest efficiency was found to be 81% at pH 6 and contact time of 300 min. Adsorption data were analysed by Langmuir, Freundlich and Temkin isotherm models. The adsorption was found to follow Freundlich adsorption isotherm ($R^2 = 0.9972$). The kinetic study of the process was done by analysing the time adsorption data with pseudo-first-order, pseudo-second-order and intraparticle diffusion models, and it was found to obey pseudo-second-order kinetics. The adsorbent showed 73% of efficiency even after four regeneration cycles. FTIR analysis revealed that the various groups present at the surface of adsorbent were responsible for uptake of dye molecules. This study reveals that leaves of *Thuja orientalis*, a common biowaste, can be used as an efficient adsorbent for the removal of RBBR dye from its aqueous solution. It provides an economical and eco-friendly way to mitigate the waste and use it as an adsorbent.

Keywords Remazol Brilliant Blue R · Adsorption · Kinetics · *Thuja orientalis*

1 Introduction

Various types of dyes that are used in leather, food processing, cosmetics, paper and pulp, dye manufacturing industries, rubber and printing industries pass into effluents running out of these industries. About 50% of the dyes used in textile industries alone go into the effluent [1]. These residual dyes are quite stable in the aquatic system and potentially hazardous to the environment and aquatic life as these interrupt light penetration, gas solubility and phytoplankton's photosynthesis [2]. Also, many of these dyes disintegrate to give toxic, carcinogenic and

mutagenic products [3]. The presence of dye in water even at a low concentration of 1 mg L⁻¹ makes it unfit for human usage [4]. Anthraquinone dyes are the second most common dyes, after azo dyes, used to impart violet, blue and green colours to the fabric [5]. Remazol Brilliant Blue R (RBBR) is an important anthraquinone dye with a chemical formula C₂₂H₁₆N₂Na₂O₁₁S₃ and molecular weight of 626.54 g mol⁻¹ [6]. Its structural formula is shown in Fig. 1. It has good solubility in water which makes it a common pollutant present in industrial effluent [7, 8]. Non-biodegradable and recalcitrant nature of RBBR makes it toxic organopollutant [9, 10].

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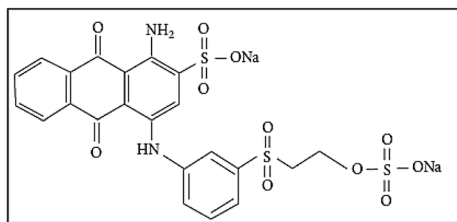


Fig. 1 Structure of RBBR

Various methods, including physical and chemical treatments, have been used to remove these dyes from the wastewater. Oxidative removal of dyes by using oxidising agents viz. H_2O_2 , chlorine, ozone, use of UV and visible radiation is the main chemical methods of dye removal [11, 12], whereas physical methods involve distillation, membrane filtration, chemical, solvent extraction ion exchange and reverse osmosis [13]. Bioremediation of wastewater using different plants and even bacteria has been also tried by researchers [14]. Among these, chemical precipitation methods include complex structural setup, high consumption of chemicals and formation of the large volume of sludge [15]. Apart from this, these methods have low efficiency at low dye concentration, particularly in the range of 1–100 mg L^{-1} [16]. Also, the stability of anthraquinone dyes towards oxidation puts a limitation on these methods [4]. Due to these limitations, researchers are oriented towards adsorption technique which has been proven as an efficient, low-cost substitute for these technologies.

Researchers have used various materials, from conventional adsorbents viz. activated carbon, silica, alumina, resins viz. Amberlite IRA-958 [16, 17], etc., to the cheaper alternatives such as biomass of crops for the adsorptive removal of the dyes and hazardous metals from the wastewater. The literature review shows the experiments with the biomass viz. *Hibiscus cannabinus* fibre modified by graft copolymerisation [18], coconut jute carbon [19], sugarcane bagasse [20], leaf mould [21], rice straw [22], coconut tree sawdust [23], pine needles and wool [24], pine cone [25], Japanese cedar bark [26], eucalyptus bark [27], tamarind hull [28], neem leaf [29], guar gum [30], banana peel [31]. Focus has been shifted to increase the efficiency of these adsorbents by chemically modifying them. Use of charcoal modified with oxidising agents [32], guar gum–nano-zinc oxide biocomposite [33] is some of these efforts. Among the various sources of biomass, preference is given to the plants available locally that are rich in biomass; their biomass has no other use and poses problems of disposal. *Thuja orientalis* plant suits best in these criteria. *Thuja orientalis* is an ornamental evergreen coniferous tree of Cupressaceae family. The literature reveals that leaves of

Thuja orientalis have various secondary metabolites which include rhodoxanthin, amentoflavone, hinokiflavone, quercetin, myricetin, carotene, xanthophylls and ascorbic acid [34]. Though *Thuja orientalis* is a common tree of high altitude which has rich biomass as dry leaves, it has been hardly studied for its use. The literature review reveals that there is some work on the use of its cone as adsorbent, but the leaves have not been explored for any such use though the plant is far rich in leaves than cones [35, 36]. Although many studies are available on the removal of RBBR, adsorptive removal of this dye by activated charcoal from leaves of *Thuja orientalis* is not yet reported.

The present work gives a detailed method of preparation of adsorbent from the leaves of *Thuja orientalis*, its activation, characterisation and its use for adsorptive removal of RBBR. The adsorbent, activated charcoal prepared from *Thuja orientalis* leaves (ACTOL), showed the high efficiency of 81% at pH 6 and contact time of 300 min. Adsorption data, analysed by Langmuir, Freundlich and Temkin isotherm models, were found to follow Freundlich adsorption isotherm ($R^2 = 0.9964$). The time adsorption data on analysis were found to obey pseudo-second-order kinetics. The leaves of *Thuja orientalis* deserve attention as new biomass which provides an eco-friendly yet highly efficient method for adsorptive removal of RBBR from its aqueous solution.

2 Experimental section

2.1 Materials

Analytical grade anthraquinone dye RBBR was procured from SRL India. HCl, H_2SO_4 , NaOH were purchased from Molychem, India. Double-distilled water was used throughout the experiments.

2.2 Preparation and activation of adsorbent

The adsorbent was prepared from the leaves of *Thuja orientalis* by a reported method [37]. The leaves were collected from the *Thuja orientalis* plants from Nainital, Uttarakhand, India. A suitable mass of the sun-dried leaves was washed thrice with double-distilled water to remove water-soluble impurities. The washed leaves were dried in a hot air oven at 100 °C for 24 h to remove moisture and volatile components. The dried leaves were then filled in a tin container for carbonisation. The container was tightly covered by the aluminium foil paper with few holes in it. Heating of tin container on fire for 3 h in the absence of oxygen resulted in carbonisation of dried mass and resulted into formation of charcoal. The charcoal thus obtained was ground in ball mill as fine as possible and

washed subsequently thrice with double-distilled water with constant stirring. This powder was mixed with 0.2 M H₂SO₄ in 1:10 (mass/volume) with constant stirring for 2 h at 25 °C to remove the organic components and activate it. After this mixing, the charcoal was washed thoroughly and a pH and conductivity of the eluant were measured with each washing. Washing was done until its pH and conductivity became constant. The stabilised charcoal was dried again in a hot air oven at 100 °C for 5 h. The dried activated charcoal from the leaves of *Thuja orientalis* (ACTOL) was sieved with mesh with different sizes and stored separately according to particle size.

2.3 Characterisation of adsorbent

Surface analysis of the activated adsorbent viz. surface area, pore diameter and pore size distribution was carried out by nitrogen gas adsorption technique using micropore analyser. The surface area was calculated by BET method. The surface texture of the adsorbent was explored by SEM (ZEISS SUPRA instrument), and functional groups present at the surface were analysed by FTIR carried out at room temperature in the spectral range varied from 4000 to 400 cm⁻¹.

2.4 Adsorption experiments

Batch experiments were performed to assess the adsorption capacity of ACTOL towards RBBR. A 250 mL of the stock solution (10⁻² M) of analytical grade RBBR was prepared in double-distilled water. This stock solution was diluted with distilled water to get solutions of desired concentrations. Adsorption study was done by taking a suitable volume (100 mL) of this solution in a 250 mL conical flask and adding a suitable mass of adsorbent to it with constant shaking for a suitable time (300 min) at 200 rpm on an orbital shaker at room temperature (25 °C). An aliquot was taken out at every 60 min till the attainment of equilibrium. The aliquot was centrifuged at 1000 rpm for 5 min to separate adsorbent from adsorbate. The centrifuged aliquot was decanted in sample tubes and analysed spectrophotometrically for the residual concentration of RBBR at its characteristic λ_{max} of 593 nm [38]. The percentage removal of dye as well as the efficiency of the adsorbent (%A) was determined using the following expression:

$$\%A = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

where C₀ is the initial concentration of RBBR solution and C_e is its equilibrium concentration.

This experimental procedure was repeated for different parameters viz. mass of adsorbent initial

concentration of dye, pH of the solution, contact time to determine their effect on % removal of dye. Finally, q_e was calculated to know the adsorption isotherm by the following formula:

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

where q_e is the adsorption capacity, i.e. mg of dye adsorbed per gram of adsorbent at equilibrium, V is the volume of solution in L and W is the mass of adsorbent in g.

2.5 Isothermal study

To analyse the equilibrium adsorptive behaviour, the isothermal study of the adsorption of RBBR on ACTOL was carried out by fitting the adsorption data to three isotherm models, namely Langmuir, Freundlich and Temkin models [39]. These three models can be represented as follows:

Freundlich Adsorption Isotherm:

$$\log q_e = \frac{1}{n} \log C_e + \log K_F \quad (3)$$

Langmuir Adsorption Isotherm:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (4)$$

Temkin Equation:

$$q_e = B \log A + B \log C_e \quad (5)$$

where C_e is the equilibrium concentration of dye in mg L⁻¹; K_F and n are Freundlich constant; K_L is the Langmuir's constant; q_m (mg/g) is the maximum adsorption capacity; A (L g⁻¹) is Temkin equilibrium binding constant; and B (J mol⁻¹) is Temkin constant related to heat of sorption.

2.6 Kinetic study

Adsorption kinetics of RBBR on ACTOL was studied by analysing the time adsorption data with pseudo-first-order [40], pseudo-second-order [41] and intraparticle diffusion models [42] according to the following equations:

Pseudo-first-order equation:

$$\log (q_e - q_t) = \log q_e - k_1 t \quad (6)$$

Pseudo-second-order equation:

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

Intraparticle diffusion:

$$q_e = \frac{k_{diff}}{t^2} + C \tag{8}$$

where q_e is the adsorption capacity, i.e. g of dye adsorbed per gram of adsorbent at equilibrium; q_t is g of dye adsorbed per gram of adsorbent at time t ; k_2 is pseudo-second-order rate constant; k_{diff} is intraparticle diffusion rate constant and C is boundary layer thickness.

3 Result and discussion

3.1 Characterisation of adsorbent

The textural properties of ACTOL obtained by micropore analysis and BET method are given in Table 1.

The surface area of charcoal of *T. orientalis* biomass reported in the literature is $27.7 \text{ m}^2 \text{ g}^{-1}$ [36]. The higher surface area obtained in the present study can be attributed to activation of charcoal by acid treatment.

The SEM images of ACTOL before and after adsorption, taken at magnification $\times 550$, are shown in Fig. 2a, b. Figure 2a shows that the adsorbent surface has an uneven morphology with valley and ridge-like structures throughout the surface. The surface has pores of different sizes of less than $20 \mu\text{m}$. The presence of uneven surface and pores adds to the efficiency of the adsorbent as these features increase the surface area. SEM image in Fig. 2b shows slightly different surface morphologies where surface is less uneven and has relatively less pores which may be due to adsorption.

The FTIR spectra of the charcoal before and after adsorption of RBBR are shown in Fig. 3. The spectrum of the charcoal after adsorption shows a broad peak at 3353 cm^{-1} which is absent in the spectrum of bare charcoal. It corresponds to moisture (H_2O), which might have crept in from the solvent. Two prominent peaks at 2960 cm^{-1} and 2949 cm^{-1} are seen in the FTIR of the charcoal before adsorption. These peaks have resulted from stretching of C-H of CH_3 and CH_2 groups. These peaks shift remarkably to higher wave numbers on adsorption of dye. The charcoal before adsorption shows two more peaks at 1600 cm^{-1} and 1058.14 cm^{-1} which correspond to N-H bending and C-N stretching, respectively. These peaks shift to higher wave numbers, 1610 cm^{-1} and 1079.65 cm^{-1} , respectively, on adsorption

Table 1 Textural properties of activated charcoal of *T. orientalis* leaves

Surface area ($\text{m}^2 \text{ g}^{-1}$)	38.4
Pore volume ($\text{mm}^3 \text{ g}^{-1}$)	63
Pore size distribution (nm)	80–600

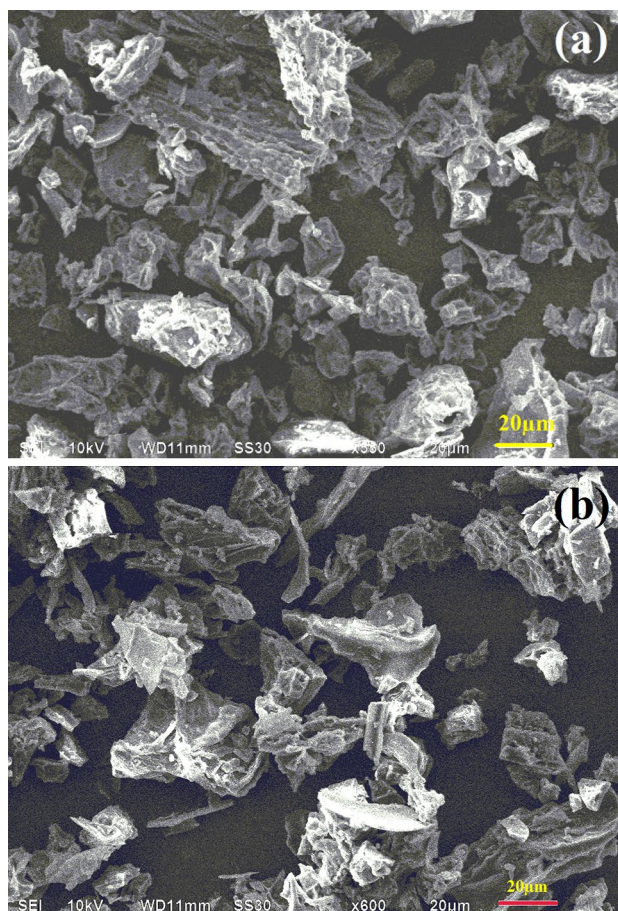


Fig. 2 SEM images of charcoal prepared from the leaves of *Thuja orientalis* **a** before adsorption **b** after adsorption

of dye. The adsorption bands around $1403, 1420$ and 1440 cm^{-1} are due to characteristic stretching of $\text{C}=\text{C}$ of aromatic hydrocarbons which are not prominent before adsorption become conspicuous after adsorption. These could be originated from the aromatic ring of the anthraquinone dye RBBR. In short, comparative study of FTIR analysis of the adsorbent, before and after adsorption, gives concrete evidence of dye absorption on the charcoal.

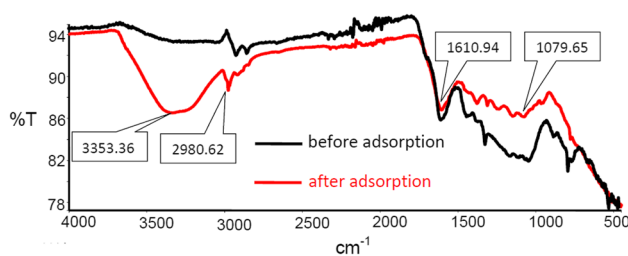


Fig. 3 FTIR peaks of adsorbent **a** before **b** after adsorption of RBBR

4 Batch experiments

4.1 Effect of pH on RBBR dye removal

The adsorption experiments were carried out in the pH range 5.0 to 9.0 taking five values namely 5.0, 6.0, 7.0, 8.0 and 9.0. The concentration of dye solution and the mass of ACTOL were kept the same throughout these six experiments. 1 M NaOH and 1 M HCl were used to vary the pH. The result of the effect of pH on adsorption is shown in Fig. 4a. The percentage adsorption increased on moving from pH 5.0 to 6.0 and decreased on further increase in pH. The literature reveals that activation of the adsorbent by treatment with H_2SO_4 increases cationic exchange sites at the surface [43]. At acidic pH, the dye molecule behaves as a cation due to protonation of the NH_2 group. It enhances its adsorption on the cation exchange sites as a result higher adsorption efficiency is seen at acidic pH which decreases sharply on moving to basic pH. All the adsorption studies in the present work were carried out at the optimum pH 6.0

4.2 Effect of contact time

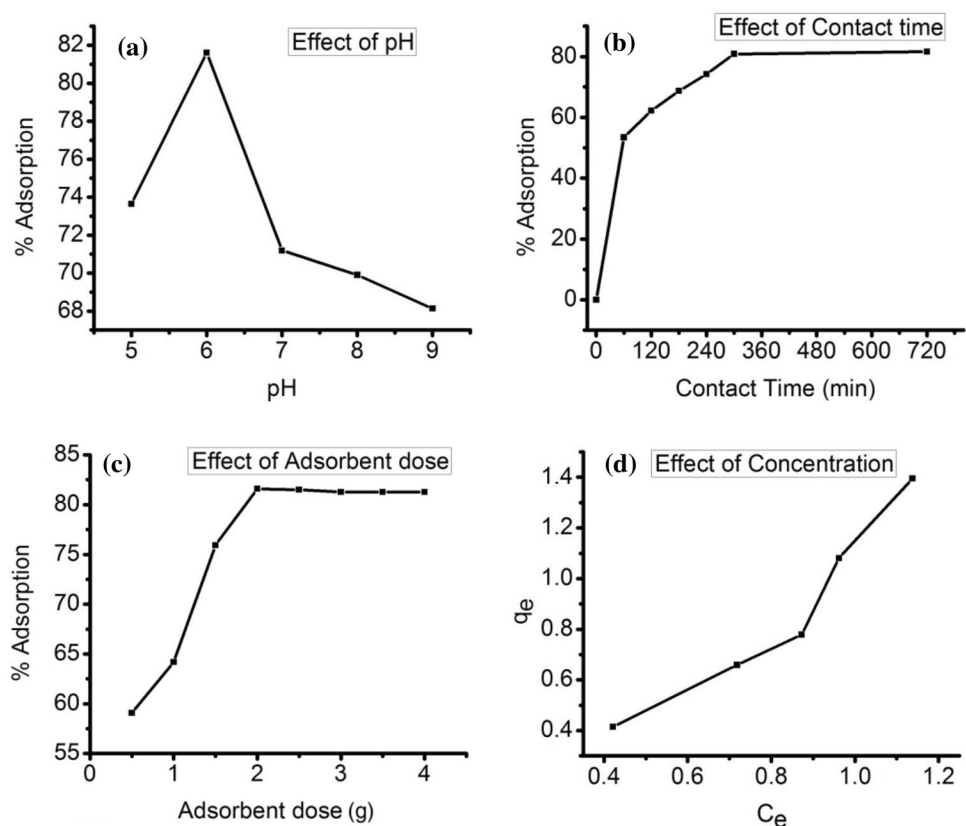
Effect of contact time on adsorption of RBBR is shown in Fig. 4b. Spectroscopic analysis of the aliquot withdrawn from the reaction mixture at regular intervals showed that

the adsorption of dye increased with increase in contact time. The adsorption rate initially increased rapidly and became almost constant after 300 min. It increased to 80.85% in 300 min and finally rose to 81.60% on an overnight stay. So, the contact time of 300 min was taken as optimum contact time for adsorptive removal of RBBR by ACTOL.

4.3 Effect of adsorbent dose on adsorption of RBBR

The effect of adsorbent dose on the adsorption of RBBR was studied in the range from 0.5 to 4.0 g of the mass of adsorbent. The adsorption efficiency increased with the adsorbent dose from 0.5 to 2.0 g beyond which mass of adsorbent did not affect the adsorption efficiency. This is shown in Fig. 4c. Similar results were obtained by Al Mammam (2014) who found that the sorption capacity of *Thuja* biomass towards the removal of Safranin O dye decreased with increase in adsorbent dose [36]. The observation can be explained on the basis of interference of adsorbent particles with the binding sites beyond a certain concentration and electrostatic interaction between cells. The observation is in agreement with the literature which asserts that the concentration gradient between adsorbate in the solution and the adsorbent surface causes the decrease in adsorption capacity [44].

Fig. 4 Effect of various parameters **a** pH **b** contact time **c** mass of adsorbent and **d** initial concentration of RBBR



4.4 Effect of concentration of dye solution on adsorption of RBBR

Effect of the initial concentration of RBBR on the adsorption efficiency was studied by varying the concentration of RBBR between 0.1 and 6.65 m mol L⁻¹ taking 2.0 g of adsorbent at 25 °C at pH 6.0. The data showed that there was a regular increase in adsorption with the increase in the concentration of dye solution. The observation was found to be in complete agreement with the literature [9, 45]. These results are shown in Fig. 4d.

The adsorption experiments, performed after optimising the various parameters viz. pH, contact time, the mass of adsorbent and concentration of RBBR, showed that 2.0 g of ACTOL showed maximum efficiency of 81.60% at pH 6.0 in 300 min when the concentration of dye was taken as 6.65 m mol L⁻¹.

Further, adsorptive efficiency of ACTOL towards the removal of RBBR has been compared with the reported adsorbents used for adsorptive removal of RBBR. The comparative results, tabulated in Table 2, showed that most of the adsorption studies have been carried out with either very dilute solution or a higher dose of adsorbent with longer contact time. The present study, carried out with highly concentrated dye solution, has proved that the removal of RBBR using ACTOL is more efficient and less time-consuming method.

4.5 Adsorption isotherms

The equilibrium data of adsorption were analysed by three different isotherm models, namely Freundlich, Langmuir

and Temkin model. The linear forms of these isotherms are shown in Fig. 5, and their correlation coefficients with respective adsorption parameters are reported in Table 3.

From the value of the correlation factor R^2 listed in Table 3, it can be evidenced that adsorption data of RBBR dye on ACTOL fit better to Freundlich isotherm. Freundlich adsorption isotherm is valid for heterogeneous surfaces and large value of n (calculated from the slope of the linear plot of $\log C_e$ vs. $\log q_e$) indicates a strong interaction between adsorbent surface and RBBR. It can be explained based on the strong affinity of cationic exchange sites with the groups' viz. NH_3^+ present on the dye at the given pH [4].

4.6 Adsorption kinetics

From the plot of adsorption efficiency and contact time, adsorptive removal of RBBR by adsorbent can be visualised as three distinct steps: initial adsorption which last for 60 min and removed almost 54% of RBBR, followed by the second step of next 180 min in which another 20% of the dye was removed. The last step that can be called as residual adsorption where adsorbate and adsorbent were in contact for more than 12 h, removed only 5% of the dye. This observation suggested a progressive saturation of the active sites. Fast adsorption, in the beginning, can be attributed to the high concentration gradient of RBBR. Adsorption of RBBR on ACTOL was studied using pseudo-first-order, pseudo-second-order and intraparticle diffusion models. These models and their linear forms are reported in Table 4. The kinetic parameters showed that the value of q_e determined from the equation of

Table 2 Comparison of various adsorbents used for the removal of RBBR from its aqueous solution

Reference	Adsorbent	Concentration of dye	Amount of adsorbent	Contact time	Adsorption efficiency (%)	Adsorption efficiency (mg g ⁻¹)
Ergene et al. [9]	Immobilised Scenedesmus quadricauda	150 mg L ⁻¹	0.5131 g L ⁻¹	24 h	17.0 ^a	48.3
Trivedi et al. [46]	Polyaluminum chloride	100 mg L ⁻¹	0.1501 g L ⁻¹	24 h	95.0	63.29 ^a
Gecgel and Kolanclar [47]	Activated carbon prepared from a pine cone	50 ppm	1 g L ⁻¹	10 min	98.7	49.35 ^a
Pelosi et al. [48]	Macrophyte <i>Salvinia natans</i>	100 mg L ⁻¹	1 g L ⁻¹	800 min	50.2 ^a	50.20
Lazim et al. [49]		1000 mg L ⁻¹	5 g	24 h	14.9	3.0 ^a
Mourid et al. [50]	Calcined [Zn ₂ AlCO ₃]	200 mg L ⁻¹	50 mg	24 h	100.0	1000
Rahmat et al. [39]	Pineapple leaf powder	50 mg L ⁻¹	5 g	24 h	> 90	9.60
Present study	Activated charcoal from leaves of <i>Thuja orientalis</i>	4.165 g L ⁻¹	2.0 g	300 min	81.60%	170

^aValue calculated from the given data in the paper

pseudo-second-order model was very close to its experimental value. Also, the R^2 value was close to 1. These facts suggested that pseudo-second-order model was the better fit than pseudo-first-order and intraparticle diffusion models. So, it could be inferred that adsorptive removal of RBBR by ACTOL followed pseudo-second-order model.

4.7 Regeneration of the adsorbent

RBBR dye acts as a cationic dye at the optimised pH, so an already reported efficient method was used to study

regeneration of the adsorbent [51]. The adsorbent was regenerated using 0.1 M HCl and reused for the adsorptive removal of RBBR from aqueous solution. It was found that the adsorbent exhibited highly stable performance towards adsorptive removal of RBBR because no significant loss in activity was observed. It showed less than 10% deactivation after 4 cycles, and it showed 73% efficiency after four number of adsorption–desorption cycles. The results are shown in Fig. 6. It showed the cost-effective nature of adsorbent.

Fig. 5 Fitting of **a** Freundlich **b** Langmuir and **c** Temkin models

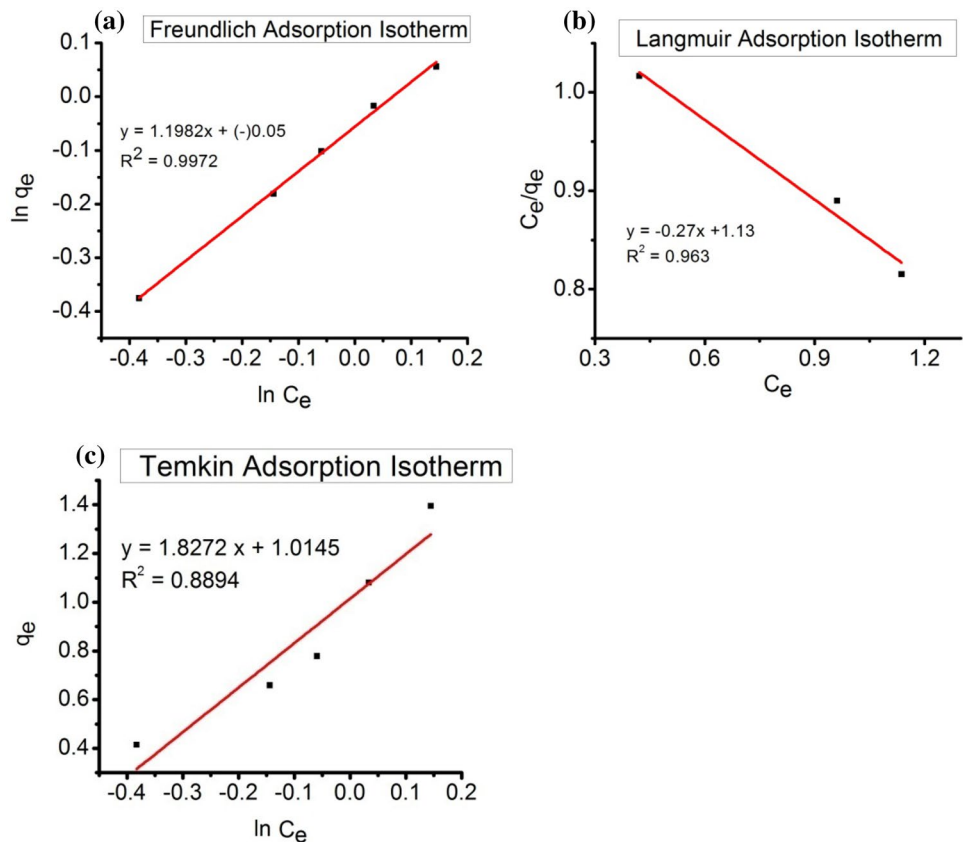


Table 3 Freundlich, Langmuir parameters and correlation coefficients for adsorption of RBBR on activated charcoal of *Thuja orientalis* leaves

Adsorbent dose (G)	Freundlich			Langmuir			Temkin		
	N	K_L (mg g ⁻¹)	R^2	Q_m (mg g ⁻¹)	K_L (mg L ⁻¹)	R^2	A	B	R^2
2.0	0.8344	0.8796	0.9972	3.8244	0.2319	0.9630	3.9862	1.8272	0.8894

Table 4 Kinetic parameters for RBBR sorption on activated charcoal of *Thuja* leaves

Adsorption kinetics	Q_e exp (g of dye per g of adsorbent)	Q_e calc (g of dye per g of adsorbent)	Rate constant (k)	R^2
Pseudo-first order	0.1699	0.1848	$k_1 = 0.00576$	0.8541
Pseudo-second order	0.1699	0.1707	$k_2 = 0.09693$	0.9978
Intraparticle diffusion model	0.1699		$k_{diff} = 0.00944$	0.9305

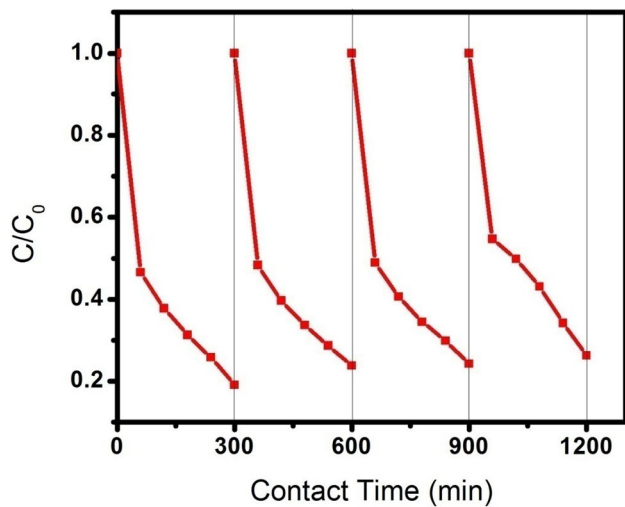


Fig. 6 Regeneration studies for the adsorption–desorption of RBBR dye onto activated charcoal of *Thuja orientalis* leaves

5 Conclusion

The waste biomass (leaves) of *Thuja orientalis* was turned into finely powdered charcoal which was activated by acid treatment. It was characterised and used as adsorbent for the removal of RBBR dye from its aqueous solution. Experiments were performed to optimise initial pH, contact time, dose of adsorbent and initial concentration of dye solution. It showed the high efficiency of 82% at a pH 6.0 and contact time of 300 towards RBBR solution of concentration $6.65 \text{ m mol L}^{-1}$. The R^2 values showed that the experimental data fitted reasonably well in the Freundlich adsorption isotherm model. The kinetic data were analysed with pseudo-first-order, pseudo-second-order and intraparticle diffusion models, and the adsorbate–adsorbent interaction was found to obey pseudo-second-order kinetics. This study highlights the use of biomass of *Thuja orientalis* as an effective adsorbent for the removal of RBBR dye from wastewater due to its high efficiency, abundance and low cost. The cost-effective nature of the adsorbent was further proved by its regeneration studies as it showed 73% efficiency after four number of adsorption–desorption cycles. The work, thus, provides a green eco-friendly way to mitigate the waste and its use as an adsorbent.

Compliance with ethical standards

Conflict of interest The authors declare that they have no competing interests.

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