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Biosorptive Removal of Congo Red Dye Using Eco-friendly Biomass from *Polyalthia longifolia*

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Authors' contributions

This work was carried out in collaboration between all the authors. The experimental work was performed by author M. Sarwar in a team comprised of authors KM, M. Ajmal and MF. Author UF supervised the work throughout the experimental and analysis stage. The first and final drafts were prepared by authors M. Sarwar, UF and M. Salman. The presentation of the study and the results was finalized by authors M. Athar and UF. All of the authors read and approved the final manuscript.

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Original Research Article

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ABSTRACT

Being toxic, Congo red is one of the most important dyes to be treated before being disposed of into waters. In the present study, dried powdered branches and leaves from an ornamental plant (*Polyalthia longifolia*) have been investigated for the biosorptive removal of Congo red dye from its aqueous solution. Infrared spectrum of the leaves and branches indicated the presence of -OH, - NH₂ and -C=O groups as the main binding sites. Influence of time of contact, pH of dye solution and initial concentration of dye was studied in batch adsorption experiments. Using nonlinear modeling, it was found that dye-leaves and dye-branches systems obeyed pseudo-second-order kinetic model with equilibrium reaching in 60 minutes at 2 g/L of biomass and 20 mg/L of dye

*Corresponding author: E-mail: umar.chem@pu.edu.pk; †Present Address concentration. Langmuir model was followed by both systems with maximum capacity as leaves (10.32 mg/g) and branches (8.57 mg/g). The biosorption capacities have been compared with a number of reported studies.

Keywords: Polyalthia longifolia; Congo red; nonlinear kinetics; langmuir; adsorption.

1. INTRODUCTION

Dyes are color substance being employed for the coloring of clothes, paper, tanning, timber, liquid ink, edible stuff and even metals. They are available both in natural and artificial forms [1]. Approximately 700,000 tons of more than 10,000 different dyes and pigments are being produced annually and a large proportion of these enter the environment through wastewaters [2]. Due to their complex structure and improper disposal. they are poisonous to human and other living creatures [3]. Organic dyes (e.g. azo dyes) are toxic because of the presence of poisonous aromatic amines that cannot be eliminated by natural biodegradation process. Congo red (Fig. 1) dye is known to be the cancer-causing in the living organisms [4]. The water contaminated with Congo red creates hazards in the environment by decreasing the photosynthetic process of aquatic plants [5]. The disposal of untreated dye-contaminated water, as a result of anthropogenic activities, in aqueous system is dangerous to the environment owing to its adverse effects on aquatic life [6,7].

A number of studies are underway to find lowcost and benign materials for the treatment of dye-contaminated waters [8]. Adsorption using activated carbon (AC) is one of the most effective processes but is limited due to cost and nonreusability of carbon. Alternative materials based on biological origin (the biosorption) are being searched for and reported for their dye removing abilities from model aqueous solutions; mainly due to their low-cost, easy availability, abundance and attractive binding potential. For instance, a number of materials have been investigated for removal of Congo red (CR) from aqueous solution; such as bentonite clay [4], rice hulls [9], rice husk [10], coir pith [11], *Azadirachata indica* leaf powder [12], orange peel [13] and jute stick powder [14]. Relatively low biosorption capacity has limited their uses. So, there is a need for some 'new' material for the removal of CR from aqueous solutions.

Polyalthia longifolia (PI), locally known as Ashok or ulta Ashok, is an evergreen ornamental plant, having a limited food value, native to Pakistan and India and found in many tropical countries also. Leaves are soft, delicate, lance-shaped and having wavy edges, with dark green color. These can be found abundantly as waste materials since the new leaves are grown; old-ones are dried down. The methanolic extract has been found to contain a number of compounds with cytotoxic properties [15]. Previously, its various parts have been reported to be used as an adsorbent for methylene blue [16], and various metal ions [17], indicating its potential efficacy as an adsorbent. So far now, no study for the removal of congo red by dried leaves and branches from Polyalthia longifolia has been reported.

The present study is an attempt to explore the removal potential of *Polyalthia longifolia* for the anionic dye CR from aqueous solutions. The effects of various factors including time of contact, solution pH, concentration and doe of material as well as equilibrium and kinetic models have been studied using nonlinear approach. The material has also been compared with other reported materials in order to show its efficiency.



Benzidine-based diazo dye, anionic Formula: $C_{32}H_{22}N_6Na_2O_6S_2$ Mol. wt: 696.665 g/mol C.I.: 22120, Direct Red 28 λ_{max} : 496 nm (water)

Fig. 1. Structure of Congo Red (CR) dye

2. MATERIALS AND METHODS

AR grade chemicals (Merck, Germany) were employed in the present work. Doubly distilled water was used for all solution preparations. Dried leaves and branches of Polyalthia longifolia were collected from the farmlands of University of the Punjab, Lahore. The leaves were washed with tap water to remove the attached solid particles and dried again in the sun. These were ground to pass 80 mesh (ASTM) screen. The obtained powder was soaked in water to remove any soluble material. It was again dried in an oven till constant mass. These were labelled as PLF. A similar procedure was adopted for branches and labelled as PLB. Both materials (PLF and PLB) were characterized using available analytical techniques to investigate their extent/potential for biosorption.

A known volume of dye solution (v, 50 mL) at a fixed concentration (C_0 , 10-50 mg/L) was allowed to be in contact with a fixed amount (m, 0.2-2.0 g) of biomass (PLF or PLB) for a fixed interval of time (t, 5-120 min) at preset conditions. Afterwards, the contents were filtered and the filtrate was subjected to analysis using UV-Vis spectrophotometer (Labomed, UVD-3500), at 496 nm, for residual dye concentration (C_e , mg/L), using a pre-designed calibration curve. The variation in time of contact, solution pH and dye concentration were studied in a similar way. The extent of adsorption (q, mg/g) was determined using the following equation (Equation 1).

$$q = \frac{C_0 - C_e}{m} \times v \tag{1}$$

Root mean square error (RMSE, equation 2) value was used as a tool for goodness-of-fit of the nonlinear model. (*N* is the number of observations and the subscripts *exp* and *cal* represent experimental and calculated (model) values for q.)

$$RMSE = \sqrt{\frac{\sum \left(q_{\exp} - q_{cal}\right)^2}{N}}$$
(2)

3. RESULTS AND DISCUSSION

3.1 Effect of Dose of Biosorbent

For a particular adsorption removal process to be cost-effective, it is very important to determine the minimum amount of material to remove maximum dye from a solution of given concentration. In order to find the minimum amount of PLF and PLB for CR removal, experiments were performed with their varying amounts for dye solution (20 mg/L). The relatively low concentration was selected due to relatively high color intensity of the dye. The results of such an experiment are shown in Fig. 2.

With increase in the dose, it was found that biosorption capacity of both materials decreased. An increase in the amount of material increased the number of available sites for the attachment of dye molecules/ions. Since dye concentration was fixed, the number of dye ions being attached was also limited/fixed. Thus increase in amount caused a decrease in q-value (Equation 1). This is in accordance with the previously reported observations [8,11,18,19]. As expected (from FTIR studies), a very small difference in the q-values for PLF and PLB, was observed. Leaves offered a relatively higher value (26.36% higher) as compared to dried branches. So, for further experiments, the amount of biomass was selected to be 0.2 g (4 g/L).

3.2 Effect of Time of Contact

Contact time study is very important in order to design a cost-effective and efficient adsorption removal process. The time taken for the maximum removal of dye species from the solution (equilibrium or optimum time) is important to scale up the process and to study the process kinetics. A shorter equilibrium time is advantageous for rapid and fast removal of species; hence, a material with fast process can be a potential candidate as an alternative to the materials in the on-going processes.

The time of contact study for the dve-PLF and dye-PLB systems is shown in Fig. 3. It can be observed that both the materials were efficient enough to rapidly attach CR species in the initial 20 minutes. Afterwards, the process slowed down and the equilibrium was reached in 60 minutes of contact, for both types of materials. With an increase of 66% in time of contact (from 20 to 60 min), q-value increased only 10% for PLF and 13.4% for PLB from that at 20 min. PLF described a better q-value as compared to that for PLB. The slower rate of adsorption at later time intervals was due to the lesser number of CR ions available for the adsorption onto the material. This time of contact (equilibrium time) was smaller than a number of reported times for CR adsorption onto various biosorbents (Table 1).

The kinetics of the CR-PLF and CR-PLB processes was also studied using pseudo- firstorder (PFO) and pseudo-second-order (PSO) kinetic models. A non-linear approach was used to test the goodness-of-fit of the model with the experimental data. The non-linear equations for PFO and PSO are given as Equations 3 and 4 respectively.

$$q_t = q_e (1 - e^{-k_1 t})$$
(3)

$$q_{t} = \frac{q_{e}^{2}k_{2}t}{1 + q_{e}k_{2}t}$$
(4)

where k_1 (min₋₁) and k_2 (g mg⁻¹ min⁻¹) are the rate constants for PFO and PSO respectively. The goodness-of-fit for the nonlinear plots was determined by RMSE (Equation 2). Fig. 3 depicted the fitting of PFO and PSO for both the systems and Table 2 represented the kinetic parameters. As far as PFO is concerned for both systems, it can be observed that the curve followed a trend similar to the experimental data set, but the difference between the experimental and model data sets was highly significant. The experimental and calculated q_e -values also showed a very high difference i.e, -47.42% for PLF and -60.24% for PLB; the negative sign indicated a value smaller than the experimental one. This indicated that PFO was unable to describe the kinetics of the systems. RMSE values also indicated a similar inference.



Fig. 2. Effect of dose of PLF and PLB on adsorptive removal of CR dye

Table 1. Adsorption of Congo red by various adsorbents

Adsorbent	Time (min)	q _{max} (mg/g)	Kinetic model*	Equilibrium model**	Reference
Carbon from Coir pith	10	6.72	PSO	Lang	[11]
Flyash	60	4.125 (30℃)	PFO	Lang	[8]
Acid activated Red mud	90	7.08		Lang	[20]
Roots of Eichhornia crassipes	90	1.58	PSO	Lang	[21]
Cicer arientinum shell	300	22.22	PSO	Lang	[18]
Hevea brasiliensis	90	9.82	PSO	Lang	[19]
Kaolin	1440	5.44	PSO	Lang	[22]
Zeolite	1440	3.03	PSO	Freund	
Rice husk ash	60	7.04	PSO	Freund	[23]
Polyalthia longifolia leaves	60	10.32	PSO	Lang	Present
Polyalthia longifolia branches	60	8.57	PSO	Lang	study

* PFO = Pseudo-first-order PSO= Pseudo-second-order ** Lang = Langmuir, Fruend = Freundlich

Table 2. Kinetic parameter for CR-PLF and CR-PLB systems (Bold-faced values indicate the deviation from experimental values)

		Leaves (PLF)	<i>q_{e (exp)}</i> (mg/g)	Branches (PLB)
Pseudo-first-order	<i>q_{e calc}</i> (mg/g)	2.0711 (-47.42%)	3.961	1.5147 (-60.24%)
model	k_1 (1/min)	0.0786		0.0514
	RMSE	1.664		1.9912
Pseudo-second-	<i>q_{e calc}</i> (mg/g)	4.076 (+2.92%)		3.9339 (+3.25%)
order model	k_2 (mg / g min)	0.0924		0.0769
	RMSE	0.0818		0.1056



Fig. 3. Contact time and kinetic modeling for CR-PLF and CR-PLB systems (L represented PLF and B represented PLB)

The fitting of PSO calculated data with the experimental data is also observable in Fig. 3. It can be seen that the model curve fitted quite well with the experimental data set. The experimental and calculated (model) qe-values showed an insignificant difference i.e, 2.92% for CR-PLF and 3.25% for CR-PLB systems. The difference is significantly less than that found in case of PFO. In addition, the RMSE values disclosed a similar trend for the goodness-of-fit. Smaller RMSE value for PSO indicated a better fit than that for PFO. This exhibited that PSO was better in describing the kinetics of the both systems. So, it can be concluded that the kinetics of CR-PLF and CR-PLB systems can be described using PSO model. This is in compliance with the previously reported adsorption removal studies [Table 1].

3.3 Effect of Initial Concentration

The adsorption removal process is highly affected by the number of CR ions available for attachment onto a fixed number of binding sites. It has been reported that a change in the initial dye concentration caused a change in the equilibrium adsorption capacity, at the studied fixed set of conditions [23]. Hence, the effect of variation in the initial dye concentration needs to be investigated. As depicted in Fig. 4, the experimental q-values for both materials, PLF and PLB, increased with CR concentration. The fixed numbers of binding sites present in the materials are saturated with increase in concentration. Greater the number of dye ions, greater is the rate of adsorption, since the concentration gradient acts as a driving force for adsorption.

The variation in the dye concentration is also used for the investigation of equilibrium models. These models express the quantitative relationship between the adsorbed and available dye concentration in the solution. Two of the most commonly used models are the Freundlich and the Langmuir models. The non-linear expressions for these are given in Equations 5 and 6 respectively.

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \tag{5}$$

$$q_e = K_F C_e^{1/n} \tag{6}$$

Here K_L is the Langmuir constant related to the free energy of adsorption, q_m is the maximum adsorption capacity at equilibrium, K_F and n are the Freundlich model parameters and indicate the extent of adsorption and degree of non-linearity.

Non-linear approach was used for the fitting of the models with the experimental data sets for CR-PLF and CR-PLB systems. The model parameters / constants are given in Table 2. Fig. 4 depicted such a relationship; L indicated the data set for leaves (PLF) and B indicated data set for branches (PLB). As evident from Figure, for PLF, Freundlich curve is rather straight and least fitting the experimental data. The difference in the fitting of Langmuir curve with that of experimental values is guite insignificant. The goodness-of-fit is indicated by the RMSE values. As can be seen, RMSE values are higher for Freundlich fitting than that for Langmuir fitting, indicating a better fit for Langmuir model. The fitting of Langmuir model is an indicative of the fact that CR ions adsorbed onto the PLF surface forming a monolayer. The thickness of this layer is equivalent to the size of ions being adsorbed. The maximum adsorption capacity (q_m) of PLF for CR, under studied set of conditions, was 10.32 mg/g. A similar trend in the equilibrium modeling can be observed in case of PLB. The maximum adsorption capacity of PLB was 8.57 mg/g. A relatively smaller qm value was observed for PLB as compared to that for PLF. The feasibility of the adsorption is given by dimensionless equilibrium parameter, the $(R_L = 1/(1+K_LC_0))$. The R_L values for PLF vary in the range of 0.1037 - 0.5365, and for PLB vary in the range of 0.1419 - 0.6232, indicating a feasible adsorption for CR-PLF and CR-PLB systems.

The adsorption removal efficiency of PLF and PLB has been compared with a number of reported studies (Table 1). It was found that under the given set of condition, PLF and PLB

were better than a number of materials of biological origin. This indicates the possible candidature of these materials as alternate to other already used materials for the effective removal of CR from aqueous solutions.

Table 3.	Equilibrium	parameters	for CR-PLF
	and CR-P	LB systems	

		Leaves	Branches
Langmuir	<i>q_m</i> (mg/g)	10.32	8.57
model	K_L (L/mg)	0.1728	0.1209
	RMSE	0.1902	0.1232
Freundlich	K_{F}	1.6211	1.1057
model	1/n	0.5838	0.5843
	RMSE	0.3857	0.2864

3.4 Effect of pH of Solution

The stability of the adsorbate structure, surface charge and disassociation of functional group from the surface of adsorbent depend upon the solution pH. Congo red is a diazo dye and is most likely influenced by a change in pH. Below a pH of 3.0. CR shows a blue color which changes to red when pH increases to 5.2. The change in color indicates a change in the stable structure of the ion present in the solution. Hence, an investigation related to the change in the solution pH on CR removal was performed. The relationship between pH of the solution and the removal efficiency of PLF and PLB is shown in Fig. 6. In order to have a realistic and more technically true information about the removal process, λ_{max} was determined at various pH values of dye solution and used to determine the respective residual dve concentration after the adsorption experiment.



Fig. 3. Variation in CR concentration and equilibrium modeling for PLF and PLB (L represented PLF and B represented PLB)

It can be observed from Fig. 5 that with an increase in the pH of solution from 4 to 6, the adsorption removal capacity of both materials increased. The maximum was observed between pH of 6 and 7 and the optimum pH was selected as 6.5. Afterwards, a gradual decrease in the removal was observed. This removal can be explained on the basis of the charges present on the materials as well as the dye ions. The dye ions are negatively charged in solution. With an increase in the pH of the solution, the concentration of OH⁻ ions also increases. Due to

a possible competitive adsorption between dye anions and hydroxyl ions (negative species), the number of adsorbed dye ions decreased. This can also be explained on the basis of negatively charged surface of the materials in basic pH range. The negative charges on the surface of materials create repulsion for the dye anions causing a decrease in the removal adsorption process [2]. Similar results have been reported for biosorption of Congo red by various materials [3,8,19,21].



Fig. 5. Effect of variation in solution pH on Cr-PLF and CR-PLB biosorption systems



Fig. 6. FTIR for PLF, PLB before and after CR adsorption

3.5 Characterization of Biosorbents

Both the materials. PLF and PLB, were characterized in order to determine their possible potential / use as an adsorbent for CR from aqueous solutions. The literature revealed that biosorbents having available / ionizable carboxyl, hydroxyl and amino groups possess good biosorption / binding capacities for dye molecules [23]. The functional groups in PLF and PLB were characterized by Fourier transform infrared spectroscopy (FTIR). The results of the FTIR spectra of PLF and PLB before and after biosorption of dye were shown in Fig. 6. The broad band between 3400-2700 cm⁻¹ indicated the presence of -OH stretching vibrations in PLF and PLB. Although -NH gives sharp peaks/bands in the same region, it might be inferred that the bands are underneath the broad -OH band. A sharp band at 2919 cm⁻¹ indicated the -CH stretching. The bands around 1735-1618 cm⁻¹ represented the -C=O, -C=C, -C=N vibrations [24]. A very insignificant difference in the bands for both materials was observed, indicating a similar variety of functional groups in both materials. The materials were found to be polyfunctional in nature.

After adsorption of dyes, a change/shift in the positions of bands was observed. CR itself contained carboxyl, sulphonic acid, amine and aromatic groups indicating their own presence through FTIR spectrum. In spite of this, the shift in the position of the bands, mostly in the fingerprint region, indicated that dye adsorbed onto the biomass at the positions where oxygen and nitrogen containing functional groups were present [25]. Based on the nature of functional groups, a similar extent of adsorption is expected for both materials.

4. CONCLUSION

Congo red, containing two azo groups and toxic to life, is one of the important dyes to be properly treated before being disposed of. Dried leaves and branches from an ornamental plant (*Polyalthia longifolia*) have been investigated in the present study for the removal of Congo red dye from aqueous solution. Infrared spectrum indicated the presence of oxygen and nitrogen containing functional groups as the binding sites. Batch experiments were performed to analyses the adsorption capacities of biomass of leaves and branches of *Polyalthia longifolia*. A nonlinear approach was used to study the kinetics of the process and pseudo-second-order model was found to be followed with equilibrium reaching in 60 minutes of cotnact. Similarly, equilibrium models, namely, Langmuir and Freundlich models, were used for the equilibrium study in nonlinear mode. RMSE analysis indicated that Langmuir model was followed with maximum capacity as leaves (10.32 mg/g) and braches (8.57 mg/g). Both the materials are easily available at almost no cost; leaves and branches of *Polyalthia longifolia* are attractive alternates to the costly adsorbents for the removal of Congo red dye from aqueous solutions.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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