Biosorption of BF-4B Reactive Red Dye by using Leaves of Macrophytes *Eichhornia crassipes*

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Abstract: *The removal potential on BF-4B reactive red dye by Eichhornia crassipes dried leaves has been investigated. Furthermore, the influence of process parameters such as solution pH, agitation, and particle size on the dye adsorption efficiency was assessed. Both kinetic and equilibrium experiments were performed in batch operation of the system. Kinetic results demonstrated that the equilibrium state was achieved after 45 min process time. The kinetic experimental data were best described by applying a pseudo-second order model that evaluated the value of rate constant 0.22 g/mg/min and an equilibrium dye concentration 8.20 mg/g. A set of isotherm models, taken from knowledge database, was tested in order to represent the equilibrium adsorption data. The Langmuir model performed the best when fitting experimental data where the maximum adsorption capacity of 20.38 mg/g was achieved. The results demonstrated that the E. crassipes has good potential to be used as a biosorbent in industrial treatment systems to remove reactive dyes from textile effluents.*

Keywords: *Eichhornia crassipes, Reactive red dye, Biosorption.*

Introduction

In the world annually are produced more than 700 thousand tons of synthetic dyes, with more than 10,000 different types of dyes and pigments used in various industries such as textiles, leather, cosmetics, paper, food, among others. Research indicates that approximately 15% of the production of synthetic dyes is lost during the production processes and management of organic compounds hazardous to human health [6].

Discharges of wastewater from textile industries in environmental bodies of water make the latest unuseful for human consumption due to high alkalinity, biochemical oxygen demand, chemical oxygen demand and total dissolved solids concentration usually below 1 g/l. Such wastes also decrease the penetration of sunlight into the water and affect the activity of photosynthesis of natural macro and micro plants. The dyes are highly hazardous to aquatic systems, due to their carcinogenic, mutagenic, toxic and allergic nature. The difficulty of biodegradation of the dyes is related to their synthetic origin and stable complex aromatic structures [22, 24]. Thus, it is necessary to remove such pollutants from industrial effluents before their release into the aquatic environments. The treatment of textile effluents can be done by different methods: physical [20], biological [8], electro-coagulation [18], advanced oxidation processes [7, 15, 19], and adsorption [2, 13].

The adsorption process has been highlighted as a promising and efficient method for treatment of effluents. There are lots of studies on adsorbents applications: alumina [5], zeolite [3], polyurethane foam [17]. Application of natural sorbents have to be mentioned as well-cellulose derivatives [4], fruit peel [1, 2], macrophytes [9, 11], among others.

Among the various biosorbents used, the macrophytes have emerged as a promising biosorbent for their high efficiency in dye removal processes [13, 21] and heavy metals [10, 12, 14, 16]. The macrophyte *Eichhornia crassipes* is native specie of Brazil, which can grow freely on the surface of fresh waters or is anchored in the mud and can cover large areas of lakes and reservoirs. Hence, that such growth prevents light penetration and development of submerged vegetation, causing problems for aquatic flora and fauna. Moreover, this macrophyte causes problems of ships' navigation, fishing, water entries for hydropower stations and irrigation systems. Thus *Eichhornia crassipes* is a plant that threatens biodiversity and it is important to limit its growth and prevent its spread [25].

The goal of this work was to study kinetic processes and equilibrium state of the removal of BF-4B reactive red dye by using dry biomass of macrophyte *E. crassipes*.

Materials and methods

Biomass preparation

The leaves of *E. crassipes* macrophyte were collected at the Center for Advanced Study in aquaculture, at Toledo (PR). In the next step, they were washed with distilled water and dried at 30°C. Further, the leaves were crushed in a mill and sieved into three different particle sizes (0.589 mm, 0.295 mm, and 0.147 mm).

Preparation of the solution of BF-4B reactive red dye

A solution of 50 mg/l BF-4B reactive red dye was used to determine the wavelength of maximum absorption. The absorption was measured by using spectrometer UV/Vis-1800 Shimadzu, and the chosen wavelength range was from 350 to 1000 nm. In this range, the authors expected to locate the maximum absorption of the dyes. To obtain the calibration curve, dye solutions with concentrations from 0 to 100 mg/l were used at wavelength of maximum absorption.

Preliminary tests

Preliminary tests were performed in order to investigate the influence of process parameters – particle size, temperature, pH, and stirring speed on the removal of reactive red dye BF-4B by using macrophyte. For all tests, 0.2 g of biomass and 50 ml of dye solution at 50 mg/l were used in 125 ml conical flasks under constant agitation in “shaker”. After 180 min, the samples were filtered through a nylon filter, centrifuged for 5 min at 3000 rpm, and the concentrations were determined by UV/Vis-1800 Shimadzu spectrometer. The pH of the dye solutions was adjusted with addition of 1M NaOH or 1M HCl. All tests were performed in triplicate.

Particle size of the adsorbent

The particle sizes of the samples were as follows: 0.589 mm; from 0.147 to 0.295 mm; and from 0.295 to 0.589 mm without separation. The initial conditions of this test were pH = 2, agitation speed 90 rpm, temperature 30°C, and reaction time of 3 hrs. Further, the particle size (0.147 to 0.295 mm) showing the highest rate of dye removal was determined, and it was set as optimal.

Temperature

The adsorption temperatures investigated were 30, 40 and 50°C respectively under the conditions mentioned above. The temperature with the highest rate of removal of the dye was set up as optimal (50°C) for the following tests.

pH of the dye solution

To obtain the optimal pH of the dye removal process, several tests were performed in the range of pHs from 1 to 12 by increasing with 1 unit. The other parameters of the process – particle size, temperature, agitation speed, and contact time were maintained as previously determined. The optimal pH (pH = 2), under which the highest dye removal was achieved, was used in further experiments.

Agitation speed

The mixing conditions of dye removal process were studied under the agitation speeds of 30, 90, and 150 rpm. The values of other process parameters were maintained as previously determined. Thus, the optimal agitation speed (150 rpm) was considered the one under which the highest removal rate was obtained.

Kinetic and equilibrium tests

Kinetic tests were carried out by using 0.3 g of adsorbent in 50 ml of dye solution in 125 ml Erlenmeyer flask, with the optimum conditions obtained in preliminary tests: particle size between 0.147 to 0.295 mm, temperature 50°C, pH = 2, and agitation speed of 150 rpm. Samples were withdrawn at predetermined time intervals (1, 2, 3, 5, 10, 15, 30, 45, 60, 120, 180, 240, 480 min) in order to obtain the standard curve of the dye concentration vs. contact time. This procedure was performed in the same manner as it was done in preliminary tests including filtration, centrifugation, and subsequent reading of optical density by using UV/Vis-1800 Shimadzu spectrometer. The equilibrium tests followed the same procedure as the kinetic tests. The contact time was set up 45 min, and the mass of adsorbent used was 0.05 to 1.00 g by an increase of 0.05 g.

Results and discussion

Preliminary tests

During the tests about the influence of particle size on biosorption process, it was found that the removal of dye by the particle size from 0.147 to 0.295 mm was approximately 93%. When a mixture of all sizes was used, a removal of 87% of the dye was obtained, while for the other sizes, the achieved removal capacity was approximately 83%. Therefore, the optimal particle size used during the tests was from 0.147 to 0.295 mm. The results of the tests investigating the effect of adsorption temperature have shown that a better removal capacity has occurred at 50°C with the value of approximately 98%.

Analysis of the tests about the influence of pH on the adsorption of dyes shows the highest removal values occurring at acidic pH between 95% and 98% at pH = 1 to pH = 2, respectively. However, it was found that when the pH increased, there was a reduction in the rate of removal of dye, starting from 66% at pH = 3. For other pH values (ranging from 4 to 12) the removal capacity was found to be around 45%. The influence of agitation speed on the process of adsorption of dye was not significant for the chosen values of agitation.

In conclusion, during the study of the process kinetic and equilibrium sorption the following parameters were chosen as optimal: biomass particle size of 0.147 to 0.295 mm; temperature of 50°C; pH = 2, and the liquid phase stirring speed of 150 rpm.

Kinetic test

The results of the effect of contact time on bioremediation of BF-4B reactive red dye by using macrophyte biomass are shown in Fig. 1. The test was executed under the following working conditions: particles size from 0.147 to 0.295 mm; process temperature of 50°C, pH = 2; agitation speed of 150 rpm. The obtained results demonstrated that the biosorption process considerably increased until 45 minutes of contact time. After this point, variation of the solution concentration was insignificant. Therefore, for the given process conditions, the biosorption process achieved its equilibrium for 45 minutes.

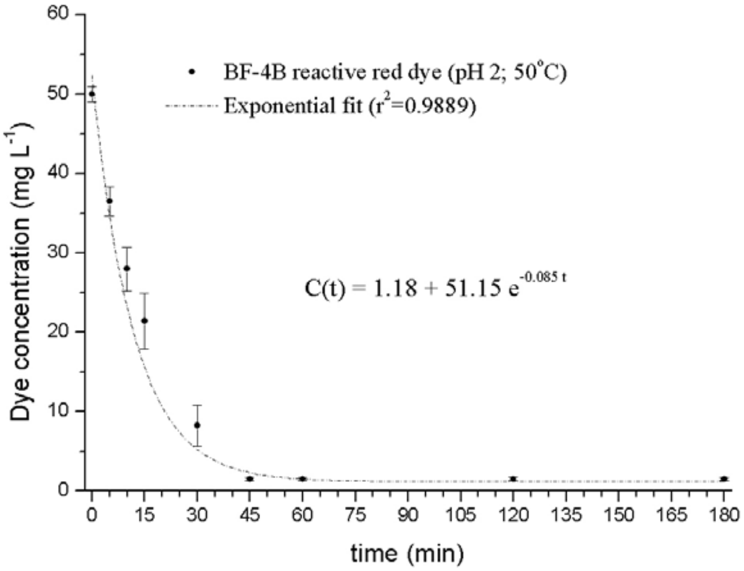

Fig. 1 Biosorption kinetic data of BF-4B reactive red dye (solid circle), obtained at pH = 2, process temperature of 50°C and by using the aquatic microphyte *E. crassipes* biomass, including an exponential fit (dashed line)

Adsorption kinetics

The concentration of the dye in the solid phase at equilibrium at time *t*, was calculated by using Eq. (1).

$$q_{eq} = \frac{V(C_0 - C_t)}{m_s} \tag{1}$$

where C_0 is the initial concentration of dye in solution (mg/l); C_t is final concentration of dye in solution (mg/l); V stands for the volume of solution (l); m_s is dry mass of the biosorbent (g). The kinetic data were evaluated by using kinetic models taken from the catalog of biosorption modeling pseudo-first order and pseudo-second order. The linear form of pseudo-first order model, shown by Eq. (2), depicts the rate of adsorption with respect to the adsorption capacity, whereas the linear pseudo-second order form (see Eq. (3)) is more flexible and can be used to describe chemisorption reactions which are more efficient in adsorption of metals and dyes.

$$\log(q_{eq} - q_t) = \log\left(q_{eq}\right) - \frac{K_1}{2,303}t \tag{2}$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_{eq}^2} + \frac{1}{q_{eq}}t \tag{3}$$

where K_1 is the rate constant (1/min); q_{eq} is the amount of dye adsorbed at equilibrium (mg/g); q_t is the amount of dye sorbed at time t (mg/g); K_2 is a rate constant (g/mg/min).

By using the software Origin ® version 8.0, the obtained model which best fit the kinetic data was the pseudo-second order as shown in Fig. 2. The values obtained for the rate constant and the amounts adsorbed at equilibrium were 0.22 g/mg/min and 8.20 mg/g, respectively.

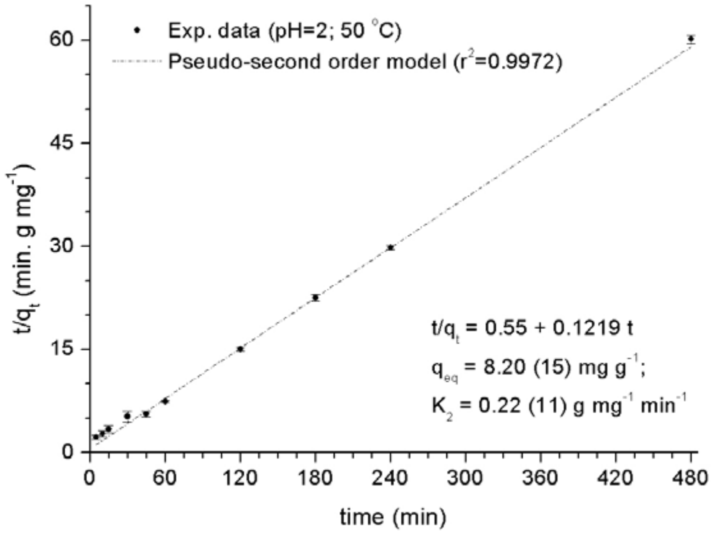

Fig. 2 BF-4B reactive red dye adsorption kinetic data (solid circle) obtained at pH = 2, process temperature of 50°C and by using the aquatic microphyte *E. crassipes* biomass and its fit by the pseudo-second order model (dot-dashed line)

Biosorption isotherm models

The adsorption isotherms were very important to estimate the maximum adsorption capacity and affinity between adsorbate and adsorbent and other physical parameters. As the adsorption phenomenon is related to the type of used material for the adsorption (physical and chemical) and the type of adsorbate as well as the experimental conditions, the kinetics and equilibrium states of each adsorption process have to be studied in detail. These isotherms represent an equilibrium relation between the concentration of the dye and the biosorbent in the liquid phase under the given working conditions: mass of biosorbent and temperature. In this context, in order to obtain the model that best describes the experimental data, we tested the Langmuir isotherm, Freundlich, Temkin, Sips and Toth ones being the most applicable in bioremediation processes using macrophytes [2, 15, 16, 23].

The adsorption isotherms were fitted to the experimental data of BF-4B reactive red dye obtained under the following conditions: particle size of 0.147 to 0.295 mm, 50°C, pH = 2, agitation speed of 150 rpm, and process time of 45 minutes.

The Langmuir isotherm (see Eq. (4)) was originally developed under the study of gas-solid equilibrium. The assumptions of this model were as follows: the adsorption sites were distributed homogeneously on the surface of the adsorbent, and there was no interaction between its molecules. This isotherm was applied only for a formed monolayer where each site was occupied by only one molecule of adsorbate. The molecules adsorbed on the surface of the adsorbent sites did not interact between them, and the energy of each site was equally distributed.

$$q_{eq} = q_{\max}\left(\frac{b_L C_{eq}}{1 + b_L C_{eq}}\right), \tag{4}$$

where q_{eq} is the adsorbed amount of dye per amount of biomass at the equilibrium; $q_{\max}$ is the maximum adsorption capacity of the dye per amount of biomass at the equilibrium conditions; b_L is the ratio of the rates of adsorption and desorption; C_{eq} is the remaining dye concentration in the solution.

The Freundlich isotherm, described by Eq. (5), was initially proposed as an empirical model, but years later was adopted as a logarithmic distribution of active sites, commonly used when the adsorbate molecules did not interact. This model does not describe the saturation of the adsorbent sites; hence, it can be used only inside the concentration range of adsorbate for which the parameters have been adjusted.

$$q_{eq} = k_F \left(C_{eq}\right)^n, \tag{5}$$

where q_{eq} is the amount of adsorbed dye per amount of biomass under the equilibrium conditions; k_F is a dimensionless constant related to the capacity of adsorption; C_{eq} is the remaining dye concentration in the solution; n is a dimensionless constant related to the strength of adsorption.

Temkin model, described by Eq. (6), considers the effects of indirect interactions between adsorbate molecules and the decrease of calories of adsorption with an increase of the removal rate. This is true under the assumption that the heat of adsorption of molecules in the layer decreases linearly with the filling of the adsorbent surface.

$$q_{eq} = \frac{RT}{b_T}\ln\left(a_T C_{eq}\right), \tag{6}$$

where q_{eq} is the amount of adsorbed dye per amount of biomass under the equilibrium conditions; C_{eq} is the remaining dye concentration in the solution at equilibrium; b_T is Temkin's constant related to the adsorption heat (kJ/mol); a_T is a Temkin equilibrium constant (l/g); R stands for the gas constant (0.00813 kJ/mol/K); T stands for the absolute temperature (K).

The Toth isotherm (see Eq. (7)) was applied in heterogeneous adsorption processes. It was derived from the potential theory and assumed a Gaussian distribution of energy, where most of the sites presented adsorption energy below the maximum energy of adsorption. The exponent of this model evaluated the experimental equilibrium data obtained for non homogeneous adsorbents.

$$q_{eq} = q_{\max}\left(\frac{b_T C_{eq}}{\left(1+\left(b_T C_{eq}\right)^{nT}\right)^{\frac{1}{nT}}}\right), \tag{7}$$

where q_{eq} is the amount of dye adsorbed per amount of biomass at equilibrium state; $q_{\max}$ stands for the maximum adsorption capacity of the dye per amount of biomass at equilibrium; b_T is a Toth's constant; C_{eq} is a remaining dye concentration in the solution at equilibrium state; nT stands for parameter heterogeneity.

The isotherm of Sips, also known as Langmuir-Freundlich, has the advantages of both models. The exponent in this model described the affinity distribution on the adsorbent surface. Analogously to Temkin's model, when the exponent tends to a value closer to 0 (zero), the model indicates the system is heterogenous, and when the value tends to unit value (one) this can be interpreted as homogeneous system (Langmuir model). Eq. (8) represents the model Sips:

$$q_{eq} = q_{\max}\left(\frac{\left(b_S C_{eq}\right)^m}{1+\left(b_S C_{eq}\right)^m}\right), \tag{8}$$

where q_{eq} is the amount of dye adsorbed per amount of biomass at equilibrium state; $q_{\max}$ is the maximum adsorption capacity of the dye per amount of biomass at equilibrium state; b_S is a constant of Slip's model; m is a parameter of heterogenity; C_{eq} is the remaining dye concentration in solution at equilibrium.

The Langmuir isotherm, Freundlich, Toth, Sips, and Temkin, represented by Eqs. (4)-(8), were fitted to experimental data of biosorption of BF-4B reactive red dye by using the biomass of macrophyte *E. crassipes* under the equilibrium conditions. The parameters of each isotherm, shown in Table 1, were estimated by using the software Origin ® version 8.0.

The experimental data on equilibrium adsorption of BF-4B reactive red dye by *E. crassipes* and model simulations at pH = 2, 50°C, and 150 rpm are shown in Fig. 3. The Langmuir isotherm showed the best fit to the equilibrium data ($r^2 = 0.9932$). The value obtained for the biosorption capacity ($q_{\max}$) was 20.38 mg/g with a ratio between the rates of sorption and desorption of 0.13 g/l. These results suggest that the adsorption of BF-4B reactive red dye occurs in heterogeneous monolayer surface. Similar results are reported by Módenes et al. (2011) [13] where the aquatic macrophyte *Egeria densa* was used for removal of blue 5G reactive dye. The authors found that the Langmuir model best described the experimental data, where the value for the biosorption capacity was of 29.12 mg/g, and the ratio of the rates of sorption and desorption was of 0.13 g/l, respectively. Vasques et al. (2011) [26] studied residual textile sludge as adsorbent, and they obtained values for maximum biosorption capacity (q_{max}) of RO16 dyes, RR2, and RR141 of 81.30, 53.48, and 78.74 mg/g, respectively.

Table 1. Modelling parameter values of five isotherms applied to BF-4B reactive red dye adsorption data obtained at pH = 2, process temperature of 50°C, 150 rpm orbital agitation, by using the aquatic microphyte *E. crassipes* biomass and under the equilibrium condition

Isotherm	Parameter	Value
Langmuir	q_{max} (mg/g)	20.38 ± 1.21
	b_L	0.39 ± 0.04
	r^2	0.9932
Freundlich	k_F	6.02 ± 0.37
	n	0.39 ± 0.04
	r^2	0.9287
Toth	q_{max} (mg/g)	21.04 ± 3.21
	b_T	0.406 ± 0.072
	nT	0.93 ± 0.30
	r^2	0.9769
Sips	q_{max} (mg/g)	20.60 ± 2.41
	b_S	0.38 ± 0.12
	m	0.98 ± 0.17
	r^2	0.9768
Temkin	b_T (kJ/mol)	4.10 ± 0.26
	a_T	4.27 ± 0.50
	r^2	0.9607

Based on the highly favorable characteristics of BF-4B red reactive dye biosorption process such as short process time for reaching the equilibrium state, high removal rate, and high adsorption capacity, the macrophyte *Eicchornia crassipes* can be considered as a very promising material to be used as a biosorbent in waste water treatment systems.

Conclusion

In this study, we evaluated the ability of BF-4B reactive red dye removal by using *E. crassipes*, aiming to use it as an alternative biosorbent in industrial effluent treatment systems. Analysis of the results of preliminary tests revealed the following conclusions: the higher agitation rate resulted in maximum removal of dye; the adsorption temperature does not significantly influence the rate of dye removal; removal of dye is inversely proportional to the adsorbent size under the acidic conditions (more specifically at pH = 2). From the tests, the authors found that the highest removal percentage was obtained with the following values of process parameters: pH = 2, 50°C, 150 rpm.

Analysis of kinetic test results verified that the adsorption occurred rapidly, reaching equilibrium in about 45 min, and the kinetic model of pseudo-second order was the best fit to the experimental data. Evaluated values of the rate constant of 0.0102 g/mg/min and the amount dye adsorbed at equilibrium 12.5 mg/g were achieved. From the data obtained in the study of equilibrium, it was found that the Langmuir isotherm was the best fit to the experimental data, where the maximum capacity of biosorption (q_{max}) was of 20.38 mg/g, and a ratio of rates biosorption and desorption was of 0.13 g/l, respectively.

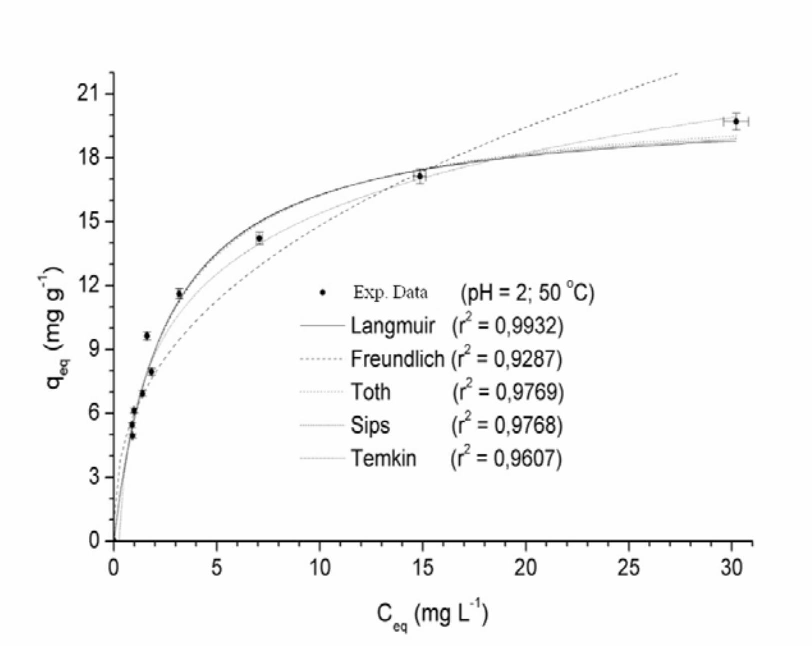

Fig. 3 Simulation results of all applied isotherms for description of experimental data of biosorption process of the BF-4B reactive red dye by using *E. crassipes* biomass at equilibrium and under the following values of parameters: pH = 2, 50°C, and 150 rpm

Finally, the macrophyte biomass of *E. crassipes* specie has shown low process time to achieve equilibrium state, as well as a good removal capacity and natural availability in large amounts in various Brazilian regions. Thus, the chosen specie has shown attractive properties to be used as an efficient biosorbent in bioremediation systems.

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