Original Article

An Investigation of the Efficacy of Cuttlefish Bone Powder in the Removal of Reactive Blue 19 Dye from Aqueous Solutions: Equilibrium and Isotherm Studies

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Received: 8/18/2012 Accepted: 10/8/2012

Abstract

Introduction: Discharge of textile wastewater causes the reduction of sunlight penetration, interferes with the receiving waters ecology and damage the environment. The aim of this study was to determine the removal efficiency of reactive blue 19 dye from aqueous solutions by cuttlefish bone powder.

Materials & Methods: This study was performed experimentally and in laboratory scale. In this research, the effect of adsorbent dose, contact time, initial dye concentration and pH was evaluated. Dye concentration in unknown samples was determined by UV/Vis spectrophotometer. In order to better understand the adsorption process, the experimental data were analysed with Langmuir and Freundlich isotherm models.

Results: Results showed that increasing the adsorbent dose up to 0.4 gr/100ml and increasing of contact time, led to an increase in the efficiency of dye removal. Increasing the initial pH had no effect on the adsorption efficiency and increasing the initial concentration of dye decreased the removal efficiency. The Removal efficacy of the dye was found to be 60%, 45%, 37.5% and 31.9% at the time interval of 3h and the initial dye concentrations of 25, 50, 75 and 100 mg/l, respectively. The experimental data were in good concordance with Langmuir isotherm model (R^2=0.993).

Conclusion: Cuttlefish bone powder is a natural and inexpensive adsorbent that can be used for the removal of environmental contaminations. The adsorption process is affected by sorbent dose, initial dye concentration and contact time but pH had no significant effect on removal efficiency.

***Keyword*:** Adsorption; Remazol Brilliant Blue R; Ecosystem; Water Pollution; Solutions/isolation and purification

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Introduction

The textile industry plays an important role in the economy of many countries. Dyeing is one of the most important production stages of textile fiber processing that depending on the degree of fixation of the dyestuffs on the fiber produces some colored wastewaters. The rate of dye material fixation depends on the nature of the fiber, the intensity of coloration and the application method [1, 2]. The wastewaters discharged from textile industries and dyeing processes are of low BOD and high COD. The discharge of colored wastewaters into receiving waters is toxic to aquatic life [1, 3].

Color is one of the most important hazardous species found in industrial effluents, which needs to be treated [4].

The colors disturb the biological activities in water bodies and are toxic, mutagenic and carcinogenic. They can threaten aquatic life by causing disturbances in the function of kidneys, reproductive system, brain and central nervous system [1, 3]. The industrial effluents containing dyes need to be treated before being released into the environment[5].

The treatment of colored wastewaters is one of the most important environmental issues. There are a number of methods for dye removals which include physicochemical and biological methods[6-10]. In each of these methods, the ability to remove color and the initial cost and operation are different[9,10].

Current studies show that the adsorption process is one of the most promising techniques for the removal of contaminants [11]. If properly designed, the adsorption process can produce effluent with proper quality [12]. The advantages of adsorption process, compared to other separation processes, are its simplicity and flexibility in design and operation. This process is inexpensive and can produce a free contaminant effluent for reuse [12, 13].

Although the activated carbon is one of the most widely studied adsorbents for the control of environmental pollution, the main disadvantage of activated carbon is its high production and treatment costs. In addition to cost, adsorption characteristics and availability are also from basic parameters in adsorbent selection. Thus, many researchers throughout the world have focused their efforts on optimizing the adsorption and developing novel alternative adsorbents with high absorptive capacity and low cost [12, 14]. Studies have shown that sorbents include clay materials (bentonite), zeolites, siliceous material, solid wastes, chitosan, rice hull, eggshell can be used as adsorbents, which could effectively remove the dyes from wastewaters [15-18].

Cuttlefish bone is rich in calcium and its powder is used in the toothpaste preparation. Cuttlefish bone powder has been used only once for the removal of fluoride from water. In Ben Nasr et al. study (2011), efficiency of cuttle fish bone in removing fluoride from water was found to be 80% at pH =7.2, 1h contact time, 15g/l adsorbent dose and 5mg/l initial fluoride concentration [19].

The objective of the present study was to evaluate the feasibility of cuttlefish bone powder for the removal of reactive blue 19 dye from aqueous solutions. Langmuir and Freundlich isotherm models were used for describing the relationship between the amount of adsorbed dye and adsorbent in solution.

Material & Methods

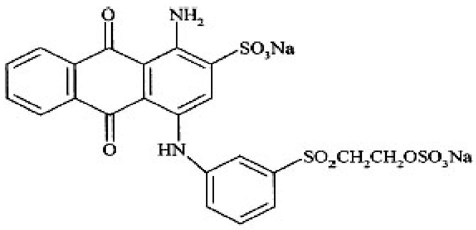
Chemicals and materials

Reactive blue 19 dyes (RB19) were obtained commercially from Dye Star Company. Some of the important physicochemical properties of RB19 are stated in Table 1 [20, 21].

The stock solution was prepared by dissolving 100 mg of RB19 in 1L distilled water. For the present analyses, the dye solutions with different concentrations were prepared by diluting the stock solution in distilled water.

The maximum absorption wavelength of the dye (λ_{max}) was determined by absorbance spectrum detection between 200 nm to 800 nm using the UV/Vis spectrophotometer (model of SP-3000 Plus). The curve of absorption plaines, λ_{max} of RB19 is 592 nm. For control of accuracy of spectrophotometer results, calibration curve was drawn every week.

Table 1. Basic properties of the reactive blue 19 dye

Chemical formula	$C_{22}H_{16}O_{11}N_2S_3Na_2$
Commercial name	Remazol Brilliant Blue R
Class	Azo
Molecular weight (g/mol)	626.5
λmax	592
Molecular structure	

Preparation of the Cuttlefish bone powder

The target adsorbent was prepared in the laboratory. Cuttlefish bone was rinsed with deionized water, boiled for 10 min.

To desorb any impurities, it was dried at 103–105 ˚C f or 24 h and allowed to cool in a desiccator [19]. In our study, cuttlefish bone was crushed and pulverized, by standard ASTM sieves (range being 60 to 100 meshes), into 150-250 µm particles and used as an adsorbent in the following experiments[22].

Adsorption experiments

All the adsorption experiments were carried out at 20 °C by batch technique. A 100 ml of dye solution at the initial concentrations of 25, 50, 75 and 100 mg/l was introduced in 250 ml Erlenmeyer's flasks. For adjusting the pH of the solution, sodium hydroxide or sulfuric acid solutions (1N) were used. The pH of the samples was measured using a pH meter (Mi 151). After adjusting the pH of solutions to the desired level, the adsorbent was added to the solution. Then, the solutions were shaken by a mechanical shaker (GFL 137 Innova, England) at the constant agitation speed (120 rpm) for 24 h. Subsequently, in order to separate the adsorbents from the aqueous solutions, all samples were filtered using a 0.45 μm membrane filter paper for measuring the remaining dye concentration in the solutions of UV-Visible spectrophotometer (Optima SP-3000 Plus model, Japan) at the wavelength of 592 nm. The amount of dye adsorption capacity and percentage of the removed dye by adsorbents was calculated using the following relationships:

$$q_e = \frac{V}{M} \times (C_0 - C_e) \qquad (1)$$

$$E = \frac{(C_0 - C)}{C_0} \times 100 \qquad (2)$$

(In equation 2 C is not defined) Where, q_e is equilibrium adsorption capacity (mg/g); V is the volume of dye solution (L); M is the mass of adsorbent (g); C_e, C_o and C are the equilibrium and initial and remaining dye concentrations (mg/l) respectively and E is the removal efficiency[11,23].

Equilibrium and Isotherm studies

Adsorption isotherms are the equations that illustrate the adsorption equilibrium between fluid and solid phases [24]. Adsorption equilibrium is established when the amount of solute being adsorbed onto the adsorbent is equal to the amount being desorbed.

At this point, the equilibrium solution concentration remains constant. To optimize the design of an adsorption system for the adsorption of pollutants, it is important to establish the most appropriate correlation for the equilibrium curves[25].

The present study investigates the Langmuir and Freundlich adsorption isotherms. Adsorption tests were performed for the optimal periods of time and pH levels.

The unlinearized form of the Langmuir isotherm is expressed as follows:

$$q_e = \frac{q_m b C_e}{1 + bC_e} \qquad (3)$$

The linear form of Langmuir adsorption isotherm can be expressed as follows:

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{1}{q_m} C_e \qquad (4)$$

Where q_e is equilibrium adsorption capacity on adsorbent (mg/g), C_e is the equilibrium concentration of the adsorbate ions (mg/L), and q_m and b are Langmuir constants related to maximum adsorption capacity (mg/g) and energy of adsorption (L/mg), respectively.

These constants can be determined from the linear plot of C_e /q_m versus C_e, which has a slope of 1/q_m and an intercept of 1/kq_m. The essential characteristic of the Langmuir isotherm can be expressed by the dimensionless constant called separation coefficient (R_L) and is calculated by the following equation:

$$R_L = \frac{1}{1 + bC_0} \qquad (5)$$

Where b is the Langmuir constant and C_0 is the initial dye concentration. The value of separation factor (R_L) indicates the nature of adsorption process. R_L values indicate the type of isotherm to be irreversible (R_L=0), favourable (0<R_L<1), linear (R_L=1) or unfavourable (R_L>1).

In Freundlich isotherm, the amount of solute adsorbed, (q_e), is related to the equilibrium concentration of solute in solution, (C_e), as follows:

$$q_e = K_f C_e^{\frac{1}{n}} \qquad (6)$$

This expression can be linearized to give the following equation:

$$Lnq_e = Ln K_f + \frac{1}{n} Ln C_e \qquad (7)$$

Where q_e is the equilibrium adsorption capacity of the adsorbent (mg/g), C_e is the equilibrium concentration of the adsorbed dye (mg/l), K_f and n are Freundlich constants, related to the adsorption capacity and intensity, respectively. The Freundlich constants, n and K_f, were obtained from the plot of $\ln q_e$ versus $\ln C_e$ that should give a straight line with a slope of 1/n and the intercept of $\ln K_f$. The 1/n values indicate the type of isotherm to be irreversible(1/n=0), favorable(0<1/n<1), and unfavourable(1/n>1)[25-29].

Results

Effect of adsorbent dose

To determine the effect of adsorbent dose, the initial dye concentration was 25 mg/l, and pH, the shaker speed and contact time were 7, 120 rpm and 24 h, respectively. The various amounts of adsorbent were in the range of 0.2-1 g/100 ml. The effect of adsorbent dose on the RB19 dye removal is shown in Figure 1.

According to the results, increasing the adsorbent dose led to an increase in the percentage removal of RB19. While For 0.2, 0.4, 0.6, 0.8 and 1 grams of the adsorbent dose of 100 ml, the remaining concentration of the dye (removal efficiency) was 5.05 mg/l (79%), 0.88 mg/l (96%), 0.23 mg/l (99%), 0.17 mg/l (99%) and 0.05 mg/l (99%), respectively.

This phenomenon is related to an increase in the number of adsorption site's availability a versus constant rate of pollutant concentration.According to the present results, the optimal and effective adsorbent dose were detected to be 0.4 g/100 ml.

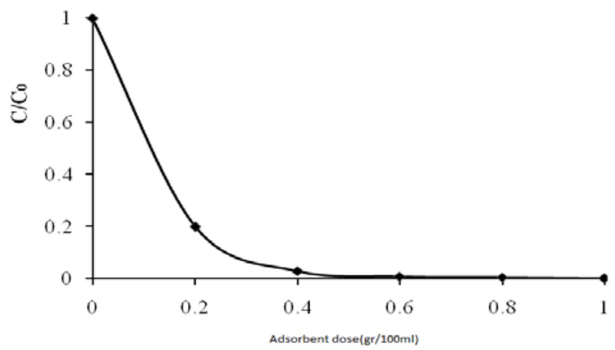


Figure 1. Effect of adsorbent dose on the removal of RB19 dye (dye initial concentration= 25 mg/l and pH=7)

Effect of pH

The pH of aqueous solutions is an important parameter in the adsorption process, which affects both the degree of dye ionization as well as the surface properties of the biosorbent. For investigating the pH, dye solutions were prepared with the initial concentration of 25 mg/l at pH= 3, 5, 7, 9 and 11, in the presence of 0.4 g/100 ml of the adsorbent dose. After the contact time of 24h, the remaining concentration of the dye was determined spectrophotometrically. The effects of initial pH on the removal of RR198 dye by sorption on cuttlefish bone powder are illustrated in Figure 2. According to the results, increasing the initial pH from 3 to 11, because of releasing carbonated compounds from the adsorbent into the reaction environment, had no effect on the adsorption efficiency [19]. The dye removal for pH= 3, 5, 7, 9 and 11 was 97%, 97%, 96%, 96% and 93%, respectively.

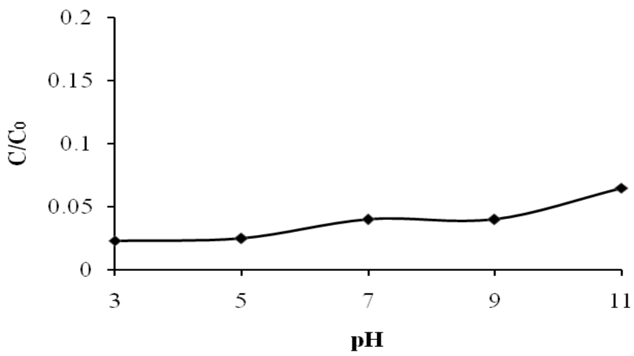


Figure 2. Effect of pH on the removal of RB19 dye (dye initial concentration= 25 mg/l and adsorbent dose=0.4 gr/100ml, contact time=24hr)

Effect of initial dye concentration and contact time

To determine the effect of contact time of adsorption equilibrium on the adsorption of RB 19, the experimental solution was administered with 25, 50, 75 and 100 mg/l concentrations and 0.4g/100 ml adsorbent dose(Figure 3).

At this stage, the environmental pH of the reaction was 7 and the adsorption efficacy was investigated in 30, 60, 90, 120, 150 and 180 min. Results showed that by increasing the initial concentration of the dye, the removal efficacy of RB19 dye decreased but the amount of adsorbed dye per unit weight of the adsorbent (qe) increased. The qe for different concentrations of dye was 6.09, 11.5, 15.62 and 18.09 mg/g, respectively.

Results showed that the removal efficacy of RB19 dye increased by increasing the contact time. Most of the efficacy was recorded at the initial 60 min of reaction.

With the passage of time, the amount of dye disruption was reduced that can be due to a decrease in the solution concentration and the active sites on the adsorbent surface [30]. The removal efficacy of the dye was found to be 60%, 45%, 37.5% and 31.9% at the time interval of 3h for the initial dye concentrations of 25, 50, 75 and 100 mg/l, respectively.

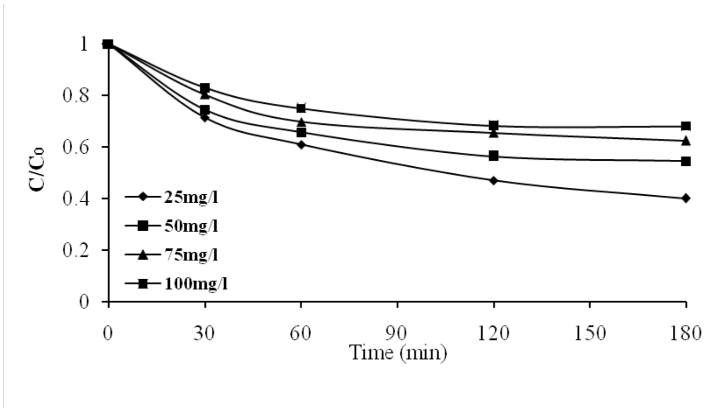


Figure 3. Effect of initial dye concentration and contact time on the removal of RB19 dye (adsorbent dose=0.4 gr/100ml and pH=7)

Adsorption isotherms

Figure 4 shows the Langmuir isotherm for 25 mg/l adsorbent, while the isotherm constants are presented in Table 2. The rates of Langmuir constant (b), adsorption capacity (q_m) and coefficients of Freundlich constant and n were obtained from Linear regression.

Table 2. Characteristics of adsorption isotherms (C_0=25 mg/l, pH=7, contact time=24hr)

Langmuir isotherm		**Freundlich isotherm**	
b (L/mg)	2.63	K(mg/g).(L/mg)$^{1/n}$	5.78
q_{max}(mg/g)	10.86	1/n	0.32
R^2	0.99	R^2	0.96
R_L	0.014		

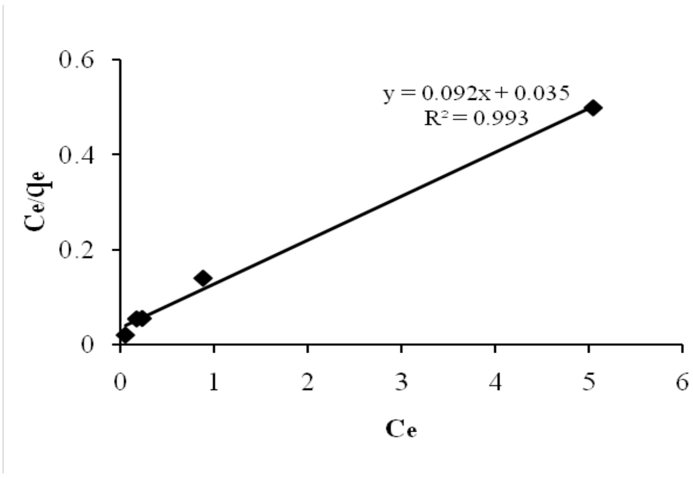


Figure 4. Langmuir isotherm for the adsorption of RB19 dye on different adsorbents (C_0=25 mg/l, pH=7, contact time=24hr)

Discussion

In the stage of the effect of adsorbent dose it was found that the increase in adsorption dose from 0.2 to 0.4 gr/100ml leads to an increase in the removal efficiency from 79% to 96%. While, an increase of adsorption dose from 0.4 to 0.6 gr/100ml and from 0.8 to 0.1 gr/100ml, has a negligible effect on the removal efficiency. Nasr et al. (2011), have investigated the adsorption of fluoride by cuttle fish bone powder and reported that an increase in adsorption with adsorbent dosage can be attributed to the increased adsorbent surface area and availability of more adsorption sites [19].

This result was also obtained by Elkady et al. who studied the removal of RR198 by eggshell biocomposite beads [14] and Moussavi et al. (2009), who studied the degradation and the biodegradability improvement of the RR198 azo dye using catalytic ozonation with MgO nanocrystals [31].

PH is one of the effective factors on adsorption rate and concentration of contaminants. By affecting on the adsorbent surface charge, pH can alter the removal efficiency. According to the results, increasing the initial pH because of releasing carbonate compounds from the adsorbent to reaction environment and change of final pH to about 9-9.5, has no effect on the adsorption efficiency. Cuttlefish bone, when added to water, will produce the following species, HCO3-, CO32-, Ca2+, CaHCO3+ and CaOH+ [19].

According to the findings of this study, the equilibrium adsorption capacity (q_e) increased by increasing the initial concentration of dye from 25 to 100 mg/l. These results showed that the amount of adsorbed dye per unit of the adsorbent is a function of initial concentration of dye, while q_e in higher concentrations is more. These results imply that the removal of RB19 with cuttlefish bone depends on dye concentration and may be related to driving forces that need to be overcome for the resistances of pollutant migration from the aqueous solutions to the biosorbent surface [32, 33]. With time, the amount of dye disruption reduced that this can be due to decreasing of solution concentration and so decreasing of active sites on the surface of adsorbent. Similar data also obtained by Ehrampoush et al. [11] and Renganathan et al. [30]. In the present study, most of the efficacy was observed in the first 60 minutes of reaction.

Based on dimensionless segregation factor (RL) on Langmuir model, the value of this parameter for adsorption of RB19 on cuttlefish bone is between 0 and 1 which confirms that the adsorption of this dye to this sorbent under the conditions of this research is favourable.

According to Freundlich isotherm, 1/n is less than 1, therefore adsorption of this dye to this material is unfavourable. Values of correlation coefficient (R2) are regarded as a measure of the goodness-of-fit of experimental data on the isotherm model. The Langmuir model for initial dye concentration of 25 mg/l had higher agreement with the experimental data. The R^2 value in Langmuir isotherm was higher than 0.99 and near to 1 (R^2=0.993) that indicates a very good fit. According to this isotherm, adsorption of RB19 is a single-layer adsorption on a homogeneous adsorbent surface. In study of Elkady et al. it was elucidated that the Langmuir equation represents the Remazol Red sorption process on the eggshell composite at different solution temperatures very well [14]. Osman Gulnaz et al. In their study found that Langmuir isotherm provides a reasonable description of the experimental data since the linear correlation coefficient of the Langmuir adsorption isotherm model is higher than the value of Freundlich adsorption isotherm model [34].In other researches, Mahmoodi et al. Investigated the kinetics and equilibrium of textile dye adsorption on Pine Cone. Their results showed that the AB26 and AG25 followed Langmuir isotherm and AB7 followed Freundlich isotherm [25].

Conclusion

Cuttlefish bone powder is a natural and inexpensive adsorbent that can be used for the removal of environmental contaminations. The adsorption process is affected by sorbent dose, initial dye concentration and contact time but pH had no significant effect on removal efficiency.

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