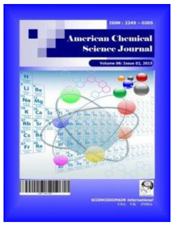

American Chemical Science Journal
12(2): 1-9, 2016, Article no.ACSJ.22821
ISSN: 2249-0205

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Biosorption of *Acid Blue 225* from Aqueous Solution by *Azolla filiculoides*: Kinetic and Equilibrium Studies

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Authors' contributions

This work was carried out in collaboration between all authors. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/ACSJ/2016/22821
Editor(s):
(1) T. P. West, Department of Chemistry, Texas A & M University-Commerce, USA.
Reviewers:
(1) P. N. Palanisamy, Kongu Engineering College, India.
(2) Anonymous, Jawaharlal Nehru Technological University, Anantapur, India.
(3) Luz María Torres Rodríguez, University of San Luis Potosi, Mexico.
Complete Peer review History: http://sciencedomain.org/review-history/12830

Original Research Article

Received 29th October 2015
Accepted 17th December 2015
Published 29th December 2015

ABSTRACT

Batch studies were conducted for kinetics and equilibrium studies on biosorption of Acid *Blue 225* (*AB 225*) dye from aqueous solution by *Azolla filiculoides*. The biomass used as adsorbent in this work was initially characterized by SEM and BET. The effect of parameter including adsorbent dose, initial pH, initial concentration and contact time were investigated. Results show that the pH value of 3 is favorable for the biosorption of dye. The dye adsorption efficiency increased with increase in adsorbent dose and contact time. The equilibrium adsorption data were analyzed using three widely applied isotherms: Langmuir,Freundlich and Tempkin. The results revealed that Langmuir isotherm are well fitted on the experimental results. The maximum dye removal efficiency was obtained to be

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31.66 at dose adsorbent 2.5 g/L and 19.94 at dose adsorbent 5 g/L. Batch kinetic experiments showed that the adsorption followed pseudo-second-order kinetic model with correlation coefficients greater than 0.997.

Keywords: Adsorption; Azolla filiculoides; Acid Blue 225; Isotherm-Kinetic model.

1. INTRODUCTION

The dye substances are usually presented in the effluent of many industries, such as textiles, leather, paper, printing, and cosmetics [1,2]. The complex aromatic structures of dyes make them more stable and more difficult to remove from the effluents discharged into water bodies [3,4]. Therefore, increasing attention is paid to remove the dyes from aqueous solution in the last few years. Various methods including aerobic or anaerobic digestion, coagulation, advanced oxidation processes and adsorption have been developed to remove color from dye-containing effluents which they had various effectiveness, economic cost and environmental impacts [5-7]. Among various treatment technologies, adsorption onto activated carbon has proven to be one of the most effective and reliable physicochemical treatment methodologies. Due to high cost of activated carbon, a lot of alternative adsorbents are developed and used for dye removal from the aqueous solutions [8-10]. The numbers of non-conventional and low-cost agricultural materials are used as adsorbents for removal of pollutants from wastewater [11-15]. New-economical, locally available and high efficient adsorbents are still in the process of development. *Azolla filiculoides* is a floating water fern which it can grow rapidly on the water surface and can form a dense mat; therefore it can lead to many negative effects to aquatic life [16,17]. Therefore, use of *Azolla filiculoides* as a biosorbent for dye removal from the industrial effluent can help to solve both problems including dye removal as well as weeds problem [18,19]. Recently, dried and modified *Azolla filiculoides* has been used as a proper biosorbent for the removing of heavy metal [20,21], phenol compounds [22] and dyes effluent [23,24]. In present work , the potential of *Azolla filiculoides* biomass as adsorbent was evaluated for the biosorption of *acid blue 225 (AB 225)* from aqueous solutions. Also, The effects of biosorbent dosage, contact time, pH and initial dye concentration on the biosorption of *AB 225 dye* onto *Azolla filiculoides* were investigated. Furthermore, the isotherms, kinetics data were evaluated.

2. MATERIALS AND METHODS

2.1 Biosorbent and Chemicals

Azolla filiculoides was collected from Anzali wetland, Iran. It was then sun dried and using a disk mill to obtain material with an average particle size of 1-2 mm. The crushed particles were then treated with 0.1 M HCl for 5 h followed by washing with distilled water and then kept for shaded dry. The resultant biomass was subsequently used in sorption experiments [25].

The specific surface area of *Azolla filiculoides* before use was determined by the BET-N_2 method using an ASAP 2000 apparatus based on nitrogen adsorption–desorption isotherms at 77 K. The morphological features and surface characteristics of dried *Azolla filiculoides* before and after use were examined using an environmental scanning electron microscopy (ESEM) instrument (Philips XL30).

The *AB 225 dye* were obtained from Alvan Sabet Company, Hamadan, Iran and used without further purification. The chemical structures and general data of this dye are displayed in Fig. 1 and Table 1. The dye stock solutions (1000 mg/L) were prepared by dissolving accurately weighted dye in distilled water and the experimental solutions concentrations were obtained by dilution of prepared stock solution.

2.2 Adsorption Experiments

All experiments were carried out with biosorbent samples (1 g/L) at 30℃ in 50 mL beakers in a magnetic stirrer operating at 200 rpm to elucidate the optimum conditions (pH, contact time, biosorbent dosage and initial dye concentration). Prior to analysis of the dye concentration, samples were centrifuged at 4000rpm for solid–liquid separation. The concentration of the dye in solution was analyzed using UV-DR5000. The maximum wavelength (λ_{max}) was determined to be 628 nm. The concentrations of dyes in solution were estimated quantitatively using the linear regression equations obtained by plotting a calibration curve for dye over a range of

Table 1. Properties of *AB 225dye* [26]

C. I. name	Type	Molecular weight (g/mol)	λ_{max} (nm)	Molecular formula
Acid blue 225	Anionic	533.99	628	$C_{26}H_{20}Br_2N_3O_6S$

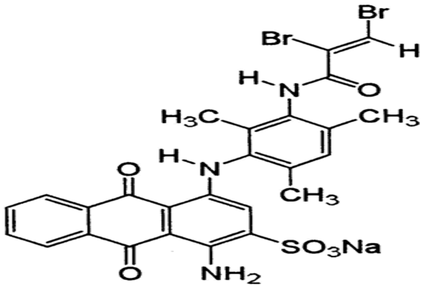

Fig. 1. Molecular structure of *AB 225dye*

concentrations. The dye adsorption capacities of biosorbent were determined at a certain time intervals (10-180 min) and at various biosorbent dosages (1–10 g/L). The effect of pH on biosorption efficiency was studied by adjusting dye solutions (25 mg/L) with different pH values (3–12) and agitated with 5 g/L of biosorbent for 90min. The pH of dye solutions were adjusted by addition of HCl or NaOH (0.1 M). Dye adsorption experiments were also accomplished to obtain isotherms at range of 25–500 mg/L dye concentrations. The amount of dye adsorbed by biosorbent, q_e (mg/g), was calculated by the following mass balance relationship [15]:

$$q_e = (C_0 - C_e)\,V/m$$

Where C_0 and C_e are the initial and equilibrium dye concentrations in solution, respectively (mg/L), V the volume of the solution (L) and m is the mass (g) of the adsorbent used.

3. RESULTS AND DISCUSSION

The specific surface area is related to the number of active adsorption sites of dried *Azolla filiculoides.* The specific surface area of the modified *Azolla filiculoides* was determined in the size of 36 m^2/g.

Dried *Azolla filiculoides* was also examined using environmental scanning electron microscopy before and after use. Fig. 2(a) clearly shows the pore textural structure of dried *Azolla filiculoides* before use. However, as shown in Fig. 2(b), clear pore textural structure is not observed on the surface of dried *Azolla filiculoides* after use which it could be due to either agglomeration on the surface or the incursion of *AB 225 dye* into the pores of dried *Azolla filiculoides*.

3.1 Effect of Contact Time

Fig. 3 shows the effect of contact time on adsorption of *AB 225dye* by *Azolla filiculoides*. The amount of *AB 225dye* adsorbed increased with the increase in contact time. The adsorption rate was greater at the first 45 min and finally equilibrium was established after 90 min. The rapid adsorption observed during the first 45 min was probably due to the abundant availability of active sites on the *Azolla filiculoides* surface. After this time, the adsorption efficiency was decreased with the gradual occupancy of these sites [27,28].

3.2 Effect of pH

The effect of pH on the adsorption of *AB 225* dye onto *Azolla filiculoides* is shown in Fig. 4. It can be seen that the adsorption of *AB 225 dye* was pH dependent. The results show that the amount of adsorbed dye onto *Azolla filiculoides* decreases by increasing of pH from 3 to 11.This can be related to the surface charge of the adsorbent. *Azolla filiculoides* has negatively charged adsorption sites, but it is positively charged at low pH values. Therefore, the electrostatic interactions increased between negatively charged SO_3^- groups in the dye molecule and the positively charged adsorbent [29,30]. As a result, the amount of dye molecules onto the *Azolla filiculoides* increases at lower pH values.

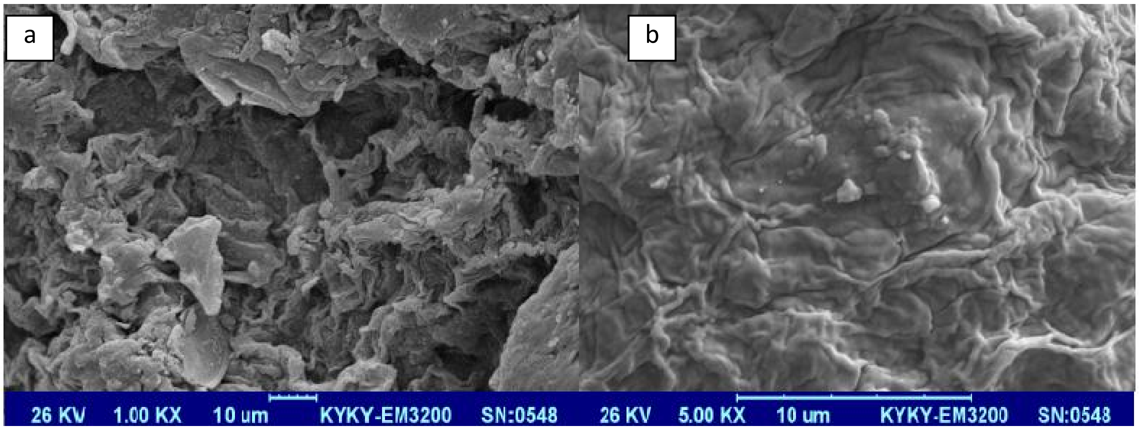

Fig. 2. SEM image of *Azolla filiculoides* a: before used b: after used

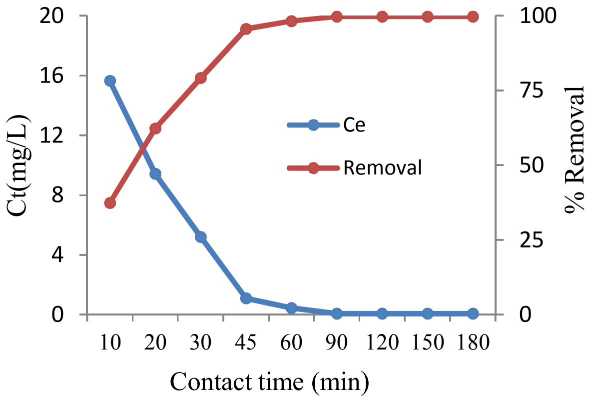

Fig. 3. Effect of contact time for *AB 225 dye* adsorption (initial concentration= 25 mg/L,pH = 3, biomass dose: 5 g/L)

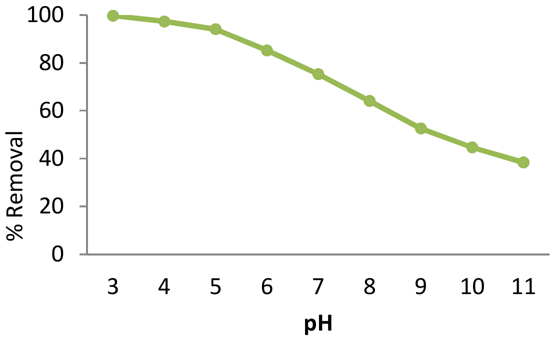

Fig. 4. Effect of pH on adsorption of *AB 225 dye* (initial concentration = 25 mg/L, contact time = 90 min, dose: 5 g/L)

3.3 Effect of Adsorbent Dose

Adsorbent dose is an important parameter in the determination of adsorption capacity. The effect of the adsorbent dose was investigated by addition of various amounts of *Azolla filiculoides* in 50 mL of 100 mg/L *AB 225 dye* aqueous solution for 90 min. The result is shown in Fig. 5. It was observed that the removal efficiency increased from 41.6% to 92.4% with an increase in adsorbent dose from 1 to 5 g/L. This can be due to the increasing of the specific surface area of adsorbent and availability of more adsorption sites [31,32]. However, the further increase in the amount of the adsorbent has not significant affect on removal efficiency. It is also observed that the adsorption capacity decreases from 41.6 to 9.24 mg/g as the adsorbent dose increases from 1 to 5 g/L. Consequently, the adsorbent dose of 5 g/L was used as optimum dosage for further experiments.

3.4 Effect of Dye Initial Concentration

The effect of initial concentration was studied with initial *AB 225 dye* concentrations ranging from 25 to 500. Fig. 6 shows the adsorption capacity and % removal versus initial *AB 225 dye* concentrations. It was clear that the adsorption of dye depends on the concentration of the dye. The adsorption capacity increased from 4.98 to 65.3 mg/g with the increase of *AB 225 dye* concentration from 25 to 500 mg/L, however the % removal decreased from 99.7 to 65.3. This probably occurs due to this fact that by increasing of the surface charge on the adsorbent, the adsorption sites of top surfaces of adsorbent are saturated and the removal efficiency decreased [33,34]. The reason for the rising of the adsorption capacity by increasing of the initial dye concentrations is the increasing of collusion and contact between adsorbent and adsorbate [35].

3.5 Adsorption Isotherms

Adsorption isotherm models are widely used to describe the adsorption process and investigate the mechanisms of adsorption. The equilibrium data was analyzed by the Langmuir, Freundlich and Tempkin isotherm models. The isotherm adsorption study was conducted for two adsorbent doses (2 and 2.5 g/L) at the pH=3 and initial dye concentration of 100 mg/L at contact time 10-180 min.

The linear form of Langmuir equation can be written as follows [36]:

$$\frac{Ce}{qe} = \frac{1}{q_m K_L} + \frac{Ce}{q_m}$$

Where C_e (mg/L) is the concentration of *AB 225 dye* at equilibrium, q_e(mg/g) is the amount of *AB 225 dye* adsorbed by the *Azolla filiculoides* at equilibrium, q_m (mg/g) is the maximum adsorption capacity corresponding to monolayer coverage, and K_L (L/mg) is the Langmuir constant. The values of q_m and K_L can be calculatedfrom plotting C_e/q_e versus C_e. In order to determine if thedsorption process is favorable or unfavorable, a dimensionlessconstant separation factor or equilibrium parameter R_L, isdefined according to the following equation [37]:

$$R_L = \frac{1}{1 + K_L C_0}$$

Where K_L (L/mg) is the Langmuir constant and C_0 (mg/L) is theinitial AB 225 dye concentration. The R_L value indicates adsorption process is irreversible for R_L equal with 0; favorable for R_L between 0 and 1; linear for R_L equal with 1; and unfavorable for R_L greater than 1.

The Langmuir plots for *AB 225 dye* adsorption onto *Azolla filiculoides* biomass are depicted in Fig. 7, and the parameters are shown in Table 2. The all of correlation coefficients of the isotherms are higher than 0.994 at the twodose adsorbent and it indicates that the Langmuir isotherm fits the equilibrium data very well.The maximum monolayer adsorption capacities of dried *Azolla filiculoides* are 31.66 mg/g at 2.5 g/L and 19.94 mg/g at 5 g/L of dose adsorbent. The whole calculated values of R_L are in the range of 0.17–0.81,thereby confirming that the three adsorption processes are favorable.

The linear form of Freundlich equation is given as [38]:

$$\text{Log } q_e = \frac{1}{n} \log Ce + \log K_F$$

Where q_e is the *AB 225 dye* concentration on *Azolla filiculoides* at equilibrium, C_e (mg/L) is the concentration of *AB 225 dye* in solution at equilibrium, K_F and 1/n are Freundlich constants related toadsorption capacity and adsorption intensity, respectively. Higher value for K_F indicates higher affinity for adsorbate and the values of the empirical parameter 1/n lie between $0 < 1/n < 1$, indicating favorable adsorption. Freundlich constants are calculated and are given in Table 2. The correlation coefficients were low and it can be said that the experimental data was not best fitted to the Freundlich isotherm model.

The Tempkin isotherm model suggests an equal distribution of binding energies over the number of the exchanging sites on thesurface. The linear form of Tempkin isotherm can be written as [39]:

$$q_e = B\ln(A) + B\ln(C_e)$$

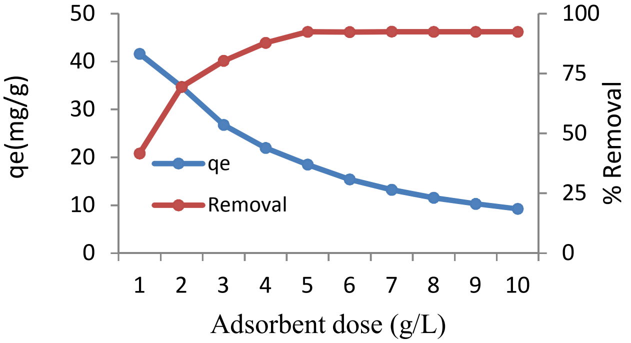

Fig. 5. Effect of adsorbent dose (contact time = 90 min, pH = 3, initial concentration: 100 mg/L)

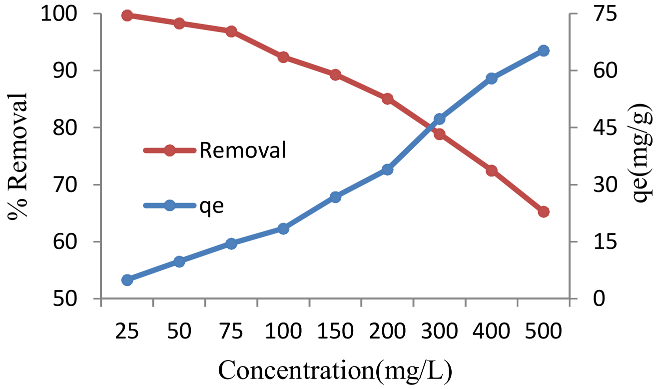

Fig. 6. Effect of initial concentration (contact time = 90 min, Biomass dose:5 gr/L,pH = 3)

Where B=RT/b, T is the absolute temperature in Kelvin and R is the universal gas constant (8.314 J/mol K). A is the equilibrium binding constant and B is corresponding to the heat of sorption. The results of the isotherm parameters/constants are displayed in Table 2. The correlation coefficients were low and it can be said that the experimental data was not fitted better to the Tempkin isotherm model.

3.6 Adsorption Kinetics

In order to examine the mechanism and rate-controlling step in the overall adsorption process, three kinetic models including pseudo-first-order, pseudo-second-order and intra-particle diffusion are adopted to investigate the adsorption process.

The pseudo-first-order equation can be expressed as thefollowing equation [40]:

$$\log (q_e - q_t) = \log q_e - k_1 t / 2.3$$

Where q_e and q_t (mg/g) are the *AB 225 dye* adsorption capacity at equilibrium and at time t (min), respectively, and k_1 (1/min) is the rate constant of the pseudo-first-order.The parameters are given in Table 3. The values of the correlation coefficient obtained at all the studied concentrations are low and in the range0.74–0.82. This suggests that the pseudo-first-order kinetic model is not suitable to describe the adsorption process.

The pseudo-second-order kinetic modelcan be expressed in linear form as follows [41]:

$$t/q_t = 1/k_2 q_e^2 + 1/q_e t$$

where k_2 (g/mg min) is the rate constant of the pseudo second order.The parameters are listed in Table 3. The values of the correlation coefficient are higher than 0.998 and they suggest that adsorption of *AB 225 dye* onto *Azolla filiculoides* has predominantly followed the pseudo-second-order kineticmodel.

Table 2. The adsorption isotherms constants for the removal *AB 225 dye*

	Langmuir model				Freundlich model			Tempkin model		
Dose (g/L)	**$q_{e\,exp}$ (mg/g)**	**q_m (mg/g)**	**K_L(L/mg)**	**R^2**	**n**	**K_F(L/g)**	**R^2**	**B**	**A(L/g)**	**R^2**
2.5	32.12	31.66	0.037	0.994	3.25	6.81	0.894	27.12	0.41	0.832
5	19.65	19.94	0.062	0.995	2.74	7.45	0.925	35.24	0.58	0.848

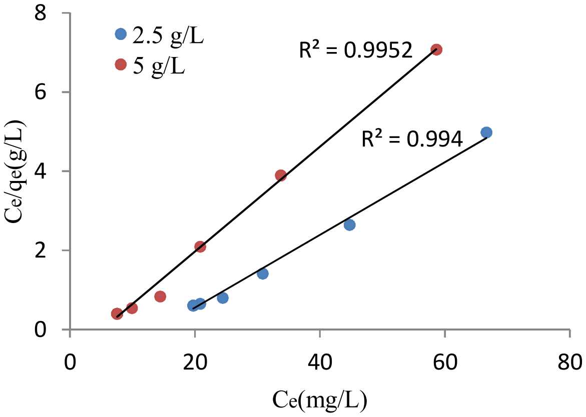

Fig. 7. Langmuir plots for the *AB 225 dye* adsorption onto *Azolla filiculoides* biomass

Table 3. Kinetic parameters for *AB 225 dye* adsorption onto *Azolla filiculoides*

	Pseudo second-order model				Pseudo first-order model			Intraparticle diffusion		
C_o (mg/L)	**$q_{e\,exp}$ (mg/g)**	**k_2 (g/mg min)**	**R^2**	**q_e (mg/g)**	**K_1 (1/min)**	**R^2**	**q_e (mg/g)**	**K (mg/gmin$^{1/2}$)**	**C**	**R^2**
25	4.98	0.045	0.999	5.61	0.141	0.873	3.84	2.07	2.14	0.847
100	19.65	0.081	0.998	22.44	0.245	0.864	12.45	4.18	1.39	0.835
500	65.3	0.112	0.999	72.17	0.468	0.881	45.17	9.71	3.65	0.812

The intraparticle diffusion equation is written as follows [42]:

$$q_t = K\,t^{0.5} + C$$

Where C is the intercept which describes the foundry layer thickness and K (mg/g min$^{1/2}$) is the rate constant of intraparticle diffusion. Based on the results, the values of coeffieient correlation which is presented in Table 3 are also low. From these results, it can conclude that the biosorption process of AB 225 dye onto the dried *Azolla filiculoides* is not only depended on intraparticle diffusion but other mechanisms might be involved. Therefore, the data is not well fitted on the intraparticle diffusion model.

4. CONCLUSIONS

The results indicated that *AB 225 dye* adsorption by *Azolla filiculoides* was strongly dependent on adsorbent dose, initial pH andcontact time. Low initial concentration and pH favor the *AB 225 dye* adsorption on the *Azolla filiculoides*. The isotherm study indicates that adsorption data fit well with the Langmuir models. $R_L$values from Langmuir model indicate that the removal of *AB 225 dye* on the *Azolla filiculoides* is favorable. The kinetic study at different initial concentrations reveals that the pseudo-second-order model yields better fit than that of the pseudo-first-order model and intraparticle diffusion. Based on these results, it is concluded that *Azolla filiculoides* could be used as a low-cost and relatively effective adsorbent for the removal of *AB 225dye* from wastewater.

ACKNOWLEDGEMENTS

The authors would like to express their gratitude toward the Kurdistan University of Medical Sciences for funding this research.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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