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Factors controlling the adsorption of acid blue 113 dye from aqueous solution by dried *C. edulis* plant as natural adsorbent

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Abstract Contamination of surface water and groundwater by organic pollutants is a serious problem due to their persistence, bioaccumulation and biomagnification through food webs. Since the removal of dyes from wastewater is considered an environmental challenge and government legislation requires textile wastewater to be treated, therefore there is a constant need to have an effective process that can efficiently remove these dyes. The aim of the present study is to evaluate the potentiality of dried *Carpobrotus edulis* plant as low-cost adsorbent for the removal of the industrial acid blue 113 dye from aqueous solutions using the batch equilibration technique. The effects of different physicochemical parameters such as adsorbent dose, contact time, initial dye concentration, solution pH and temperature on adsorption rate of anionic AB113 dye on microparticles of dried *C. edulis* plant were investigated. The experimental data were analyzed by using mathematical models to determine the thermodynamic parameters. The negative values of free energy change indicated the spontaneous nature of the adsorption and negative value of enthalpy change suggested the exothermic nature of the adsorption process. These results indicate that dried *C. edulis* plant as an environmentally friendly adsorbent could be potentially used for the removal of anionic dyes from aqueous solutions.

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Introduction

Contamination of surface water and groundwater by organic and inorganic pollutants is a serious global problem due to their persistence, bio-accumulation and bio-magnification through food webs. Dyes are widely used in textile, paper, rubber, plastic, leather, cosmetic, and pharmaceutical and food industries. The discharge of these dyes in to effluents even at very low concentrations from these industries into natural streams has caused many significant problems such as increasing toxicity and chemical oxygen demand of the effluent, and also reducing light penetration, which has a derogatory effect on photosynthetic phenomena. Dyes are mutagenic, toxic, allergenic, carcinogenic, non-degradable, stable towards light and oxidizing agents and resistant to aerobic digestion thereby creating serious problem (Golka et al. 2004; Christie 2007; Das et al. 2009; Ong et al. 2010; Chiban 2011). Anionic dyes includes many compounds from the most varied classes of dyes, which exhibit characteristic differences in structure (e.g. azoic, anthraquinone, triphenylmethan and nitro dyes) but possess as a common feature, water-solubilizing, ionic substituents. Acid dyes are used with silk, wool, polyamide, modified acrylic and polypropylene fibres. Acid dyes have good water solubility, and they have a harmful effect on human beings since they are organic sulphonic acids (Attia et al. 2006; Gupta and Suhas 2009).

Many studies concerning the remove of anionic dyes from water as well as industrial effluents were reported such as membrane techniques, ozonation, coagulation and filtration, advanced processes of oxidation and adsorption (Gupta and Suhas 2009; Gupta et al. 2016; Akar et al. 2006; Chiban

2007). However, these methods have several disadvantages which include incomplete pollutant removal and the high cost and the disposal of the residual metal sludge. Adsorption is an effective technique used in the water and wastewater treatment; the granular or powdered activated carbon is the most widely used adsorbent (Gupta and Suhas 2009), but their use is usually limited due to their high cost. That is why an increasing attention and this problem has led many workers to search for cheaper adsorption substitutes including peat, plum kernels, wood, coal, resin, coir pith, chitosan fibres, pine cone, mango seed, sepiolite, bamboo and various industrial wastes (Dávila-Jiménez et al. 2009; Mui et al. 2010; Mahmoodi et al. 2011; Tabak et al. 2009; Gupta and Suhas 2009; Errais et al. 2011), due to all their local abundance and low cost. There are large numbers of studies in the literature in which various adsorbents are used for removal of arsenic from aqueous solution. The review reported by Gupta and Suhas (2009) can be referred for the other possible adsorbents for the removal of anionic dyes. Although these materials are regarded as cheap and effective adsorbents, there are several problems (their impurities, unknown stability and regeneration, low adsorption capacity and slow kinetics) associated with their use. For the past few years, the focus of the research is to use cheap materials as potential adsorbents, and the processes developed so far are based on exploring those natural adsorbent, which can prove economic and bring cost effectiveness (Mohan and Pittman 2007; Gupta and Suhas 2009; Gupta et al. 2011; Chiban et al. 2012a, 2013; Shirzad-Siboni et al. 2014; Valliammai et al. 2015; Lee et al. 2015).

In the present study, we aimed to evaluate the potentiality of dried *Carpobrotus edulis* plant as low-cost adsorbent for the removal of acid blue 113 from aqueous solutions using the batch equilibration technique. The effects of adsorbent dose, contact time, initial dye concentration, solution pH and temperature on adsorption rate of anionic dye on microparticles of dried *C. edulis* plant were investigated. The experimental data were analyzed using mathematical models.

Materials and methods

Adsorbent preparation

The adsorbent used in this study was obtained from dried plant of *C. edulis* which is a Mediteranean plant. The microparticles of *C. edulis* plant were dried in oven at 35 °C during 24 h and crushed with an electric grinder to get fine powders. The obtained microparticles (<250 μm) were used as bioadsorbent material in batch experiments without any other pretreatment.

A detailed characterization of *C. edulis* plant adsorbent has been done by BET at 77 K adsorption, scanning electron microscopy, energy dispersive X-ray spectroscopy, Fourier transform infrared spectroscopy and phytochemical screening as it was described in our previous reports (Chiban et al. 2007, 2011b). The result has shown that the major functional groups on *C. edulis* plant are polar hydroxyl, aldehydic and carboxylic groups, which could be responsible to pollutants removal from aqueous solutions.

Preparation of dye solutions

Synthetic dye solution of acid blue 113 (AB113) was the sorbate used in this study, and the anionic dye was used without further purification. Properties of AB113 dye are presented in Table 1. The synthetic dye used in this study was purchased from Sigma-Aldrich. A stock solution of 500 mg l^{-1} in anionic dye was prepared by dissolving accurately weighed quantity of AB113 dye in double distilled water. All the compounds used to prepare reagent solutions were analytic reagent grade and all experiments employed double distilled water.
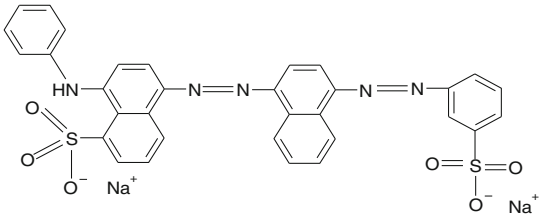
Batch adsorption experiments

The adsorption of acid blue 113 was performed by batch technique at room temperature. Well established amounts of dried *C. edulis* plant were placed in Erlenmeyer glass flasks of 100 ml containing 40 ml of dye solution of known concentration and pH. The solutions were vigorously stirred for a given time period to reach equilibrium. The agitation speed was kept constant for each run to ensure equal mixing. After different contact times (T_c), the resulting solutions were centrifuged at 5000 rpm for 10 min, and the supernatant was filtered through a 0.45 μm membrane filter, and the filtrate was analyzed. All experiments were conducted in duplicates, and the results are reported as average (error ±1–2 % for percentage removal and ±0.005–0.01 mg/g for amount adsorbed).

Adsorption equilibrium studies were conducted at initial dye solution pH of ~6.3. pH was not fixed but measured before and after each experiment. Equilibrium data were obtained by adding 0.1–1 g of dried plant into a series of 100 ml Erlenmeyer each filled with 40 ml of AB113 solution at 20 mg/l, and the contact time was fixed to 6 h.

The effect of contact time on the removal of AB113 was studied as follows: A series of 100 ml Erlenmeyer each containing 0.8 g adsorbent and 40 ml of acid blue 113 solution of different concentrations were shaken with varying contact time at room temperature (23 ± 2 °C) and at natural pH (~6.3).

Adsorption experiments for the effect of initial solution pH were conducted as follows: 0.8 g of adsorbent was suspended in 40 ml of acid blue 113 solutions containing 20 mg/l. The pH of the solution was adjusted to 2–12 by adding dilute solution of hydrochloric acid or sodium hydroxide (0.1 M NaOH or 0.1 M HCl); the agitation time was fixed to 6 h, and the temperature was 23 ± 2 °C.

Table 1 Properties of acid blue 113 dye

Molecular structure	
Molecular formula	$C_{32}H_{21}N_5Na_2O_6S_2$
Molar mass	681.65 g/mol
C.I. number	26360
CAS number	3351-05-1
Type	Anionic
λ_{max}	566 nm

Analysis of AB113 dye

The residual (final) concentration of dye in the filtrate was analyzed by using UV-Vis spectrophotometer (TechComp UV 2300 Spectrophotometer) at a wavelength of 566 nm. The concentration of AB113 removed (C_r) from the aqueous solutions was calculated as the difference between the initial concentration, C_0, and the concentration at different contact time ($C_r = C_0 - C_t$). The amount removed per mass unit of the bioadsorbent at time 't' (q_t, mg/g), was calculated by the following:

$$q_t = (C_0 - C_t) \times \frac{V}{m} \qquad (1)$$

and the removal dye percentage was calculated as follows:

$$\% \text{ adsorption} = \frac{(C_0 - C_t)}{C_0} \times 100\% \qquad (2)$$

where C_0 (mg/l) is the initial concentration of anionic dye in aqueous solutions; C_t (mg/l) is the concentration of anionic dye left in aqueous solutions at time 't'; q_t (mg/g) is the amount removed per mass unit of the bioadsorbent at time 't'; C_e (mg/l) is the equilibrium concentration of anionic dye in aqueous solutions; q_e (mg/g) is the amount removed per mass unit of the bioadsorbent at equilibrium; m (g) is the amount of the bioadsorbent; and V (l) is the working solution volume.

Results and discussion

The adsorption of anionic dyes from aqueous solution onto adsorbent surface can be well described as a reversible reaction under an equilibrium condition established between the two phases. The adsorption rate at which the molecules are removed from the solution onto an adsorbent surface is an important factor for designing treatment plants. Thus, in order to characterize the adsorption process of anionic dye on dried plant microparticles, we have discussed the effect of various parameters such as contact time, initial dye concentration, initial solution pH, solution temperatures and ratio mass/volume of the solution on the removal rate of acid blue 113 onto dried *C. edulis* plant from aqueous solution.

Effect of adsorbent dosage

Adsorbent dosage is an important parameter because this factor determines the capacity of an adsorbent for a given initial concentration of the adsorbate (Benhima et al. 2008; Chiban et al. 2011a; Chiban 2011). The effect of adsorbent dosage on the removal of AB113 at $C_0 = 20$ mg/l was studied, and the results are depicted in Fig. 1. The removal of acid blue 113 increases up to 67 % by increasing amount of the adsorbent from 0.1 to 0.6 g and stays almost constant up to 0.6 g of adsorbent dosage. Thus, increasing the amount of the adsorbent makes a large number of sites available leading to an increase in adsorption. The dye removed remains almost unchanged when the used adsorbent concentration was higher than 20 g/l (0.8 g/40 ml). For adsorbent dosage of 20 g/l, the incremental acid blue 113 removal becomes very low on the adsorbent surface, and the dye concentration comes to equilibrium with each other. For higher adsorbent concentration of 20 g/l, the removal efficiency becomes almost constant for the
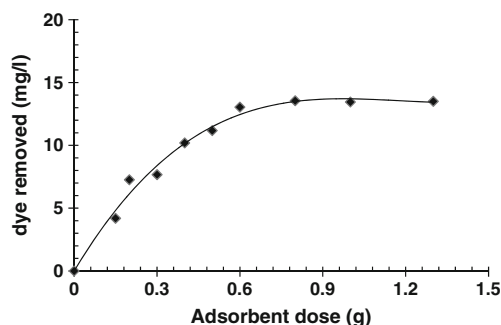

Fig. 1 Effect of *C. edulis* amount on the removal of AB113: $C_i = 20$ mg/l, $T = 23 \pm 2$ °C, pH = 6.3 and $T_c = 6$ h
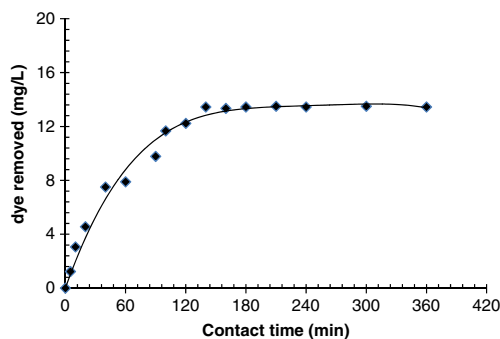
Fig. 2 Effect of contact time on the removal of acid blue onto dried *C. edulis* plant: m/V = 20 g/l, C_i = 20 mg/l, pH_i = 6.3 and T = 23 ± 2 °C

Fig. 4 Effect of initial pH on adsorption of AB113: C_i = 20 mg/l, m/V = 20 g/L, T = 23 ± 2 °C and T_c = 3 h

removal of AB113 onto microparticles of dried plant. Similar behaviour for the effect of adsorbent concentrations on anionic dyes adsorption capacity was observed and discussed in the literature for a variety of adsorbents and dyes (Gupta and Suhas 2009). In order to achieve a maximum adsorption of the acid blue 113, the m/V ratio of 20 g/l (0.8 g/40 ml) was selected for further studies.

Effect of contact time

Equilibrium time is another important operational parameter for an economical wastewater treatment process. Figure 2 indicates the effect of contact time on the batch adsorption of dye solution containing 20 mg/l of acid blue at room temperature (~23 ± 2 °C) and initial pH value 6.3. Percent adsorption of acid blue increases from 6 to ~68 % at contact time of 140 min. This represents the time at which equilibrium of AB113 concentration is presumed to have been attained. The rate of uptake is rapid in the early stages, gradually decreases and becomes constant when equilibrium is attained. This result is important for considering the treatment of wastewaters and the other similar applications. With these results, dried *C. edulis* plant with highest binding sites for dye requires shorter time to achieve a high acid blue removal efficiency. Greater availability of various functional groups on the surface of the dried plant, which are required for interaction with
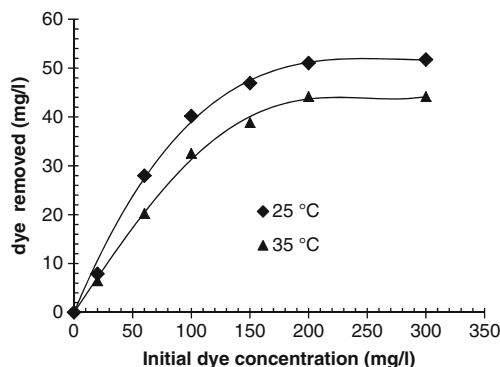
anionic dyes, significantly improved the binding capacity of an adsorbent and adsorption proceeds rapidly. Taking into account these results, the contact time was fixed to 3 h for the rest of the batch experiments. It is revealed that the contact time is an important parameter for the adsorption of acid blue on dried *C. edulis* plant.

Effect of initial dye concentration

In adsorption process, the initial concentration of pollutant in the solution plays a key role as a driving force to overcome the mass transfer resistance between the adsorbent and the adsorbate. Therefore, the amount of AB113 adsorbed was expected to increase as a function of the initial concentration. This was indeed the case as can be seen in AB113 equilibrium values removed at room temperature and pH 6.3 versus the initial concentration. The results are illustrated in Fig. 3. At equilibrium (after 3 h of contact time), the adsorption capacity of dried plant increased with the initial AB113 concentration in the solution which may be due to more availability of acid dyes molecules, and the active sites of the adsorbent have not reached saturation even at initial dye concentrations more than 200 mg/l. This result indicates a great potential application of dried *C. edulis* particles, as an adsorbent, to the treatment of wastewaters containing anionic dyes at high concentrations. The removal percentage decreases, and the

Fig. 3 Effect of initial dye concentration on the removal of AB113: ratio m/V = 20 g/L, pH = 6.3, T_c = 3 h

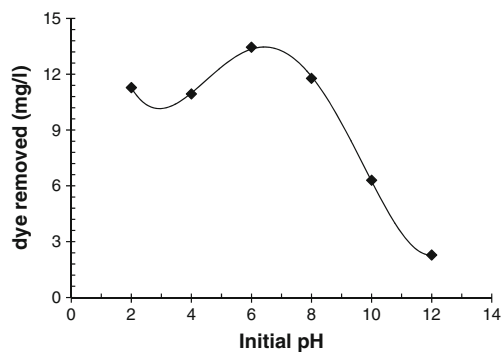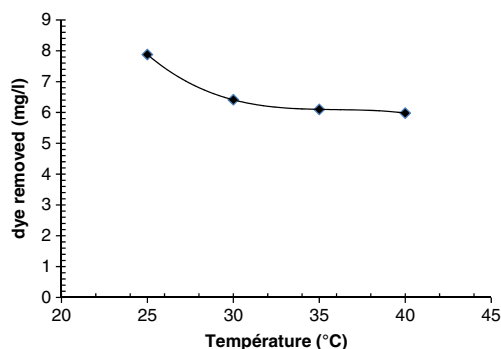**Fig. 5** Effect of temperature on AB113 adsorption by dried *C. edulis* plant: C_i = 20 mg/l, m/V = 20 g/L, pH = 6.3 and T_c = 3 h
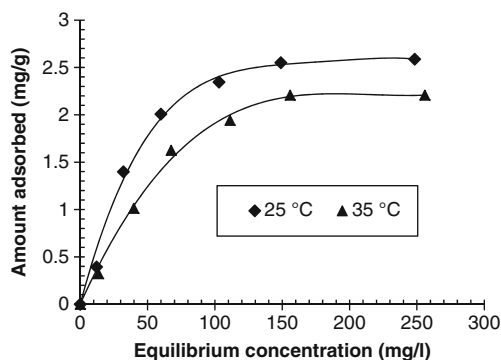
Fig. 6 Adsorption isotherm of AB113 on dried *C. edulis* plant for two temperatures: m/V = 20 g/L, pH = 6.3 and T_c = 3 h

adsorption capacity increases with an increasing in the initial acid dye concentration. The equilibrium adsorption capacity of acid blue 113 from aqueous solution by using dried *C. edulis* plant was found to be 2.6 mg/g.

Effect of initial pH

For anionic dyes adsorption, experiments were carried out by varying initial pH values from 2 to 12. The temperature of the solution was ~23 ± 2 °C, and the contact time fixed at 3 h. The results presented in Fig. 4 indicate that acid blue 113 removal is considerably affected by the initial pH of solution. From these results, the acid blue 113 uptake, first increased slightly from pH 2 to 6 and then after decreased with further increase in the pH up to 12. The further decrease in AB113 uptake with the increasing of the pH (6–12) may be explained as at higher pH values; the surface of the adsorbent may be negatively charged by adsorbing hydroxyl ions (OH⁻) on the surface or by ionization of very weak acidic functional groups of the adsorbent, or both. A repulsive force may develop between the negatively charged surface and the anionic dyes molecules. This result showed that AB113 uptake is lower at higher pH values. Favourable adsorption of AB113 by dried *C. edulis* plant was found to take place at about pH 6 (Fig. 4).

Effect of temperature

The effect of temperature on the adsorption process can be carried out by prepare adsorbent–adsorbate solution with different initial dye concentration then shaken

together until equilibrium time at different temperature (Chiban 2011; Chiban et al. 2012a). Temperature is an indicator for the adsorption nature whether it is an exothermic or endothermic process (Jain et al. 2003, Chiban et al. 2012b). The rates of AB113 dye adsorption were studied in the temperature range of 25 to 40 °C for dried *C. edulis* plant as shown in Fig. 5. It is observed that the adsorption is slower at higher temperatures, and the decrease of adsorption capacity with increasing temperature indicates that the adsorption is an exothermic process. Increasing temperature may decrease the adsorptive forces between the dye molecules and the active sites on the adsorbent surface as a result of decreasing adsorption capacity as found by Ofomaja and Ho (2007). The adsorption data were fitted and found to conform best to the Langmuir equation as follows (Langmuir, 1916):

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_{max}KC_e} \tag{3}$$

where q_e (mg/g) is the amount adsorbed at equilibrium concentration C_e (mg/l), q_{max} is the Langmuir constant related to maximum monolayer capacity and K (l/mg) is the Langmuir constant related to energy of adsorption. Values of q_{max} and K were calculated from the plot of $1/q_e$ against $1/C_e$ at different temperatures (Fig. 6).

The values of maximum adsorption capacity (q_{max}) and equilibrium constant (K) are reported in Table 2. The monolayer capacity (q_{max}) of *C. edulis* adsorbent is comparable to the maximum adsorption observed. As expected, the q_{max} values decrease with increasing temperature. The influence of adsorption isotherm shape to know whether the adsorption is favourable or not, has been discussed in terms of a dimensionless constant R_L, referred to as separation factor (Weber and Chakkravorti 1974), defined as follows:

$$R_L = \frac{1}{1 + K_L C_0} \tag{4}$$

where K (l mol⁻¹) is Langmuir constant and C_0 (mol l⁻¹) is the highest initial anionic dye concentration. According to the value of R_L, the isotherm shape may be interpreted as shown in Table 3 (Bulut et al. 2007). The calculated value of parameter R_L for this study was found to be 0.155, indicating that the

Table 2 Langmuir constants and thermodynamic parameters for AB113 dye adsorption of on dried plant adsorbent at different temperatures

Temperature (°C)	q_{max}		K (l mol⁻¹)	ΔS° (J K⁻¹ mol⁻¹)	ΔG° (Kj mol⁻¹)	ΔH° (Kj mol⁻¹)
	(mg g⁻¹)	(mmol g⁻¹)				
25	8.20	12.02 × 10⁻³	29.72 × 10²	41.13	−19.82	−7.57
35	6.49	9.52 × 10⁻³	26.91 × 10²	38.99	−19.58	

Table 3 Type of isotherm for different values of R_L

Value of R_L	Type of isotherm
$R_L > 1$	Unfavourable
$R_L = 1$	Linear
$0 < R_L < 1$	Favourable
$R_L = 0$	Irreversible

adsorption of AB113 onto dried *C. edulis* particles was favourable.

According to thermodynamics, the Gibbs free energy change ($\Delta G°$) is also related to the enthalpy change ($\Delta H°$) and entropy change ($\Delta S°$) at constant temperature by the following equations:

$$\Delta G° = -RT\, Ln(K_L) \tag{5}$$

$$Ln\left(\frac{K_2}{K_1}\right) = -\frac{\Delta H°}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \tag{6}$$

$$\Delta G° = \Delta H° - T\Delta S° \tag{7}$$

where T is temperature in kelvin, R is the gas constant having value as 8.314 J mol^{-1} K^{-1} and K is the equilibrium constant obtained from Langmuir isotherm. The obtained results for thermodynamic parameters are reported in Table 2. The small negative value of $\Delta H°$ suggests the adsorption to be physical and exothermic process which is consistent with experimental observations. The negative $\Delta G°$ values show spontaneous nature of adsorption process, and the positive value of entropy ($\Delta S°$) revealed that the degrees of free active sites increased at the solid–liquid interface during the adsorption of anionic dye onto dried *C. edulis* plant.

Comparison with published data

The percentage removal of AB113 dye adsorbed onto dried *C. edulis* plant was compared with the percentage removal by other materials reported in the literature. Differences in anionic adsorption capacities are due to the properties of each adsorbent such as structure, polyphenolic groups, functional groups and surface area. For example, Valliammai et al. (2015) examined the adsorption of AB113 dyes from aqueous solution by using activated carbon of Varagu millet husk (*Paspalum scrobiculatum*) and found 80 % AB113 percentage removal after 3 h of contact time. Overripe (*Crocus sativus*) peel as an adsorbent for anionic AB113 dye removal gave a maximum adsorption capacity of 80 % (Lee et al. (2015)). Shirzad-Siboni et al. (2014) used activated red mud for AB113 dye adsorption from aqueous solution and found about 80 % AB113 percentage removal. In another study by Gupta et al. (2011), AB113 removal efficiency varied ranging from 2.19 to 7.84 mg/g for CAC and from 2.28 to 9.72 mg/g for

RTAC depending on the adsorbent type, contact time and the particle size. The results indicated that *C. edulis* natural biosorbent has a great potential to be used in the treatment of dye-contaminated wastewater without any pretreatment.

Conclusion

This study showed that microparticles dried *C. edulis* plant could be used as adsorbents for the treatment of wastewater contaminated with anionic dyes. The adsorption process has been shown to be affected from experimental conditions such as temperature, contact time, adsorbent dose, pH of solution and initial dye concentration. Batch adsorption studies under varying experimental conditions showed that the adsorption of AB113 is fairly rapid in the first 60 min and thereafter increases slowly to reach equilibrium in about 140 min. The maximum AB113 removal was observed at about pH 6. The data obtained from Langmuir adsorption isotherm at different temperatures were used to calculate thermodynamic parameters such as $\Delta G°$, $\Delta H°$ and $\Delta S°$ of adsorption. A negative value of the standard enthalpy change suggests that the interaction of AB113 adsorbed by plant is of exothermic nature. The negative values of the standard free energy changes indicate that the adsorption reaction is a spontaneous process. The natural materials used in this work are suitable candidates as adsorbents in anionic dye removal techniques, considering that these adsorbents are naturally abundant and relatively low-cost materials.

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