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Removal of Acid Red 18 (Azo-Dye) from Aqueous Solution by Adsorption onto Activated Charcoal Prepared from Almond Shell

ABSTRACT

One of the most important environmental pollutants is azo dyes in textile wastewater. In this study, the removal of azo dye Acid Red 18 (AR 18) with initial concentration of 25-100 mg L^{-1} in aqueous solution by the adsorption process onto activated charcoal prepared from almond shell (AC-AS) was investigated. The effects of initial pH (2-12), contact time (15-120 min), adsorbent (0.2-2 g L^{-1}) and initial concentration of AR 18 (25-100 mg L^{-1}) on the removal efficiency of AR 18 solution were investigated. All experiments were performed on a synthetic wastewater. The efficiency of dye removal and Freundlich and Langmuir adsorption isotherms were compared as a function of decolorization and adsorption and kinetic behavior of AR 18. The maximum removal efficiency of AR 18 (i.e., 90.83%) was occurring with pH 2, contact time of 60 min, initial dye concentration of 75 mg L^{-1} and the adsorbent dose of 0.8 g L^{-1}. The removal of AR 18 followed the Frundlich isotherm model (r^2>0.994). Fitting of obtaining data onto kinetic models showed the pseudo second-order reaction kinetics with respect to the dye concentration. This investigation revealed adsorption onto activated charcoal prepared from almond shell presents good efficiency for solution decolorization.

Key words: *Adsorption, Acid Red 18, Activated charcoal, Kinetic models, almond shell*

Akram A. Najafi Chaleshtori[1]
Fazel Mohammadi Meghadddam[1]
Mehraban M. Sadeghi[1*]
Rohollah R. Rahimi[1]
Sara Hemati[1]
Ali A. Ahmadi[2]

[1] Department of Environmental Health Engineering, School of Public Health, Shahrekord University of Medical Sciences, Shahrekord, Iran
[2] Department of Epidemiology and Biostatistics, School of Health, Shahrekord University of Medical Sciences, Shahrekord, Iran

*Corresponding author:
sadeghi@skums.ac.ir

INTRODUCTION

Increasing development in industries and discharge industrial wastewater into the environment has caused a series of new challenges due to groundwater pollution. The textile industry is one of the most polluting industries because its wastewater contains great amounts of dyestuff, chemical compounds and nonionic surfactants (*Ozcan and Ozcan 2004*). Among the dye compounds; azo dyes are the most used because of easy to make and the diversity in chemical structure (*Konstantinou and Albanis 2004*). This group of the dye is cheap to purchase and has high stability and solubility and may have one or more nitrogen-nitrogen band (*Crini 2006*). In addition, these dyes are non-degradable, resistant to light, chemicals, heat and oxidizing agents. It may cause health and environmental problems such as renal, central nervous system, brain, liver dysfunctions and even cancer if discharge to the environmental (*Robinson et al. 2002*). Hence, the dye removal from textile industry wastewater is absolutely necessary before discharge into the environment. Various techniques based on the biological, physical, and chemical methods such as trickling filter, activated sludge, electrocoagulation, photochemical and membrane processes has recently used in order to remove dyes (*Wong et al. 2004*). Each of these methods have problems such as overpriced facilities, the production of toxics, the high maintenance and operation costs, the need to energy and pretreatment. In comparison with these methods, the adsorption method has known as an effective process and cost benefit for removing dye, pigments and also chemical oxygen demand (*Konicki et al. 2014*). Different adsorbents such as zeolite, activated carbon, agricultural wastes residual, bagasse ash, ketone, ketazon and almond shell (AS) are used in this method due to the low cost, easy to design, the high efficiency, availability and ability to desiccate an spectrum of chemical compounds from aqueous solutions (*El-Bindary et al. 2014; Tor and Cengeloglu 2006; Gupta and Suhas 2009*). During the recent decades, using the activated charcoal prepared from agricultural wastes such as AS has been used to remove pollutants due to the abundance and the low cost. In the study performed by *Zhang and Ou* (*2013*) in order to adsorb crystal violet dye by the activated carbon prepared from almond crust demonstrated that the maximum dye adsorption rate of the dye was occurred at 100 min which follows Langmuir isotherm and pseudo second order kinetic

model (*Zhang and Ou 2013*). *Al-othman et al.* (*2012*) used the activated carbon prepared from almond crust for chrome removal. The results showed that chrome removal rate increased with increasing in temperature and resulted data followed both Langmuir and Freundlich isotherms (*Al-Othman et al. 2012*). *Wilson et al*. (*2006*) obtained to the sorption of 0.05-0.95 m mol g^{-1} of metal types from aqueous solutions using the activated carbon prepared from almond crust (*Wilson et al. 2006*).

One of the most important environmental pollutants is azo dyes in textile wastewater and there is not any suitable control in developing countries on the effluent discharged from these industries to the environment. In addition, a significant amount of agricultural wastes (especially AS) is produced annually. In this study, AS is used as base material for the preparation of activated carbon because it is cheap and easily available in Iran. In fact, it has no commercial usage and is incinerated by farmers. Almond shells are a low-cost, relatively abundant agricultural by-product. Research has shown that almond peel has a good ability to produce activated carbon due to lignocellulosic material. The almond shell cell wall contains silicates, cellulose, carbohydrates and many hydroxyl groups (*Plaza et al. 2010*). According to the conducted studies, almond shell coal and magnetic nanoparticles have very good ability to remove organic compounds and toxic heavy metals from aqueous solutions (*Mohan et al. 2011, Arbabi et al. 2016*).

In order to optimize the consumption of these materials, the present investigation is carried out to determine the optimum conditions for using the activated carbon prepared from AS for the removal AR 18 from textile wastewater.

Materials and Methods

Preparation of activated carbons

Hard shells of almond are agricultural by-product that in this study is obtained in the autumn season in Saman. It is grinded with mesh size of 2.5-3 mm and carefully washed with tap water in order to remove the wastes. Then, it is rinsed with deionized water and dried in air oven at 105°C (2 h). The gravels crushed were submersed in phosphoric acid (1 N) for 24 h. The shells saturated with acid were activated at 700 °C (1 h). The activated carbon produced before stage was graveled by screener with mesh of 20-100 until diameter range of the gravels remained between tow screeners adjusted among of 0.15-0.85 mm. Characteristics of this biopolymer was evaluated by Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electronic Microscope (SEM).

Adsorption experiments. The dye used in this study is azo dye C.I. Acid Red 18 ($C_20H11N_2Na_3O10S_3$ MW=604.47 g mol^{-1}) obtained from the Textile Nahangol Company (Industrial Centers, 75 km west Isfahan) which is characterized by difficult biodegradation due to their complex structure and synthetic nature and high toxicity (*Popli and Patel 2015*)(**Figure 1**). The AR18 dye was made with high purity of dye and was applied without further purification. The dye stock solution was prepared by dissolving 500 mg of acid red 18 (AR18) in 1000 mL deionized water. The lesser concentrations of dye was fabricated with dilution of stock dye (*Shirmardi et al. 2012*).

The initial AR 18 concentration (25-100 mg L^{-1}), pH (2-12), adsorbent dosage (0.2-2 g L^{-1}) and temperature (20±1°C) are selected as the experimental parameters ranges. Each sample is shacked (120 rpm, 60 min) for the better contact between the adsorbent and adsorbate and then, the residual AR 18 concentration is analyzed by a spectrophotometer (UV/vis., Unico 2100) at 506 nm. The following mass balance equation is used to calculate the AR 18 adsorbed per unit mass of adsorbent qe (mg g^{-1}):

$$q_e = \frac{(C_0 - C_e)}{W} \times V \quad (1)$$

Where C0 and Ce are the initial and equilibrium concentrations of AR 18 (mgL^{-1}), respectively. V the volume of the solution (L) and W the mass of the adsorbent of AC-AS (g). The efficiency removal (ɳ%) of AR 18 is calculated from the equation:

$$\eta\% = \frac{C_0 - C_t}{C_0} \times 100 \quad (2)$$

Ct (mg $L^{-1)}$ is the concentration of AR 18 at the time (t).

Adsorption isotherms and kinetics. Different isotherm and kinetic models are used to optimize the design of an adsorption system to remove AR 18 and to survey of the adsorption mechanism and to control reaction rate such

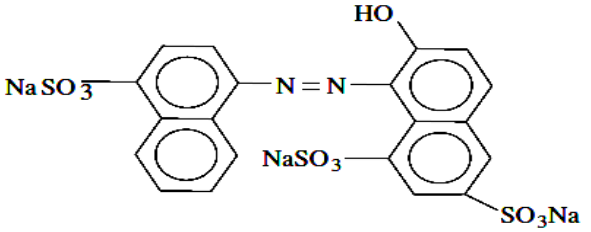

Figure 1. Chemical structure of Acid Red 18 (AC 18).

as mass transfer and development of the chemical reaction. Freundlich and Langmuir adsorption isotherms and kinetics of pseudo first-order and second-order reactions are used to evolution of kinetic behavior of AR 18 adsorption process. The mathematical equations of the adsorption isotherms and kinetics are given by:

$$\mathrm{Log}q_e = \mathrm{Log}k_f + \frac{1}{n} \times \mathrm{Log}(C_e) \quad \text{(Freundlich's isotherm equation)}$$

$$C_e/q_e = C_e/Q_{max} + 1/(Q_{max}.k_L) \quad \text{(Langmuir's isotherm equation)}$$

$$\log(q_e - q_t) = \log q_e - \frac{k_{ad}t}{2/303} \quad \text{(Pseudo first-order reaction)}$$

$$t/q_t = 1/k_{2ad.q_e^2} + (1/q_e)t \quad \text{(Pseudo second-order reaction)}$$

Where qe, Ce and qt= adsorption capacity at balance situation (mg g^{-1}), effluent concentration (mg L^{-1}) and adsorption rate (mg g^{-1}) at time of t, respectively.

q_m= adsorption capacity maximum (mg g^{-1}).
n, k_f and k_l= adsorption isotherm coefficients.
k_1 and k_2= adsorption kinetic coefficients.

RESULTS AND DISCUSSION

Adsorbent Characteristics

The chemical structure of the adsorbent is very important in understanding the adsorption process (*Daffalla et al. 2011*) because sorption capacity of an absorbent is affected by this structure. The FTIR technique is an important tool to identify the chemical structure and functional groups. The analysis showed that the bands obtained in ranges of 3400-3500 Cm^{-1}, 1500-1600 Cm^{-1}, 1000-1200 Cm^{-1}, 800-900 Cm^{-1}, and at 500-700 Cm^{-1} are attributed to the functional groups such as hydroxyl, carboxyl, aromatic ring, amine, ether and ester. In addition, the FTIR analysis showed that further of the functional groups of activated carbon have been involved in the adsorption. The results are similar to the observations of *Tan et al.* (*2008*); *Cuhadaroglu and Uygun* (*2008*); *Wasewar et al.* (*2009*); *Ould Brahim et al.* (*2014*).

It should not be ignored that the surface chemistry of adsorbents plays an effective role for determining the adsorption properties (*Solum et al. 1995; Kaouah et al. 2013*). The surface of AC-AS confirms substantial changes provoked by heat and the adsorbent surface clearly shows the porous nature with a predominant microporous character, while the surface of the other one (i.e., AS) is smooth with no porosity (**Figure 2**). FTIR spectra gives an important information about the chemical structure and functional groups of AC-AS **(Figure 3).** The FTIR spectrum shows the adsorptions at 3440.39 Cm^{-1} correspond to N-H and O-H groups, at 1563.99 Cm^{-1} correspond to secondary amid band of N-H, at 1116.5 Cm^{-1} and 1058.73 Cm^{-1} correspond to stretch vibration band of C-O-H and at 830-870 Cm^{-1} correspond to stretch band of N-O (**Figure 3-a**). Infrared spectroscopy of the adsorption with the activated carbon demonstrated a change in the spectrums between 3300-3500 Cm^{-1} ascribed to hydroxyl and amine ($-NH_2$) groups and 1050-1200 Cm^{-1} attributed to C-O stretching vibration band (*Kaouah et al. 2013*). The adsorption bands in these regions showing the decrease of energy band due to the adsorbate dye ions by AC-AS.

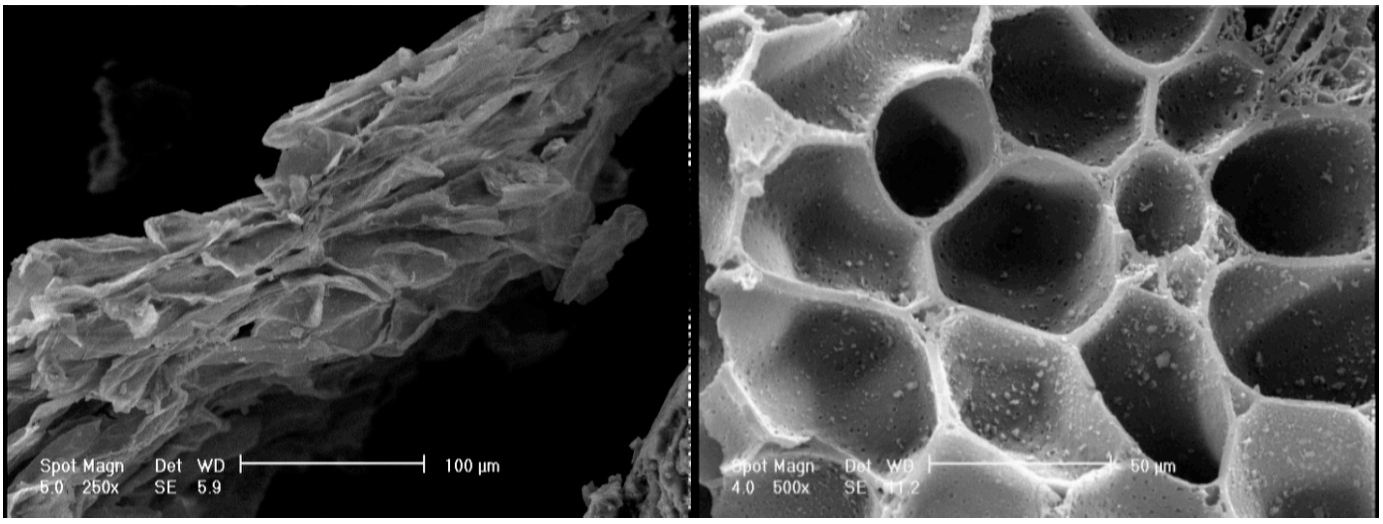

Figure 2. SEM micrographs of (a) AS and (b) AC-AS.

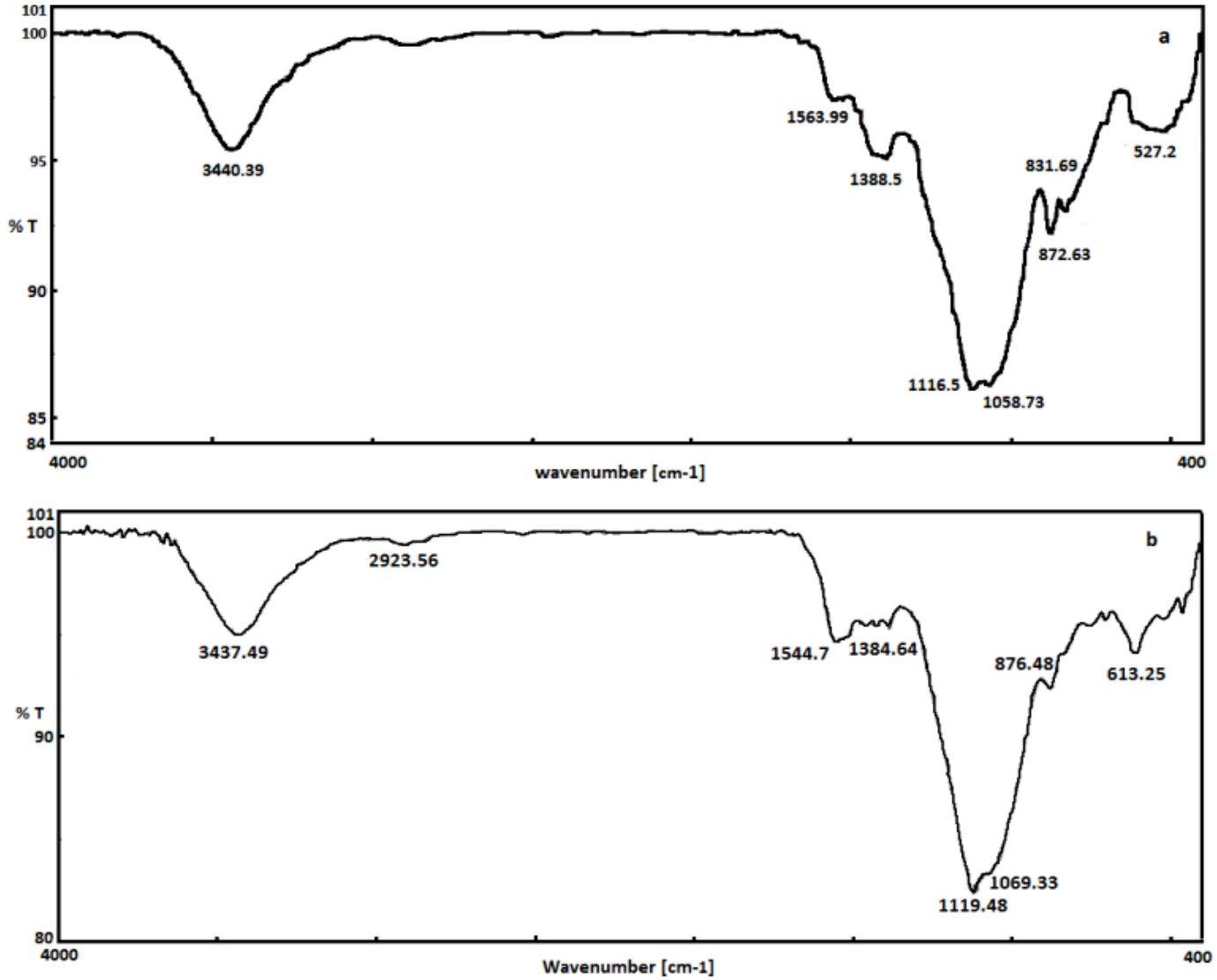

Figure 3. Infrared spectra of AC-AS (a) before adsorption, and (b) after adsorption.

Adsorption Experiments

Effect of pH. The pH is an important operating parameter in the dye adsorption (*Kaouah et al. 2013, Sathishkumar et al. 2012*) due to its effect on the adsorbent properties and the adsorbate ionization. The pH is effective on pollutant ionization degree, the mechanism interaction between the adsorbent with the dye and ionic charge of the functional groups (*Annadurai et al. 2002*). The effect of the pH on AR 18 adsorption onto AC-AS is studied over the pH range (2-12) (**Figure 4**). The adsorption capacity of AR 18 onto AC-AS increased with decreasing pH, and shows a maximum value of qe,cal (40.4 mg g^{-1}) at pH 2 (with maximum the removal percentage =90.83%) and decreases up to pH 12 (0.18 mg g^{-1}) (**Table 1**).

The high adsorption capacity of AR 18 at acidic pHs is due to the electrostatic sorption between the

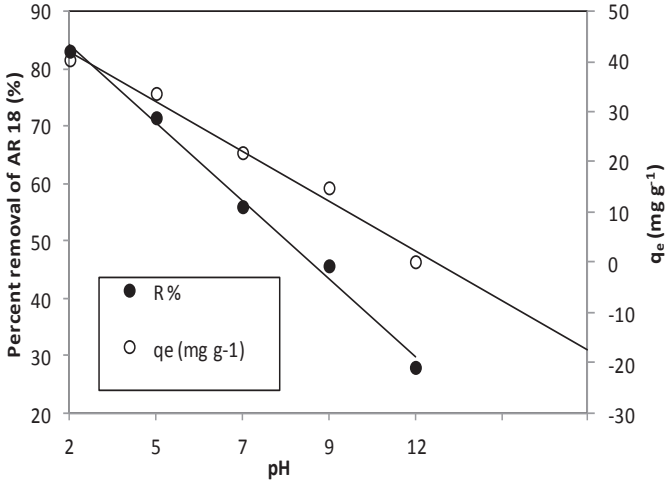

Figure 4. Effect of solution pH on AR 18 adsorption (adsorbent dose: 0.8 gL^{-1}, contact time: 60 min, dye concentration =75 mg L^{-1}, temperature: 20±1°C).

Table 1. The removal percentages and the quantity adsorbed of AR 18 at equilibrium (at 20±1°C).

Parameters	Percentages removal (R %)	qe, cal (mg g^{-1})
Co (mg^{-1})		
25	99.7	15.6
50	95.7	29.3
75	90.81	42.7
100	82.5	51.6
pH		
2	90.83	40.4
4	83	33.7
7	77.03	21.9
9	61.6	14.9
12	39.5	0.18
Adsorbent dose (g L^{-1})		
0.8		

surface positive charge on the adsorbent and negative charge on the dye molecules. While, at higher pHs the number of the positive charge on adsorbent is low and there is a compete between hydroxyl and the dye molecules for the functional groups of the adsorbent which causes the decrease in the dye removal. The results is similar to the observation of *Schimmel et al.* (*2010*). Furthermore, in the studies performed by *Demiral et al.* (*2008*); *Ishaq et al.* (*2014*), pH=2 was selected as an optimum condition in order to dye removal by the activated carbon prepared from industrial wastes. In the study of *Hamoudi et al.* (*2017*), the optimal pH for nitrate adsorption derived was 6, which is much higher compared to the activated almond carbon (*Hamoudi et al. 2007*).

Effect of the adsorbent dosage. The increase in the adsorbent dose leads to the increase of the removal percentage of AR 18 from 61.4 to 96.5 (**Figure 5**). This is due to the increase in availability of surface active sites resulting from the increased in the adsorbent dosage (*Shirmardi et al. 2012, Kaouah et al. 2013, Moussavi and Mahmoudi 2009, Konaganti et al. 2010*). Similar results were reported in 2013 by Mike and colleagues (*Xing et al. 2011*). But Arbabi et al. in their study reported that by increasing in adsorbent dose, no significant increase in the nitrate removal efficiency and adsorbent dose in the removal of nitrate was not significant (*Arbabi et al. 2016*). The relationship between the adsorbent dosage and the removal efficiency isn't constant as we can see the adsorption rate initially increased rapidly, and the optimal removal efficiency is reached within about 0.8 g L^{-1} (**Figure 5**). Further, the adsorbent concentration does not show significant change in equilibrium dosage. This might be due to a competition of active sites of the adsorbent in excess of equilibrium concentration of AC-AS.

Effect of AR 18 concentration. In the sorption process the initial concentration of adsorbent ions plays a key role as driving force to overcome the mass transfer resistance between the liquid and solid phases (*Kaouah et al. 2013, Dogan et al. 2006*). The results obtained from the effect of this factor in the range (25-100 mg L^{-1}) showed that the percentage of AR 18 removal by sorption decreases from 99.7 to 82.5% with increasing the initial dye concentration (**Figure 6**). The low removal efficiency at higher concentrations of AR 18 is due to the saturation of sorption sites (*Kaouah et al. 2013, Ozer et al. 2005*). This result is similar to the observations of *Garg et al.* (*2003*) on the removal of dye from aqueous solutions using treated sawdust; as well as with the stusy of *Doulati Ardejani et al*. (*2008*) on the removal Direct Red 80 dye from aqueous solution onto almond shells. While, the adsorption equilibrium qe, cal increases from 15.6 to 51.6 mg g^{-1}.

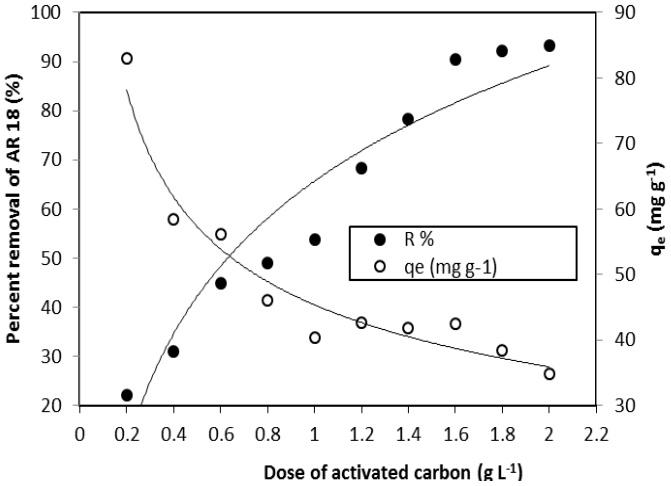

Figure 5. Effect of adsorbent dose on AR18 adsorption (pH: 2, contact time: 60 min, dye concentration = 75mgL^{-1}, temperature: 20±1°C).

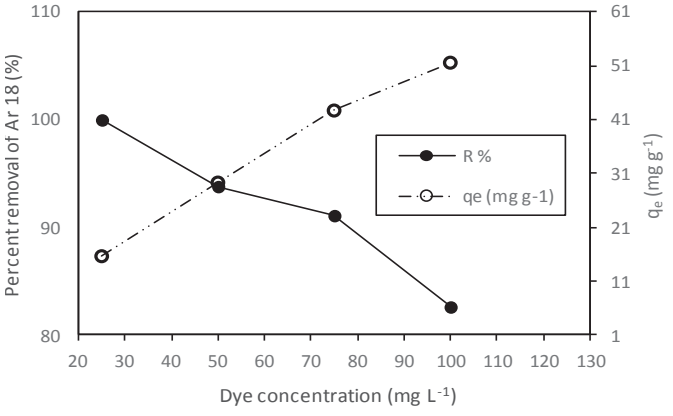

Figure 6. Effect of the initial concentration of dye on AR18 adsorption (adsorbent dose: 0.8 gL^{-1}, contact time: 60 min, pH: 2, temperature: 20±1°C).

Effect of contact time. The results of this work confirmed the effect of contact time on the adsorption rate. The removal percentage is increased from 39.9 to 96.07% with increasing the contact time that is similar to the observations of *Akl et al.* (*2013*) and *Arami et al.* (*2006*) (**Figure 7**). It was observed that the maximum adsorption capacity (44 mg g^{-1}) occurred at 2 h contact time and at equilibrium time of 60 min, the adsorption capacity of 42.4 mg g^{-1} is achieved. The curve revealed that once the equilibrium is reached, further increase in contact time does not have significant effect on AR 18 adsorption and the removal rate becomes almost constant (**Figure 7**). This is due to the occupation of all the active sites of the adsorbent by AR 18 molecules.

Equilibrium adsorption models. Isotherms are functions which connect the amount of adsorbate on the adsorbent and these mechanisms are usually applied in order to describe the adsorption process. The isotherm models such as Langmuir and Freundlich may be used to describe distribution of adsorbate between the liquid phase and the solid phase at equilibrium. One of the important steps for the design purpose is the fitting of data with different models. In addition, the adsorption isotherm is fundamentally important to describe how the dye interacts with the adsorbent. In this research the equilibrium experiments are performed in optimal conditions that were; pH 2, AR 18 concentration of 75 mg L^{-1}, contact time of 60 min, adsorbent dosage of 0.8 g L^{-1} and temperature of 20±1 ℃. The adsorption of AR 18 over AC-AS followed the pseudo-second-order kinetics with an R2 of 0.981 and was fitted better by the Freundlich isotherm model with an R2 of 0.994 (**Table 2** and **Figure 8**). This result is similar to the observations of *Shirmardi et al.* (*2012*) on the adsorption of AR 18 using multiwall carbon nanotubes from aqueous solution and on the adsorption of acid dyes from aqueous solutions onto acid-activated bentonite (*Ozcan and Ozcan 2004*).

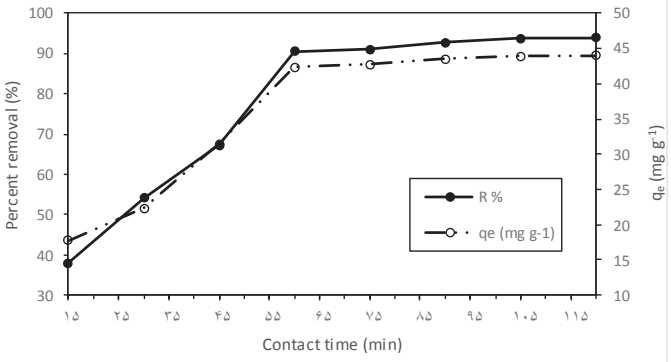

Figure 7. Effect of contact time on AR18 adsorption (adsorbent dose: 0.8 gL^{-1}, dye concentration =75 mgL^{-1}, pH: 2, temperature: 20±1°C).

CONCLUSION

The current investigation has shown that satisfactory removal efficiency of AR 18 can be obtained by using almond shells treated with phosphoric acid and activated with heat process. The activated carbon prepared from almond shells is successfully used for the removal of AR 18 in batch scale. The adsorption of AR 18 is also found to increase by increasing the adsorbent dose, contact time and by decreasing the concentration of dye and pH. The SEM micrographs indicate that the activation of the almond shells produces a possible porosity for the adsorption. In general, removal efficiency between 82.6-100% could be achieved after activating the almond shells in the adsorption equilibrium conditions (i.e. pH 2, contact time=60 min, adsorbent dose=0.8 g L^{-1}) with dye initial concentration of 25-100 mg L^{-1}. The pseudo-second-order is the best model to describe the adsorption of AR 18 onto the surface of heat activated almond shells as the R2 values were 0.981 and the data was fitted better by the Freundlich isotherm model with R2 of 0.994.

Table 2. Equilibrium constants for AR 18 adsorption.

Langmuir		
KL (L mg^{-1})	q_{max} (mg g^{-1})	R^2
0.861	10.752	0.988
Freundlich		
KF ($mg^{-1}(1/nf)g^{-1}$ L1/nf)	nF	R^2
5.372	4.564	0.994

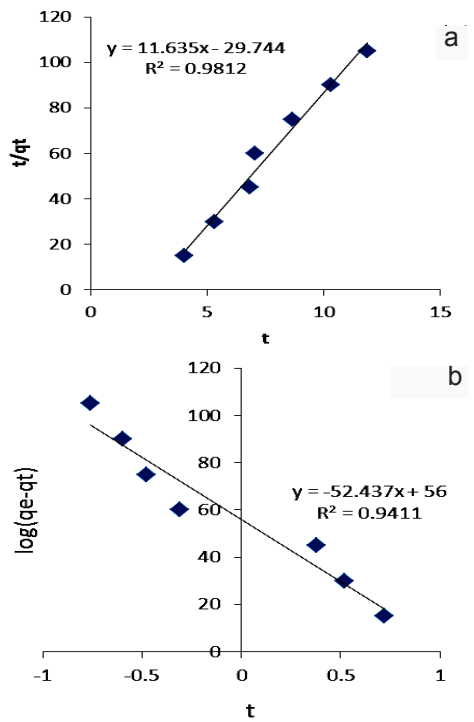

Figure 8. The linear plots of (a) pseudo-first-order, (b) pseudo-second-order kinetic models.

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