Journal of Food Engineering 111 (2012) 490–495

Contents lists available at SciVerse ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Removal of dark colored compounds from date syrup using activated carbon: A kinetic study

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ARTICLE INFO

Article history:
Received 25 December 2011
Received in revised form 18 February 2012
Accepted 25 February 2012
Available online 7 March 2012

Keywords:
Date syrup
Powdered activated carbon
Adsorption
Isotherm
Kinetic

ABSTRACT

Dark colored compounds in date syrup have been removed using powdered activated carbon (PAC) at different operating conditions including different temperatures (30, 40, 50 and 60 °C), as well as different PAC concentrations (0.04, 0.06, 0.08, 0.1 and 0.12 g ml^{-1}). Adsorption isotherms of Langmuir, Freundlich, Tempkin and Harkins–Jura were selected to fit the equilibrium data at different operating temperatures. The isotherms were correlated based on the absorbance intensity and the concentration of a colored compound, which gives the same absorbance intensity as the real date syrup. Among the adsorption models, the Langmuir isotherm best fitted the experimental data with coefficient of 0.99. To study the adsorption kinetics, the pseudo-first order and the pseudo-second order models were evaluated to fit the experimental data. The pseudo-second order equation fitted the experimental data very well.

1. Introduction

The dates (*Phoenix dactylifera* L.), very nutritious and healthy foods, are one of the most widely product fruits in the world that play an important role in the daily life of people for several thousands years ago (Al-Shahib and Marshall, 2003). They are served mainly as a source of calories with about 78% carbohydrates, 2–3% proteins and 1% fat. Most of the carbohydrates in dates are in the form of fructose and glucose, which are easily absorbed by the human body. Dates play an important role in nutrition, as research has indicated the clear contribution of them to human health when consumed with other food constituents (Sidhu, 2006). The inferior grade dates can be considered as good sources of sugars, minerals and some other substances (Ramadan, 1995). Industrially they are utilized to produce several products such as syrup, jam, date-jelly, date butter, vinegar, wine and single cell protein (El-Sharnouby et al., 2009). Liquid sugar and high fructose syrup can also be produced from date syrup. One of the most important steps in production of these products is clarification and discoloration of date extract in order to remove its brown color. The color of date syrup has been originated from the phenolic compounds present in dates which act as precursors for melanin formation, the brown-colored products. Roufegari-nejad (2002), indicated that melanins and color substances resulted from non-enzymatic browning reactions, particularly melanoidins were the major colorants of date syrup. In order to prevent fruit juices from browning and haze formation, reduction of melanoidins and phenolics is necessary. Typical adsorbents such as activated carbon, bentonite, casein, ion-exchange resins and polyvinylpolypyrrolidone, have been studied for the removal of polyphenols and brown color compounds from fruit syrups such as date syrup (Giovanelli and Ravasini, 1993). Out of all mentioned adsorbents, the activated carbon is used more than the others due to its higher adsorption capacity (El-Geundi, 1995).

Several researches have been carried out on the adsorption isotherm of colored compounds in different syrups. In this respect, Simaratanamongkol and Thiravetyan (2010) studied the adsorption behavior of synthetic melanoidin solution employing activated carbon at the temperatures of 5, 25 and 60 °C. They found that the equilibrium adsorption capacities, fitted well with Langmuir and Frendlich models, increased at higher temperatures. Koyuncu et al. (2007) analyzed the effect of bentonite concentrations and various temperatures on adsorption equilibrium of apple juice dark-colored compounds. They discovered that the adsorption efficiency was improved by increasing the clay concentration from 2 to 8 g clay/dm^3 of apple juice, in temperature range of 296–336 K. Arslanoglu et al. (2005) investigated the adsorption of pulp peach dark colored compounds at different PAC concentrations (0.5, 1, 3 and 5 kg PAC/m^3 peach pulp) in the temperature range from 20 to 60 °C. They studied the adsorption isotherms and kinetics to evaluate the effect of adsorbent concentration and contact time and showed that with increase of PAC concentration and temperature, the rate of adsorption increased. Al-Farsi (2003)

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http://dx.doi.org/10.1016/j.jfoodeng.2012.02.037

investigated five different treatments for clarification of date syrup and reported that PAC along with filtration gives the highest decolorization and leaves the lowest ash. Subsequently, it improves the syrup purity up to 99.2% in the powder form and up to 91.4% in the granular form. Roufegari-nejad (2002) studied the effect of activated carbon, bone char and Amberlit IRA 410 (a strongly anionic resin) on date syrup decolorization at different time intervals (10–60 min) and different decolorizing substance qualities (2–30%). The results indicated that activated carbon has the highest decolorizing ability so that about 99% reduction can be achieved compared to bone char (89%) and amberlite resin (87.6%).

In this work, adsorption behavior of date syrup dark colored compounds on PAC is investigated experimentally and theoretically. The effect of different conditions, such as temperature, PAC concentration and contact time are studied. Different adsorption isotherms are evaluated to fit with the experimental values. In order to predict the rate of the adsorption process, some kinetic models are compared to fit with the experimental data at different operating temperatures from 40 to 60 °C and various PAC concentrations of 0.6 and 0.1 g ml^{-1}.

2. Materials and methods

2.1. Materials

Date syrup was purchased from Khorma Bon-e Jonoob factory (Bandar Abbas, Hormozgan). It was packed in sealed cans and stored at room temperature (22 °C). Table 1 shows the chemical properties of the above date syrup. Wood-based PAC (ColorSorb M5) supplied by Jacobi Chem Co. (Sweden), was the adsorbent utilized in this work. Table 2 shows some physical and chemical properties of this product. Bentonite from Xinjiang Bentonite Co. (China) was used to facilitate filtration. Deionized water (3.4 µs/cm at 23 °C) was used as a solvent to prepare stock solutions.

2.2. Experimental set-up

To study the effect of important parameters such as temperature, adsorbent concentration and contact time on the removal of colored compounds in date syrup, the following study was conducted. Adsorption experiments were carried out with mixtures containing different PAC concentrations (4, 6, 8, 10 and 12 g) in 100 ml of date syrup (brix 20) in glass flasks. Then they were placed in a thermostated water bath (Fan Azma Gostar, Iran) at constant temperatures and mixed at 400 rpm with mechanical stirrer (IKA RW-20, Germany) for 1.5 h. Afterward, the samples were clarified via filter-press and the absorbance of the supernatant was measured colorimetrically using spectrophotometer (UNICO, UV-2100) at 520 nm. The experiments were conducted at 30, 40, 50, and 60 °C to study the effect of temperature on the adsorption capacity and to evaluate the adsorption thermodynamic parameters. It should be taken into account that to reduce the possibility of evaporation at high temperature, the flasks and water bath were sealed thoroughly.

Table 1
General properties of date syrup used in this research.

Dry substance (%)	76.28
Soluble solids (°Brix at 20 °C)	76.1
pH	4.25
Ash (% w/w)	2.63
Acidity (as citric acid) (% w/w)	0.69
Color (ICUMSA units)	1145
Conductivity (µs/cm)	350
Fructose (%)	45.7
Dextrose (%)	43.3
Saccharose (%)	0.5
Mineral matters	
Na (mg/kg)	1573
K (mg/kg)	13109
Ca (mg/kg)	1573
Mg (mg/kg)	852

Table 2
The physical and chemical characteristics of PAC used in this study.

Parameter	Quantity
Iodine number (mg/g)	800
Molasses number	216
Total ash content (%)	7
Moisture content (%)	8
Surface area (BET) (m^2/g)	760
Acid soluble phosphate (%)	1.7
Total pore volume (cm^3/g)	2.1
pH	6–7
Particle size distribution:>200 USS (0.075 mm) (%)	20
<200 USS (0.075 mm) (%)	80

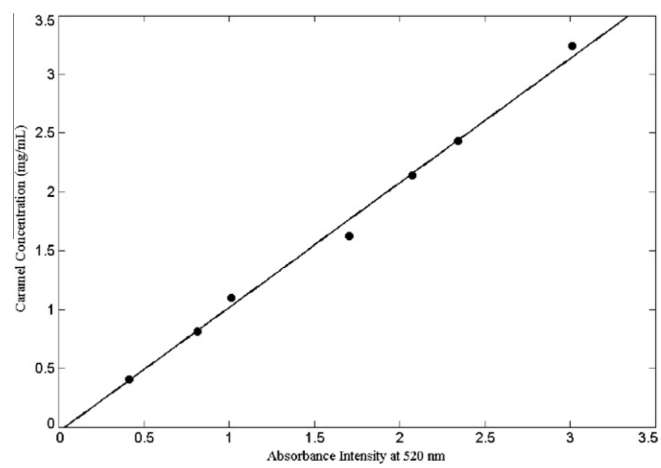

Fig. 1. Standard curve of absorbance intensity of caramel at different concentrations at 520 nm.

To verify the effect of contact time on the adsorption, 100 ml date syrup was mixed with 6 and 10 g of PAC. In each experiment, samples were withdrawn at appropriate time intervals: 5, 10, 15, 20, 30, 45, 60 and 90 min. Adsorption kinetics were investigated by determining the adsorptive uptake of the colored compounds in date syrup solution at the mentioned time intervals and temperatures of 50 and 60 °C. In order to convert the absorbance intensity to concentration of colored compounds, caramel has been used as an indicator. Standard curve of caramel has been shown in Fig. 1.

3. Mathematical modeling

3.1. Adsorption isotherm models

Equilibrium behavior of the adsorption system is necessary for modeling of adsorption kinetics. Adsorption data are usually described by adsorption isotherms. These isotherms indicate the relation between equilibrium impurities (colorants) uptake per unit weight of adsorbent (q_e) and the equilibrium adsorbate concentration in the bulk fluid phase (C_e) at a given temperature. The amount of dye adsorbed onto carbon, q_e (mg g^{-1}), was calculated by the following mass balance relation (Eq. (1)):

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

where C_0 and C_e (mg ml^{-1}) are the initial and equilibrium liquid-phase concentrations of colored compounds, respectively, V (ml) is the volume of the solution and W is the weight of the PAC (g) employed.

Out of several equations, which have been proposed for modeling of equilibrium adsorption data, the Langmuir, Freundlich, Tempkin and Harkins–Jura isotherms are the most common ones used in the case of mono component systems. Langmuir isotherm is shown in the Eq. (2) with the following presumptions (Langmuir, 1918):

- Monolayer adsorption onto a completely homogenous surface with a finite number of identical sites.
- Negligible interaction between adsorbed molecules.

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_m} + \frac{C_e}{Q_m} \quad (2)$$

where K_L is the Langmuir adsorption constant (ml mg^{-1}) and Q_m is the theoretical maximum adsorption capacity (mg g^{-1}).

The essential characteristics of the Langmuir isotherm can also be expressed in terms of a dimensionless constant separation factor or equilibrium parameter (R_L), which is defined by Eq. (3).

$$R_L = \frac{1}{(1 + K_L C_0)} \quad (3)$$

where C_0 is the highest initial solute concentration and K_L is the Langmuir's adsorption constant (ml/mg). The R_L value confirms the adsorption to be unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$) (Mckay et al., 1989).

The empirical Freundlich isotherm is based on the equilibrium relationship between heterogeneous surfaces. This isotherm is derived from the assumption that the adsorption sites are distributed exponentially with respect to the heat of adsorption. Freundlich model is defined by Eq. (4).

$$q_e = K_f C_e^{1/n} \quad (4)$$

where K_f is Freundlich constant and n is the heterogeneity parameter of the adsorption process. Higher n value shows the non-linearity of the data, which n values above 10 indicate that the adsorption process is irreversible (Crini et al., 2007).

The linear form of the Freundlich equation is given by Freundlich (1906) as follows (Eq. (5)):

$$Ln(q_e) = Ln(K_F) + \frac{1}{n} Ln(C_e) \quad (5)$$

The Tempkin isotherm usually has been applied in the following form of Eq. (6).

$$q_e = \frac{RT}{b_T} Ln(K_T C_e) \quad (6)$$

and can be expressed in its linear form by Eq. (7).

$$q_e = B_T Ln(K_T) + B_T Ln(C_e) \quad (7)$$

where $B_T = (RT)/b_T$, T is the absolute temperature in Kelvin, R is the universal gas constant, 8.314 J/mol K, K_T is the equilibrium binding constant (ml/mg) which depends on the maximum binding energy, and B_T is related to the heat of adsorption (Tempkin, 1940).

The Harkins–Jura adsorption isotherm can be expressed (Amin, 2009) by Eq. (8).

$$\frac{1}{q_e^2} = \left(\frac{B}{A}\right) - \left(\frac{1}{A}\right) \log(C_e) \quad (8)$$

This isotherm explains the multilayer adsorption in the presence of a heterogeneous pore distribution. A and B can be calculated from the intercept and slope of the plot, $1/q_e^2$ vresus log C_e.

3.2. Adsorption kinetic models

For evaluation of the adsorption kinetics of the colored compounds of date syrup onto PAC, the pseudo-first and the pseudo-second order kinetics models are performed. The adsorption parameters of these equations are necessary for designing and modeling the adsorption processes. The pseudo-first order model is described by Lagergren (1898) by Eq. (9):

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad \text{I.C.}: \ @t = 0 \quad q_t = 0 \quad (9)$$

where q_e and q_t refer to the amount of adsorbed dye (mg g^{-1}) at equilibrium and at any time, t (min), respectively. Also, k_1 is the pseudo-first order adsorption rate constant (min^{-1}). Eq. (10) is obtained by integrating Eq. (9) with respect to time.

$$Ln(q_e - q) = Ln(q_e) - k_1 t \quad (10)$$

The pseudo-second-order model is represented by Eq. (11). (Ho et al., 2000)

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad \text{I.C.}: \ @t = 0 \quad q_t = 0 \quad (11)$$

where k_2 is the pseudo-second-order adsorption model constant (g mg^{-1} min^{-1}). By integrating Eq. (11) with respect to time Eq. (12) is obtained.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (12)$$

4. Results and discussions

4.1. The effect of temperature and PAC dosage on the adsorption efficiency

Temperature and PAC dosage are the most effective parameters on the adsorption efficiency of date syrup dark colored compounds. The adsorption efficiencies were evaluated by Eq. (13).

$$\text{Adsorption efficiency} = \frac{A_0 - A_E}{A_0} \times 100 \quad (13)$$

where A_0 and A_E are the initial and equilibrium absorbance intensities at 520 nm, respectively. Fig. 2 shows the adsorption efficiency as a function of PAC concentration at different temperatures. As adsorbent dosage is increased, the adsorption efficiency increases under all temperature conditions. The ability of activated carbon

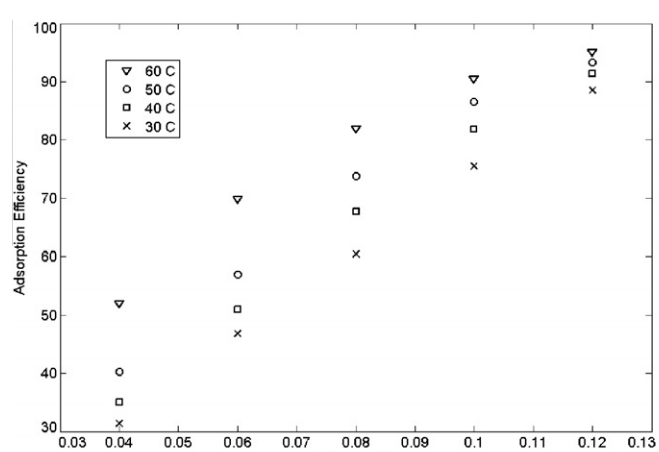

Fig. 2. The effect of PAC concentrations and temperatures on the adsorption efficiency in date syrup at 520 nm.

Table 3
Estimated Langmuir, Freundlich, Tempkin and Harkins–Jura constants with their fitting quality parameters (RMSE and R^2) at 30, 40, 50 and 60 °C.

		Temperature (°C)			
		60	50	40	30
Langmuir	K_L (ml/mg)	5.407	11.427	14.142	15.125
	Q_m (mg/g)	43.824	32.216	27.952	25.081
	R_L	0.056	0.027	0.022	0.021
	R^2	0.992	0.998	0.999	0.999
	RMSE	1.25E-3	9.69E-4	7.66E-4	9.19E-4
Freundlich	K_f (ml/mg)	36.669	29.094	25.993	23.645
	$1/n$	0.215	0.109	0.064	0.034
	R^2	0.997	0.979	0.951	0.895
	RMSE	0.012	0.017	0.014	0.010
Tempkin	K_T	2.2338E + 2	1.6259E + 4	9.0684E + 6	6.6247E + 12
	B_T	6.803	3.007	1.624	0.801
	R^2	0.985	0.981	0.956	0.891
	RMSE	0.873	0.427	0.327	0.229
Harkins–Jura	A	2214.787	3408.863	4980.806	8203.218
	B	1.666	4.068	7.400	14.688
	R^2	0.991	0.964	0.940	0.902
	RMSE	4.646E-5	5.870E-5	4.739E-5	3.295E-5

to adsorb organic substances is essentially related to its extended surface area, high adsorption capacity, microporous structure and special surface reactivity. Indeed, the adsorption efficiency increases at higher activated carbon concentrations due to availability of functional groups on the surface of activated carbon. Arslanoglu et al. (2005) and Serpen et al. (2007) reported similar results for the removal of dark colored compounds from peach pulp and Maillard reaction products from sugar syrups, respectively.

Furthermore, the adsorption efficiency is increased with increasing of the temperature at constant PAC concentration due to viscosity reduction. The viscosity reduction affects the adsorbate diffusion toward the adsorbent. This behavior was also reported for the adsorption of dark colored compounds of peach juice and apple juice using various types of activated carbon and adsorbent resin (Koyuncu et al., 2007; Arslanoglu et al., 2005). The above results were also observed when activated palm ash and chitosan biopolymer were used for the adsorption of acid dye and reactive dye from aqueous solutions. (Hameed et al., 2007; Annadurai et al., 2008).

4.2. Adsorption isotherm

In this work, the adsorption equilibrium data of date syrup colored compounds are fitted by several well-known isotherm models containing Longmuir, Freundlich, Tempkin and Harkins–Jura at different temperatures. The model parameters are determined based on the square of the data errors in predicting the equilibrium. These parameters are shown in Table 3 along with the root mean square error (RMSE) and the coefficient of determination (R^2), calculated by Eqs. (14) and (15).

$$\mathrm{RMSE} = \left(\sum_1^N \left(\frac{q_{e,\exp} - q_{e,\mathrm{calc}}}{q_{e,\exp}}\right)^2 / N\right)^{\frac{1}{2}} \quad (14)$$

$$R^2 = \frac{\sum_1^N (q_{e,\mathrm{calc}} - \bar{q}_e)^2}{\sum_1^N (q_{e,\exp} - \bar{q}_e)^2} \quad (15)$$

$$\bar{q}_e = \frac{1}{N}\sum_1^N q_{e,\exp}$$

where $q_{e,\exp}$ and $q_{e,\mathrm{calc}}$ are experimental and calculated q_e values, respectively and N is the number of data points. All the parameters in Table 3 are the average values of three replicates with ±95% confidence intervals. The fitting quality parameters (RMSE and R^2) indicate that Langmuir model gives the smallest RMSE and the highest R^2 and so better describes the adsorption data of darked colored compounds in date syrup. Furthermore, the R_L values, the essential characteristics of the Langmuir isotherms at different temperatures, are shown in Table 3. All of the R_L values are less than 1 for constant initial concentration of 3.101 mg ml^{-1} confirming a favorable adsorption type. These results are comparable to those reported by Carabasa et al. (1998) and Luisa Soto et al.,(2008). They reported that Lamgmuir and Freundlich models had good ability to fit the experimental adsorption data of fruit juices as well as distilled-grape-pomace dark colored compounds by activated carbon, respectively. While Arslanoglu et al. (2005) reported that Tempkin and Langmuir models described the adsorption isotherm of dark colored substances of peach pulp at different PAC concentrations and temperatures well. Furthermore, Langmuir model predicted colored compounds adsorption well in the five different following processes: adsorption of sugar beat vinasse colored compounds (Caqueret et al., 2008), removal of basic dye from durian shell (Chandra et al., 2007), adsorption of remazol black 5 on the palm kernel shell activated carbon (Zawani et al., 2009) and removal of direct blue-106 dye from aqueous solutions (Amin, 2009). Fig. 3 shows the equilibrium adsorption data (C_e/q_e) at different PAC concentrations and different temperatures. In this figure the experimental data are fitted with the best model, Langmuir. Also, it shows that temperature has positive role on the adsorption with activated carbon, corresponding to endothermic nature of adsorp-

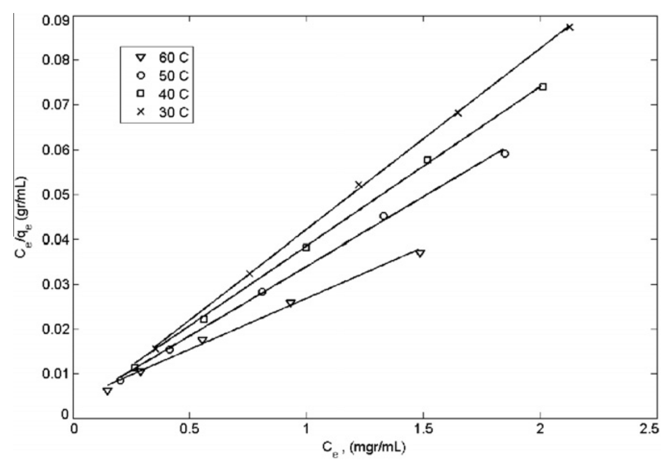

Fig. 3. Langmuir isotherm plots of colored compounds of date syrup on PAC at different temperatures.

Table 4
The adsorption kinetics model constants of the colored compounds of date syrup at two different temperatures (50 and 60 °C) and two different PAC concentrations (0.06 and 0.1 g ml^{-1}).

T (°C)	PAC (g/ml)	$q_{e(exp)}$	Pseudo first order				Pseudo second order			
			k_1	$q_{e(calc)}$	R^2	RMSE	k_2	$q_{e(calc)}$	R^2	RMSE
50	0.06	33.194	0.049	5.491	0.830	0.370	0.025	33.551	0.999	0.021
	0.1	26.876	0.043	5.237	0.910	0.226	0.036	27.420	0.999	0.031
60	0.06	36.120	0.020	4.682	0.758	0.186	0.021	36.458	0.999	0.029
	0.1	28.082	0.085	4.082	0.964	0.272	0.049	28.257	0.999	0.010

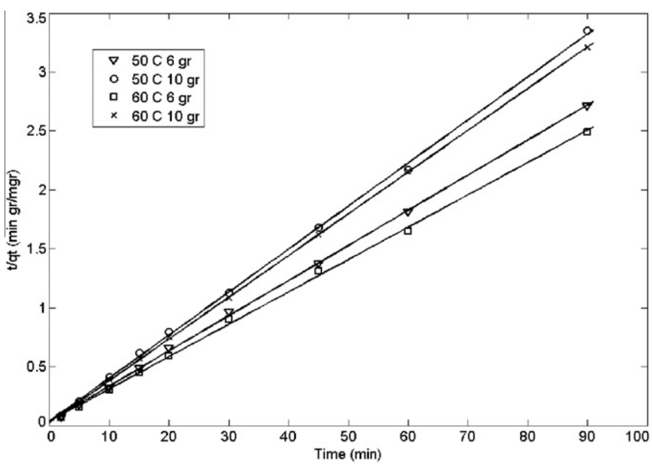

Fig. 4. Plot of the pseudo-second order model at different initial PAC concentrations (0.06 and 0.1 g ml^{-1}) and temperatures (50 and 60 °C).

tion phenomenon. The increase of temperature may contribute to the better diffusion of adsorbate in the porous microstructure of activated carbon, as both temperature-dependent dispersion forces and electrostatic interactions affect the adsorption process (Terzyk Artur, 2004). The adsorption of phenolic compounds, having major role in formation of dark colored compounds in date syrup, on activated carbon can be explained in term of dispersive interactions between the aromatic ring of phenol and the basic groups on the surface of PAC. Basic functional groups on the carbon surface increase π electron density on the graphene layers of activated carbon, and so the stronger the dispersive interactions between π–π electrons (Ania et al., 2002). These results were previously confirmed by other investigators such as Garcia-Araya et al. (2003), chingombe et al. (2006), aksu and kabasakal (2004) and Michailot et al. (2008).

4.3. Adsorption kinetic

In order to investigate the adsorption behavior of colored compounds on activated carbon, the pseudo-first and pseudo-second order kinetic models were used. The values of adsorption rate constants such as k_1, k_2 and q_e for the two mentioned kinetic models were calculated by the method described in Section 3.2. Table 4 shows these adsorption rate constants, with the two kinetic models relevant regression coefficients (R^2 and RMSE) at two different temperatures (50 and 60 °C) and two different PAC concentrations (0.06 and 0.1 g ml^{-1}). The magnitude of the determination coefficient (R^2) and the root mean square error (RMSE) values are the model selection criteria. Regarding the values of R^2 and RMSE of the pseudo-second order and the pseudo-first order one, the pseudo-second order model is more appropriate. The plot of t/q_e versus t for the best model, pseudo-second order, at temperatures of 50 and 60 °C with PAC concentrations of 0.06 and 0.1 g ml^{-1} is shown in Fig 4. As seen, the kinetic experimental data have been predicted well with this model and the correlation coefficient values for all experiments were more than 0.99. Previous studies have also shown that the pseudo-second order rate equation predicts the experimental data trend more reasonably (Serpen et al., 2007; Amin, 2009; Luisa Soto et al., 2008; Hameed et al., 2007; Mane et al., 2007).

5. Conclusions

PAC is found to be a suitable adsorbent for the dark colored compounds removal from date syrup. Moreover, this process had not any undesirable effects on the sensory characteristics of finished product. The adsorption quantity depends on the temperature and adsorbent dosage. The experimental results have proved that, the maximum of colored compounds removal (95%) is obtained at the temperature of 60 °C. Equilibrium experiments are conducted at 30, 40, 50 and 60 °C and the kinetic data are taken at 50 and 60 °C with PAC dosage of 0.06 and 0.1 g ml^{-1}. The equilibrium data show perfect fit with the Langmuir isotherm, which confirms that the adsorption process is a monolayer adsorption of colored compounds at the outer surface of PAC. The kinetic study of dark colored compounds adsorption on PAC is investigated by using the pseudo-first order and pseudo-second order equations. The results indicate that the adsorption kinetics data are fitted well by the pseudo-second order rate.

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