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Equilibrium and kinetic modeling of acid dye removal from aqueous solution by polymer inclusion membrane (PIMs)

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ABSTRACT

In this study, polymer inclusion membranes (PIMs) consisting of cellulose triacetate (CTA) as the base polymer, 2-nitrophenyl octyl ether as plasticizer, and tricaprylmethylammonium chloride (Aliquat 336) as carrier are used for removing the acid dye (red bordeaux acid and yellow erionyl) from aqueous solution. Extraction efficiency was studied under various experimental conditions, such as pH (2–9) of the aqueous solution, the concentration of extractant in the membrane (5–30 μmol/cm^2), initial dye concentration (50–250 ppm), and the stirring speed (250–400 rpm). Under the optimized experimental conditions, 99% of dyes were extracted. The Langmuir, Freundlich, and Temkin isotherm models were used to study the equilibrium extraction data. The Langmuir equations have better regression coefficient (0.95, 0.95) than Freundlich (0.96, 0.904) and Temkin (0.873, 0.909) equation describing the acid dye extraction by the matrix CTA-Aliquat 336. The kinetic data were evaluated using pseudo-first-order, pseudo-second-order and intra-particle diffusion model. The kinetic study's results indicate that the process of removing acid dye by PIMs is described by pseudo-second-order model.

Keywords: Polymer inclusion membrane (PIM); Aliquat 336; Acid dye removing; Kinetic modeling

1. Introduction

Dyes have become one of the main sources of severe water pollution due to the rapid development of textile, leather, tanning, paper, rubber, plastics, cosmetics, pharmaceutical products, and food industries. The synthetic dyes classified by their chromophores have different and stable chemical structures to meet various coloring requirements, and they are not degraded and/or removed by conventional physical and chemical processes [1,2].

In the last few years, applications of membrane separations of organic pollution control have been extensively researched [3–5]. Membrane separation is a relatively new type of separation process which is predicted ultimately to replace a majority of the conventional separation systems. Recently, several researchers have studied the extraction of dye membranes. Muthuraman et al. [6,7] and Hu et al. [8] reported the recovery of dyes by liquid–liquid extraction. Liquid membranes have been developed

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for the extraction of dyes from wastewater. Some workers studied the extraction of anionic and cationic dyes from aqueous solutions by emulsion liquid membrane [9–11]. Removal of anionic reactive dyes from water using anion-exchange membranes as adsorbers was reported by Liu et al. [12]; Wu et al. [13] used a cation-exchange membrane for treating water containing a cationic dye methyl violet 2B.

The kinetic elimination of dyes is important information in the process of adsorption, extraction of dyes. Many searches have realized in this context E. Demirbas et al. studied the adsorption of reactive blue 21 (RB21) onto fly ash (FA) and sepiolite from aqueous solutions. The kinetic study shows that the adsorption of RB21 dye onto FA and sepiolite were best described by the pseudo-second-order kinetic model [14]. The pseudo-first-order and pseudo-second-order used to describe the kinetics data. The kinetic adsorption of RR120 onto AOPA described by the pseudo-second-order kinetic equation [15]. Although the pseudo-first-order is applicable in the adsorption of lac dyeing of silk R^2 between 0.9603 and 0.9939, the pseudo-second-order is more representative of the kinetic system, R^2 which is between 0.9921 and 0.9996. The pseudo-second-order kinetic model indicated with the activation energy of 47.5 kJ/mol. It is suggested that the overall rate of lac dye adsorption is likely to be controlled by chemical process [16].

Adsorption of azocarmine B (acid dye) by the microspheres contents pullulan-graft-poly (3-acrylamidopropyl trimethylammoniumchloride), P-g-p AP-TAC, was used as a model to demonstrate the removal of anionic dyes from aqueous solutions. Constantin et al. [17] found that the mechanism of adsorption is the interaction of the polymer quaternary ammonium groups with the sulfonate groups of dyes. This exchange follows the pseudo-second-order mechanism. Luo et al. [18] prepared the novel molecularly imprinted polymers (MIPs) for toxic and carcinogenic dyes adsorption. It was found that their kinetic rates were exported by the pseudo-first-order model. The kinetic studies of removing eosin yellow from its aqueous solutions by adsorption onto the waste material, de-oiled soya revealed that the pseudo-second-order kinetics is operative during the adsorption process and the rate constant for the process was close to $1.10^{-9}\,\mathrm{g\,mg^{-1}\,s^{-1}}$ at both the temperatures studied ($T_1 = 30$°C and $T_2 = 50$°C). The treatment of kinetic data revealed that the ongoing adsorption proceeds via film diffusion process and adsorption of the dye is taking place mainly on the external surface of the de-oiled soya [19]. The intra-particle diffusion model described the selective adsorption of a reactive red and basic red dye by molecularly imprinted polymers (Mag–MIPs) [20].

The aim of this work was the application of polymer inclusion membranes (PIMs) consisting of plasticizer cellulose triacetate (CTA) containing tricaprylmethylammonium chloride (Aliquat 336**)** as the carrier for removing the anionic dyes (red bordeaux acid (RBA) (acid violet 90) and yellow erionyl (JEA) (acid yellow 127)) from aqueous solution. The effects of time pH, the stirring speed, and concentration of the carrier and dyes have been optimized. The kinetic parameters and adsorption isotherm were also evaluated.

2. Experimental

2.1. Reagent

CTA, 2-nitrophenyl octyl ether (2NPOE) obtained from Aldrich. Tricaprylmethylammonium chloride (Aliquat 336**)** obtained from Albright and Wilson. Chloroform ($CHCl_3$) acquired from Fluka.

RBA (acid violet 90) and JEA (acid yellow 127) was purchased from China11 and chem.net, respectively.

The structures of these dyes are given in Fig. 1. The aqueous phases were prepared by dissolving the different reagents in distilled water**.**

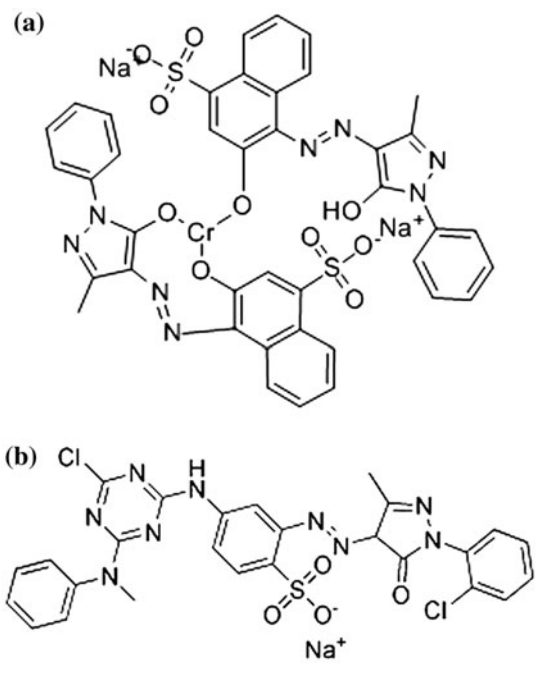

Fig. 1. The structure of the dyes: (a) RBA and (b) JEA.

2.2. PIMs preparation

PIMs were prepared using the same procedure described by Kebiche-Senhadji et al. [3,21]. A chloroform solution of CTA (200 mg) in 20 ml, the appropriate plasticizer (0.3 ml), and the carrier (Aliquat 336) in variable amounts was prepared. After vigorous stirring, the mixture is poured into a 9.0 cm diameter flat-bottom glass petri dish. The solvent was evaporated slowly overnight to obtain a polymer film. A small quantity of distilled water was deposited on the film to help its unstitching of the glass support.

2.3. Membrane characterization

Scanning electronic microscopy (SEM) images of PIMs were acquired using a HITACHI S4500 microscope that can reach a resolution of 1.5 nm.

X-ray diffraction (XRD) analyses were obtained by a diffractometer type Epert Prof. Panalytical.

2.4. Membrane solid–liquid extraction

Solid–liquid extraction experiments undertaken at fixed temperature and with stirred solution using a magnetic stirrer in range of 200–500 rpm. Dilute HNO_3 and NaOH were used to adjust pH of aqueous solution. The weight of the membrane used in each experiment is in order of 0.05 g.

The wavelengths of maximum adsorption $\lambda_{\max}$ of the dyes RBA and JEA are 525 and 440 nm, respectively.

The percentage extraction (E) calculated as per the following equation.

$$E = \frac{[\text{dye}]_{\text{aq0}} - [\text{dye}]_{\text{aq}}}{[\text{dye}]_{\text{aq0}}} \times 100 \qquad (1)$$

$[\text{dye}]_{\text{aq0}}$: initial dye concentration in the aqueous phase (mg/l).

$[\text{dye}]_{\text{aq}}$: dye concentration in the aqueous phase after extraction (mg/l).

3. Results and discussion

3.1. Membrane characterization

3.1.1. SEM

The SEM images of all membranes (Fig. 2) show uniform surfaces and appear dense with no apparent porosity.

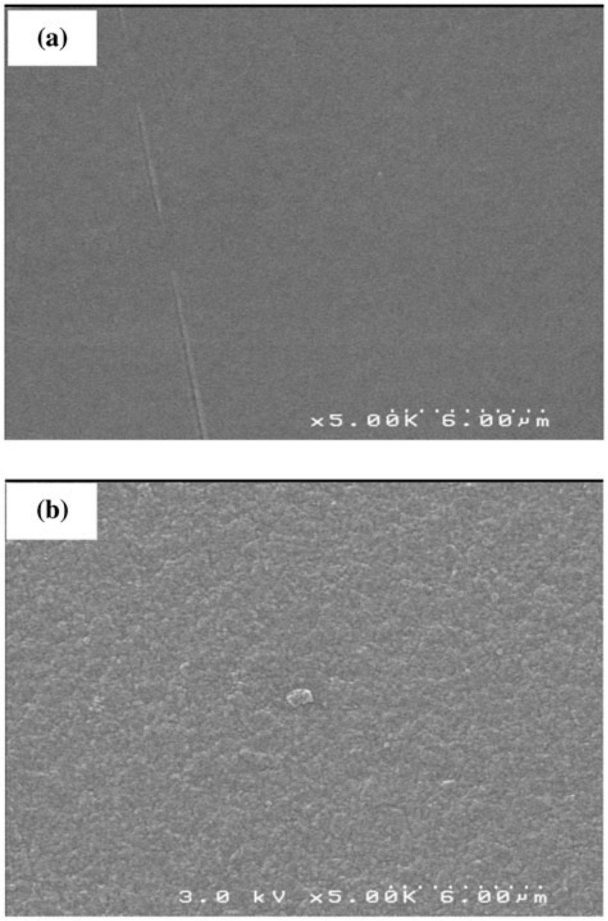

Fig. 2. SEM micrographs of polymer membranes before extraction. (a) CTA alone and (b) CTA + 2NPOE + Aliquat 336.

3.1.2. XRD

XRD spectra show that the dyes studied in this work are crystalline form. The membranes CTA-Aliquat 336, Fig. 3 indicates the presence of a broad peak in the vicinity of the angle ($2\theta = 30°$), this means that the membrane is semi-crystalline or amorphous. The same structure is observed after extraction of dyes RBA and JEA.

3.2. Solid–liquid extraction

3.2.1 Effect of the carrier concentration

Carrier in the membrane is varying in this range of 5–30 µ mol/cm^2. The results of different concentrations are given in Fig. 4(a). The percentage of extraction increase with increase of carrier concentration up to 20 µmol/cm^2 then stabilized in a higher concentration.

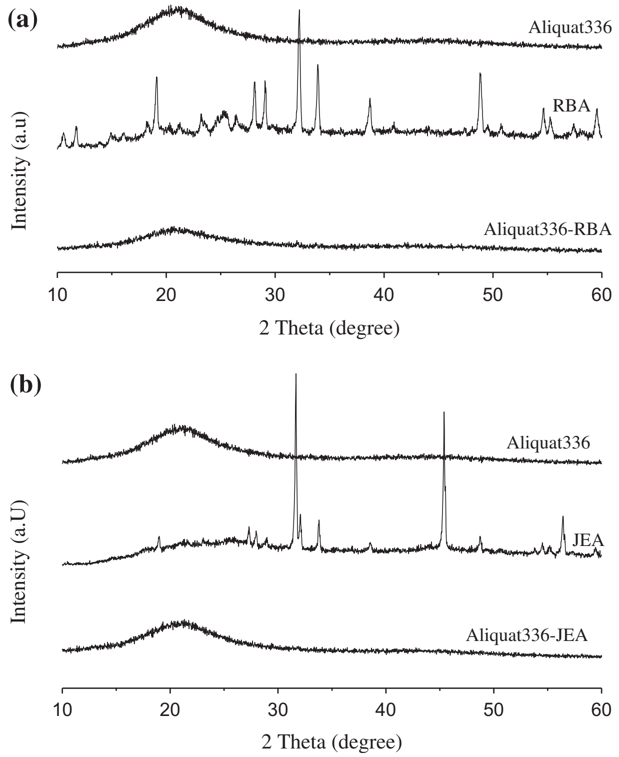

Fig. 3. Comparison of XRD spectra of samples: membrane CTA-Aliquat 336 before and after extraction of dyes separately: (a) RBA and (b) JEA.

3.2.2. Effect of the pH in aqueous solution

pH of the aqueous solution is one of the important parameters which controls the process of extraction of dyes. To study the effect of pH on the extraction of dye in the aqueous solution, pH is varied between 2 and 9, whereas carrier concentration in PIMs and dye in aqueous phase were kept constant. The relationship between the extraction efficiency and the pH is given in Fig. 4(b). The results show that pH had only a slight change in the pH interval [3–7], indicating that the electrostatic mechanism was not the only mechanism for dye adsorption in the solution, but was also affected by the chemical reaction between the dye molecules and the membrane CTA-Aliquat 336 [22].

3.2.3. Effect of initial dye concentration

The variation of the dye concentration at the optimum condition has been done over the range of 50–250 ppm. Fig. 5(a) shows that the extraction of the anionic dye is constant in the interval 50–200 ppm. The efficiency of extraction is getting 98%, after this value (200 ppm) and a decrease of the extraction efficiency is observed with the increase of the dye concentration. This may be due to membrane saturation and lower effective membrane area. This behavior was also found in the study concerning the removal of methylene blue by PIMs (CTA-D2EHPA) [23].

3.2.4. Effect of stirring speed

Fig. 5(b) shows the influence of stirring speed on the extraction of the RBA and JEA by the matrix CTA-Aliquat 336. The result indicates that the extraction efficiency increases by the increasing speed, the maximum was obtained at 350 rpm. For the speed faster than 350 rpm, we obtained the decrease of the extraction efficiency. This is caused by the decrease of permeability which is the consequence of the turbulence created by stirring. The same result was obtained by Muturaman and Teng [24], who studied the transport of rhodamine B (cationic dye) across supported liquid membrane using vegetable oil as carrier. The same in our previous work we obtained 350 rpm [23].

3.3. Adsorption isotherms

The adsorption isotherm provides important models in the description of adsorption behavior. It describes how the adsorbate interacts with the adsorbent and provides insight into the nature and mechanism of the adsorption process. When the adsorption reaction reaches a state of equilibrium, the adsorption isotherm may indicate the site of molecules of adsorbate between the solid phase and the liquid phase [25]. Therefore, it is essential to establish the most appropriate correlation of equilibrium curves to optimize the conditions for the design of adsorption systems. In the present work, the isotherms of Langmuir, Freundlich, and Temkin were used to study the adsorption behavior and we found that all three could be used to explain the adsorption behavior.

3.3.1. Freundlich isotherm

The linear equation represented by the Freundlich equation:

$$\log(Q_e) = \log(K_f) + \frac{1}{n} \times \log(C_e) \tag{2}$$

Q_e: the amount of the dye adsorbed per unit mass of the membrane (mg/g) at equilibrium.

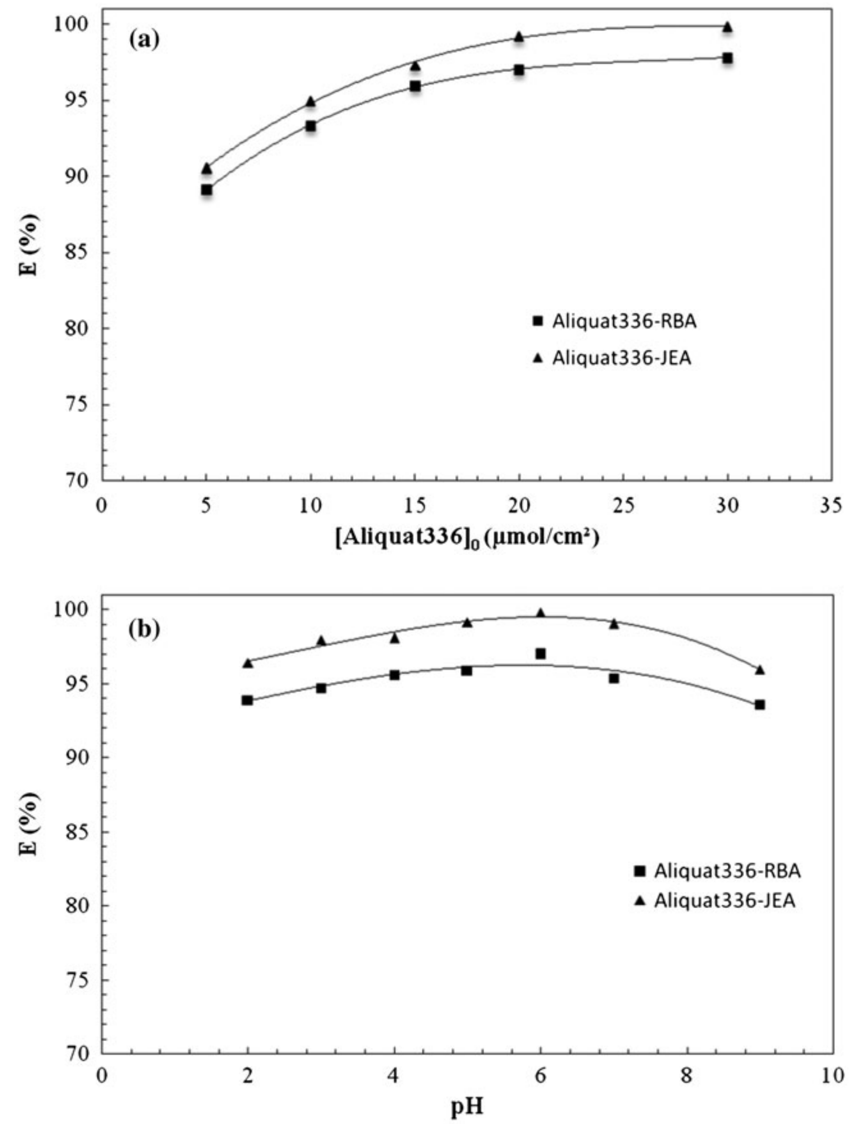

Fig. 4. (a) Effect of carrier concentration onto the extraction efficiency. Stirring time = 6 h, stirring speed 350 rpm, pH = 6, and concentration of the dye 100 ppm. (b) Effect of the pH of the aqueous solution onto the extraction efficiency: stirring time = 6 h, stirring speed 350 rpm, [Aliquat 336] = 27 μmol/cm^2, and concentration of the dye 100 ppm.

C_e: the concentration of the dye equilibrium in the solution (mg/l).

K_f: constant of the Freundlich adsorption linked to the adsorption capacity of the adsorbent.

n: dimensionless constant.

log(Q_e) vs. log(C_e) for the different cases studied are presented in Fig. 6. Constants K_f, n, and R^2 are summarized in Table 1. The model is applicable in the case of extraction of acid dyes with CTA-Aliquat 336. The correlation coefficients obtained with the acid dyes are in the range of 0.904–0.96.

3.3.2. Langmuir isotherm

The Langmuir adsorption isotherm assumes that adsorption occurs at specific homogeneous sites within the adsorbent and found successful application in many studies of monolayer adsorption. The Langmuir equation is given in the following equation [26]:

$$q_e = \frac{q_m K_L C_e}{(1 + K_L C_e)} \tag{3}$$

Eq. (3) can be rearranged to a linear form:

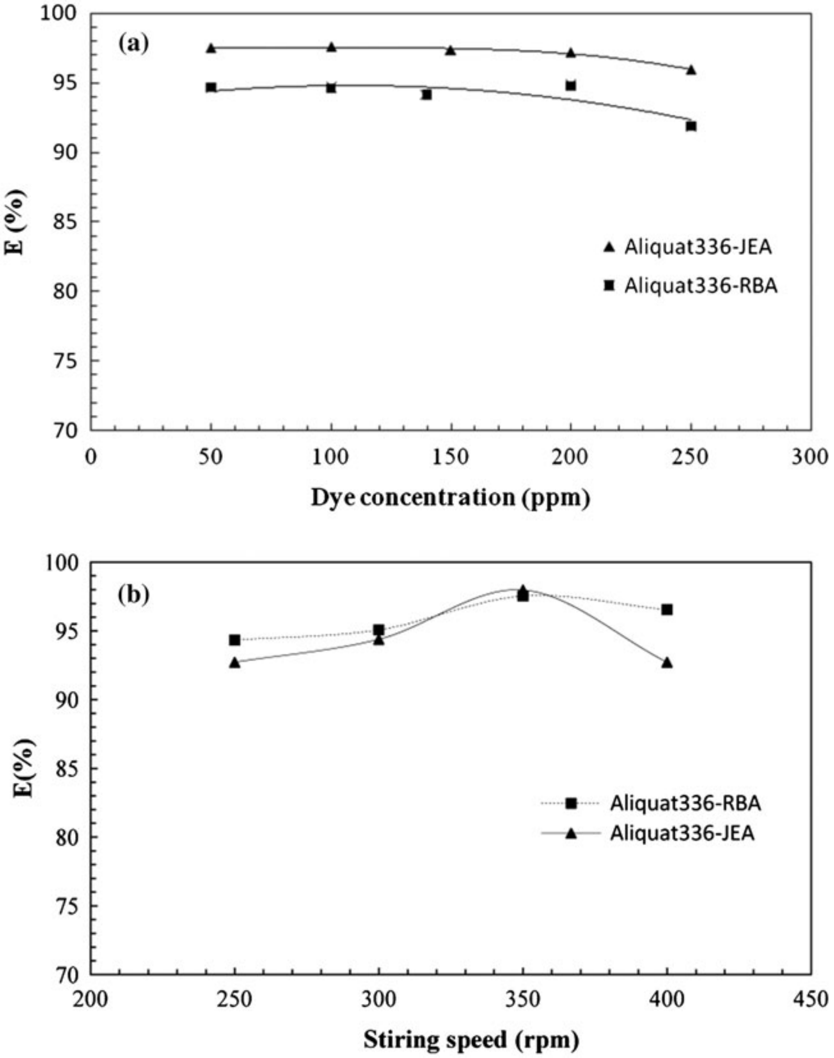

Fig. 5. (a) Effect of the concentration of the dye onto the extraction efficiency: Stirring time = 6 h, pH = 6, stirring speed 350 rpm, and [Aliquat 336] = 27 µmol/cm^2. (b) Effect of the stirring speed onto the extraction efficiency: stirring time = 6 h, pH = 6, stirring speed 350 t /min, [Aliquat 336] = 27 µmol/cm^2, and the concentration of the dye 100 ppm.

$$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{K_L q_m} \tag{4}$$

q_m is the maximum adsorption capacity corresponding to complete monolayer coverage on the surface (mg/g).

q_e is the solid phase adsorbate concentration in equilibrium (mg/g).

C_e is the concentration of adsorbate at equilibrium (mg/l).

K_L is the Langmuir constant (mg/l).

q_m and K_L are Langmuir constants related to adsorption capacity and rate of adsorption, respectively.

The constants can be evaluated by the intercepts and the slopes of the linear plots of C_e/q_e vs. C_e.

The Langmuir isotherm model was used to describe the relationship between the amount of dye adsorbed and its equilibrium concentration in solutions. The Langmuir isotherm is valid for monolayer adsorption on a surface containing same number of identical sites. The model assumes that a uniform adsorption occurs on the surface and transmigration in the plane of the surface [27].

Fig. 7 shows a linear relationship between (C_e/q_e) and (C_e). This linearity suggests the applicability of Langmuir isotherm model for the present system. The values of Q_m and K_L obtained from the linear plot and their values are given in Table 1.

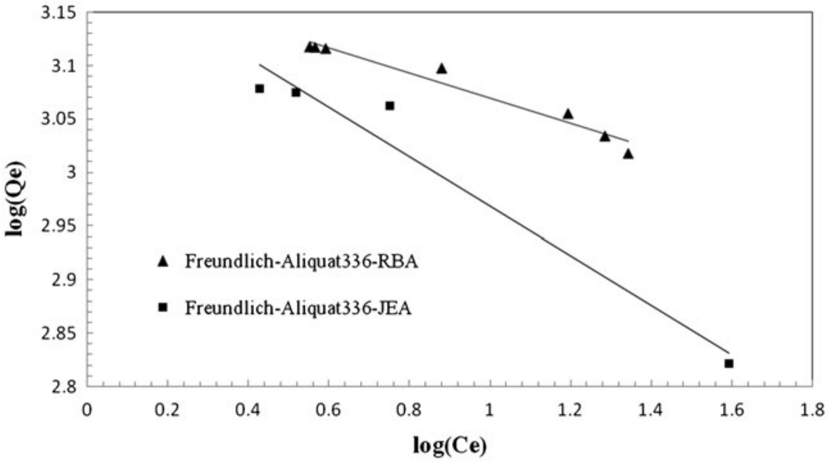

Fig. 6. The linear equation Freundlich variation log (Q_e) vs. log (C_e). In optimal conditions for extracting the acid dyes with the PIMs CTA-Aliquat 336.

Table 1
Constants of adsorption isotherms, Freundlich, Langmuir, and Temkin

	Freundlich			Langmuir				Temkin		
Carrier-Dye	n	K_f	R^2	q_m	K_L	R_L	R^2	A	B	R^2
Aliquat 336-RBA	8.47	1,541	0.96	500	2	0.006	0.95	0.866	242.1	0.873
Aliquat 336-JEA	4.34	1,581	0.904	1,000	1	0.011	0.956	0.864	207 .8	0.965

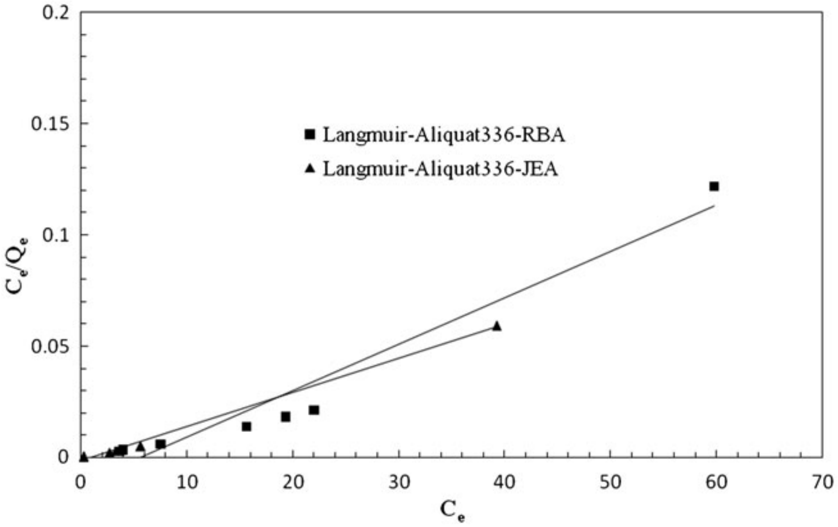

Fig. 7. The linear equation Langmuir variation (C_e /Q_e) vs. (C_e). In optimal conditions for extracting the acid dyes with the PIM CTA-Aliquat 336.

The most important characteristics of the Langmuir isotherm can be expressed in term of a dimensionless separation factor (R_L), defined as:

$$R_L = \frac{1}{(1 + K_L C_0)} \tag{5}$$

K_L is the Langmuir constant and C_0 is the highest dye concentration (mg/l).

The separation factor R_L indicates that the adsorption is favorable if the R_L value lies between 0 and 1. Unfavorable if $R_L > 1$, linear if $R_L = 1$, and irreversible if $R_L = 0$ [28]. Values of R_L were found to be 0.006 and 0.073 for the RBA and JEA, respectively, and confirmed that the adsorption of RBA and JEA in to the matrix CTA-Aliquat 336 is favorable under studied conditions.

3.3.3. Temkin isotherm

The Temkin isotherm assumes that the heat of adsorption of all the molecules in layer decreases linearly with coverage due to adsorbent–adsorbate interactions and that the adsorption is characterized by a uniform distribution of the bonding energies, up to some maximum binding energy [29]. The Temkin equation is given as:

$$q_e = A \ln(B \times C_e) \tag{6}$$

A is the equilibrium binding constant (l/mg) corresponding to the maximum binding energy, and constant B is related to the heat of adsorption. A plot of q_e vs. $\ln(C_e)$ (Fig. 8) enables the determination of the isotherm constants A and B from the slope and intercept of the straight line plot. The values of the parameters are given in Table 1.

3.4. Kinetics of the dyes removal

3.4.1. Pseudo-first-order

The easiest model to analyze the kinetics of the extraction of acid dyes by membrane CTA-Aliquat 336 is the pseudo-first-order. The linearized form of the pseudo-first-order equation of Lagergren is expressed as following [30]:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2,302} \times t \tag{7}$$

According to Low et al. [31] the equation can be written as following:

$$\ln\left(1 - \frac{q_t}{q_e}\right) = -k_1 t \tag{8}$$

where q_e and q_t (mg/g) are the amounts of dye adsorbed at equilibrium and at time t (min), respectively. k_1 (min^{-1}) is the rate constant of adsorption. The model is connected to the process of removing the dye by the inclusion membrane polymer. The values of k_1 were calculated by the plots of $\ln(1 - q_t/q_e)$ vs. t (Fig. 9). The linear plots must pass through the origin $(0, 0)$. The values of k_1 obtained in the extraction of the two dyes with the membranes CTA-Aliquat 336 are summarized in Table 2.

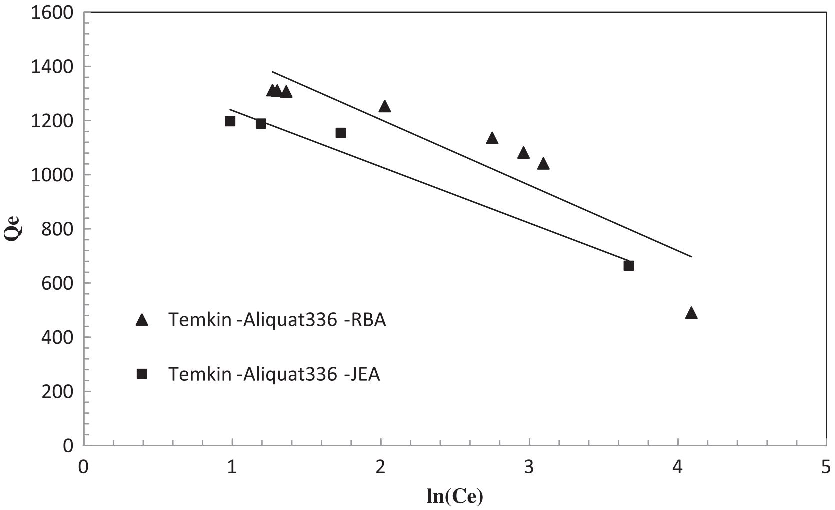

Fig. 8. The linear equation Temkin variation (Q_e) vs. $\ln(C_e)$. In optimal conditions for extracting the acid dyes with the PIM CTA-Aliquat 336.

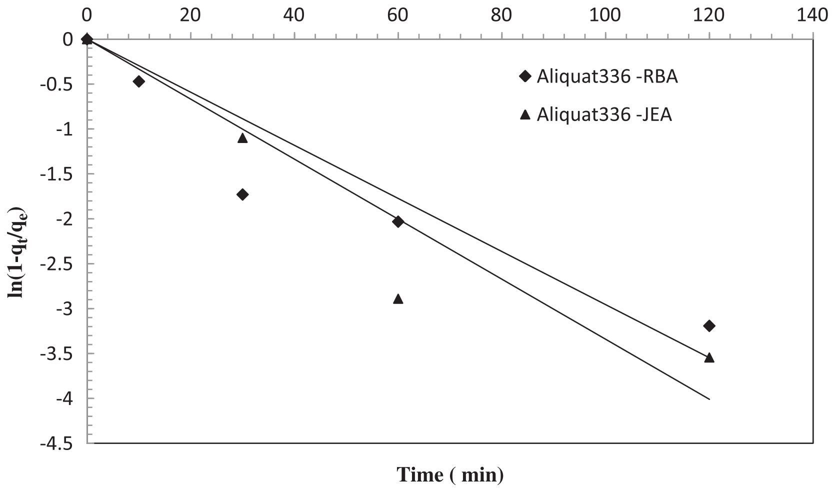

Fig. 9. Pseudo-first-order kinetics for the extraction of RBA and JEA dyes onto the PIMs at the optimum conditions of extraction.

3.4.2. Pseudo-second-order

The pseudo-second-order model is one of the most used in the analysis of dye removal [28,32,33], it is expressed by the following equation:

$$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + t\frac{1}{q_e} \tag{9}$$

In [31], it is expressed by the following equation:

$$\frac{1}{q_t} - \frac{1}{q_e} = \frac{1}{k_2 {q_e}^2 t} \tag{10}$$

where the equilibrium adsorption capacity (q_e) and the pseudo-second-order constants k_2 (g/(mg min)) can be determined experimentally from intercept of the plots $\frac{t}{q_t}$ vs. t, otherwise directly from the slope of plot $\frac{1}{q_t} - \frac{1}{q_e}$ vs. $\frac{1}{t}$ Fig. 10. The rate constant k_2 of the model and the regression R^2 of the two dyes are summarized in Table 2. The R^2 values of pseudo-second-order are relatively higher so the latter is preferable than the pseudo-first-order.

From the rate constant of the pseudo-second-order, we have calculated the activation energy of the reaction of dye extraction with membrane CTA-Aliquat 336.

The rate constant of the second order constant pseudo-adsorption is expressed in function of temperature by the following Arrhenius relationship [34]:

$$\ln(k_2) = \ln(A') - \frac{E_a}{RT} \tag{11}$$

The values of E_a obtained are 42.53 and 47.77 kJ/mol for Aliquat 336-RBA and Aliquat 336 –JEA, respectively. The values are above 40 kJ/mol. It can be concluded that the adsorption of RBA and JEA onto CTA-Aliquat 336 follows a chemisorption mechanism.

3.4.3. Intra-particle diffusion

The adsorbate species are probably transported from the bulk of the solution into the solid phase through intra-particle diffusion/transport process, which is often the step-limiting rate in many

Table 2
The kinetic parameters of the extraction of anionic dyes by PIMs (CTA-Aliquat336)

	First order		Second order		Intra-particle diffusion			
	k_1 (min^{-1})	R^2	k_2 (mg^{-1} mn^{-1})	R^2	K_{p1} (mg/mn$^{0.5}$)	R^2	K_{p2} (mg/mn$^{0.5}$)	R^2
Aliquat336-RBA	0.029	0.856	0.0021	0.975	550.9	0.962	0.604	0.738
Aliquat336-JEA	0.033	0.872	0.0025	0.973	1243	0.908	0.074	0.662

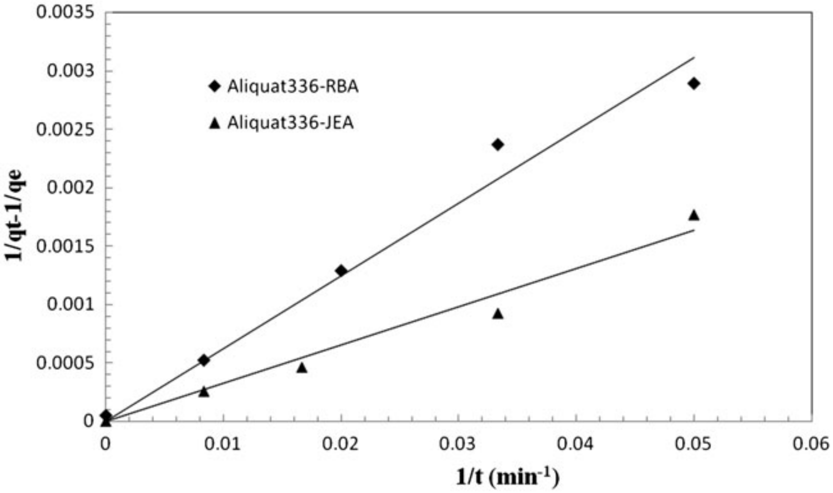

Fig. 10. Pseudo-second-order kinetics for the extraction of RBA and JEA dyes onto the PIMs at the optimum conditions of extraction.

adsorption processes, especially in a rapidly stirred batch reactor [35]. In the process of extraction of anionic dyes by PIM, there is intra-particle diffusion. To verify the existence of this type of dye transfer, we have used the model of intra-particle diffusion. This model can be written by the following:

$$q_t = k_\text{p} \times t^{0.5} + C \qquad (12)$$

where q_t is the amount of dye adsorbed at time t, C is the intercept, and k_p is the intra-particle diffusion rate constant (mg min$^{1/2}$/g). The values of q_t found vary linearly with the values of $t_{1/2}$.

It has been documented that if the line passes through the origin the intra-particle diffusion is the only rate-determining step. Otherwise, some other mechanisms along with the intra-particle diffusion are also involved which is reflected in multi-linearity of the q_t vs. $t^{1/2}$ plot [36].

Fig. 11 shows multi-linearity represented by two different stages, the first stage represents the mass transfer of dyes through boundary layers of liquid and adsorption of dyes in the available sites on the

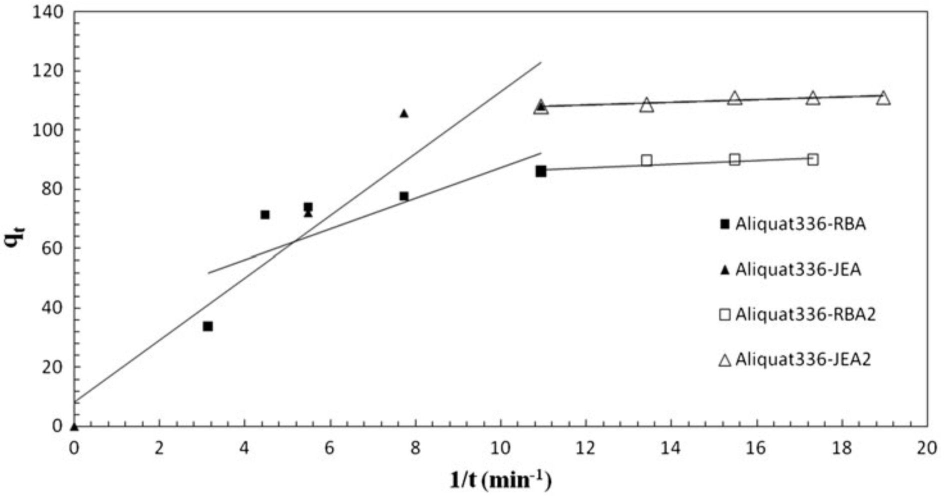

Fig. 11. The intra-particle diffusion plots for adsorption of RBA and JEA dyes onto the PIMs at the optimum conditions of extraction.

external surface of the PIMs. The second stage represents the penetration of dye molecule in the porous structures of the membrane. Finally, the dye is adsorbed on the active sites of the internal surface of the membrane.

The transportation of dye molecules from external surface to bulk of the adsorbent shows an increasing diffusion resistance, which reflects the slow of the second-stage adsorption.

The slopes of the straight line of the two stages called the speed setting k_{p1} and k_{p2}. Speed parameters of the various steps listed in Table 2 shows that the rate constant of the first adsorption step (k_{p1}) is greater than that of the second adsorption step (k_{p2}) for the two dyes. Neither linear profiles passes through the origin, confirming that the intra-particle diffusion is not the only process that controls the extraction of dye (RBA, JEA) by the matrix CTA-Aliquat 336.

4. Conclusion

Aliquat 336 incorporated into the PIMs is an excellent extractant for acid dyes RBA and JEA. Optimization of the parameters studied allowed us having greater than 98% extraction efficiency. The Langmuir model was more suitable to describe the removal of acid dyes onto the PIMs with maximum monolayer adsorption capacity of 1,000 mg/g for remove of the JEA with the membrane CTA-Aliquat 336. The pseudo-second-order kinetic model fits very well with the kinetic data. The activation energy of remove of RBA and JEA into the matrix CTA-Aliquat 336 exceeds 40 kJ/mol, which suggests that the dye is chemically bonded to the membrane.

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