



Lactic acid recovery from water by Amberlite IRA-400

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ABSTRACT

The aim of this investigation is to recover lactic acid, which widely produced in aqueous solutions by fermentation processes, from water by Amberlite IRA-400. This separation is very important for the production of lactic acid because of the accumulation of lactic acid must be prevented for the fermentation process to be continued continuously. Amberlite IRA-400 is an ion-exchange resin used as an adsorbent in this separation study. Adsorption time, initial concentration of lactic acid, amount of Amberlite IRA-400 and temperature have been selected as the parameters affecting the adsorption process. The adsorption capacities of Amberlite IRA-400 under various conditions were detected and compared with each other. It has been determined that a significant lactic acid amount was recovered by Amberlite IRA-400. Langmuir, Freundlich and Temkin isotherm models were utilized to explain the adsorption mechanism. Kinetic models such as pseudo-first-order, pseudo-second-order kinetic models were used, and also, Weber-Morris intra-particle diffusion model was performed. Among the isotherms used, Freundlich isotherm model was found to fit the experimental results. Besides, the most favorable kinetic model was determined as pseudo-second-order kinetic model.

Keywords: Lactic acid; Amberlite IRA-400; Adsorption; Recovery

1. Introduction

Lactic acid is also known as alpha-hydroxy acid. It contains carboxylic and hydroxyl groups. These groups play significant roles in various chemical production processes. Lactic acid has been utilized as a raw material for polylactic acid production which is a biodegradable polymer. Furthermore, lactic acid is also used in the manufacture of many chemicals like pyruvic acid, acrylic acid, propionic acid, propylene glycol, acetaldehyde and, 2,3-pentanedione. It has also been utilized in various industries including food, pharmaceutical, cosmetic and textile industries [1].

Lactic acid can be acquired by either biological or chemical synthesis. However, it has been broadly produced in aqueous solutions by fermentation processes [2]. During the production of lactic acid by fermentation, pH change is

very significant. Because, lactic acid accumulation during fermentation decreases pH of the fermentation broth and microorganism activity is reduced. Therefore, the accumulation of lactic acid must be prevented for the fermentation process to be continued continuously [3]. Also, lactic can be present in waste streams or effluents in numerous chemical industries. Because of these reasons, it is very significant to recover lactic acid from the water. To this end, different techniques are frequently utilized to recover lactic acid from the water like solvent extraction [4,5], reactive extraction [6,7], direct distillation [8], reactive distillation [9], reverse osmosis [10,11], electrodialysis [12,13], ultrafiltration [13,14] and adsorption [15,16]. Among these recovery techniques, adsorption has many benefits like a higher efficiency, easy handling, low capital, operating costs, quick response [17,18],

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absence of sludge and harmful by-product generation [19]. This technique also can be integrated easily with the fermentation process [2]. The adsorption of lactic acid was examined by using various adsorbents like multiwall carbon nanotube [1], zeolite [20], activated carbon [2,21] and layered double hydroxide [16].

Amberlite IRA-400, a macroporous and strong base anion-exchange resin, can be used for the removal of certain ions from a solution by replacing them with other ions [22,23]. In other words, Amberlite IRA-400 can take place the adsorption by means of an ion exchange mechanism. So, it has been preferred in the adsorption process under different operating conditions. Besides, anion-exchange resins are non-toxic for microorganisms; hence they can be utilized directly in the fermenter [24]. Various materials such as heavy metals [22,25], clavulanic acid (a beta-lactam antibiotic) [26,27] have been tested by Amberlite IRA-400. Many of the published studies were examined by the removal of lactic acid using Amberlite IRA-400 by column method [28–32]. Moldes et al. [33] studied the resins (Amberlite IRA-900, IRA-400, IRA-96 and IRA-67) for lactic acid recovery in simultaneous saccharification and fermentation (SSF) processes. Ataei and Vasheghani-Farahani [34] examined in situ separation of lactic acid from fermentation broth using Amberlite resin. Pradhan et al. [24] investigated the adsorption behavior of lactic acid on anionic resins (Amberlite IRA-92, IRA-400 and IRA-425) by batch method. However, little research has been done on the adsorption mechanisms of lactic acid under different operating conditions by batch method.

In the current study, adsorption based on ion-exchange was examined for lactic acid separation from the water using Amberlite IRA-400. Some parameters like adsorption time, the initial lactic acid concentration, Amberlite IRA-400 amount and temperature were investigated. The acquired experimental data were modelled using the adsorption isotherm models including Langmuir, Freundlich and Temkin isotherm models, and kinetic models including the pseudo-first-order, pseudo-second-order kinetic models and Weber-Morris intra-particle diffusion model.

2. Materials and methods

The lactic acid (>90%) was supplied by Merck (USA). The lactic acid solutions were prepared by dissolution of the initial acid concentration of about 2% (w/w) (pH = 2.11), 4% (w/w) (pH = 1.90), 6% (w/w) (pH = 1.81), 8% (w/w) (pH = 1.76) and 10% (w/w) (pH = 1.66) in distilled water. Amberlite IRA-400 resin (chloride form) was acquired from Sigma-Aldrich (USA) and was utilized without further treatment. The characteristic of the Amberlite IRA-400 was summarized in Table 1.

In the adsorption experiments, the volume of lactic acid solution of 5 mL was utilized. Aqueous lactic acid solution and a known quantity of Amberlite IRA-400 were added to glass Erlenmeyer. Experiments were conducted by shaking glass Erlenmeyer in thermostatic water bath shaker (Nüve ST 30, Turkey) for the determined optimum time. The shaker speed was 150 rpm. Amberlite IRA-400 was separated from the solution by the filtration method. The concentrations of lactic acid before and after adsorption were analyzed by means of an automatic titrator (SI Analytics, Schott Titroline, Germany) using 0.1 N NaOH solution.

Adsorption capacity (q_e , mg g⁻¹), the adsorbed quantity of lactic acid by Amberlite IRA-400 at equilibrium, was calculated by means of Eq. (1):

$$q_e = \left[\frac{(C_0 - C_e)}{M} \right] \times V \quad (1)$$

where C_0 and C_e (mg L⁻¹) represent the concentrations of initial and equilibrium lactic acid, M (g) implies the quantity of adsorbent and V (L) signifies the volume of aqueous lactic acid solution.

3. Results and discussion

3.1. Determination of optimum adsorption time

At the beginning of adsorption experiments, the optimum adsorption time was determined by using Amberlite IRA-400 of 0.1 g and the initial concentration of lactic acid solution of 10% (w/w) at 25°C. Fig. 1a depicts the effect of time on the lactic acid adsorption by Amberlite IRA-400. It was obvious from Fig. 1a that the adsorption capacity of lactic acid increased with increasing time at the initial stages, then, the adsorption became slow until the equilibrium was reached. At the start, the adsorption is rapid due to the adsorption sites on the adsorbent surface are free. Then, the adsorption becomes slow owing to decreasing in the number of available adsorption sites [35,36]. As illustrated in Fig. 1a, the equilibrium attained about 180 min. Consequently, the optimum adsorption time was determined as 180 min for the next experiments.

3.2. Effect of different parameters on the adsorption of lactic acid by Amberlite IRA-400

In the second stage of experimental investigation, the effect of initial lactic acid concentration was examined at the optimum adsorption time. In these experiments, different acid concentrations of about 2% w/w (20.3 g L⁻¹), 4% w/w (39.9 g L⁻¹), 6% w/w (60.55 g L⁻¹), 8% w/w (80.96 g L⁻¹) and 10% w/w (100.81 g L⁻¹) were used. The adsorbent amount is 0.1 g and the temperature is 25°C. After that, the effect of the amount of adsorbent was detected by using Amberlite IRA-400 of 0.1, 0.2, 0.3, 0.4 and 0.5 g and initial lactic acid concentration of 10% (w/w) 25°C. Finally, the effect of temperature was investigated at the various temperatures of 25°C, 45°C and 65°C. In these experiments, the amount

Table 1
Characteristic of Amberlite IRA-400 resin

	Properties
Matrix	Styrene-divinylbenzene (gel)
Functional group	Quaternary ammonium
Ionic form	Cl ⁻
Capacity	1.4 meq mL ⁻¹ by wetted bed volume
Particle size	600–750 μm
Operating pH	0–14

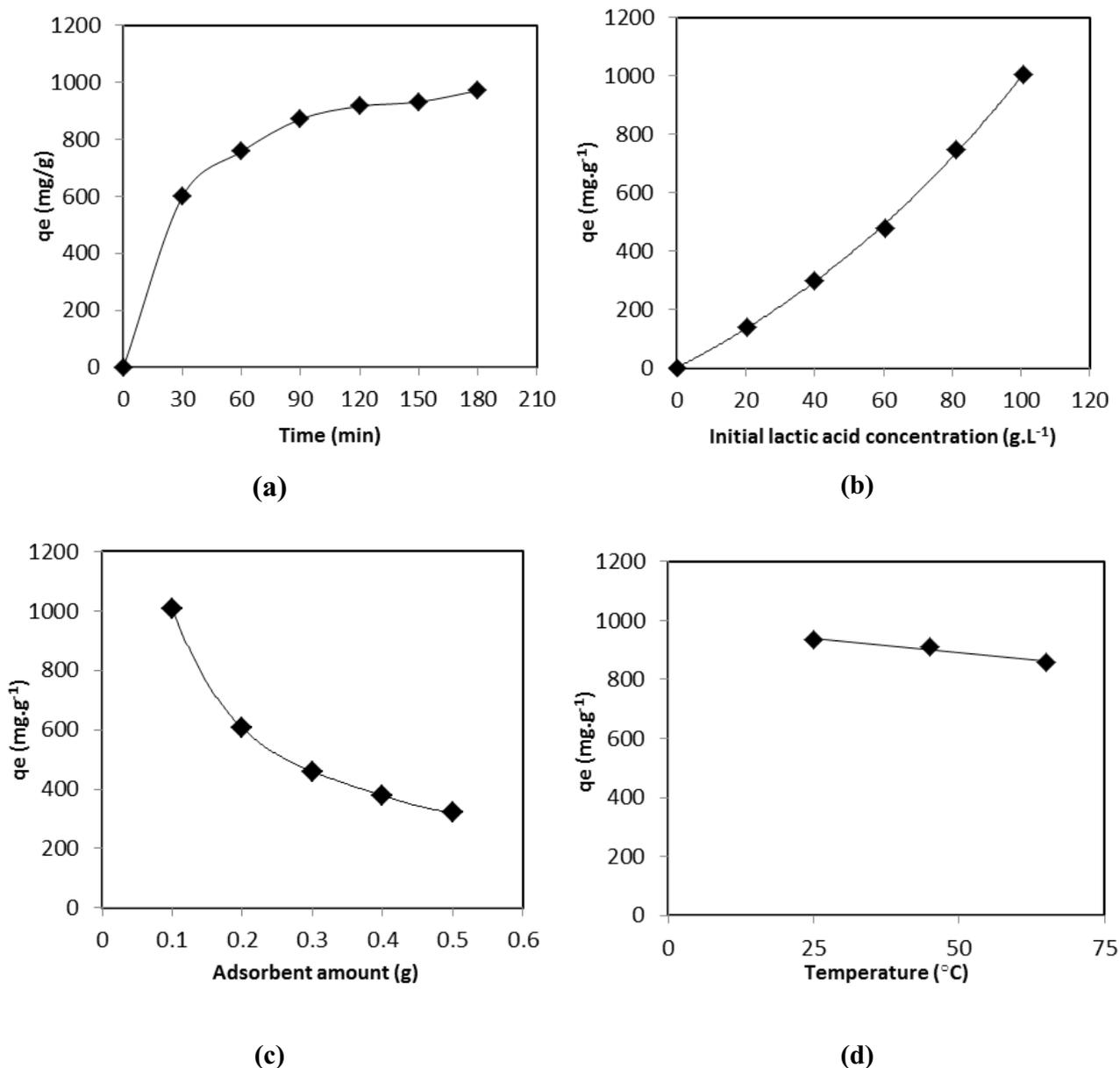


Fig. 1. (a) Effect of contact time, (b) initial acid concentration, (c) adsorbent amount and (d) temperature on the adsorption of lactic acid by Amberlite IRA-400.

of adsorbent is 0.1 g and the concentration of lactic acid is 10% w/w.

Fig. 1 represents the effect of initial lactic acid concentration, adsorbent amount and temperature on the adsorption. From Fig. 1b, the adsorption capacity was increased by increasing the initial concentration of lactic acid. It can be explained by the driving force for mass transfer. As the initial acid concentration increases, the driving force for mass transfer becomes greater, so the adsorption capacity increases [37]. A similar result was obtained by Uslu and Majumder [38] for lactic acid adsorption by Amberlite XAD-7. As can be seen in Fig. 1c, the increase in the amount of Amberlite IRA-400 resulted in decreasing the adsorption capacity. The maximum value of adsorption capacity

was obtained as $1,010 \text{ mg g}^{-1}$ when the smallest amount of Amberlite IRA-400 (0.1 g) was used. Polowczyk et al. [39] was used Amberlite IRA-400 for the removal of molybdenum(VI) and vanadium(V), and they observed that the adsorption capacity decreased with the increase in resin amount. Furthermore, as shown in Fig. 1d, the adsorption capacity decreased slightly with the increasing temperature. It shows that the lactic acid adsorption is exothermic. Similarly, Fu et al. [40] have investigated the adsorption of fumaric acid by Amberlite IRA-400 and they found that the values of equilibrium adsorption capacity decreased with the increasing temperature.

Amberlite IRA-400 is a strong base resin containing a positive charged quaternary ammonium functional group.

The quaternary ammonium functional group can adsorb both the lactate ion and molecular lactic acid. In this case, the adsorption mechanism of lactic acid varies depending on the degree of ionization of lactic acid in the solution. This is related to the relationship between the pKa value and the pH of the solution. The degree of ionization of lactic acid in the solution can be calculated by the following equation [24,41]:

$$\text{Ionized\%} = \frac{100}{1 + \exp^{(\text{pKa} - \text{pH})}} \quad (2)$$

From Eq. (2), the degree of ionization of lactic acid can be obtained as 13.5%, 29.7%, 46.5%, 24.2%, 10.5%, 4.2% and 1.6% for pH 2.0, 3.0, 4.0, 5.0, 6.0, 7.0 and 8.0.

At the pH value (2.0, 6.0, 7.0 or 8.0), the undissociated form of lactic acid is predominant and Amberlite IRA-400 predominantly binds to the molecular lactic acid. At the pH range (3.0–5.0), the dissociated form of lactic acid is predominant, in this case, Amberlite IRA-400 can form ionic bonds with the lactate ion as follows:



In this equation, L signifies the lactate ion and R symbolizes a cationic quaternary ammonium functional group of the resin. Chloride form (Cl⁻) of quaternary ammonium of Amberlite IRA-400 has a weak base characteristic. This property facilitates the adsorption of acid through acid-base interaction. In this case, lactic acid adsorption with Amberlite IRA-400 takes place by means of an ion-exchange mechanism [24,42,43]. In this study, under the pH range studied (1.66–2.11), the degree of ionization of lactic acid (9.9%–14.8%), the molecular lactic acid (predominant) and the lactate ion binds to Amberlite IRA-400. Cao et. al [43] studied the adsorption of lactic acid on IRA-400, at the pH above (5.0) and below (2.0) the pKa (3.86) of lactic acid with column separation process. They reported that Amberlite IRA-400 was successfully applied for the separation of lactic acid at pH above and below the pKa (3.86). Raya-Tonetti et al. [32] was also used Amberlite IRA-400 in an upstream fluidized bed column to recover lactic acid directly from fermentation at pH 8.0.

Table 2 shows a comparison of the values of adsorption capacity of Amberlite IRA-400 and various resins for the lactic acid adsorption.

3.3. Adsorption isotherm models for lactic acid adsorption by Amberlite IRA-400

The adsorption mechanism is generally described by an isotherm model that correlates the equilibrium concentration of the solid phase (adsorbent) with the equilibrium concentration of the liquid phase (adsorbate). In the current work, the most used isotherm models, Langmuir, Freundlich and Temkin isotherm models were utilized to express and model the adsorption characteristic of lactic acid by Amberlite IRA-400. These isotherms models and equations summarized in Table 3 [17,45].

Fig. 2 illustrates the linear plots of Langmuir, Freundlich and Temkin isotherm models for the adsorption of lactic acid by Amberlite IRA-400. Table 4 shows the parameters of Langmuir, Freundlich and Temkin isotherm models for calculated from the linear plots of isotherm models. Langmuir isotherm model approach assumes that the adsorption occur monolayer on the surface, indicating that the adsorbent surface is homogeneous and has energetically equivalent adsorption sites [46]. As can be seen in Table 4, Langmuir isotherm parameters have negative values, which suggesting that the adsorption mechanism of the studied system does not explain sufficiently by the Langmuir approach [47,48]. Since the Langmuir isotherm model approach is not sufficient to explain the adsorption mechanism, non-linear Langmuir–Freundlich combination isotherm was employed. Langmuir–Freundlich combination isotherm model can be expressed below as [41,49]:

$$\text{Non-linear form: } q_e = \frac{Q_m K C_e^{\frac{1}{n}}}{1 + K C_e^{\frac{1}{n}}} \quad (10)$$

where Q_m is Langmuir adsorption constant related to the adsorption capacity (mg g^{-1}), K is equivalent to the Langmuir isotherm constant (L mg^{-1})^{1/n}, and n is equivalent to Freundlich adsorption constant related to adsorption intensity. As shown in Table 4, the obtained isotherm model parameters were positive values, which means this isotherm

Table 2
Comparison of adsorption capacities of ion exchange resins for lactic acid adsorption

Ion exchange resins	Type	Initial lactic acid concentration	Adsorbent dose (g)	Temperature (°C)	Adsorption capacity (mg g ⁻¹)	References
Amberlite IRA-400	S	10% w/w (100.81 g L ⁻¹)	0.1	25	1,010	This study
Amberlite IRA-400	S	18 g L ⁻¹	0.5	30	48	[24]
Amberlite IRA-96	W	100 g L ⁻¹	25	25	210.46	[42]
Amberlite IRA-402	S	160 mg lactic acid/5 ml	1	25	119	[44]
Amberlite IRA-67	W	160 mg lactic acid/5 ml	1	25	126	[44]
Amberlite IRA-67	W	6.66 g L ⁻¹	5	25	60.10	[15]
Amberlite IRA-67	W	208.98 g L ⁻¹	1	25	743.20	[2]

S: Strong anion exchange resin.
W: Weak anion exchange resin.

Table 3
Adsorption isotherm models

Isotherm models	Equations
Langmuir isotherm model	Non-linear form: $q_e = \frac{Q_m K_L C_e}{1 + K_L C_e}$ (4)
	Linear form: $\frac{C_e}{q_e} = \frac{C_e}{Q_m} + \left(\frac{1}{K_L Q_m} \right)$ (5)
	Plot: $\frac{C_e}{q_e}$ vs. C_e
	K_L : Langmuir isotherm constant (L mg ⁻¹) Q_m : Maximum adsorption capacity (mg g ⁻¹)
	Non-linear form: $q_e = K_f C_e^n$ (6)
Freundlich isotherm model	Linear form: $\log q_e = \log K_f + \frac{1}{n} \log C_e$ (7)
	Plot: $\log q_e$ vs. $\log C_e$
	K_f : Freundlich isotherm constant related to adsorption capacity (mg g ⁻¹)(L mg ⁻¹) ⁿ n : Freundlich isotherm constant related to adsorption intensity
	Non linear form: $q_e = B \ln(K_i C_e)$; $B = \frac{RT}{b_i}$ (8)
Temkin isotherm model	Linear form: $q_e = B \ln K_i + B \ln C_e$ (9)
	Plot: q_e vs. $\ln C_e$
	K_i : Temkin isotherm constant (L g ⁻¹) b_i : Temkin isotherm constant related to adsorption heat (J mol ⁻¹)

model can be used to explain the adsorption. However, as can be seen in Table 4, Freundlich isotherm model was better fitted to the experimental adsorption results due to its having the highest regression coefficient ($R^2 = 0.9935$). Freundlich isotherm model describes the multilayer adsorption on the heterogeneous adsorbent surface. Furthermore, Fig. 3 depicts the comparison of the values of experimental adsorption capacity with the values of adsorption capacity obtained from the parameters of isotherm models. The adsorption capacity of the isotherm models was calculated from non-linear isotherm equations by placing the calculated isotherm parameters given in Table 4. As depicted in Fig. 3, the experimental data were well characterized by Freundlich isotherm model.

3.4. Adsorption kinetics models for lactic acid adsorption by Amberlite IRA-400

Several kinetic models including pseudo-first-order, pseudo-second-order kinetic models and Weber-Morris intra-particle diffusion model were performed to appraise the experimental results of lactic acid adsorption. These kinetics models and their equations were summarized in Table 5 [1,50]. To examine the adsorption kinetics of lactic acid by Amberlite IRA-400, was studied for a period of 180 min for an initial lactic acid concentration of 10% (w/w) at 25°C.

The amount of Amberlite IRA-400 was 0.1 g. The values of adsorption capacity were calculated by means of Eq. (1) and also shown in Fig. 1a. After that, the kinetics model parameters were calculated by using experimental adsorption capacity results by means of the equations summarized in Table 5. Fig. 4 demonstrates the linear plots of pseudo-first-order, pseudo-second-order and Weber-Morris intra-particle models for the adsorption of lactic acid by Amberlite IRA-400. The kinetic parameters calculated from the linear plots of models for the adsorption of lactic acid on Amberlite IRA-400 are shown in Table 6. The results given in Table 6 indicated that among these three models, pseudo-second-order kinetic model had the highest R^2 value. So, pseudo-second-order kinetic model was well fitted the experimental data owing to the highest R^2 value of 0.9992. Pseudo-second-order kinetic model indicates that the adsorption mechanism depends on the adsorbent and adsorbate [46,51].

4. Conclusion

Amberlite IRA-400 resin has been successfully performed for the recovery of lactic acid from the water. The current adsorption study showed that the strong base resin Amberlite IRA-400 had a high affinity for lactic acid uptake. The adsorption experiments indicated that the adsorption capacity increased with the increasing the initial lactic acid

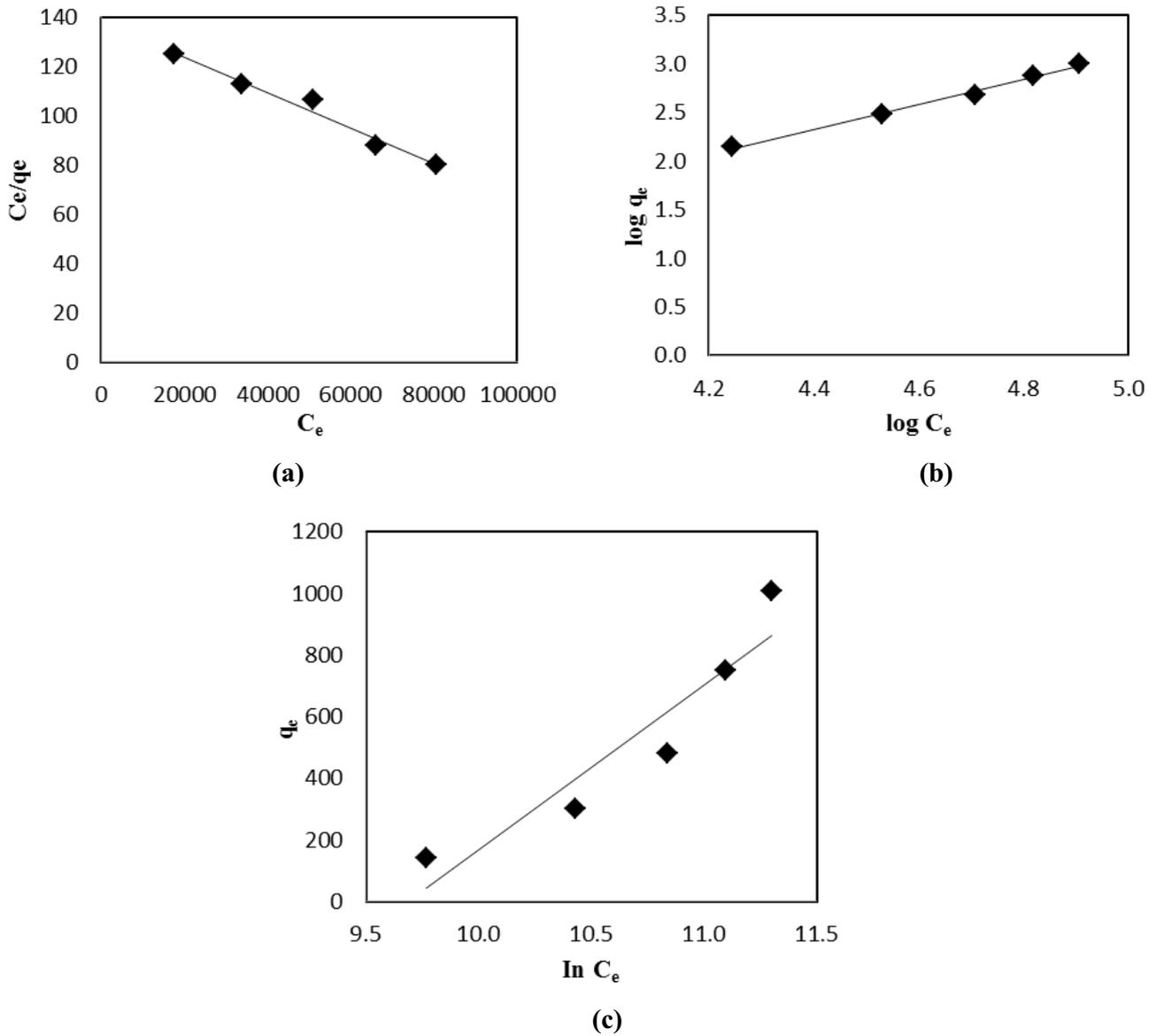


Fig. 2. Linear fits of (a) Langmuir, (b) Freundlich and (c) Temkin isotherm models for the adsorption of lactic acid by Amberlite IRA-400.

Table 4
Parameters of Langmuir, Freundlich, Langmuir–Freundlich and Temkin isotherm models for lactic acid adsorption by Amberlite IRA-400

Isotherm models	Isotherm parameters and regression coefficients			
Langmuir isotherm model	Q_m (mg g ⁻¹)	K_L (L mg ⁻¹)	R^2	
	-1,428.5	-5.06×10^{-6}	0.9763	
Freundlich isotherm model	K_f (mg g ⁻¹)(L mg ⁻¹) ⁿ	n	R^2	
	4.9×10^{-4}	0.78	0.9935	
Langmuir–Freundlich combination isotherm model	Q_m (mg g ⁻¹)	K (L mg ⁻¹) ^{1/n}	n	R^2
	1,005.5	1.202×10^{-13}	0.36	0.9898
Temkin isotherm model	K_t (L g ⁻¹)	b_t (J mol ⁻¹)	R^2	
	6.19×10^{-5}	4.62	0.8799	

Table 5
Adsorption kinetic models

Kinetic models	Equations
	Non-linear form: $\frac{dq_t}{dt} = k_1(q_e - q_t)$ (11)
Pseudo-first-order model	Linear form: $\ln(q_e - q_t) = \ln q_e - k_1 t$ (12) Plot: $\ln(q_e - q_t)$ vs. t k_1 : Kinetic rate coefficient of pseudo-first-order (min^{-1})
	Non-linear form: $\frac{dq_t}{dt} = k_2(q_e - q_t)^2$ (13)
Pseudo-second-order model	Linear form: $\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$ (14) Plot: $\frac{t}{q_t}$ vs. t k_2 : Kinetic rate coefficient of pseudo-second-order ($\text{g mg}^{-1} \text{min}^{-1}$)
	$q_t = k_{id} t^{\frac{1}{2}} + C$ (15)
Weber-Morris intra-particle diffusion model	Plot: q_t vs. $t^{\frac{1}{2}}$ k_{id} : Intra-particle diffusion rate coefficient ($\text{mg g}^{-1} \text{min}^{-1/2}$) C : Intercept related to the thickness of boundary layer
Notes	q_e : Adsorption capacity of the adsorbent at the equilibrium (mg g^{-1}) q_t : Adsorption capacity of the adsorbent at the time (t) (mg g^{-1})

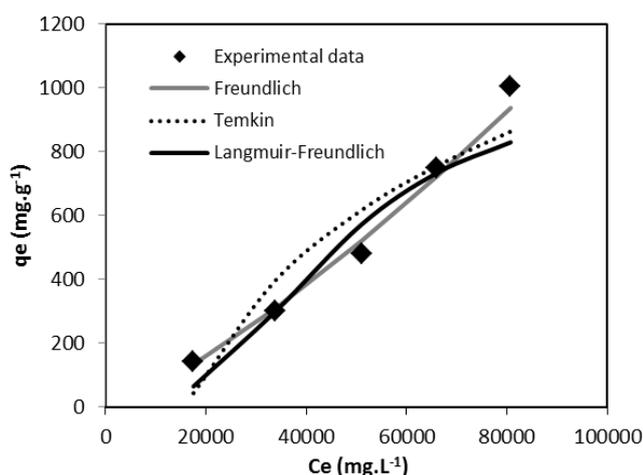


Fig. 3. Comparison of the values of experimental adsorption capacity with values of the adsorption capacity calculated from isotherm models.

concentration, and also the adsorption capacity decreased with the increasing Amberlite IRA-400 amount. In addition to, the increase in temperature resulted in decreasing adsorption capacity, indicating the lactic acid adsorption with Amberlite IRA-400 is exothermic. The maximum adsorption

Table 6
Kinetic model parameters for lactic acid adsorption by Amberlite IRA-400

Kinetic models	Kinetic parameters and regression coefficients		
Pseudo-first-order model	k_1 (min^{-1})	q_e (mg g^{-1})	R^2
	0.0194	641	0.9848
Pseudo-second-order model	k_2 ($\text{g mg}^{-1} \text{min}^{-1}$)	q_e (mg g^{-1})	R^2
	3.4×10^{-5}	1,111	0.9992
Weber-Morris intra-particle diffusion model	k_{id} ($\text{mg g}^{-1} \text{min}^{-1/2}$)		R^2
	45.76		0.9375

capacity for lactic acid removal was obtained to be 1,010 mg g^{-1} at the experimental conditions conducted as the initial acid concentration of 10% w/w (1.12 mol L^{-1}), the amount of Amberlite IRA-400 of 0.1 g and the temperature of 25°C. Freundlich adsorption isotherm model fitted experimental results quite well. Lactic acid recovery was well characterized by the pseudo-second-order kinetic model.

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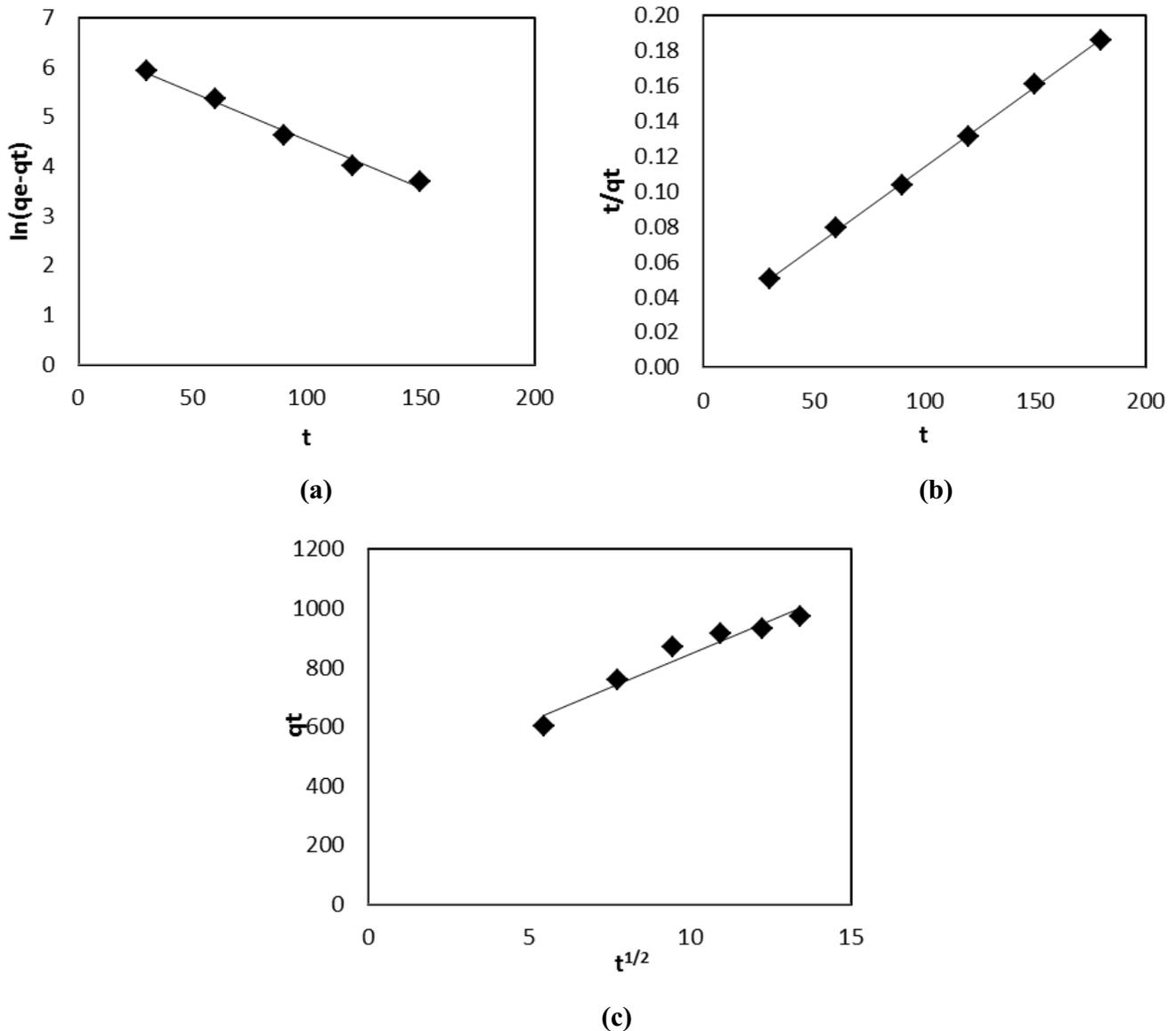


Fig. 4. Linear fits of (a) pseudo-first-order kinetic model, (b) pseudo-second-order kinetic model and (c) Weber-Morris intra-particle diffusion model for the adsorption of lactic acid by Amberlite IRA-400.

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