

## Separation of lactic acid by multiwall carbon nanotube adsorption from water

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### ABSTRACT

This study aims to present adsorption data for the separation of lactic acid from water by multiwall carbon nanotube (MWCNT). The effects of the equilibrium time, the initial lactic acid concentration and the amount of MWCNT on lactic acid adsorption have investigated experimentally. The adsorption capacity of MWCNT at different conditions were determined and compared. It has been found that the considerable amount of lactic acid has removed by MWCNT. Langmuir, Freundlich and Temkin isotherms have been used to define the mechanism of adsorption. To describe adsorption kinetic of lactic acid on MWCNT, Pseudo-first order, Pseudo-second order, Elovich kinetic models and Weber-Morris intra-particle diffusion model have been applied. Langmuir isotherm has been found as the best fit the equilibrium data for lactic acid adsorption with value of R square 0.99. Also as the most suitable models, Elovich kinetic model and Weber-Morris intra-particle diffusion model have been determined with values of R square 0.95.

*Keywords:* Lactic acid; Adsorption; Carbon nanotube

### 1. Introduction

Lactic acid is classified as an alpha-hydroxy acid containing the carboxylic acid and hydroxyl groups that plays an important role in many chemical processes. It has been used as a monomer for the production of biodegradable polylactic acid. However, it has attracted attention for use as a raw material to produce many chemical substances like pyruvic acid, propionic acid, acrylic acid, propylene glycol, acetaldehyde and 2,3-pentanedione. Lactic acid has also very large using in different industrial applications such as pharmaceutical, food, cosmetic and textile industries [1–3].

Lactic acid is produced by biological and chemical synthesis. Generally it is largely obtained in aqueous solutions by fermentation processes [4]. In this process, the broth obtained from fermentation including lactic acid is averagely not more than 10% by mass. Lactic acid has been in wastewaters and effluents in many industrial facilities. Thus, it is very important to separate this substance from water. For the purpose, various methods are often used to separate lactic acid from water such as solvent extraction,

electrodialysis, distillation, ion-exchange, ultrafiltration, reverse osmosis, membrane separation and adsorption [5–10]. Adsorption has some advantages such as high efficiency, simple handling and low-cost effectiveness with respect to other methods [11,12]. Other separation methods are required a big amount of energy or high amount of solvent that can be harmful for environment [13,14].

Carbon nanotubes are well known one of very effective adsorbents because of high adsorption capacity. They have porous-rich structure and very large surface area that provides interaction varieties between adsorbent and adsorbate [15,16]. They can be used for separation of many various substances such as heavy metal [17,18], enzyme [19], gas molecule [20], dyes [21,22], phenol [23], amino acid [24] and carboxylic acids such as tannic acid [25], fulvic acid [26] and humic acid [27].

Several researchers have investigated the adsorption of lactic acid from water by using different adsorbents such as zeolite [28], ion exchange resin [12], polymeric resin [29] and activated carbon [30,31]. Pradhan et al. have investigated adsorption of lactic acid on granular activated carbon and anionic ion exchanger [32]. Yousuf et al. have used an ion exchanger resin, Amberlite IRA-67 for adsorption of

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carboxylic acid such as lactic acid [33]. However, it has not used multiwall carbon nanotube (MWCNT) and thus, there is not enough study in the literature about the adsorption of lactic acid on multiwall carbon nanotube (MWCNT).

The goal of this research is to examine separation of lactic acid from water by multiwall carbon nanotube (MWCNT) adsorption. For this purpose, batch adsorption experiments have been performed in a routine way. The effects of some parameters such as the equilibrium time, the initial concentration of lactic acid and the different MWCNT amount on adsorption have investigated experimentally. The adsorption capacity of MWCNT at different conditions were determined and compared. Langmuir, Freundlich and Temkin isotherms were used to describe the adsorption equilibrium data. Pseudo-first order, pseudo-second order, Elovich kinetic models and Weber–Morris intra-particle diffusion model were applied to determine adsorption kinetic model.

## 2. Materials and methods

MWCNTs were purchased from Sigma-Aldrich. MWCNT's diameter and length used in this study is 10–20 nm and 1–2 mm, respectively. The purity of MWCNT is >98%. Lactic acid (>99%) was obtained from Merck. Solutions of lactic acid have been prepared by dissolving an initial concentration of approximately 2%, 4%, 6%, 8% and 10% (w/w) in distilled water.

In the experiments, firstly, the equilibrium adsorption time was found for the adsorption of lactic acid by using 0.01 g MWCNT, and sample volume has taken as 5 mL. The contact time has determined for the equilibrium as 210 min. Then, with using this equilibrium contact time and same MWCNT dose (0.01 g), the effect of initial lactic acid concentrations on adsorption was determined. Finally, the effect of MWCNT amount on adsorption was investigated. 0.01 g, 0.05 g, 0.10 g, 0.15 g, 0.20 g, 0.25 g amounts have used for determining the effect of MWCNT amount on adsorption. The adsorption experiments were made at 150 rpm and 298.15 K in a thermostatic shaker bath (Nüve ST30), MWCNT was separated from solution by filtration. The lactic acid concentrations of the solutions before and after the adsorption process were detected with by volumetric NaOH titration method.

At equilibrium, the adsorbed amount of lactic acid by MWCNT, the adsorption capacity ( $q_e$ , mg/g) was calculated by using Eq. (1).

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

In the equation,  $C_0$  and  $C_e$  (mg/L) show initial and equilibrium concentrations of the lactic acid solution, respectively.  $M$  (g) is the amount of the MWCNT and  $V$  (L) is volume of the lactic acid solution.

## 3. Results and discussion

### 3.1. Effect of equilibrium adsorption time

In the initial section of experiments of this work, the effect of equilibrium adsorption time was investigated and

then, the optimum adsorption time of lactic acid adsorption on MWCNT was determined. For the purpose, 0.01 g MWCNT and 5 mL of the initial concentration of about 10% (w/w) lactic acid solutions were used in the experiments. The results of these experiments were summarized in Table 1. The effect of equilibrium adsorption time can be seen in Table 1 and Fig. 1. As can be seen from Fig. 1 and Table 1, the adsorption process accelerated in the first 90 min, and then it slowed near the equilibrium. Because, at the first stage of adsorption process, the existed surface sites of MWCNT are vacant and available. As the time progresses, the remained surface sites of MWCNT can be more difficult to occupy because of the driving force increases between the solute molecules on the bulk solution and the surface of adsorbent [34]. The optimum adsorption time for lactic acid adsorption on MWCNT was found as 210 min from Fig. 1.

### 3.2. Effect of initial lactic acid concentration

The initial concentration of the adsorbate solution is an important parameter in the batch adsorption process that affects the adsorbent adsorption capacity value (amount of acid adsorbed per unit mass of adsorbent). In general, the adsorption capacity increases by increasing the initial acid concentration. Because, at higher initial concentrations, more acid molecules occur in the adsorbate solution and so, more acid molecules are adsorbed by adsorbent [35,36]. In order to determine the effect of initial lactic acid concentration on lactic acid adsorption, 0.01 g MWCNT and 5 mL of the different initial lactic acid concentration of about 2%, 4%, 6%, 8% and 10% (w/w) were used. These experiments were performed in the optimum adsorption time (210 min). The experimental results are shown in Fig. 2 and Table 2. It is obvious from Fig. 2 that the adsorption capacity are increased by increasing the initial lactic acid concentration in the adsorbate. The adsorption capacity of MWCNT increased from 1010 to 9420 mg/g by increasing initial lactic acid concentration from 2% to 10% (w/w). These lactic acid adsorption values obtained with MWCNT in this study are better than obtained with other adsorbents in the literature. The adsorption capacity of activated carbon for lactic acid adsorption was found as 213.95 mg/g with the 99.08 g/L initial lactic acid concentration, the adsorption capacity of

Table 1  
The effect of equilibrium adsorption time on the adsorption of lactic acid by MWCNT

Initial acid concentration $C_0$ (g/L)	Amount of MWCNT (g)	Time (min)	Equilibrium acid concentration $C_e$ (g/L)	Adsorption capacity $q_e$ (mg/g)
99.60	0.01	30	94.90	2350
99.60	0.01	60	93.00	3300
99.60	0.01	90	86.24	6680
99.60	0.01	120	85.20	7200
99.60	0.01	150	82.80	8400
99.60	0.01	180	81.40	9100
99.60	0.01	210	80.76	9420

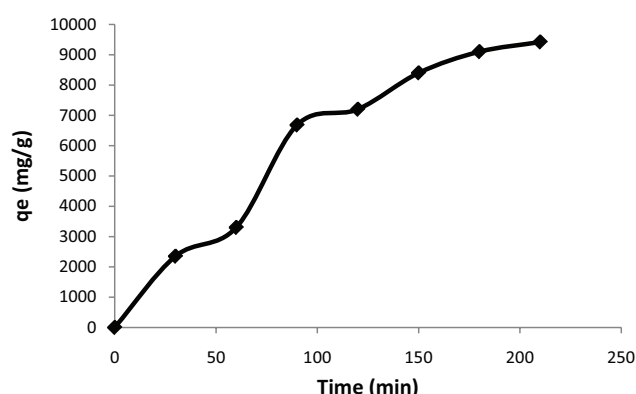


Fig. 1. The effect of equilibrium adsorption time on the adsorption of lactic acid by MWCNT.

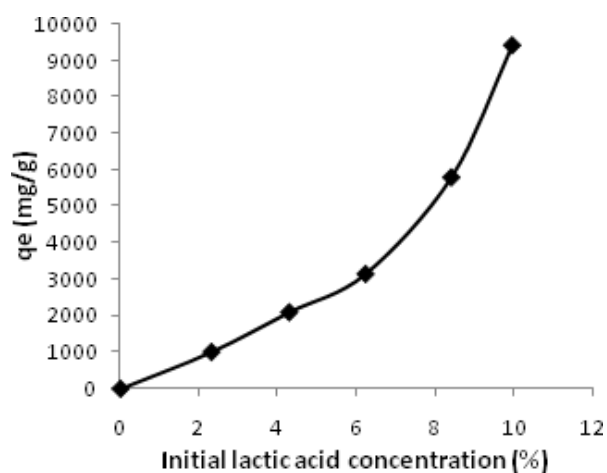


Fig. 2. The effect of initial acid concentration on the adsorption by MWCNT.

Amberlite IRA-67 was determined as 574.23 mg/g in the same conditions (with 99.08 g/L initial lactic acid concentration) [30]. But in this study MWCNT has given very higher adsorption capacity value (9420 mg/g) with 99.60 g/L initial lactic acid concentration.

### 3.3. Effect of MWCNT amount

The effect of adsorbent amount was investigated by using the selected various amount of MWCNT (0.01, 0.05, 0.10, 0.15, 0.20 and 0.25 g) at the initial lactic acid concentration of about 10% (w/w) with the period of optimum adsorption time. The experimental results are shown in Table 3 and Fig. 3. It is seen from Table 3 and Fig. 3 that the adsorption capacity of MWCNT are decreased by increasing the amount of MWCNT. It is also obtained from Fig. 3, at the optimum amount of 0.01 g MWCNT was obtained as the best adsorption capacity value (9420 mg/g). This study has showed that MWCNT is very efficient for the adsorption of lactic acid. The experimental results obtained from this study were further validated by the observations from similar works, where MWCNT

Table 2

The effect of initial lactic acid concentration on the adsorption by MWCNT

Initial acid concentration $C_0$ (g/L)	Amount of MWCNT (g)	Equilibrium acid concentration $C_e$ (g/L)	Adsorption capacity $q_e$ (mg/g)
23.10	0.01	21.08	1010
42.90	0.01	38.70	2100
62.30	0.01	56.00	3150
84.10	0.01	72.50	5800
99.60	0.01	80.76	9420

Table 3

The effect of MWCNT amount on the adsorption of lactic acid by MWCNT.

Initial acid concentration $C_0$ (g/L)	Amount of MWCNT (g)	Equilibrium acid concentration $C_e$ (g/L)	Adsorption capacity $q_e$ (mg/g)
99.60	0.01	80.76	9420
99.60	0.05	93.20	640
99.60	0.10	92.60	350
99.60	0.15	89.00	353
99.60	0.20	87.20	310
99.60	0.25	83.70	318

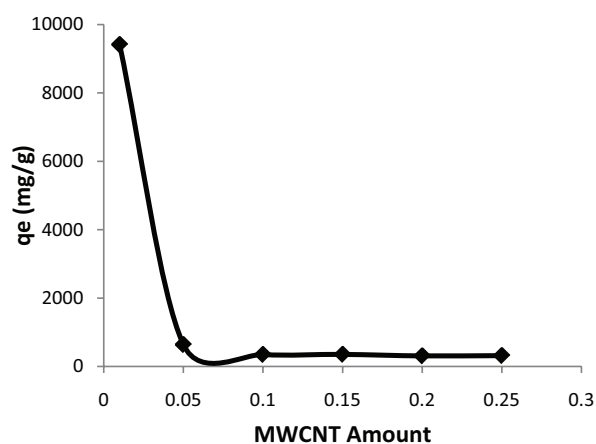


Fig. 3. The effect of MWCNT amount on the adsorption of lactic acid by MWCNT.

as well as carbon derived materials is efficient in adsorption of lactic acid [30,32].

### 3.4. Adsorption isotherms

In this examination, Langmuir, Freundlich and Temkin isotherms summarized in following Eqs. (2)–(7) were used to evaluate adsorption characteristic of lactic acid on MWCNT [37–39].

$$\text{Langmuir isotherm equation: } q_e = \frac{Q_0 * b * C_e}{1 + b * C_e} \quad (2)$$

$$\text{Linear form: } \frac{1}{q_e} = \frac{1}{Q_0} + \left( \frac{1}{b * Q_0} \right) * \left( \frac{1}{C_e} \right) \quad (3)$$

$$\text{Freundlich isotherm equation: } q_e = K_f * C_e^{\frac{1}{n}} \quad (4)$$

$$\text{Linear form: } \log q_e = \log K_f + \frac{1}{n} \log C_e \quad (5)$$

$$\text{Temkin isotherm equation: } q_e = B * \ln(K_t * C_e); B = \frac{R * T}{b_t} \quad (6)$$

$$\text{Linear form: } q_e = B * \ln K_t + B * \ln C_e \quad (7)$$

In these equations,  $C_e$  (mg/L) is the concentration of acid at equilibrium of the solution,  $q_e$  (mg/g) is the adsorption capacity of the adsorbent at the equilibrium.  $b$  (L/mg) is constant of Langmuir isotherm and  $Q_0$  (mg/g) is the maximum adsorption capacity of the adsorbent. The values of  $b$  and  $Q_0$  were determined by using the plot of  $C_e/q_e$  against  $C_e$  in given in Fig. 4a. Freundlich isotherm constants,  $K_f$  (L/g) is the adsorbent adsorption capacity and  $1/n$  is energy heterogeneity factor (dimensionless). The values of  $K_f$  and  $n$  were found from the plot of  $\log q_e$  against  $\log C_e$  in given in Fig. 4b.  $K_t$  (L/mg) and  $b_t$  (J/mol) are constants of Temkin isotherm,  $B$  represents the adsorption heat (dimensionless). The values of these isotherm constants were obtained from the straight line plot of  $q_e$  against  $\ln C_e$  in given in Fig. 4c. These isotherms constants and regression coefficients for the adsorption of lactic acid on MWCNT were calculated from the equilibrium experimental data in given in Fig. 4. The calculated results were presented in Table 4.  $R^2$ , the regression coefficient, was used to compare the best fit isotherm models. As summarized in Table 4, the regression coefficients clearly show that Langmuir isotherm are the best fitted to the experimental data. Among these isotherm models examined, Langmuir isotherm model ( $R^2 = 0.99$ ) has determined as more suitable in explaining lactic acid adsorption mechanism with MWCNT compared to the Freundlich ( $R^2 = 0.95$ ) and Temkin isotherms. Temkin isotherm was investigated in this study for lactic acid adsorption with MWCNT, but this model was not able to explain adsorption of lactic acid with MWCNT ( $R^2 = 0.74$ ). In a similar study, adsorption of lactic acid onto Amberlite IRA-67 and activated carbon followed the Langmuir isotherm under similar experimental conditions [30]. Langmuir isotherm shows that the lactic acid adsorption onto MWCNT is a monolayer adsorption, and lactic acid homogeneously distributed over the surface of the MWCNT. Also it shows that there is a restricted interaction between the lactic acid molecules [33].

### 3.5. Adsorption kinetics

Adsorption kinetic models have been used to evaluate adsorption experimental data. Many kinetic models have been developed and given in the literature. In this adsorption work, pseudo-first order, pseudo-second order, Elovich kinetic models and Weber–Morris intra-particle diffusion

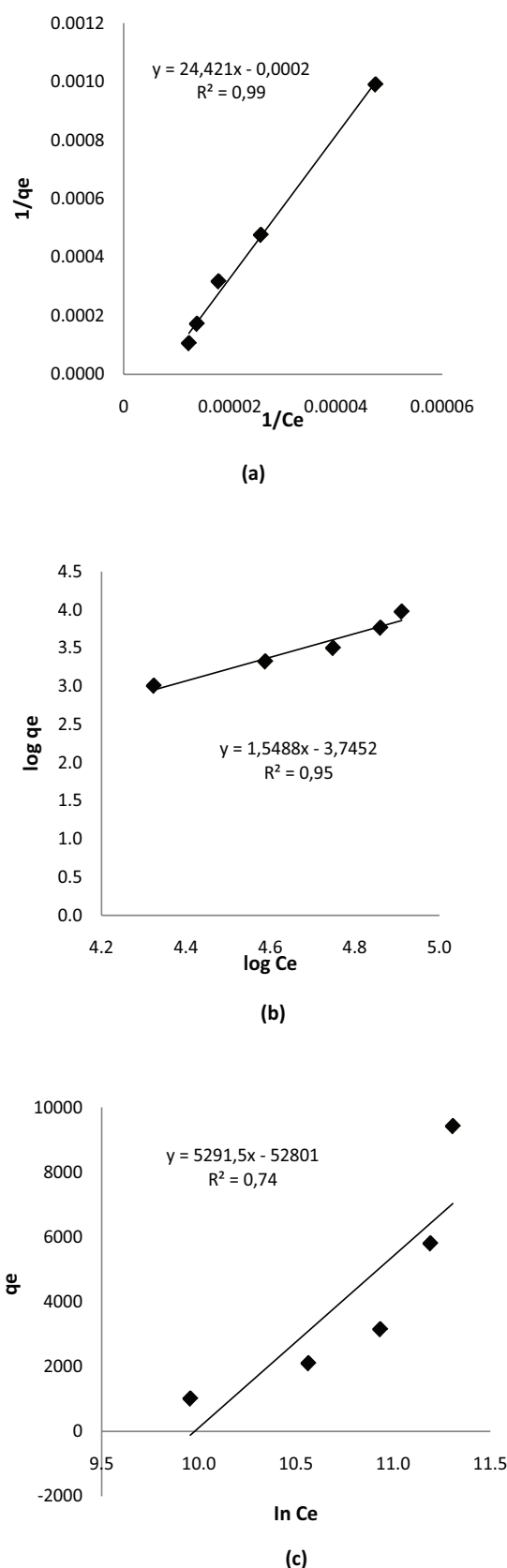


Fig. 4. (a) Langmuir isotherm (b) Freundlich isotherm (c) Temkin isotherm for the adsorption of lactic acid on MWCNT.

Table 4  
Langmuir, Freundlich and Temkin isotherm constants and regression coefficients for the adsorption of lactic acid on MWCNT

Langmuir isotherm				Freundlich isotherm				Temkin isotherm				
$Q_0$	$b$	$R^2$	$q_e$	$K_f$	$n$	$R^2$	$q_e$	$B$	$K_t$	$b_t$	$R^2$	$q_e$
-5000	$-8 \times 10^{-6}$	0.99	9127	$2 \times 10^{-4}$	0.65	0.95	7089	5292	$4.6 \times 10^{-5}$	0.47	0.74	6991

Table 5  
The calculated kinetic and diffusion model parameters for the adsorption of lactic acid on MWCNT

Pseudo-first order kinetic model			Pseudo-second order kinetic model			Elovich kinetic model			Intra-particle diffusion model	
$k_1$	$R^2$	$q_e$	$k_2$	$R^2$	$q_e$	$\alpha$	$\beta$	$R^2$	$k_{id}$	$R^2$
0.02	0.94	17262	$1.3 \times 10^{-7}$	0.69	25000	208	$2.5 \times 10^{-4}$	0.95	850	0.95

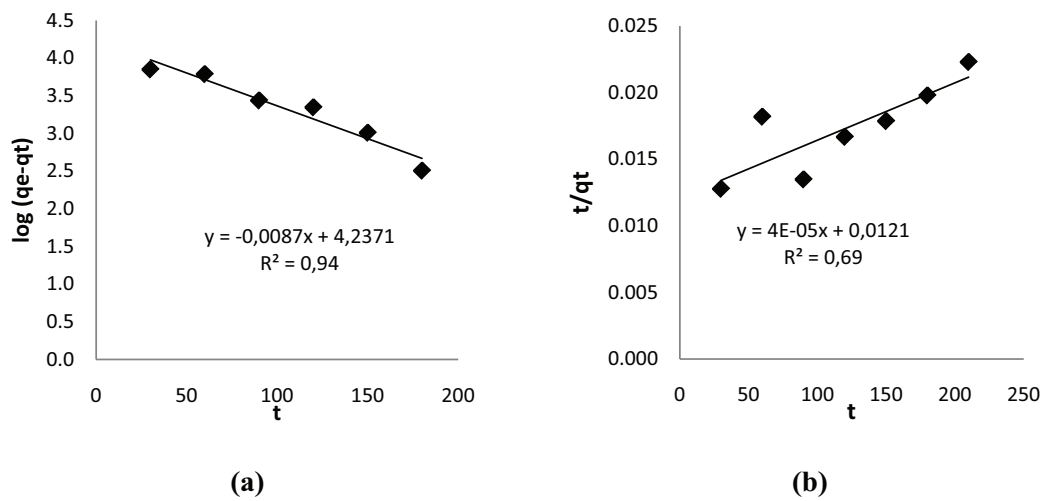


Fig. 5. (a) Pseudo-first order kinetic model (b) Pseudo-second order kinetic model plots for the adsorption of lactic acid on MWCNT.

model as expressed in Eqs. (8)–(14) were applied to define the adsorption kinetics of lactic acid on MWCNT [34,40,41].

Pseudo-first order model equation:  $\frac{dq_t}{dt} = k_1 * (q_e - q_t)$  (8)

Linear form:  $\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} * t$  (9)

Pseudo-second order model equation:  $\frac{dq_t}{dt} = k_2 * (q_e - q_t)^2$  (10)

Linear form:  $\frac{t}{q_t} = \frac{1}{k_2 * q_e^2} + \frac{1}{q_e} * t$  (11)

Elovich model equation:  $\frac{dq}{dt} = \alpha * \exp(-\beta * q_t)$  (12)

Linear form:  $q_t = \frac{1}{\beta} * \ln(\alpha * \beta) + \frac{1}{\beta} * \ln t$  (13)

Weber-Morris intra-particle diffusion model equation:  $q_t = k_{id} * t^{1/2} + C$  (14)

where  $q_e$  (mg/g) and  $q_t$  (mg/g) are the value of adsorption capacities of the adsorbent at the equilibrium and time (t), respectively. However,  $k_1$  (1/min) is the kinetic rate coefficient of pseudo-first order and  $k_2$  [g/(mg·min)] is the kinetic rate coefficient of pseudo-second-order model. In order to find the values of  $k_1$  and  $k_2$  were used the plots of  $\log(q_e - q_t)$  and  $(t/q_t)$  vs.  $t$ , as shown in Fig. 5, respectively. In Eqs. (12)–(13),  $\alpha$  [mg/(g·min)] is the initial adsorption rate,  $\beta$  (g/mg) is constant of desorption. These parameters can be obtained from the linear graph of  $q_t$  against  $\ln(t)$  in given in Fig. 6a. In Eq. (14),  $k_{id}$  [mg/(g·min<sup>1/2</sup>)] is known as the intra-particle diffusion rate coefficient and C is the intercept related to the thickness of boundary layer. The values of  $k_{id}$  and C can be obtained from the plot of  $q_t$  vs.  $t^{1/2}$ . As can be shown in Fig. 6b, the plot of  $q_t$  vs.  $t^{1/2}$  is linear, it is indicated that adsorption mechanism progresses the intra-particle diffusion process. The calculated parameters of adsorption kinetic and diffusion models are listed in Table 5. It is obviously seen from Table 5 that Elovich kinetic equation provided the best representation to the data obtained from experiments. Pseudo-second order kinetic model

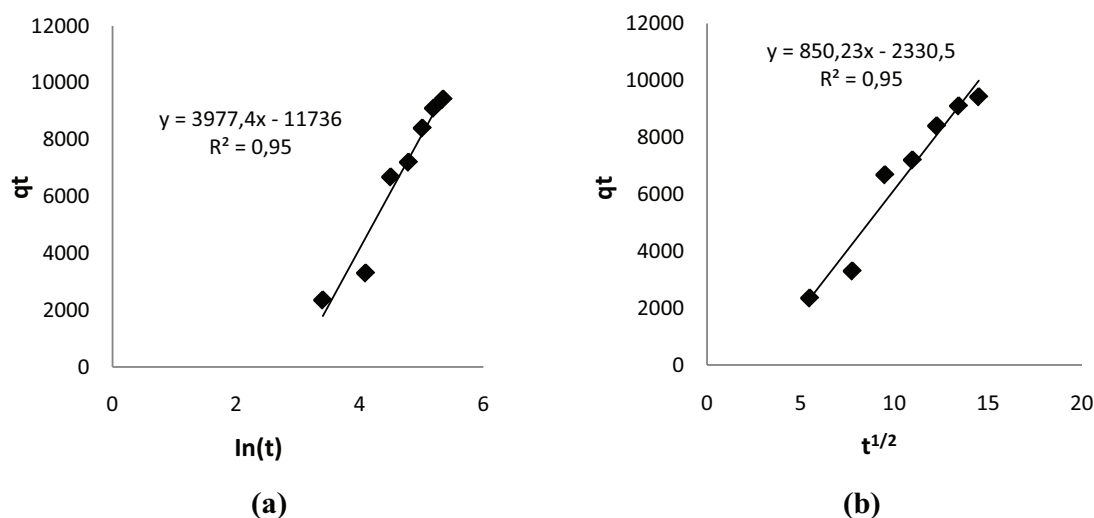


Fig. 6. (a) Elovich kinetic model (b) Intra-particle diffusion model plots for the adsorption of lactic acid on MWCNT.

was investigated in this study for lactic acid adsorption with MWCNT, but this model was not able to explain adsorption of lactic acid with MWCNT ( $R^2 = 0.69$ ).

#### 4. Conclusions

It has been shown that the considerable amount of lactic acid has separated from water by adsorption method with using MWCNT in this study. It has been determined that by increasing initial acid concentration the adsorption capacity of MWCNT is increased. This situation can be explained as higher initial concentrations means that more acid molecules occur in the adsorbate solution and so, more acid molecules are adsorbed by adsorbent. On the other hand with increasing the amount of MWCNT the capacity of adsorption ( $q_e$ ) of MWCNT is decreased. It is the result of mathematical expression of  $q_e$ . Capacity of adsorption ( $q_e$ ) is the amount of adsorbed solute per unit adsorbent that is higher adsorbent amount means lower  $q_e$ . At the optimum MWCNT amount is 0.01 g and 210 min, it was obtained as the best adsorption capacity of 9420 mg/g. Langmuir isotherm model has been found as the best fit the equilibrium data for lactic acid adsorption with value of R square 0.99. Also as the most suitable kinetic models, Elovich kinetic model and Weber–Morris intra-particle diffusion model have been determined with values of R square 0.95.

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