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A novel and inexpensive method for producing activated carbon from waste polyethylene terephthalate bottles and using it to remove methylene blue dye from aqueous solution

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ABSTRACT

The current study aims to present a novel and inexpensive method for producing activated carbon using waste polyethylene terephthalate (PET) bottles in order to remove methylene blue (MB) dye from aqueous solution. Here, a novel method for producing activated carbon is developed by utilizing KOH and combining the carbonation and activation processes. Besides, the inert gases are not used in the production process. In order to determine the characteristics of the produced activated carbon texture, N_2 gas adsorption at 77 K and scanning electron microscope are used. The adsorption isotherms of MB have been studied using Langmuir, Freundlich, Dubinin–Radushkevich, and Temkin models. The specific surface area and the total volume of the generated activated carbon pores are 353.307 m^2/g and 0.288 cc/g, respectively. In addition, q_{max} for adsorption of MB dye is obtained as 404.089 mg/g. Obtained results show that the Langmuir isotherm model has the best fitness. The values of R^2, the sum of squares of the errors percentage, and χ^2 for this model are measured as 0.996, 3.878, and 0.109, respectively. The present study shows that PET bottles are appropriate precursors for producing activated carbon. Besides, the present proposed method is effective and inexpensive.

Keywords: Waste polyethylene terephthalate bottles; Activated carbon; Methylene blue; Isotherm

1. Introduction

Dyes are important industrial pollutants in such a way that their accumulations of even lower than 1 ppm in water are identifiable by naked eyes [1]. Reports show that about 10,000 different commercial dyes and pigments are known [2]. The presence of this pollutant in water bodies is undesirable for aquatic ecosystem, because they block the penetration of the

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sunlight essential for photosynthesis of aquatic flora [2–4]. Methylene blue (MB) (Fig. 1) is one of the most widely used materials in the dye industry, which is most commonly used for dyeing cotton, wood, and silk [5,6]. Most of the dyes discharged into the water bodies by industries are toxic and even carcinogenic to both animals and humans [4]. In addition to the mentioned problems which dyes can generally create to the environment, any connection with MB can cause a lot of health problems as well; hence, it is necessary to remove them from the aquatic environments [5]. Among different physicochemical and biological methods of removing dyes from wastewater such as coagulation, oxidation, filtration, and ozonation; adsorption has been found to be an effective removal method due to its efficiency, simplicity, sludge-free, and applicability [2,4,7–9]. Activated carbon is highly important due to its unique surface properties which make it able to adsorb various materials from different environments [10]; besides, it is used in the adsorption process in order to remove different pollutants from the wastewater, especially colored contaminants [11]. For years, oil products, natural charcoal, and different woods were considered as the major precursors for producing activated carbon and since most of these materials are imported and expensive, the commercial activated carbon is also highly expensive [6,12]. Therefore, cheap and particularly disposed materials are used as the precursor for producing activated carbon in a large number of studies. For instance, Stavropoulos and Zabaniotou, Amri et al., Bansode et al., and Kadirvelu et al. succeeded to produce activated carbon from olive kernel, waste rubber, almond skin, and coconut fiber, respectively [13–16]. In the 1940s, the first industrial unit for producing synthetic polymers started to work. Consequently, production, consumption, and disposal of polymeric products have considerably increased particularly in the recent years [17,18]. Among the polymers, polyethylene terephthalate (PET) (Fig. 2) is considered as the best packaging material around the world due to its unique characteristics such as low weight, transparency, resistance against passage of oxygen and humidity, excellent tensile property, chemical resistance, nontoxicity, and flexibility [19–21]. Nonetheless, every year, large amounts of used PET create a considerable volume of waste solid which cannot be biodegraded [20]. To date, especially in developing countries, landfilling is the main method of removing the waste PET; however, rise of prices, lack of landfills, and contaminations of the landfills' soil have caused the communities to take other alternatives into account [22]. For instance, recycling might be one of the appropriate methods for reducing the landfilling of PET; however, it has not been highly welcomed due to the low cost–benefit ratio of its related technology, hence, the present study aims to produce activated carbon using this material through a novel and inexpensive method (without the utilization of inert gasses) in order to remove MB dye from aquatic environments.

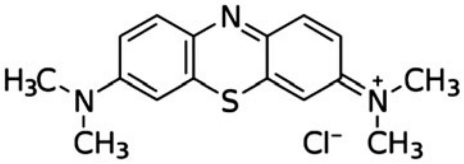

Fig. 1. Chemical structure of methylene blue.

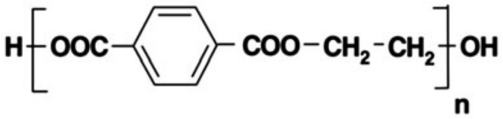

Fig. 2. Polyethylene terephthalate unit.

2. Materials and methods

2.1. Materials and apparatuses

KOH, NaOH, HCl, and MB dye were used from Merck brand (Germany, Darmstadt). pH was measured using Metrohm pH meter (Swiss, model-827) and HACH spectrophotometer (USA, DR-5000) was used in order to determine the concentration of MB dye in the aqueous solution. In addition, the prepared solutions were mixed by a shaker (Rotator R430) and ATASH furnace (Iran, Model 1200) was used for performing the carbonation process.

2.2. Producing activated carbon

Overall, there are two methods for producing activated carbon: chemical and physical. In the chemical method, the precursor is saturated by an appropriate amount of the chemical material, such as H_3PO_4, $ZnCl_2$, KOH, and NaOH, and it is carbonized in the presence of inert gasses, such as N_2 and Ar, at 450–900°C. In the thermal or physical method, first the precursor is carbonized and then, the obtained carbon is activated by water vapor, carbon dioxide, or air at 600–1200°C [11,23,24]. Of course, using the above-mentioned gasses in the activated carbon production process can cause a considerable increase in the final cost of the activated carbon. Therefore, the activated carbon utilized in the current study was produced through a novel method in which activation and carbonation processes were simultaneously performed and no inert gas was used in the production process. First, 10 g PET which is used in drinks packages and washed with double distilled water was divided into smaller parts (approximately 5 × 5 mm) in order to increase the contact area; then, they were mixed with 40 g KOH. KOH to PET weight ratio was also considered as 1:4. After obtaining a complete mixture of two materials, it was put into a completely sealed glass reactor; then, it is put into a furnace. Heating was performed at 1℃ per 5 s. After reaching 500℃, the sample was kept in this temperature for 2 h. After heating, the sample was brought back to the room temperature and the produced activated carbon (PAC) was taken out of the furnace. Next, it was completely grinded by a mortar and washed with deionized water on the filter paper until the pH of the effluent has reached 7.6. Afterward, the obtained samples were put in the oven at 120℃ for 1 h to be completely dried. Finally, samples were passed through a 0.15 mm sieve and kept in the desiccators.

2.3. Characterization of the PAC

In order to determine the characterization of the PAC texture, N_2 gas adsorption/desorption at 77 K was utilized [12,13,25,26]. Moreover, using surface analyzer (Quantachrome Company, model 2000 NOVA), NOVA Win software (v. 2.11), the BET isotherm, the specific surface, the average diameter of the pores, and the volume of the pores on the PAC surface were measured [25,27,28]. It is proper to mention that volumes of the micropores on the PAC were determined through t-plot isotherm [1,12,29]. Besides, the scanning electron microscope performed using a Philips XL-20 electron microscope. Percentage yield of the activated carbon produced by the intended precursor was assessed using Eq. (1).

$$\text{Yield of PAC (\%)} = \frac{\text{Weightoftheproducedactivatedcarbon}}{\text{Weight of the used PET}} \times 100 \tag{1}$$

Moreover, the MB dye adsorption was used as a proper index for determining the capacity of PAC [30,31].

For equilibrium studies of the MB adsorption on PAC, the experiments were conducted in a batch reactor by changing the pH, the contact time, and the initial dye concentration. First, 100 cc of various concentrations of MB dye solution (80–120 mg/L) was contacted by 0.02 g of the produced adsorbent in a flask on a shaker at 130 rpm. Then, in order to determine the equilibrium concentration, in different times (5, 20, 40, 60, 80, 100, and 120 min), 4 cc of the solution was taken and centrifuged using a special tube in 8,000 rpm. Afterward, 2 cc of the centrifuged solution was taken as a sample and the concentration of the remained MB was determined at the wavelength of 620 nm. Based on the wavelength scanning, $\lambda_{\max}$ of MB was determined to be 620 nm. For decreasing the experimental error in the equilibrium study, the sample was returned to the system. Next, to determine the effective pH, the rate of dye adsorption by the adsorbent was assessed in 6 initial pH (3, 5.5, 7, 8, 9, and 10) and the study was continued with the effective pH. Finally, the initial pH of the solutions was adjusted by 0.1 N NaOH and HCl solutions.

When the system reached a steady state, the amount of the adsorbed MB in the unit mass of the adsorbent was calculated through Eq. (2).

$$q_e = \frac{V(C_0 - C_e)}{W} \tag{2}$$

where C_0 (mg/L) is the initial dye concentration in the solution, C_e (mg/L) is considered as the dye concentration when the system reached the equilibrium state, W (g) is the weight of the adsorbent, and V (L) is the volume of the dye solution.

2.4. Adsorption isotherm

Adsorption isotherms can be used to understand the nature of and the interaction between the adsorbent and the adsorbate [32]. In order to investigate the adsorption isotherm of the PAC, Langmuir, Freundlich, Dubinin–Radushkevich, and Temkin isotherm models were used (Table 1).

In the equations presented in Table 1, $q_{\max}$ (mg/g) is the maximum adsorption capacity of the adsorbent, K_L (L/mg) is the Langmuir constant, n and K_F (1 mg g^{-1} (L $mg^{-1})^n$) are the Freundlich constants and the intensity of adsorption, respectively, Q (mg/g) is the amount of MB adsorbed in the unit mass of the adsorbent, Q_m (mg/g) is the capacity of the intended adsorbent, k (mol^{-2} kJ^2) is the constant related to the adsorption energy of the absorbent, E (kJ mol^{-1}) is the mean free energy of the adsorption, R (8.314 J mol^{-1} K^{-1}) is the universal gas constant, T (K) is the absolute temperature, and A $((RT/b_t)\ln a_t)$ and B (RT/b_t) are considered as constants of the Temkin isotherm.

Table 1
Isotherm equations and their linear relationships [33–35]

Model	Nonlinear form	Linear form	Equation number
Langmuir	$q_e = \frac{q_{\max} K_L C_e}{1+K_L C_e}$	$\frac{C_e}{q_e} = \left(\frac{1}{bq_{\max}}\right) + \frac{1}{q_{\max}} C_e$	(3)
Freundlich	$q_e = K_F C_e^{1/n}$	$\log q_e = \log K_F + \left(\frac{1}{n}\right)\log C_e$	(4)
Dubinin–Radushkevich	–	$\ln Q = \ln Q_m - k[RT \ln (1 + (1/C_e))]^2$, $E = -(2k)^{-0.5}$	(5)
Temkin	$q_e = \frac{RT}{b_t} \ln (a_t C_e)$	$q_e = A + B \ln C_e$	(6)

R_L is a dimensionless factor which predicts the appropriateness of the adsorption by the constants obtained from Langmuir model. This model is expressed in Eq. (7).

$$R_L = \frac{1}{1 + bC_0} \tag{7}$$

where b (L/mg) is the Langmuir constant. If $R_L > 1$, the used adsorbent is not appropriate for adsorption of the adsorbate. If $R_L = 0$, the adsorption on the adsorbent will be reversed. If $R_L = 1$, the isotherm is linear type and if $0 < R_L < 1$, the utilized adsorbent is appropriate [31–33,36,37].

Using k constant obtained from Dubinin–Radushkevich model, the mean free energy of adsorption (E) was calculated by Eq. (8); then, this calculated quantity was used for estimating the type of adsorption [35].

$$E = -(2k)^{-0.5} \tag{8}$$

2.5. Error analysis

In order to determine the error of each used models and to compare the obtained data with those obtained in experimental studies, the sum of square error (SSE%) (Eq. (9)) and χ^2 (Eq. (10)) methods were used [32,34].

$$\text{SSE\%} = \sqrt{\frac{\sum (q_{e,\exp} - q_{e,\text{cal}})^2}{N}} \tag{9}$$

$$\chi^2 = \sum \frac{(q_{e,\exp} - q_{e,\text{cal}})^2}{q_{e,\text{cal}}} \tag{10}$$

where $q_{e,\exp}$ is the value of the measured q_e, $q_{e,\text{cal}}$ is considered as the value of the q_e predicted by the intended model, and N is regarded as the number of $q_{e,\exp}$.

3. Results and discussion

3.1. Characterization of the PAC

Using Eq. (1), the percentage yield of the activated carbon produced from PET was obtained as 19%. Moreover, the morphology of PAC and the development of porous on the PAC are shown in Fig. 3. The pore structure of the activated carbon is usually characterized using physical adsorption of various gases among which the nitrogen adsorption at the liquid nitrogen temperature, 77 K, has been used most often [23]. By applying BET isotherm, the specific surface and the total volume of the PAC pores were measured as 353.307 m^2/g and 0.288 mL/g, respectively. Fig. 4 shows the graph of the adsorption and desorption of N_2 at 77 K. Besides, using the t-plot method, the volume of the micropores (V_{micro}) was calculated as 0.0795 mL/g. Then, by subtracting the volume of the micropores from the total volume of the pores (V_{TOTAL}), the volume of the mesopores (V_{meso}) was estimated as 0.2092 mL/g. Also, the mean diameter of the pores on the surface of the produced carbon was obtained as 3.269 nm.

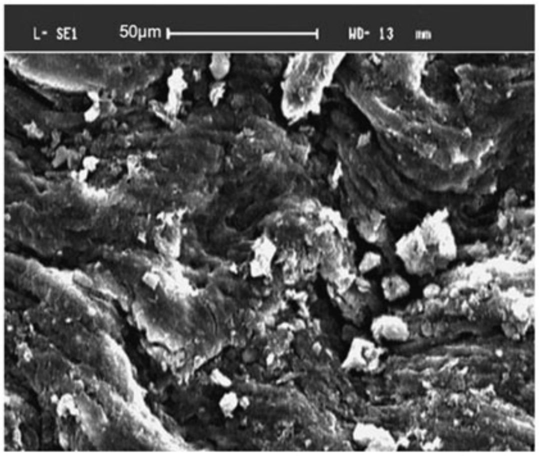

Fig. 3. Scanning electron micrograph of PAC.

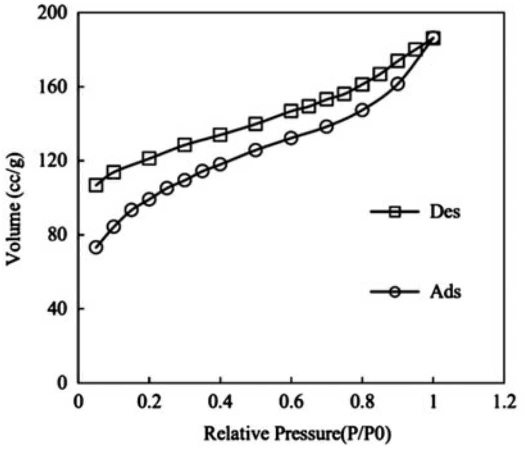

Fig. 4. Adsorption/desorption of N_2 in 77 K.

In general, the activated carbon is produced through carbonation of lingnocellulosic precursors, such as coconut skin, charcoal, and peat [38]. Since these materials have heterogeneous structures, they produce a kind of activated carbon which shows a polymorphic distribution of the pores. It is apt to mention that the micropores are highly effective in the adsorption behavior of the activated carbon. Therefore, using precursors with homogeneous structures such as polymers and copolymers can lead to the production of the activated carbon which has homogeneously structured micropores; however, these materials are highly expensive [39]. Due to the homogeneous structure as well as the high content of the carbon in PET polymer, this material is considered as an appropriate alternative for producing activated carbon [40]. In addition to the selected precursor, the carbonation and the activation processes have irrefutable influences on pores size distribution in the activated carbon [23]. Table 2 shows that in comparison to the activated carbon generated in other studies by usual method, the activated carbon produced in the present study has a relatively large specific surface area which proves the appropriate capacity for the adsorption of various materials, however, it is worth mentioning that in the present study, the inert gases are not used for the carbonation and the activation processes; hence, the remaining molecular oxygen at the reactor atmosphere consumes carbon that causes ignition and burning from the outside surface and it does not penetrate into the interior of the carbon. As a result, no proper enhancement of porosity may be occurred; therefore, as seen in Table 2, in comparison to some studies (e.g. Refs. [40,41]) which used inert gases in their production processes, the PAC used in this study has less S_{BET} and V_{TOTAL}. In general, the activated carbon with high V_{mic} is mostly appropriate for adsorption of gasses, while the activated carbon with high V_{meso} is more appropriate for adsorption of larger molecules [38]. In the current study, 72.63% of the pores on the PAC were related to mesopores and, consequently, this adsorbent is appropriate for the adsorption of large molecules such as MB molecules [23]. Also, as observed in Fig. 3, the proposed method for producing AC made large cavities and rough surface on the PAC; the white spots observed in this figure can be the salt residue.

3.2. *The effect of contact time on MB adsorption*

Fig. 5 shows the results related to the MB adsorption relative to the time. As the Fig. 5 depicts, the sharpest slope of the line is related to the MB removal between 0 and 20 min.

The process of transferring the adsorbate mass on the adsorbent consists of three stages: (1) the diffusion of the adsorbate into the liquid layer around the adsorbent, (2) the diffusion of the adsorbate into the adsorbent surface, and (3) the adsorption of the adsorbate on the internal surface of the adsorbent pores.

Table 2
The comparison of the characteristics of the activated carbon produced in this study with other studies

Precursor	Activation method	Methylene blue adsorption capacity (mg/g)	S_{BET} (m^2/g)	V_{Total} (m^3/g)	References
Sunflower oil cake	Chemical activation	15.798	114.77	0.073	[29]
Bituminous coal	Physical activation	588	857.1	0.45	[33]
Citrus fruit peel	Chemical activation	25.51	526	–	[26]
Bamboo	Physical activation	454.2	1896	1.109	[32]
PET	Physical activation	–	575	0.23	[39]
PET	Physical activation	–	1,170	0.625	[40]
PET	Chemical activation	838.012	2,831	1.44	[41]
Waste PET bottle	Chemical activation	404.089	353.307	0.288	Present study

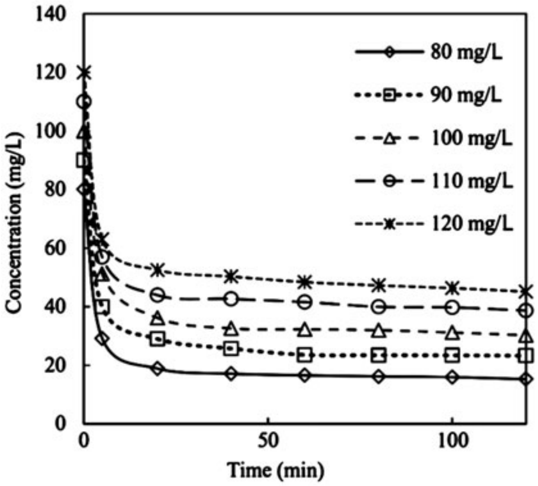

Fig. 5. The rate of MB removal relative to time.

This process is quite time-consuming [32] and, as a result, by increasing the contact time, the adsorption rate of MB dye increases, as well until the system reaches equilibrium and the adsorption reaction gets equaled to the desorption reaction [42]. As Fig. 5 depicts, the system reached the equilibrium at the 80th minute.

3.3. The effect of initial concentration on the removal efficiency

According to Fig. 6, as the initial concentration of MB dye is increased, the percentage of dye removal by the adsorbent is decreased; such a way that by increasing the initial concentration from 80 to 120 mg/L, the removal efficiency decreased from 80 to 60%. As the adsorbate concentration increases in the environment, the number of active adsorption sites on the adsorbent surface is relatively decreased and, consequently, the adsorbate molecules have less chance for being adsorbed on the activated carbon and the adsorption rate is decreased [6].

3.4. The effect of pH on the removal efficiency

As Fig. 7 depicts, as pH increased, the dye removal rate increased as well; hence, the pH 10 was selected as the most effective pH. In the present study, as the pH value increased, the MB removal rate increased, as well (Fig. 7). By increasing the pH, concentration of OH^- ions is increased in the solution and the negative charge of the adsorbent surface is amplified. Therefore, the electrostatic attractive force between MB dye which has the positive charge and the adsorbent surface are increased and, consequently, the rate of dye adsorption is increased, as well [34,43]. This finding is in line with the findings of the studies conducted by Bao and Zhang [43], Bestani et al. [30], Karagoz et al. [29], and Chen et al. [44].

3.5. Adsorption isotherm

In order to extract the coefficients of Langmuir, Freundlich, Dubinin–Radushkevich, and Temkin models, graphs of C_e/q_e versus C_e, log q_e versus log C_e, ln q_e versus ln $[1+(1/C_e)]$, and q_e versus ln C_e were drawn, respectively. Besides, using the linear forms of these models presented in Table 1, the coefficients of each of the models were extracted and the results are

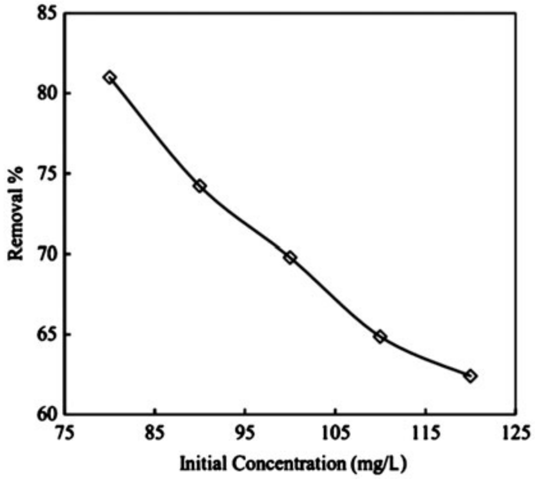

Fig. 6. Changes in the percentage of MB removal relative to the initial concentration.

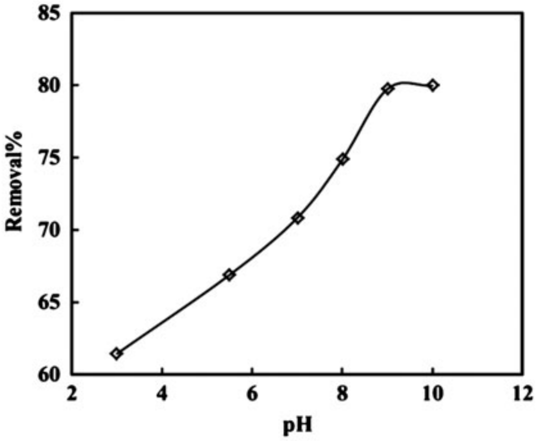

Fig. 7. pH value versus MB removal percentage.

shown in Table 3. It should be mentioned that all the experiments were performed at 25 ± 6°C. The results obtained from R_L model are presented in Fig. 8.

In designing the adsorption systems, Langmuir equation is the most commonly used isotherm model utilized for describing the behavior of an adsorbent [31]. In this model, which was first proposed in 1916, it is hypothesized that the adsorbate is adsorbed on the adsorbent through a monolayer mechanism while no interaction occurs between the adsorbate molecules, and once an adsorbate molecule is adsorbed on the adsorbent, another molecule cannot be adsorbed on the same site [6,12,37,45]. According to Table 3, the high value of R^2 obtained for the Langmuir model shows that the MB dye adsorption on the activated carbon produced from PET probably follows the monolayer mechanism and the surface of the produced adsorbent is homogeneous [33]. Moreover, considering the value of $q_{\max}$ obtained from Langmuir model and its comparison with other studies (Table 2), it is possible to conclude that the capacity of the activated carbon produced in this study is appropriate for the MB dye adsorption. Also, according to Fig. 8, values of R_L for different concentrations were calculated between 0 and 1, which shows that the adsorbent produced from PET is appropriate for the MB dye adsorption.

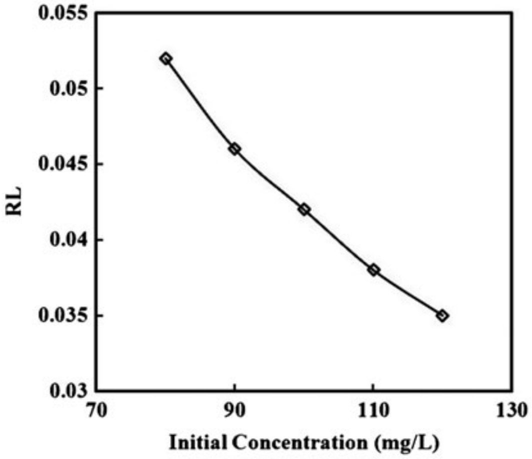

Fig. 8. Rate of RL versus the initial MB concentration.

Freundlich isotherm model is an experimental model which can be utilized for the adsorption on heterogeneous surfaces with interaction between the adsorbate molecules [6,12,29,36,45]. According to Table 3, the value of R^2 for Freundlich model was equal to 0.944, which shows that the dye adsorption on the produced adsorbent does not follow this equation and, consequently, the surface of the PAC is not heterogeneous. In this study, $1/n$ was obtained as

Table 3
Isotherm coefficients obtained from isotherm models

Model	Parameter	Value
Langmuir	$q_{\max}$ (mg/g)	404.089
	b (L/mg)	0.226
	R^2	0.996
	SSE%	3.878
	χ^2	0.109
Freundlich	$1/n$ (mg/g) (L/mg)$^{1/n}$	0.127
	N	7.845
	K_F (1 mg g^{-1} (L mg^{-1})^n)	226.583
	R^2	0.944
	SSE%	4.207
	χ^2	0.250
Dubinin–Radushkevich	$q_{\max}$ (mg/g)	366.657
	K (mol^2 kJ^{-2})	5.561×10^{-6}
	E (kJ mol^{-1})	0.299
	R^2	0.774
	SSE%	8.670
	χ^2	1.080
Temkin	A ((RT/b_t)ln a_t)	199.706
	B (RT/b_t)	44.180
	R^2	0.935
	SSE%	4.489
	χ^2	0.286

0.127, which shows the desirable adsorption of the MB dye on the produced adsorbent [32].

Langmuir and Freundlich isotherms do not provide any information about the adsorption mechanism. Therefore, other isotherm models, such as Dubinin–Radushkevich, are used for estimating the mechanism of adsorption. Radushkevich in 1949 and Dubinin in 1965 asserted that the adsorption curve depends on the structure of the adsorbent pores [46]. In this model according to Table 1, E is the mean free energy of adsorption, defined as the free energy change when one mole of the ion is transferred to the solid surface of the adsorbent from infinity in solution, which can be used in estimating of the adsorption type [35,46]. If E is between 8 and 16 kJ/mol, adsorption is assumed as the ion exchange type, on the other hand, if E is below 8 kJ mol^{-1}, the adsorption mechanism is assumed to be physical [35]. In the present study, E (at 25 ± 6˚C) was obtained as 0.299, which shows the physical mechanism of the MB dye absorption on the produced adsorbent.

Temkin isotherm is a simple isotherm model whose mechanism has been assumed by considering the chemical adsorption [36]. According to Table 3, the small value of R^2 obtained from the Temkin isotherm model shows that the MB dye adsorption on the produced adsorbent does not occur through a chemical mechanism, which had been proved by Dubinin–Radushkevich model, as well.

3.6. Error analysis

Fig. 9 shows the fitness of the utilized isotherm models in comparison to the data obtained in experimental studies. Moreover, in order to evaluate the accuracy of the utilized isotherm models, SSE% and χ^2 indexes were used and the results are presented in Table 3.

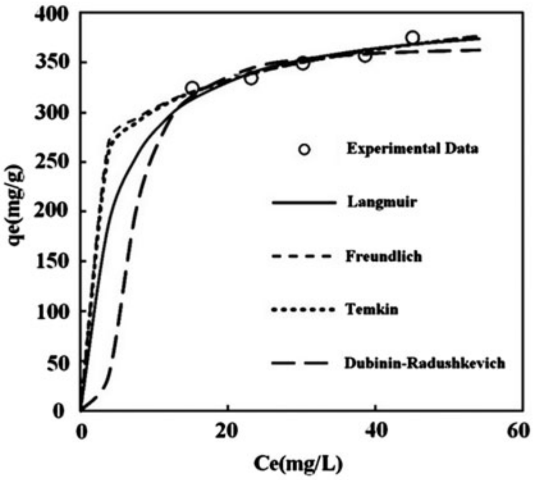

Fig. 9. Fitness of Langmuir, Freundlich, Dubinin–Radushkevich, and Temkin isotherm models compared to the values obtained from experimental studies.

Finally, considering values of SEE% and χ^2 for the utilized isotherm models (Table 3), and Fig. 9, it can be concluded that the Langmuir isotherm model had the best fitness and accuracy.

4. Conclusion

In the present study, a new method was presented for producing activated carbon from PET by applying KOH and combining the carbonation and activation processes without utilizing inert gasses. Results showed that the specific surface area was 353.307 m^2/g and the total volume of pores was calculated as 0.288 cc/g. Besides, MB dye adsorption was used as a proper index for determining the capacity of the PAC. Among different isotherm models which utilized for the adsorption of the MB, according to χ^2 (0.109), SSE% (3.878), and R^2 (0.996), the Langmuir isotherm had the best goodness of fit. Moreover, the value of $q_{\max}$ was obtained as 404.089 mg/g in Langmuir model which showed the high capacity of the produced adsorbent for removing the MB dye from aqueous solutions. Therefore, producing the activated carbon from the waste PET bottles through the method proposed in this study can be economically and environmentally taken into account.

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