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Bisphenol A Removal From Aqueous Phase via Polymeric Microbeads

Polimerik Mikroküreler ile Bisfenol A Uzaklaştırılması

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ABSTRACT

The main objective of this study is to investigate the effectiveness of poly(divinylbenzene-N-methacryloyl-L-tryptophan methyl ester) [poly(DVB-MATrp)] microbeads (average diameter = 150-200 μm) to remove bisphenol A (BPA) from aqueous phase. The poly(DVB-MATrp) microbeads were synthesized by copolymerizing N-methacryloyl-L-tryptophan methyl ester (MATrp) in the presence of divinylbenzene (DVB). The poly(DVB-MATrp) beads were characterized by Fourier transform infrared spectroscopy (FTIR), elemental analysis, scanning electron microscopy (SEM) and swelling test. The efficiency of poly(DVB-MATrp) microbeads for adsorption of BPA from aqueous medium was evaluated by investigating the effects of pH, initial concentration, contact time and temperature. The adsorption capacity of the microbeads was determined to be 171.1 mg/g at pH 7.0, 25°C. The Langmuir and Freundlich isotherm models were used to fit adsorption data. The adsorption process obeyed pseudo-second-order kinetic model. The prepared microbeads can be repeatedly used for adsorption of BPA without a significant change in the adsorption capacity.

Key Words

Bisphenol A; removal; polymeric microbeads; endocrine disruptor.

ÖZ

Bu çalışmanın amacı, poli(divinilbenzen-N-metakriloil-L-triptofan metil ester) [poli(DVB-MATrp)] mikrokürelerin (ortalama çap: 150-200 μm) sulu fazdan bisfenol A (BPA) uzaklaştırılmasındaki etkinliğinin araştırılmasıdır. Poli(DVB-MATrp) mikroküreler N-metakriloil-L-triptofan metil ester (MATrp) monomerinin divinilbenzen (DVB) varlığında polimerleştirilmesi ile sentezlendi. Poli(DVB-MATrp) mikroküreler Fourier dönüşüm infrared spektroskopisi (FTIR), elementel analiz, taramalı elektron mikroskobu (SEM), ve şisme testi ile karakterize edildi. Poli(DVB-MATrp) mikrokürelerin sulu fazdan BPA adsorplamadaki etkinliği pH, başlangıç derişimi, temas süresi ve sıcaklığın etkisinin araştırılması ile incelendi. Mikrokürelerin BPA adsorpsiyon kapasitesi pH 7.0 ve 25°C sıcaklıkta 171.1 mg/g olarak belirlendi. Elde edilen adsorpsiyon verilerinin Langmuir ve Freundlich izoterm modellerine uygunluğu araştırıldı. Adsorpsiyonun yalanı-ikinci-derece kinetik modele uygun olduğu belirlendi. Sentezlenen mikroküreler, BPA adsorpsiyon kapasitesinde önemli bir değişiklik olmaksızın tekrar tekrar kullanıldı.

Anahtar Kelimeler

Bisfenol A; uzaklaştırma; polimerik mikroküreler; endokrin bozucu.

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INTRODUCTION

Bisphenol A (BPA) is a monomer used in the manufacturing of epoxy resins, polysulphone, polycarbonated plastic, and polyacrylate resins. BPA is often contained in environmental water and recently, attracts considerable attention as an endocrine disrupter [1,2]. The non-polymerized monomer residues and the hydrolysis of the ester bonds of BPA based polymer result in its widespread contamination [3]. In aquatic environment, exposure of living organisms to endocrine distrupters has become of great public concern during recent years. It has recently been reported that trace amounts of BPA concentration at a level of 0.23 ng L^{-1} will exhibit the oestrogenic affect [4].

Up to the present, various techniques have been used to remove BPA from aqueous solutions such as adsorption [5-7], biodegradation [8-10], electrochemical techniques [11,12] and advanced oxidation techniques [13-15]. Adsorption is very simple and effective due to its ease of operation and relatively low cost. In addition, adsorption technique generates fewer harmful secondary products. A permanent organic pollutant management can be possible by developing new adsorbents with high adsorption capacities for target contaminants. An extensive effort has been made to prepare effective adsorbents to decrease the concentration of BPA in the aqueous environment. Zeolites [5,6], activated carbon [16-20], carbon nanotubes/nanomaterials [21-23], various cross-linked polymers [24,25], and molecularly imprinted polymers [26-28] have been widely used in the treatment of aqueous solutions.

In this study, we synthesized an adsorbent to remove BPA from aqueous solution. To prepare the adsorbent, N-methacryloyl-L-tryptophan methyl ester (MATrp) monomer was polymerized in the presence of divinylbenzene (DVB). Suspension polymerization enabled to obtain poly(divinylbenzene-N-methacryloyl-L-tryptophan methyl ester) poly(DVB-MATrp) microbeads. The prepared microbeads were characterized by using SEM, FTIR, elemental analysis and swelling test. Batch adsorption experiments were conducted to determine the adsorption mechanism. The efficiency of the poly(DVB-MATrp) microbeads was evaluated by investigating the effect of various experimental parameters such as initial concentration, operation pH, temperature and contact time. Adsorption mechanism was clarified by fitting the adsorption data to isotherm and kinetic models. Thermodynamic parameters (ΔHo, ΔSo and ΔGo) were also calculated.

MATERIALS and METHODS

Chemicals

Bisphenol-A (BPA) (≥99%) was purchased from Sigma-Aldrich. The divinylbenzene (DVB) was obtained from Merck (Darmstadt, Germany). Methacryloyl chloride and L-tryptophan methyl ester were purchased from Sigma Chemical Co. (St. Louis, USA). All other chemicals were of reagent grade. All water used in the binding experiments was purified using a Elga Flex3 ultrapure water instrument (Veloia Water Solutions & Technologies, France).

Synthesis of poly(DVB-MATrp) Microbeads

N-methacryloyl-L-tryptophan methyl ester (MATrp) was used as a monomer and synthesized as previously described [29]. The poly(DVB-MATrp) microbeads were prepared by suspension polymerization technique as described below. 200 mg of poly (vinyl alcohol) was dissolved in 50 ml of the purified water to prepare aqueous medium. 3.77 mL of DVB and 0.88 mL of MATrp solution (in ethanol) were added to toluene (3.5 mL) and stirred for 15 min at room temperature. Then, 100 mg of 2,2’-azobisisobutyronitrile (AIBN) was dissolved in the prepared homogeneous organic phase. The organic phase was added and dispersed in the aqueous medium by stirring (400 rpm) in a sealed cylindrical pyrex polymerization reactor. The mixture was heated to polymerization temperature (i.e., 80°C) and the polymerization was conducted for 8 h with a 400 rpm stirring rate. The obtained poly(DVB-MATrp) microbeads were washed with ethanol and water to remove unreacted monomers and then dried at 50°C in a vacuum oven.

Characterization studies

FTIR spectrum of the poly(DVB-MATrp) microbeads were recorded in the range of 400-4000 cm^{-1} (Perkin Elmer, Spectrum 100, USA). A scanning electron microscope (SEM) (Jeol, JEM 1200EX, Tokyo, Japan) was used to investigate the surface morphology and internal structure of the microbeads. Elemental analysis was performed to determine the amount of C, H and N in poly(DVB-MATrp) microbeads (Leco, CHNS-932, USA). The swelling ratio of the microbeads was determined as follows. Firstly, dry microbeads were weighed and put into a 50 mL of distilled water. The vial including the poly(DVB-MATrp) microbeads was incubated in the water bath with a fixed temperature (25 ± 0.5°C) for 8 h. The microbeads taken from the water were dried with filter paper and weighed. The dry weight and wet weight of the poly(DVB-MATrp) microbeads were used to calculate the swelling ratio.

Adsorption studies

BPA adsorption onto the poly(DVB-MATrp) microbeads was examined by varying solution pH, initial concentration, temperature and contact time. 0.02 g of the poly(DVB-MATrp) microbeads was used in the adsorption experiments. The pH of BPA solutions (20 mg/L initial concentration) was varied in the range of 2.0-12.0 to determine the effect of pH. 0.1 M HCl and 0.1 M NaOH solutions were used for pH adjustment. The effect of initial BPA concentration to adsorption capacity of the poly(DVB-MATrp) microbeads was investigated by varying the concentration of BPA solutions in the range of 1-300 mg/L at pH 7.0. The experiments were conducted at three different temperatures (25°C, 35°C, 45°C). The concentrations of BPA in the medium were determined spectrophotometrically at 276 nm wavelength by UV spectrophotometer (Shimadzu UV-1700). The amount of adsorbed BPA was calculated by using Eq 1

$$Q = \frac{[(C_0 - C)V]}{m}$$

where Q is the adsorption capacity (mg/g), V is the solution volume (L), m is the adsorbent dosage (g), C0 and C are the initial and equilibrium concentrations of BPA (mg/L), respectively.

0.02 g of adsorbent was mixed with a BPA solution (50 mg/L, pH 7.0) in an erlenmeyer to carry out kinetic experiments. BPA adsorption from the solution was analysed for 180 min. Then, the adsorbed amounts of BPA were calculated by using Eq1. The experiments were conducted at three different temperatures (25°C, 35°C, 45°C).

Kinetic and Isothermal Analyses

The following models were applied to determine the adsorption kinetics at 298 K, 308 K and 318 K: pseudo-first-order model, pseudo-second-order model and intraparticle diffusion model. The pseudo-first order model is performed for the adsorption of solid/liquid system [30]. The linear form of the applied model can be given as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303}$$

where qe and qt are the amounts of BPA adsorbed at equilibrium (mg/g) and at time t, respectively, and k_1 is the rate constant of pseudo-first order sorption (min^{-1}).

According to the pseudo-second order model, the rate-determining step can be chemical reactions. It is expressed as [31]

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

where k_2 is the rate constant (g/mg min).

Intra-particle diffusion model figures out the rate determining step and the equation is [32]

$$q_t = k_i t^{1/2}$$

where i is the number of each adsorption stage; ki (mg/g min) is the intra-particle rate constant. The plot of qt versus t1/2 passed through the origin shows that the mass transfer rate is controlled by the intra-particle diffusion; if not, the adsorption rate is controlled by other processes like chemical reaction or film diffusion.

Isotherm models are used to analyse the adsorption data. In the present study, the Langmuir and the Freundlich models were fitted to experimental data to describe the adsorption process. The linear form of the applied model can be given as:

Langmuir $$\frac{C_e}{q_e} = \frac{1}{Q_L K_L} + \frac{C_e}{Q_L}$$

Freundlich $$h\ q_e = h\ K_f + \frac{1}{n} h\ C_e$$

where Ce is the concentration of BPA solution (mg L^{-1}) at equilibrium. The constant Q_L gives the theoretical monolayer/maximum adsorption capacity (mg g^{-1}) and K_L is Langmuir constant related to the affinity of binding sites (L/mg). K_f and 1/n are the constants. K_f is relevant to adsorption capacity while 1/n is relevant to the surface heterogeneity or the intensity of adsorption.

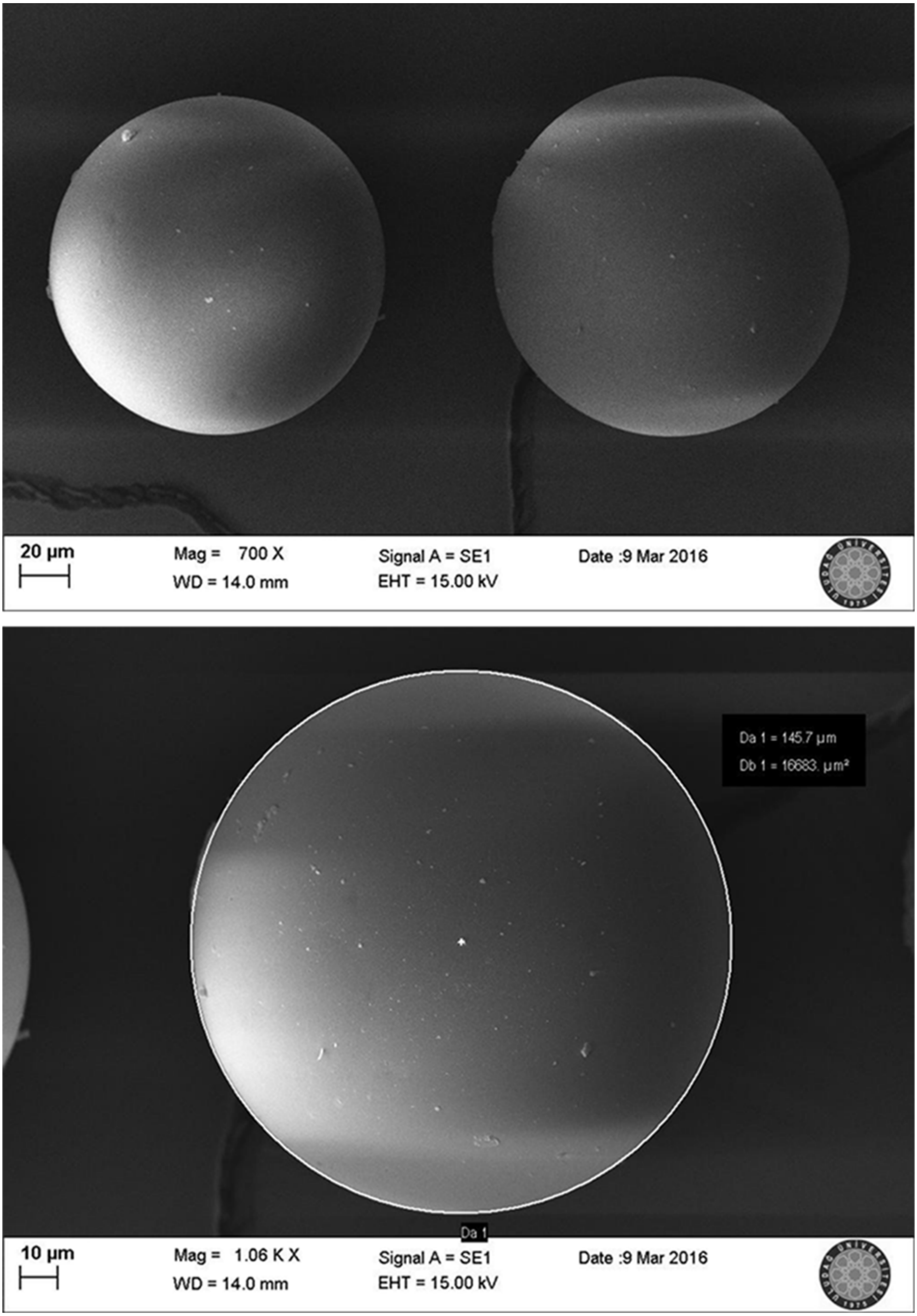


Figure 1. SEM photographs of the poly(DVB-MATrp) microbeads with different magnifications.

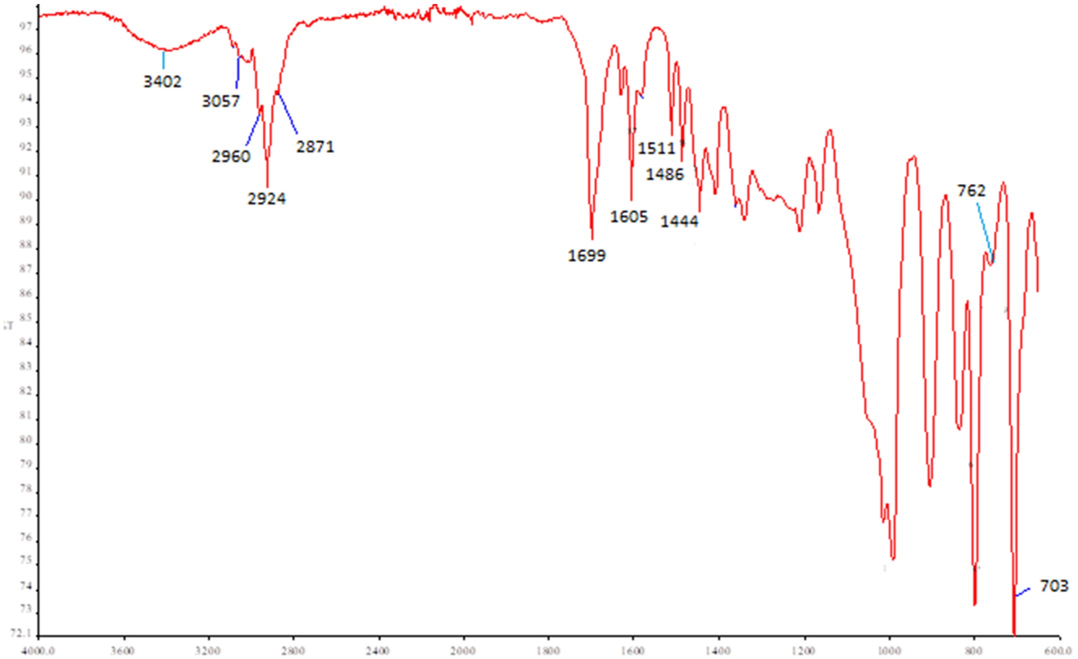


Figure 2. FTIR spectrum of the poly(DVB-MATrp) microbeads.

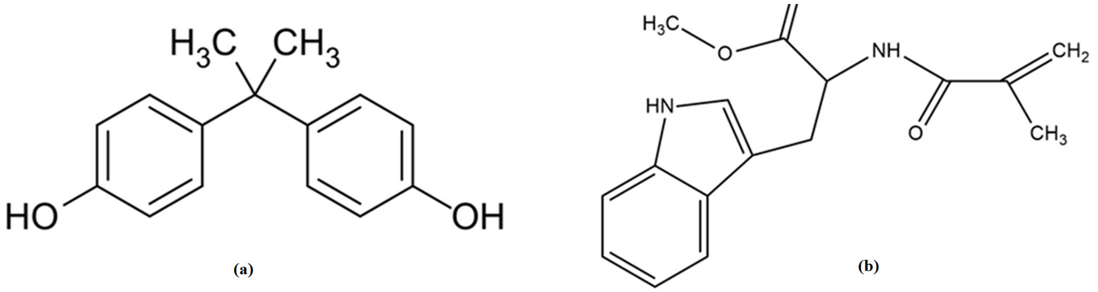
RESULTS and DISCUSSION

Properties of Poly(DVB-MATrp) Microbeads

Physical morphology of the cross-linked the poly(DVB-MATrp) microbeads was elucidated by scanning electron microscopy. The SEM images of the microbeads are shown in Figure 1. It can be clearly seen that the suspension polymerization procedure was successfully provided the microbeads in spherical form. The size of the poly(DVB-MATrp) microbeads ranges 150-200 µm in diameter. The equilibrium swelling ratio for the poly(DVB-MATrp) microbeads is 17%.

The FTIR spectrum of the poly(DVB-MATrp) microbeads is depicted in Figure 2. The characteristic adsorption bands regarding the stretching of C=C were observed at 1444 cm^{-1}, 1486 cm^{-1}, 1511 cm^{-1}, 1605 cm^{-1} and 1699 cm^{-1}. The characteristic absorption peaks at 700-750 cm^{-1} were benzene ring deformation vibration. Aliphatic C-H stretching vibrations appeared at 2870-3000 cm^{-1}. Aromatic C-H stretching vibration was observed at 3057 cm^{-1}. The FTIR spectrum of poly(DVB-MATrp) microbeads has N-H stretching vibration at 3400 cm^{-1} resulting from MATrp monomer.

Elemental analysis of the poly(DVB-MATrp) microbeads showed that the microbeads contain 81.5% C, 7.69 % H and 0.61% N. The presence of nitrogen (N) shows that MATrp was successfully polymerized with DVB. The existence of MATrp monomer is very significant for BPA adsorption due to the hydrophobic natures of MATrp and BPA. The chemical structures of MATrp and BPA were given in Figure 3.



(a) (b)

Figure 3. The chemical structures of (a) BPA and (b) MATrp monomer.

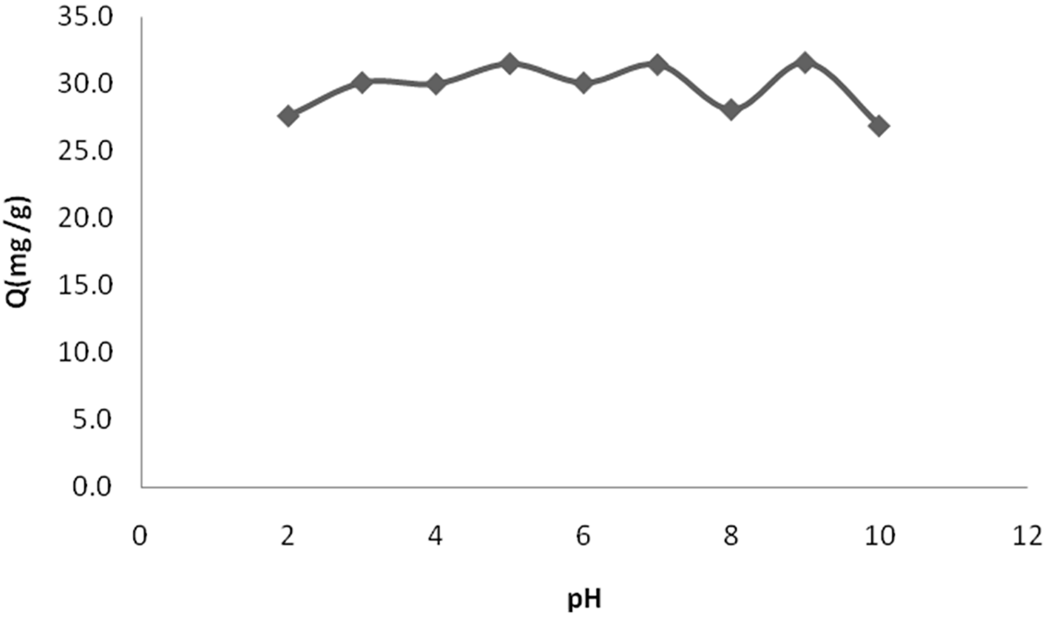


Figure 4. Effect of pH on BPA adsorption onto the poly (DVB-MATrp) microbeads (initial BPA concentration: 20 mg/L).

Effect of pH

The pH of the solution is an effective parameter on the adsorption processes. If the adsorbent and/or adsorbate have ionisable groups, the pH of the adsorption medium considerably affects the adsorption process. Batch-type experiments at various pHs (2.0-10.0) were carried out to determine the effect of pH on the adsorption of BPA and obtained data are illustrated in Figure 4.

According to the results, no significant change occurs in the adsorbed amount of BPA at the pH values ranging from 2.0 to 10.0. However, the amount of adsorbed BPA was slightly lower at pH 2.0 and 10.0. BPA has two phenolic hydroxyls and has a tendency to lose its protons in highly acidic solution. Therefore, BPA molecule has a negative charge at pH values higher than 10 [33]. A slight decrease in the adsorbed amount of BPA in acidic and basic mediums shows that adsorption mainly occurs with dispersive interaction instead of electrostatic attraction or repulsion.

Kinetic, Isotherm and Thermodynamic Analyses

Kinetic Studies

The kinetic study is fundamental to describe the adsorption behaviours such as the rate of adsorbate uptake. In addition, it controls the equilibrium time of adsorption and mass transfer. The equilibrium concentration versus time (t) curves at 298 K, 308 K and 318 K are shown in Figure 5. The adsorption kinetic curves for BPA adsorption onto the poly(DVB-MATrp) microbeads showed that the adsorption process reached equilibrium after 90 min. The decrease in the adsorbed amount of BPA with the increase in temperature from 25°C to 45°C demonstrates that the adsorption process is exothermic.

The pseudo-first-order, the pseudo second order kinetic models, and intraparticle diffusion model were applied to the experimental data and the results are represented in Table 1. Although the correlation coefficients (R^2) were high, the pseudo-first order model qe values remarkably differed from experimental values in all temperatures. Therefore, the pseudo-first order model was not appropriate to describe BPA adsorption onto the poly(DVB-MATrp) microbeads. When the experimental data were treated with the pseudo-second order equation, it can be seen that the theoretical values calculated from the model qe, values are nearly the same with the experimental data (25°C qe,cal = 76.6 mg/g and qe,exp = 74.7 mg/g; 35°C qe,cal = 67.5 mg/g and qe,exp = 67.0 mg/g; 45°C qe,cal = 62.9 mg/g and qe,exp = 61.6 mg/g). In addition, the correlation coefficients are high (0.9490-0.9942). These results demonstrate that the second-order kinetic model sufficiently describes the adsorption behaviour for the system.

An adsorption process can be controlled by more than

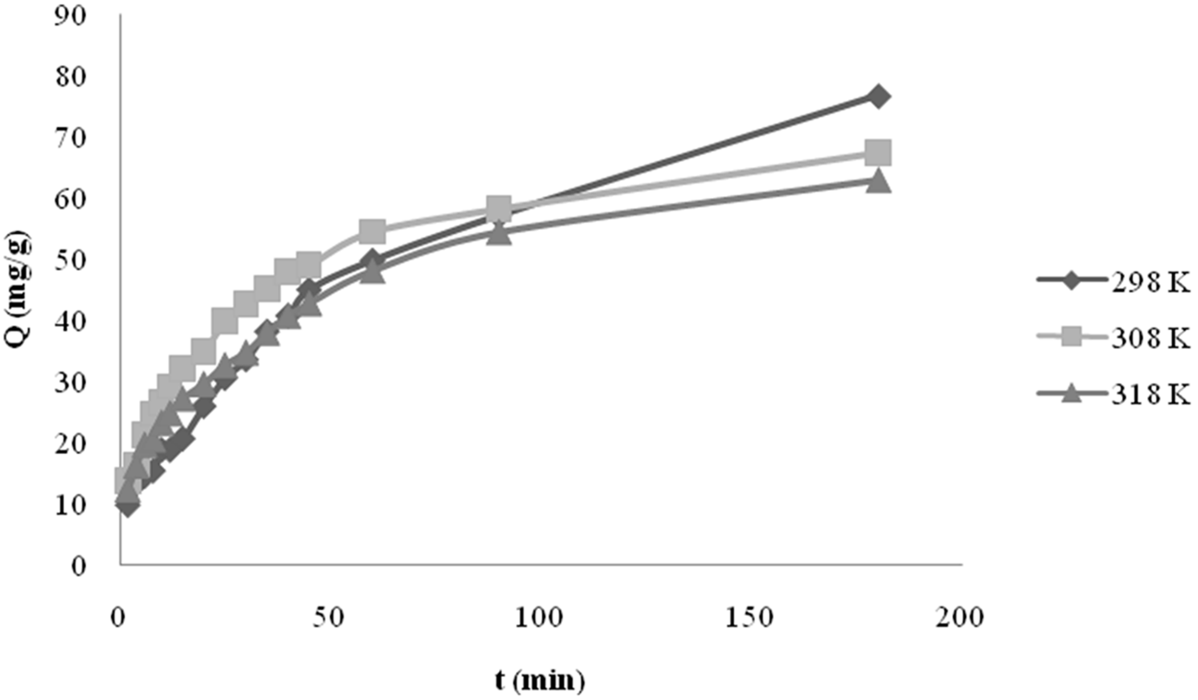


Figure 5. Effect of contact time on BPA adsorption onto the poly(DVB-MATrp) microbeads (initial BPA concentration:50 mg/L, pH 7.0).

one step, e.g. adsorption on the pore surface, surface diffusion, pore diffusion and film diffusion or external diffusion. To describe the sorption kinetics of BPA, the intraparticle diffusion model was also fitted to the experimental data. According to the intraparticle diffusion model, the plot of qt versus t1/2 with a straight line shows that the sorption process is solely controlled by intra-particle diffusion. On the contrary, the multilinear plots demonstrate the influence of two or more steps to the adsorption process. Moreover, if these lines pass through the origin then intra-particle diffusion is the rate-controlling step [34]. In this study, the intraparticle diffusion model fitted to the experimental data with a straight line but the plots do not pass through the origin. The result shows a boundary layer control in BPA adsorption. Thus, other kinetic models operating simultaneously may control the rate of adsorption.

Isothermal Studies

The relationship between the equilibrium BPA concentrations and the adsorbed amount of BPA was evaluated at three different temperatures. The results are depic-

Table 1. Kinetic parameters for the adsorption of BPA onto the poly(DVB-MATrp) microbeads.

Parameters Temperature (K)	Experimental qe(mg/g)	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model			Intraparticle diffusion model	
		$k_1 \times 10^{-2}$ (1/min)	q_{eq}(mg/g)	R²	k_2 x 10-4 ((g/mg)/min)	q_{eq}(mg/g)	R²	k_i ((mg/g)/min$^{0.5}$)	R²
298	76.7	1.47	67.32	0.9864	4.22	74.68	0.9490	5.95	0.9847
308	67.5	1.98	49.71	0.9722	9.07	67.00	0.9942	4.81	0.9227
318	62.9	2.09	49.37	0.9974	9.74	61.60	0.9743	4.48	0.9636

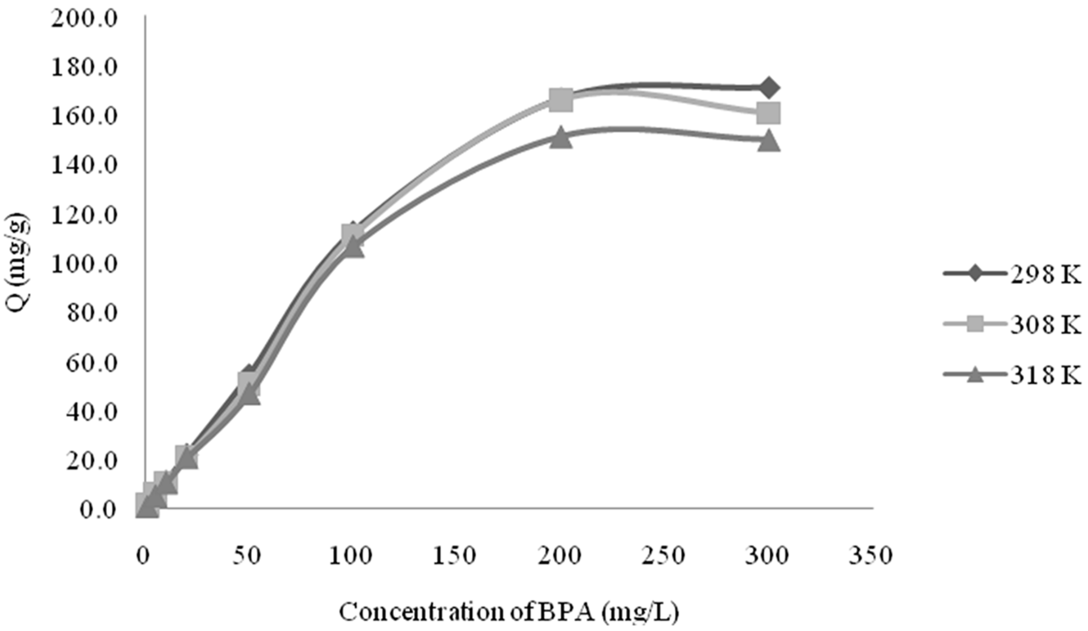


Figure 6. Effect of initial concentration on adsorption of BPA onto the poly(DVB-MATrp) microbeads.

Table 2. Parameters of Langmuir and Freundlich isotherm models, for the adsorption BPA onto the poly(DVB-MATrp) microbeads.

Parameters Temperature (K)	Langmuir isotherm constants			Pseudo-second-order kinetic model		
	K_Lx10^{-3} (L/mg)	Q_L (mg/g)	R²	K_F (mg/g) (L/mg)$^{1/n}$	n	R²
298	1.581	224.7	0.9013	5.584	1.501	0.9756
308	1.241	228.3	0.9409	3.197	1.254	0.9794
318	1.240	212.8	0.9399	2.863	1.238	0.9822

ted in Figure 6.

The results showed that an increase in the initial concentration of BPA causes an increase in the amount of adsorbed BPA. The maximum BPA adsorption was 171.1 mg/g at pH 7.0, 25°C. The adsorption capacity of the poly(DVAB-MATrp) microbeads decreased to 160.8 mg/g and 149.8 mg/g at 35°C and 45°C, respectively. The decrease in the adsorbed amount of BPA at higher temperatures showed that the adsorption process is exothermic. Two basic isotherm models; Freundlich and Langmuir were used to fit adsorption data. Related model parameters are given Table 2. When the correlation coefficients are compared, it is concluded that the Freundlich model fitted to the experimental data better than Langmuir model for poly(DVB-MATrp) microbeads, indicating the adsorption of BPA onto the adsorbent tended to heterogeneous adsorption. The values of 1/n were calculated in the range of 0.6662- 0.8078 implying that the BPA adsorption poly(DVB-MATrp) is favourable.

Thermodynamic Analysis

The internal energy changes of adsorption process are predicted by thermodynamic parameters e.g. the enthalpy (ΔH0), the entropy (ΔS0) and the Gibbs free energy (ΔG0) changes. The van’t Hoff equation was used to determine the enthalpy (ΔH0) and entropy (ΔS0) changes of the adsorption process. The van’t Hoff equation is given as:

$$\ln K_F = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{R}(\frac{1}{T})$$

The free energy change is then obtained as:

$$\Delta G^0 = \Delta H^0 - T\,\Delta S^0$$

Table 3. Thermodynamic parameters of BPA adsorption.

ΔH ° (kJ/mol)	ΔS° (kJ/mol.K)	ΔG° (kJ/mol)		
		298 K	308 K	318 K
26.491	-0.07517	-4.091	-3.339	-2.588

Thermodynamic parameters are provided in Table 3. The negative values of ΔHo show the exothermic nature of the adsorption process. The free energy changes (ΔGº) of the BPA adsorption process have negative values in the 298 K to 318 K temperature range, demonstrating the spontaneous adsorption. The adsorption is less favourable at high temperatures since an increase in temperature from 298 to 318 K causes a decrease in the negative value of ΔGº. Meanwhile, the value of ΔS° was found to be negative which indicated a decreasing of randomness at the solid–solution interface.

Reusability studies

The regeneration and reusability of an adsorbent play an important role in practical applications. The BPA adsorption capacities were determined as 171.1 mg/g, 169.7 mg/g, 168.9 mg/g, 165.5 mg/g and 163.1 mg/g for the first, second, third, fourth and fifth adsorption/desorption cycle, respectively. After fifth cycle, no significant change was observed in the adsorption capacity of poly(DVB-MATrp) microbeads. The physical nature of adsorption enables the desorption of the adsorbed BPA on poly(DVB-MATrp) microbeads. These results showed that the poly(DVB-MATrp) microbeads can be repeatedly used in BPA adsorption.

CONCLUSIONS

BPA is attracting attention as an endocrine disrupter. Therefore, the removal of BPA from water is of great importance for the protection of human and environmental health. In this study, we synthesized polymeric microbeads to remove BPA from aqueous phase. Firstly, the cross-linked poly(DVB-MATrp) microbeads were synthesized and characterized by SEM, IR, elemental analysis and swelling test. The poly(DVB-MATrp) microbeads are very hydrophobic in nature which cause specific interaction with BPA molecules in aqueous solution. The maximum BPA adsorption capacity of the prepared microbeads was determined to be 171.1 mg/g at pH 7.0, 25ºC. The BPA adsorption studies were conducted at three different temperatures (25ºC, 35ºC and 45ºC). The pseudo-second-order kinetic model fits the experimental data with high correlation coefficients for all temperatures. Adsorption data fitted with the Freundlich isotherm model, which demonstrates heterogenous binding sites. BPA adsorption onto the poly(DVB-MATrp) microbeads was spontaneous and fast. In addition, the poly(DVB-MATrp) microbeads can be regenerate easily and used for BPA adsorption repeatedly. The poly(DVB-MATrp) microbeads can be used in BPA removal processes due to their superior characteristics.

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