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Desorption of bisphenol-A (BPA) and regeneration of BPA-spent granular activated carbon using ultrasonic irradiation and organic solvent extraction

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ABSTRACT

Desorption of bisphenol-A (BPA) from granular activated carbon (GAC) by ultrapure water and regeneration of BPA-spent GAC using ultrasonic irradiation and organic solvent extraction are investigated in this paper. The results showed that the pseudo-second-order kinetic model more appropriately described the BPA desorption pattern, and BPA desorption well followed the classical Arrhenius equation at a temperature range of 288–308 K with an activation energy of −56.47 kJ mol^{-1}. Desorption rates of BPA were significantly favored by increasing acoustic intensity within a range of 17–100 W at 20 kHz frequency. Certain fraction of methanol or ethanol, particularly methanol, in the extraction solution enhanced the BPA desorption from the spent GAC. The optimal volumetric fraction of methanol in the extraction solution was 75% at which 80% of BPA on the spent GAC was desorbed within 400 min. A synergetic enhancement could be observed when ultrasonic irradiation was coupled with methanol for the GAC regeneration, and the regeneration efficiency was improved with the increasing irradiation intensity. Results demonstrate that the proposed ultrasonic irradiation and organic solvent extraction technology is a promising alternative to regenerate the spent GAC with BPA.

Keywords: Bisphenol-A (BPA); Granular activated carbon (GAC); Regeneration; Ultrasonic irradiation; Organic solvent

1. Introduction

Recently, endocrine-disrupting chemicals (EDCs) that are increasingly detected in wastewater, surface water, groundwater, and even drinking water [1–4] have caused more and more concerns on the public health [5,6], because they could lead to dysfunction of human endocrine systems even at very low levels [7]. As a representative of EDC [8], bisphenol-A (BPA) has been widely used as a key intermediate in the production of epoxy resins, polycarbonate plastics and certain polyester resins [9]. BPA has been frequently found in treated wastewater [10]. For example, BPA was typically detected at a range of 0.16–0.36 μg L^{-1} in secondary effluent of Canada [11], 0.033–36.7 μg g^{-1} BPA was reported in municipal sewage sludge in Canada [12], 30–330 μg g^{-1} in Germany [13], and even 28.3 μg g^{-1} in

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China [14]. These findings demonstrate that BPA appears not to be effectively eliminated in wastewater treatment facilities. Consequently, direct reuse of such treated wastewater, a new and encouraging global response to water shortage, may be of great risk [15].

Granular activated carbon (GAC)-based adsorption is a widely used water treatment technology for the removal of a broad spectrum of pollutants in water and wastewater, due to its extremely high specific surface area, tailored pore distribution, and high degree of surface reactivity [16]. Once GAC is saturated with contaminants of concern, the exhausted GAC should be regenerated to lower the operational cost and reduce the amount of waste GAC required for disposal [17]. However, effective and low-cost regeneration of exhausted GAC is always a great challenge because of the high affinity of pollutants to the sorbent surface. In practice, GAC regeneration techniques such as thermal regeneration [18], solvent regeneration [19], catalytic wet oxidation regeneration [20], and biological regeneration [21] have been studied and applied over the past few decades. Nevertheless, these techniques have been, more or less, restricted by their respective limitations such as too high energy consumption, costly chemical reagents, potential secondary pollution, and low regeneration efficiencies. Therefore, there is a clear demand to develop innovative, technically, and economically sound GAC regeneration methods. As a response, more recently, a few new GAC regeneration methods have been proposed and attempted, including microwave regeneration [22], electrochemical oxidation [23], supercritical or subcritical regeneration [24,25], and ultrasonic regeneration [26].

In this study, we proposed a new regeneration method for BPA-spent GAC using ultrasonic irradiation coupled with organic solvent extraction. The objective of this study was to characterize BPA desorption from spent GAC, and evaluate the technical feasibility of three BPA-spent GAC regeneration methods, including ultrasonic irradiation alone, organic solvent extraction, and combination of both.

2. Materials and methods

2.1. Materials

BPA (purity greater than 99%) was purchased from Sigma-Aldrich, whose physical and chemical characteristics are shown in Table 1. All solutions were prepared using the ultrapure water produced from a Mill-Q ultrapure water purification system (Billerica, Massachusetts, USA). Methanol and ethanol (high performance liquid chromatography (HPLC) grade) were obtained by Fisher Sci. (USA). All the chemicals used were of analytical grade, except as noted. A commercial coal based GAC was purchased from Calgon Carbon Corp. China, whose characteristics are shown in Table 2. Prior to all experiments, the carbon was washed repeatedly with tap water and ultrapure water to remove any fine particles and soluble materials, and then dried in an oven at 105°C until the weight was constant, and finally stored in a desiccator.

2.2. Experimental procedures

2.2.1. GAC saturation with BPA

A series of 500 mL beakers containing 200 mL of 300 mg L^{-1} BPA solution were installed in a shaker with a temperature control at 25°C. The adsorption was initiated once 200 mg GAC was added. A rapid shaking (150 rpm) ensured a complete mixing solution state. After 24 h, the exhausted GAC was separated by filtration, then rinsed repeatedly with ultrapure water, and finally dried in an oven at 105°C for 24 h. The dried BPA-spent GAC weighed 235.2 ± 0.2 mg. Therefore, the adsorbed BPA on the GAC was 0.176 ± 0.001 g g^{-1}.

2.2.2. Desorption tests

Batch desorption experiments were carried out in the ultrasonic reactor as shown in Fig. 1. Ultrasonic transducer (Cole-parmer cp130), at a frequency of 20 kHz, was used at different powers of 17, 30, and 100 W. Two hundred and fifty milliliters of sample was dispensed in the vessel surrounded by a glass cylindrical jacket, which prevented the increase of reaction temperature (25.0 ± 1.0°C). A rapid mixing provided by a shaker ensured a complete solution mixing. Acoustic power dissipated in the reactor was measured using the standard calorimetric method [27]. At predetermined time intervals, 1.0 mL sample was withdrawn in duplicate for further HPLC analysis. The standard deviations of all the measured data were less than 5%.

2.2.3. Regeneration of GAC

At the volume ratio of 75/25% (organic solvents/water), 200 mL methanol and ethanol aqueous solutions were used to regenerate the BPA-exhausted GAC in the shaker, respectively. Different volume ratios of aqueous methanol solution (25/75%, 50/50%, 75/25%, and 100/0%, respectively) were used for regeneration of GAC. And then the aqueous methanol

Table 1
Physical and chemical characteristics of BPA

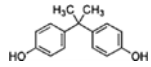
Molecular weight	Melting point (°C)	Boiling point (°C)	Henry's constant (10^{-6})	Vapour pressure (10^{-6})	Water solubility (mg mL^{-1})	Log K_{OW}	Molecular structural formula
228.3	155	398	4.03	5.3	<1 (21.5°C)	3.4	

Table 2
Physical and chemical characteristics of GAC

Average particle size (μm)	Iodine (mg g^{-1})	Methylene blue value (mg g^{-1})	Intensity (%)
550	1,092	225	92.9

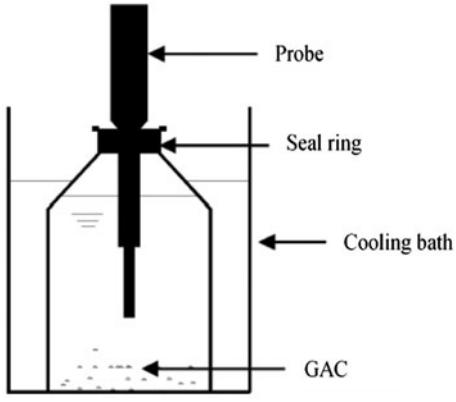


Fig. 1. Schematic description of the ultrasonic reactor.

solution (25/75%) was used in regeneration at different powers (17, 30, and 100 W) under 20 kHz of ultrasound.

2.3. Analytical methods

BPA was analyzed by HPLC (Shimadzu LC-2010AHT) with shim-pack VP-ODS column (150 × 4.6 mm id). The mobile phase was acetonitrile-water at 70:30 (v/v), and the flow rate of mobile phase was 0.8 mL min^{-1} for BPA. UV detection was performed at 278 nm. Minimum detectable level was 1.0 μg L^{-1}.

2.4. Desorption kinetic models

BPA desorption from GAC to bulk solution can be generally written as Eq. (1).

$$dC/dt = -k(C_e - C_t)^n \quad (1)$$

where C_e and C_t (μg L^{-1}) are the BPA concentrations at chemical equilibrium and any specific time t, respectively; k and n are the reaction order and rate constant, respectively. It would be noted that $C_e - C_t$ is the driving force for BPA desorption, and represents the deficiency of actual BPA concentration to saturated concentration in bulk solution. After integration, Eq. (1) can be written as Eq. (2) (n =1) or Eq. (3) (n = 2) [28].

$$\mathrm{Ln}\,(C_e - C_t) = \mathrm{Ln}\,(C_e) - k_{d1}t \quad (2)$$

$$t/C_t = 1/(k_{d2}C_e^2) + t/C_e \quad (3)$$

where k_{d1} (min^{-1}) and k_{d2} (L μg min^{-1}) are rate constants of pseudo-first-order and pseudo-second-order desorption kinetic models, respectively.

3. Results and discussion

3.1. Desorption kinetics

3.1.1. Comparison of different desorption kinetic models

BPA desorption with time is shown in Fig. 2. As shown, the desorbed BPA plateaued within 400 min. Experimental data were fit by pseudo-first- and second-order kinetics models, respectively. Key kinetics parameters are summarized in Table 3. Results demonstrated that both models were acceptable ($R^2 > 0.95$), and the pseudo-second-order kinetic model ($R^2 > 0.99$) was slightly better than the other one.

3.1.2. Effect of the temperature on BPA desorption

Effect of temperature on the BPA desorption in ultrapure water is illustrated in Fig. 3, and the

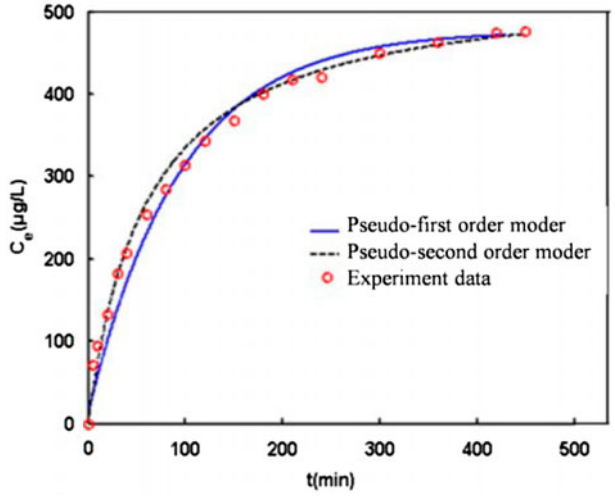


Fig. 2. Comparison of different desorption kinetic models (conditions: initial BPA concentration = 0 μg L^{-1}, spent GAC concentration = 1.176 ± 0.001 g L^{-1}, and temperature = 298 K).

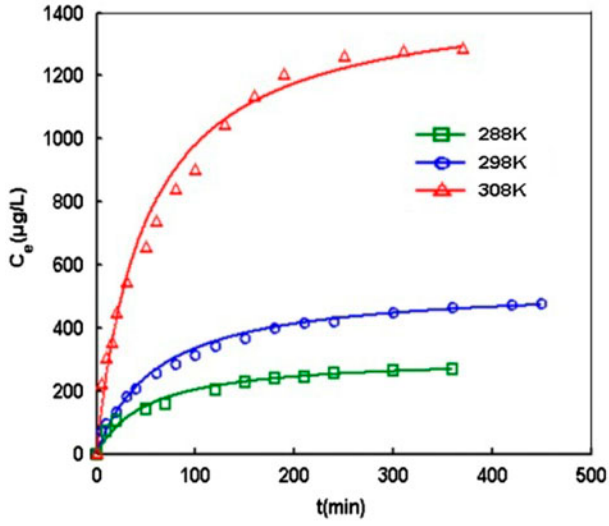


Fig. 3. Effect of temperature on the BPA desorption from GAC to water (conditions: initial BPA concentration = 0 μg L^{-1}, spent GAC concentration = 1.176 ± 0.001 g L^{-1}, temperature = 288, 298, and 308 K; scattered symbols and smooth lines denote the experimentally measured and modeled (pseudo-second-order reaction) data, respectively).

Table 3
Parameters of different desorption kinetic models

Kinetic models	Coefficient	R^2
Pseudo-first order	$k_{d1} = 0.0106\ min^{-1}$	0.9647
Pseudo-second order	$k_{d2} = 3.1202 \times 10^{-5}$ L μg min^{-1}	0.9958

Table 4
Parameters of desorption kinetic models (pseudo-second-order) at different temperatures

Temperature (K)	k_{d2} (L μg min^{-1})	R^2
288	6.4708×10^{-5}	0.9927
298	3.1202×10^{-5}	0.9958
308	1.3973×10^{-5}	0.9932

corresponding kinetic parameters are summarized in Table 4. As seen, a high desorption rate was achieved at a high temperature. Of noted, the rate increase was more pronounced at a higher temperature range. For example, at 360 min, the BPA concentration in bulk solution was increased from 210 to 430 μg L^{-1} with the increasing temperature from 288 to 298 K, and then was significantly improved to 1,280 μg L^{-1} when the temperature was further increased to 308 K. Generally, at high temperature, the activation energy required to accomplish the desorption process is reduced, and the BPA solubility in water is enhanced. The observed pseudo-second-order rate constants well followed the classical Arrhenius equation (Fig. 4), and the estimated activation energy (E_a) of the adsorption was −56.47 kJ mol^{-1}.

3.1.3. Effect of ultrasonic irradiation on the BPA desorption

Guo and Feng have proved that BPA could be significantly decomposed under the ultrasonic irradiation at the frequency of 20 kHz in aqueous solution [10]. The effect of ultrasonic irradiation at different powers on the BPA desorption from GAC is shown in Fig. 5. With the increasing irradiation power from 0 to 30 W, the BPA desorption rate was significantly increased. The enhancement effect was mainly ascribed to cavitations produced by ultrasonic irradiation. Micro-bubbles generated during cavitation were energy carriers, and could immediately collapse their production. Such significant changes could locally generate extremely high temperatures and pressures. Therefore, the bonds between BPA and GAC sorption sites might be weakened or even broken during the so-called cavitation events. The intensity of the rapid whirlpool and high pressure oscillation generated by cavitation mainly depended on the energy transferred into water by ultrasonic sensor. With the increasing ultrasonic power, the intensity of cavitation correspondingly increased, and thus more BPA was desorbed from GAC.

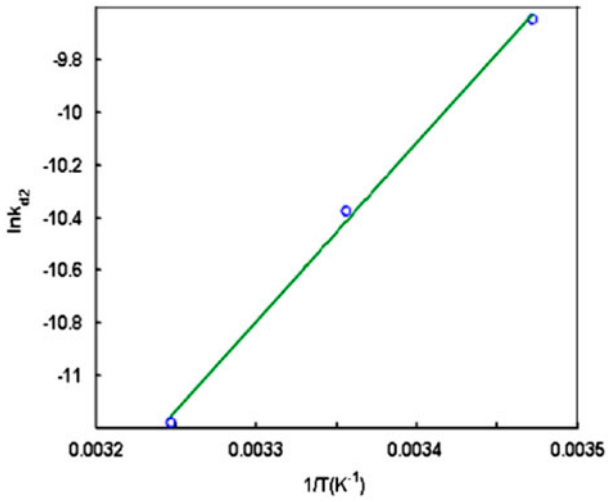


Fig. 4. The relationship between temperature and the k_{d2} value of desorption kinetic models (conditions: initial BPA concentration = 0 μg L^{-1}, spent GAC concentration = 1.176 ± 0.001 g L^{-1}, and temperature = 288, 298, 308 K).

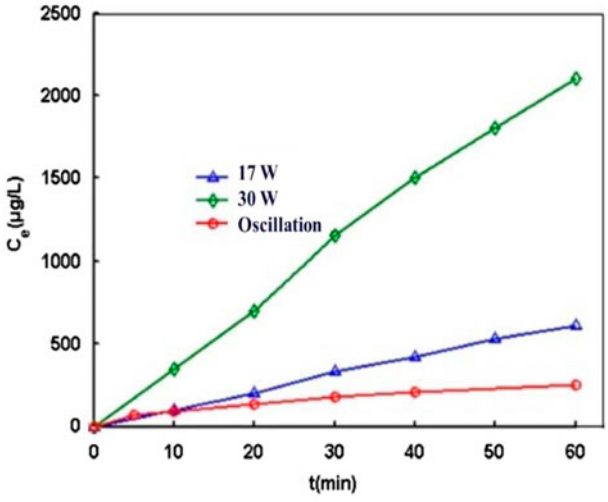


Fig. 5. Rates of BPA desorption from GAC to water under shaking or ultrasonic irradiation conditions (conditions: initial BPA = 0 μg L^{-1}, spent GAC concentration = 1.176 ± 0.001 g L^{-1}, temperature = 298 K, ultrasound power = 17, 30 W, and ultrasound frequency = 20 kHz).

3.2. Regeneration of GAC by organic solvents

3.2.1. Comparison of different organic solvents for GAC regeneration

Ethanol was demonstrated to enhance the p-chlorophenol desorption from GAC [29]. In this study, two common organic solvents, methanol and ethanol, were employed for the BPA desorption. The desorption data well fit the pseudo-second-order kinetic models, as shown in Fig. 6. Key desorption kinetic parameters and corresponding coefficients of determination are listed in Table 5. As shown, at any specific time, the BPA desorption rate achieved by methanol was slightly higher than those achieved by ethanol. Within 400 min, methanol desorbed 76% of BPA, followed by ethanol (73%). The difference of GAC regeneration behaviors in the two different organic solvents might be ascribed to the fact that methanol has higher polarity than ethanol. Given that GAC is non-polar, a more polar solvent in the extract solution typically has the stronger affinity with non-polar BPA from GAC.

3.2.2. Effect of volumetric fraction of organic solvent on GAC regeneration

The impact of the volumetric fraction ratios (25, 50, 75, and 100%) of methanol in Milli-Q water upon

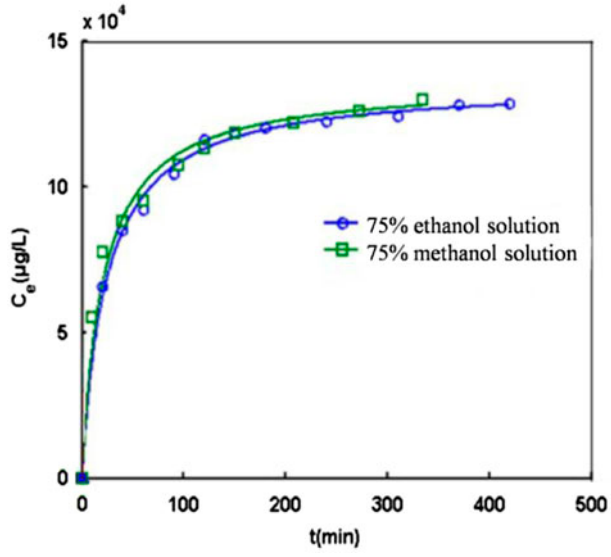


Fig. 6. BPA desorption vs. time in different organic extraction solutions (conditions: initial BPA concentration = 0 μg L^{-1}, spent GAC concentration = 1.176 ± 0.001 g L^{-1}, temperature = 298 K, and volume ratio of organic solvent solution = 75%).

Table 5
Kinetics parameters (pseudo-second-order reaction) of BPA desorption from GAC to water in different organic extraction solutions

Solvent	k_{d2} (L μg min^{-1})	R^2
Methanol	3.5366×10^{-7}	0.9984
Ethanol	3.0979×10^{-7}	0.9996

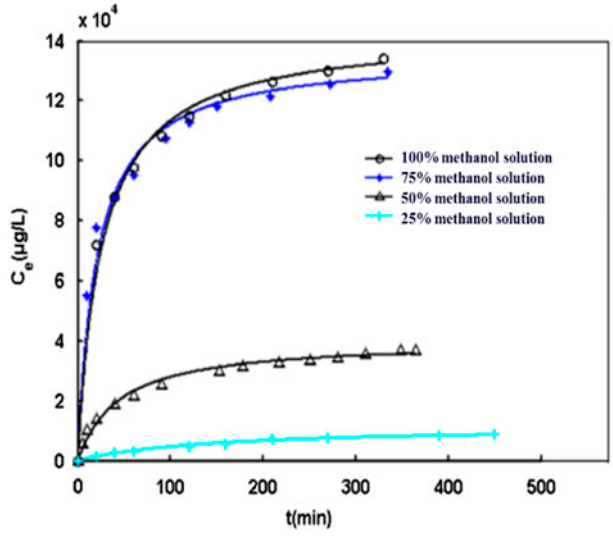


Fig. 7. Effect of methanol fraction in the extraction solution on the BPA desorption from GAC (conditions: initial BPA concentration in methanol solution = 0 μg L^{-1}, spent GAC concentration = 1.176 ± 0.001 g L^{-1}, temperature = 298 K, and volumetric fraction of methanol = 100, 75, 50, and 25%).

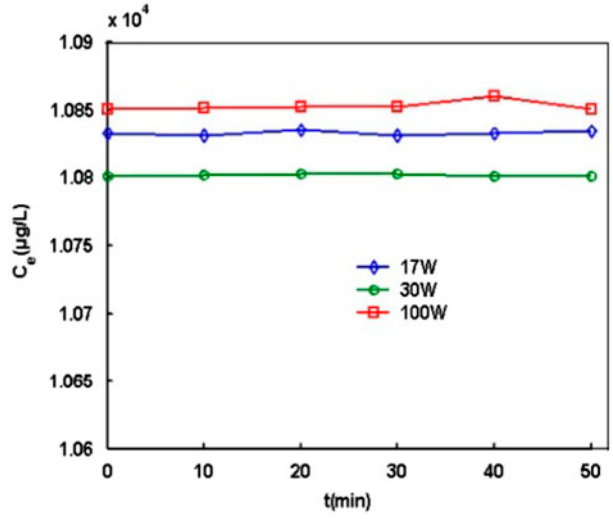


Fig. 8. Ultrasonic irradiation of BPA in methanol solution under different powers (conditions: volume fraction of methanol in water = 25%; initial BPA concentration in ultrapure water = 10.80, 10.83, 10.85 mg L^{-1}, the corresponding ultrasound powers = 17, 30, and 100 W, ultrasound frequency = 20 kHz, and temperature = 298 K).

GAC regeneration is shown in Fig. 7. All the data well fit pseudo-second-order desorption kinetic models, and their kinetic data are summarized in Table 6. As shown, the desorption rate was significantly increased with the increasing volumetric ratio from 0 to 75%. For example, at 100 min, the BPA concentrations in the extraction solution containing 25, 50, and 75% methanol were 4,000, 30,000, and 118,000 μg L^{-1}, respectively. However, the augment in the BPA desorption was almost marginal when the methanol fraction further increased from 75 to 100%, thus suggesting 75% was the optimal volumetric methanol fraction for the BPA desorption.

3.3. Regeneration of GAC by ultrasound combined with organic solvents

3.3.1. Ultrasonic degradation of methanol BPA solution

Results in ultrasonic irradiation of BPA in a methanol solution are presented in Fig. 8. Within 50 min, BPA was not significantly reduced at an increasing irradiation power from 17 to 100 W, thereby suggesting that BPA could not be significantly decomposed under

Table 6
Parameters of desorption kinetics models (pseudo-second-order) in methanol solution of different volume ratios

Volume ratio (methanol (%)/water (%))	k_{d2} (L μg min^{-1})	R^2
25/75	5.6708×10^{-5}	0.9917
50/50	4.1279×10^{-7}	0.9952
75/25	3.5366×10^{-7}	0.9984
100/0	2.6866×10^{-7}	0.9991

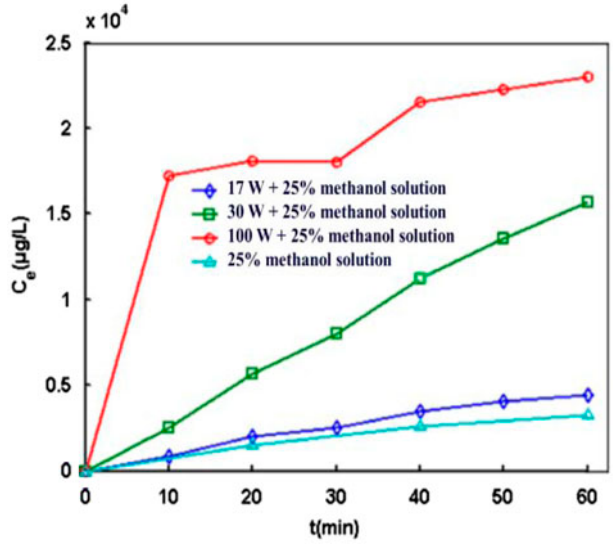


Fig. 9. Rates of BPA desorption from GAC in methanol extraction solution under ultrasonic irradiation or shaking (conditions: initial BPA concentration in methanol solution = 0 μg L^{-1}, spent GAC concentration = 1.176 ± 0.001 g L^{-1}, temperature = 298 K, volume ratios of methanol solution = 25%, ultrasound power = 17, 30, and 100 W, and ultrasound frequency = 20 kHz).

the experimental conditions. The phenomenon of BPA could not be significantly decomposed under ultrasonic irradiation in methanol solution due to the interference of methanol, a more volatile organic compound with a much higher concentration, which is greatly easier to be decomposed by sonolysis.

3.3.2. Regeneration of GAC by different power ultrasound combined with organic solvents

Results in the BPA-exhausted GAC regeneration under ultrasonic irradiation in the presence of 25% methanol are shown in Fig. 9. Obviously, the BPA desorption rate was significantly increased with the increase in ultrasonic irradiation power. For example, after 60 min, the BPA concentrations in bulk solution at 0, 17, 30, and 100 W were 3,000, 4,000, 16,000, and 22,500 μg L^{-1}, respectively. The results demonstrated that the effect of ultrasonic irradiation for the BPA desorption was greatly enhanced in an organic solvent solution.

The desorption of BPA into water and methanol solution under ultrasonic irradiation is compared in Fig. 10. For any specific irradiation power, the BPA desorption rate in a 25% methanol solution was significantly higher than that in water solution. For example, within 60 min, the BPA concentration in the organic solvent solution was 15,800 μg L^{-1}, much higher than 1,800 μg L^{-1} achieved in water at 100 W.

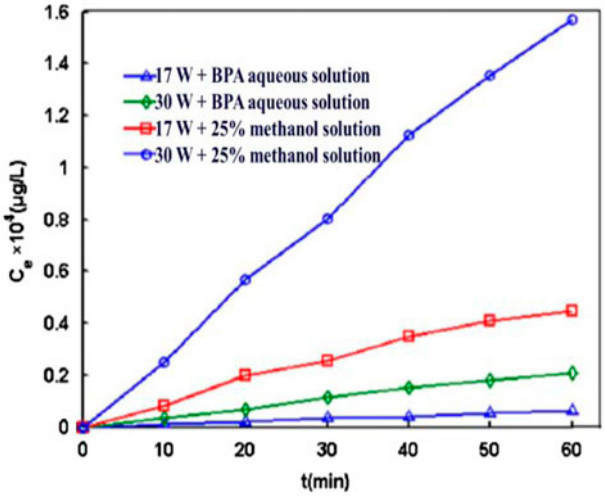


Fig. 10. Rates of BPA desorption in water and methanol extraction solutions (conditions: initial BPA concentration in methanol solution = 0 μg L^{-1}, spent GAC concentration = 1.176 ± 0.001 g L^{-1}, temperature = 298 K, volume ratios of methanol solution = 25%, ultrasound power = 17 and 30 W, and ultrasound frequency = 20 kHz).

4. Conclusions

This study was an effort to explore desorption kinetic characteristics of BPA from BPA-spent GAC, and also to evaluate the feasibility of ultrasonic irradiation and/or organic solvent for the GAC regeneration. Our results show that the combined ultrasonic irradiation and organic solvent extraction can significantly improve the BPA desorption rate, advantageous over either of ultrasonic irradiation or organic solvent extraction. This finding provides a low-cost, viable, and potential alternative for regeneration of spent GAC in practice.

Acknowledgments

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