

Research Article

Adsorbability Enhancement of Macroporous Resin by Dielectric Barrier Discharge Plasma Treatment to Phenol in Water

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In order to enhance the adsorption efficiency and economize the use of macroporous resin, we have treated it with the dielectric barrier discharge (DBD) plasma to improve its adsorbing capacity for phenol. The effects of operation conditions, for instance, applied voltage, treated time, and air flow rate on resin, were investigated by adsorption kinetics and isotherms. Results showed that the adsorption data were in good agreement with the pseudo-second-order and Freundlich equation. Experimental results showed that the modified resin was 156.5 mg/g and 39.2% higher than the untreated sample, when the modified conditions were conducted for discharge voltage 20 kV, treatment time 45 min, and air flow rate 1.2 L/min. The resin was characterized by FTIR and nitrogen adsorption isotherms before and after the DBD processes. It was found that the reason for the enhancement of resin adsorbability was attributed to the DBD plasma changing the surface physical and chemical structure.

1. Introduction

Adsorption on resin is a simple technology that has found widespread application in the treatment of industrial effluents [1, 2]. In order to improve the adsorption efficiency and economize the use of macroporous resins, many studies on surface modifications have been carried out, and they mainly focus on chemical modifications [3–6].

Recently, nonthermal plasma technology for material modification becomes one of the most active fields [7–9]. Nonthermal plasma surface treatment has lots of features, including no change to the thermal and mechanical properties of the adsorbents, but altering the various interfacial characteristics, such as the surface chemical and physical structure properties [10, 11].

Dielectric barrier discharge (DBD) could produce spatially uniform and stable plasma under atmospheric pressure easily [12, 13]. Using oxygen as gas source in DBD plasma, various kinds of active species including high-energy electron, ozone, and many other strong oxidized free radicals have been generated [12, 13], which could be beneficial to the material surface modification. However, to our knowledge,

systematic studies on surface modification of resin by DBD plasma are still limited.

Phenol is a kind of highly toxic and refractory pollutant commonly emitted from industrial effluents. This paper presents a new approach that macroporous resin is modified in a double dielectric barriers discharge reactor. The effects of operation parameters for phenol removal, including applied voltage, treatment time, and gas flow rate on adsorption kinetics and adsorption isotherms, have been investigated. FTIR and N₂ adsorption isotherms were employed to evaluate the surface chemical and physical characteristics of macroporous resin treated with DBD plasma. The obtained experimental data demonstrate the DBD could enhance the phenol adsorption of resin effectively.

2. Experimental

2.1. Materials. Macroporous resin (XDA-1, diameter 0.3–1.2 mm) was acquired from Xi'an Sunresin and Technology Co. Ltd. (Shanxi province, China). Before adsorption, the resins were under extraction filtration process using ethanol for at least 8 hours and then dried in an oven at 333 K

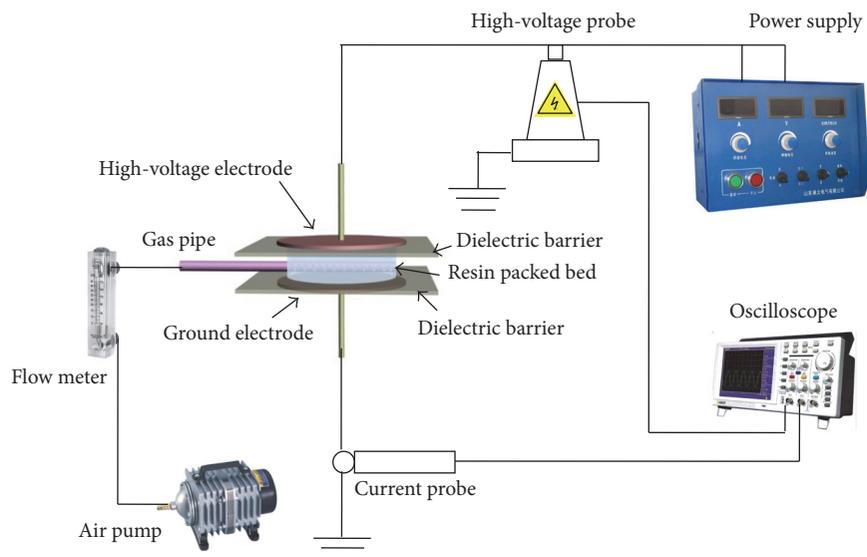


FIGURE 1: Schematics of the experimental system.

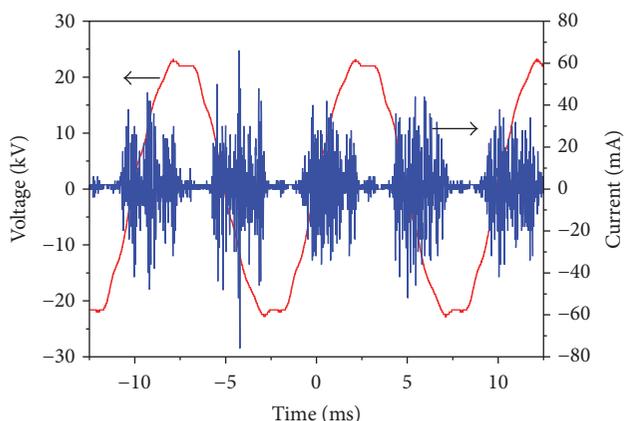


FIGURE 2: Typical voltage and current waveforms of the DBD reactor.

overnight. Phenol and other chemical reagents were of analytical grade and purchased from the Tianjin Kermel Co., Ltd. (Tianjin, China). The phenol solution was prepared with deionized water.

2.2. Experimental System. Figure 1 illustrated the DBD treatment system. The DBD reactor was driven by an alternating current (AC) high-voltage power. The reactor was comprised of two parallel-plate stainless steel electrodes as the high-voltage and ground electrodes, respectively, and each electrode was covered by one quartz glass dielectric, and the detailed dimensions were given in our previous articles [14]. The AC power frequency was 100 Hz, and the peak voltage was adjustable in a range of 0–50 kV. In every batch experiment, before the DBD treatment began, 2.0 g resin was filled in the packed bed of the reactor. Typical voltage and current waveforms of the DBD reactor were shown in Figure 2, which were recorded by a digital oscilloscope

(Tektronix TDS2014, USA) equipped with a voltage probe (Tektronix P6015A, USA) and a current probe (Tektronix P6021, USA).

2.3. Adsorption Experiments. After the DBD plasma treatment, 0.05, 0.1, 0.2, 0.3, and 0.4 g of resins were added into the phenol solution (50 mL, 100 mg/L) in a series of sealed flasks. These flasks were placed in the water bath shaker with a 150 rpm of agitation speed.

The concentrations of phenol were determined by a UV-vis spectrophotometer (UV-2102C, Unico (Shanghai, China) Instrument Co., Ltd.). The equilibrium adsorption capacity of phenol onto the adsorbent (q_e , mg/g) was calculated from the following equation:

$$q_e = (C_o - C_e) \frac{V}{m}, \quad (1)$$

where C_o and C_e (mg/L) are the initial and equilibrium concentration of phenol in solution, q_e is the adsorption capacity of resin, V (L) is the volume of solution, and m (g) is the adsorbent mass.

Langmuir isotherm is used to describe a type of monolayer adsorption, which has a finite number of identical localized sites:

$$q_e = \frac{Q_o K_L C_e}{1 + K_L C_e}, \quad (2)$$

where Q_o (mg/g) is the initial amount of adsorbate and K_L (L/g) is a Langmuir constant related to the affinity of the binding site.

Freundlich sorption isotherm is the most widely used equation describing the nonideal and reversible adsorption, and the equation has the form:

$$q_e = K_F C_e^{1/n}, \quad (3)$$

TABLE 1: Effect of applied voltage on adsorption kinetic parameters of virgin and DBD treated resins.

Sample	Exp $q_{e,exp}$ (mg/g)	Lagergren first-order			Pseudo-second-order		
		k_1 (1/h)	$q_{e,cal}$ (mg/g)	R^2	k_2 (g/mg/h)	$q_{e,cal}$ (mg/g)	R^2
Untreated	112.4	0.510	102.3	0.923	0.002348	117.2	0.984
16 kV	123.4	0.453	111.1	0.976	0.004471	126.4	0.995
20 kV	133.1	0.443	124.2	0.961	0.004788	137.8	0.996
24 kV	114.7	0.429	101.3	0.893	0.002669	117.8	0.988
28 kV	109.0	0.461	94.5	0.942	0.002567	112.1	0.986

TABLE 2: Effect of applied voltage on Freundlich and Langmuir parameters of virgin and DBD treated resins.

Adsorbents	Freundlich parameters			Langmuir parameters		
	$1/n$	K_F ($\text{mg}^{(1-1/n)}\text{L}^{1/n}/\text{g}$)	R^2	q_m (mg/g)	K_L (L/mg)	R^2
Untreated	0.7681	1.966	0.977	115.3	0.01053	0.926
16 kV	0.7766	2.174	0.978	126.0	0.01106	0.936
20 kV	0.7778	2.318	0.989	133.8	0.01119	0.934
24 kV	0.7736	2.035	0.980	119.0	0.0108	0.928
28 kV	0.7913	1.581	0.986	114.0	0.00884	0.937

where K_F ($\text{mg}^{(1-1/n)}\text{L}^{1/n}/\text{g}$) is a Freundlich constant related to adsorption amount and n is also a Freundlich constant to measure the adsorption intensity.

For the purpose of comparison the adsorption capacity before and after DBD plasma treatment, the same method was used to determine the adsorption kinetic of the virgin resin.

2.4. Analysis Method. The surface characteristics of virgin and DBD treated resins were measured from the N_2 adsorption at 77 K by NOVA 1200 (Quanta Chrome) equipment. Surface areas were identified according to the BET method. FTIR spectroscopy (EQUINOX5 spectrophotometer) was applied to characterize the chemical properties of the resin samples.

3. Results and Discussion

3.1. DBD Modified Resins Adsorption. In this study, the comparison of adsorption dynamics and isotherms of the resins has been done before and after DBD treatment under different operation parameters, including applied voltage, treatment time, and air flow rate, which allow us to obtain valuable information for the adsorption process of these resin samples.

Adsorption kinetic is an important physical characterization of adsorption rate, because it could indicate the removal rate of the pollutant in aqueous solutions and provide valuable data for a good understanding of the sorption reactions mechanism. Therefore, Lagergren first-order and pseudo-second-order kinetic models were utilized in the analysis of the experimental data in this study.

Analysis of isotherm data is very important for predicting adsorption capacity and adsorption behavior of adsorbent. Several models have appeared in the literature to evaluate adsorption isotherm. The Langmuir and Freundlich [15] models are the models often used. In the Langmuir model it

is assumed that the adsorption is totally homogeneous in the surface of adsorbent, whereas it is a highly heterogeneous surface for Freundlich model. Thus, Langmuir and Freundlich isotherm models were tested and made the comparison of the goodness of fit based on the experimental data.

3.1.1. Effect of Applied Voltage. The strength of electric field obviously affects the generation of active species (such as O_3 , $\cdot\text{OH}$, etc.) in DBD plasma and thus can influence the resin modification. The other viable run parameters were included below: treatment time 30 min and air flow rate 0.8 L/min. Figure 3 presented the adsorption kinetics of virgin and DBD treated resins under different applied voltages. For both samples, phenol adsorption was quick in the initial 7 h, and the equilibrium plateau was achieved in 12 h. After that time point, the adsorption amount kept constant.

The fast adsorption is probably because the pore structure of the resins offered sufficient available adsorption sites in the early stage of the adsorption. Thereafter the active sites reduced with increasing contact time, and then the observed plateau appeared.

Table 1 showed the kinetics of phenol adsorption on the resin samples. The Lagergren first-order and pseudo-second-order models were used to investigate the adsorption mechanisms. Clearly, the correlation coefficients of the pseudo-second-order model were much closer to 1, and the calculated q_e accorded well with the experimental data, proving that this model could be adopted for the simulation of the resins adsorption to phenol.

The fitting results of the Freundlich and Langmuir models were presented in Table 2 and Figure 4. The contrasted results showed that the theoretical calculations of Freundlich model could be better fitted to the experimental data than Langmuir.

In summary, as shown in Figures 3 and 4 and Tables 1 and 2, the DBD treated resins adsorption capacities upgraded firstly and then decreased with the rising peak voltages. When the applied voltage was 20 kV, the optimal adsorption

TABLE 3: Effect of treatment time on adsorption kinetic parameters of virgin and DBD treated resins.

Sample	Exp $q_{e,exp}$ (mg/g)	Lagergren first-order			Pseudo-second-order		
		k_1 (1/h)	$q_{e,cal}$ (mg/g)	R^2	k_2 (g/mg/h)	$q_{e,cal}$ (mg/g)	R^2
Untreated	112.4	0.510	102.3	0.923	0.002348	117.2	0.984
15 min	113.6	0.431	101.7	0.898	0.002545	116.4	0.986
30 min	133.1	0.443	124.2	0.961	0.004788	137.8	0.996
45 min	145.8	0.430	134.5	0.877	0.005135	147.1	0.997
65 min	103.2	0.374	97.5	0.906	0.002077	104.6	0.966

TABLE 4: Effect of applied voltage on Freundlich and Langmuir parameters of virgin and DBD treated resins.

Adsorbents	Freundlich parameters			Langmuir parameters		
	$1/n$	K_F ($\text{mg}^{(1-1/n)}\text{L}^{1/n}/\text{g}$)	R^2	q_m (mg/g)	K_L (L/mg)	R^2
Untreated	0.7681	1.966	0.987	115.3	0.01053	0.923
15 min	0.7657	2.093	0.975	117.4	0.01107	0.912
30 min	0.7778	2.318	0.982	133.8	0.01119	0.934
45 min	0.7864	2.485	0.974	148.9	0.01103	0.901
60 min	0.7599	1.822	0.982	106.5	0.01018	0.945

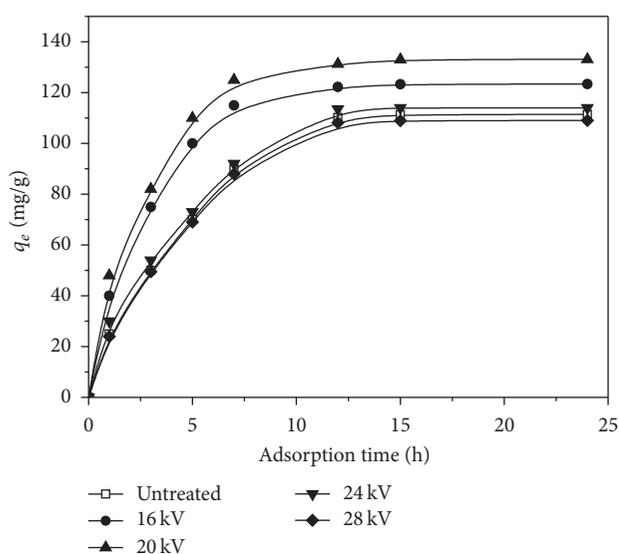


FIGURE 3: Effect of applied voltage on adsorption kinetics of virgin and DBD treated resins.

capability was 133.1 mg/g and increased 19.4% than untreated one. In contrast, the adsorption capability of 28 kV was 109.0 mg/g and lower than virgin one. It is generally believed that the excessive voltage produces more active species in plasma, which could affect the surface properties of resins, resulting in the fact that their hydrophilicity was enhanced. Under this circumstance, it was helpful for the adsorption of resin for phenol. However, the energetic species could also collapse the adsorption channels and framework of resin under higher voltage, leading to the decline of adsorption capacity of resin.

3.1.2. Effect of Treatment Time. The DBD plasma treatment time would also directly affect the generation of active species

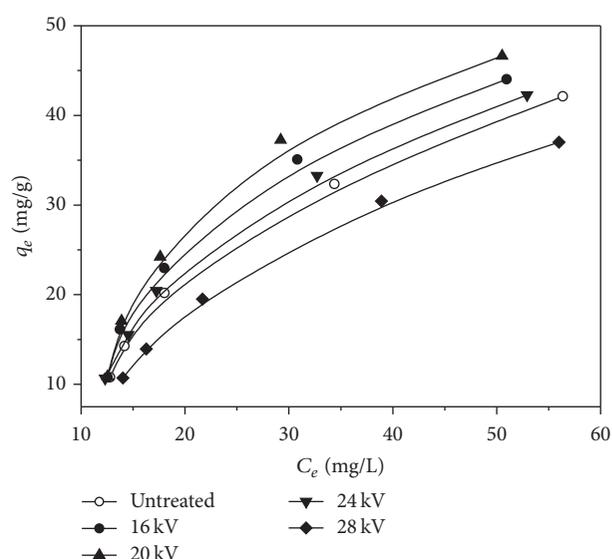


FIGURE 4: Effect of applied voltage on adsorption isotherm of virgin and DBD treated resins.

and the influence of the modification of the resins, so the effects of treatment time on resin adsorption should be clarified. The other practical run parameters included applied voltage 20 kV and air flow rate 0.8 L/min. Figure 5 indicated the adsorption kinetics of virgin and DBD treated resins under different treatment times. Combining the determination coefficient in Table 3, it is obvious that the pseudo-second-order model can imitate the phenol adsorption on resins more accurately than the Lagergren first-order.

The fitting results of the Freundlich and Langmuir isotherms were shown in Table 4 and Figure 6. By analysis and comparison, the Freundlich model could be better used to simulate the phenol adsorption onto virgin resin and DBD

TABLE 5: Effect of air flow rate on adsorption kinetic parameters of virgin and DBD treated resins.

Sample	Exp $q_{e,exp}$ (mg/g)	Lagergren first-order			Pseudo-second-order		
		k_1 (1/h)	$q_{e,cal}$ (mg/g)	R^2	k_2 (g/mg/h)	$q_{e,cal}$ (mg/g)	R^2
Untreated	112.4	0.510	102.3	0.923	0.002348	117.2	0.984
0.4 L/min	111.4	0.454	101.7	0.937	0.002592	115.6	0.985
0.8 L/min	145.8	0.430	134.5	0.877	0.005135	147.1	0.997
1.2 L/min	156.5	0.410	144.9	0.984	0.004462	157.8	0.997
1.6 L/min	127.1	0.387	117.4	0.931	0.002796	130.5	0.987

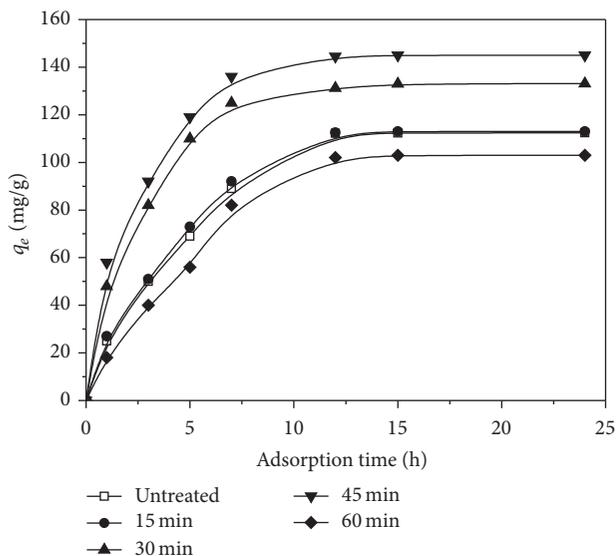


FIGURE 5: Effect of treatment time on adsorption kinetics of virgin and DBD treated resins.

treated resins under different treatment times than Langmuir model.

The adsorption quantity of phenol firstly rose and then decreased with increasing treatment time (as shown in Figure 5 and Table 3). When the discharge time increased to 45 min, the corresponding adsorption amount was 145.8 mg/g, which was enhanced to 29.0% compared to the untreated sample, whereas when discharge time extended to 60 min, the adsorption capability was 8.4% lower than virgin one. This phenomenon may be attributed to the following reasons: the increase of the treatment time resulted in the increase in the collision and even reacting probabilities between the active species and the resin, which was favorable to enhance the variety of oxygen-containing functional groups on the resin surface, strengthening the electrostatic and hydrogen bonding interactions during the phenol adsorption process. On the contrary, prolonging the reaction time would oxidize the surface structure of resin, resulting in the fact that its textural structure collapses. So it could be suggested that a longer treatment time is harmful to the adsorption of resin.

3.1.3. Effect of Air Flow Rate. Gas source is essential for non-thermal plasma production. In this research, we employed air

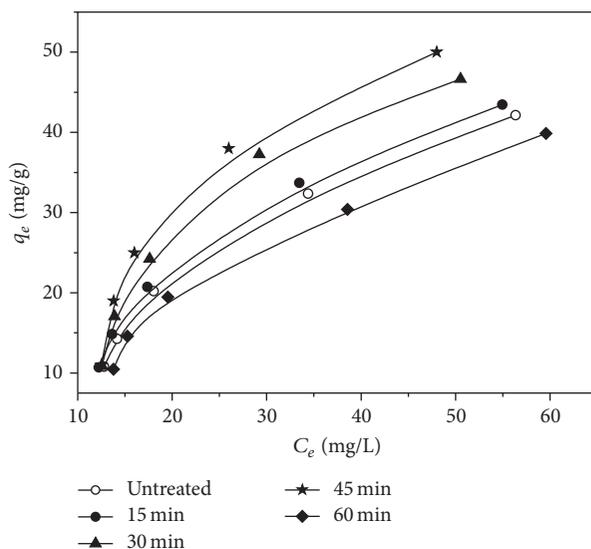


FIGURE 6: Effect of treatment time on adsorption isotherm of virgin and DBD treated resins.

as the gas source for the DBD reactor. Other experimental conditions included applied voltage 20 kV and treatment time 45 min.

Figure 7 shows the sorption kinetics of virgin and DBD treated resins under different air flow rates. Combining Table 5, it is obvious that the pseudo-second-order model has higher goodness of fit than the Lagergren first-order. Moreover, combining Figure 8 and Table 6, it can be inferred that the Freundlich model can fit better the phenol adsorption data for virgin resin and DBD treated resins.

From these figures and tables, it can be seen that the adsorption was increased with air flow rate. However, the improvement of the adsorption at 0.4 L/min was not so promoted compared with the untreated one. The maximal adsorption was achieved 156.5 mg/g when air flow rate was 1.2 L/min, which was 39.2% higher than the untreated one. It could be suggested that, with the increase of air flow, the amount of active species produced by DBD plasma is increased, which is benefit to improve the surface chemical properties. Conversely, when the air flow rate is too high (1.6 L/min), the residence time of active species in DBD reactor is shortened and their utilization efficiencies are correspondingly decreased. Hence, the combined effects of above

TABLE 6: Effect of air flow rate on Freundlich and Langmuir parameters of virgin and DBD treated resins.

Adsorbents	Freundlich parameters			Langmuir parameters		
	$1/n$	K_F ($\text{mg}^{(1-1/n)}\text{L}^{1/n}/\text{g}$)	R^2	q_m (mg/g)	K_L (L/mg)	R^2
Untreated	0.7681	1.966	0.977	115.3	0.01053	0.936
0.4 L/min	0.7597	2.123	0.974	114.2	0.01140	0.924
0.8 L/min	0.7864	2.485	0.964	148.9	0.01103	0.909
1.2 L/min	0.7918	2.574	0.953	156.1	0.01108	0.887
1.6 L/min	0.7549	2.615	0.965	127.2	0.01273	0.895

TABLE 7: Structural properties of the virgin and DBD treated resin.

Resin samples	S_{BET} (m^2/g)	$S_{\text{Micropore}}$ (m^2/g)	$V_{\text{Micropore}}$ (m^3/g)	$V_{\text{Total pore}}$ (m^3/g)	Average pore diameter (nm)
Untreated	944.6	715.8	0.377	0.586	1.428
DBD 1	1056.4	838.9	0.593	0.658	1.428
DBD 2	937.7	605.1	0.301	0.610	1.418

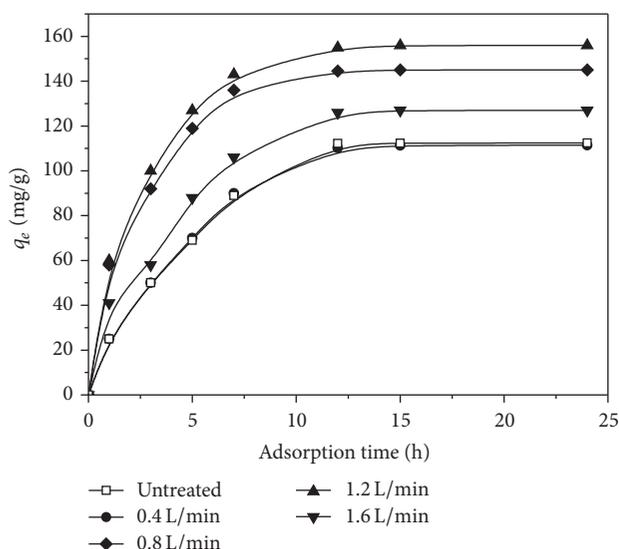


FIGURE 7: Effect of air flow rate on adsorption kinetics of virgin and DBD treated resins.

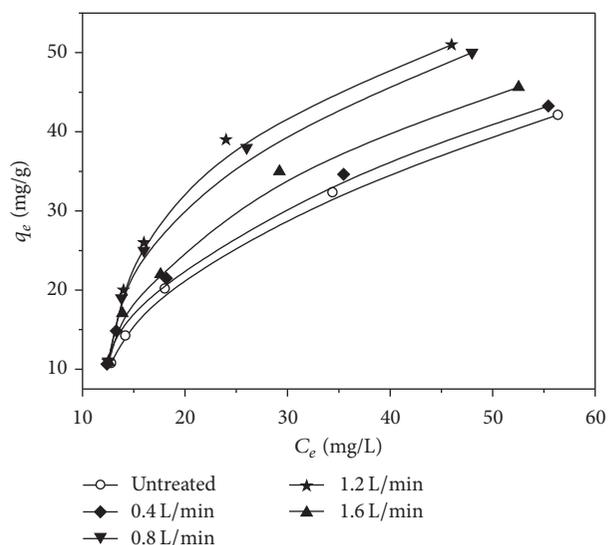


FIGURE 8: Effect of air flow rate on adsorption isotherm of virgin and DBD treated resins.

two aspects resulted in the best adsorption performance achieved when the air flow rate was 1.2 L/min.

3.2. FTIR Spectra Analysis. Figure 9 illustrates the FTIR spectra obtained from the virgin and DBD plasma treated resins. The sample presenting the highest adsorption capacity, which was treated under the condition of applied voltage 20 kV, treatment time 45 min, and air flow rate 1.2 L/min, was named as DBD 1. In contrast, DBD 2 was the lowest result at applied voltage 20 kV, treatment time 60 min, and air flow rate 0.8 L/min. From Figure 9, DBD 1 and DBD 2 had two strong representative peaks at 1604 and 2922 cm^{-1} similar to untreated one but were both greatly weakened. DBD also results in some new changes for the IR spectrum of treated samples. Firstly, a moderate C-O stretching band involving hydroxyl groups came back at 1105 cm^{-1} , and appearance of

this band might result from DBD treatment [16]. Secondly, the peak at 3600 cm^{-1} disappeared through DBD treatment, which could be assigned to O-H [4].

3.3. Surface Textural Properties. Table 7 mainly generalized the relevant surface structural properties of virgin and DBD treated resin. Based on the information observed in Table 7, it could be noticed that the various data of the DBD treated samples were considerable different from the virgin one. Compared with the untreated resin, DBD 1 resin had a bigger BET surface area, micropore area, micropore volume, and total pore volume. Conversely, all data of DBD 2 has little change compared to the virgin resin. It can be indicated that the surface area and pore volume of DBD 1 were upgraded by the suitable plasma treatment. It is generally believed that the surface textural properties of resin were improved under conditions of DBD 1; at the same time, along with the DBD

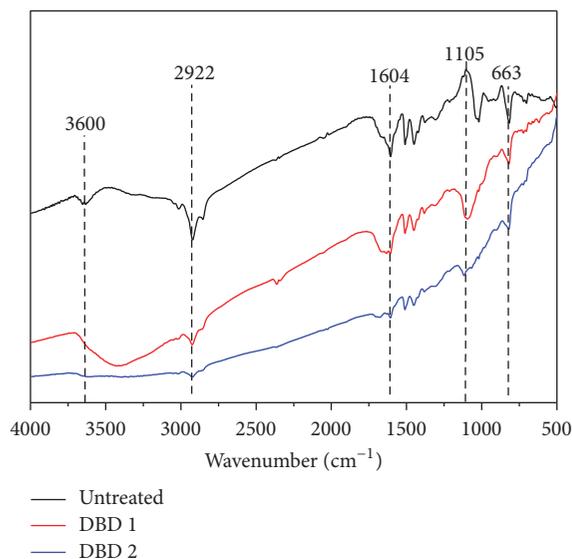


FIGURE 9: FTIR spectra of untreated and DBD treated resin.

chemical effects, such as electrostatic and hydrogen bonding interactions, the enhancement of adsorption capacity reached.

4. Conclusions

The DBD treatment for surface modification of macroporous resin had been performed for improving its adsorbability for phenol. Significant influences of applied voltage, treatment time, and air flow rate on resin adsorption capacity were observed. The experimental results show that the adsorbability of DBD treated resin was 156.5 mg/g and 39.2% higher than the untreated resin when the treatment was conducted in these conditions, such as discharge voltage 20 kV, treatment time 45 min, and air flow rate 1.2 L/min. The phenol adsorption on resins was fitted well with the pseudo-second-order and Freundlich model, indicating the homogeneity of the resin surface. At the optimum conditions, the DBD treatment increased the surface area and pore volume of the resin but slightly changed the surface chemical properties. The obtained experimental data has demonstrated that the DBD could enhance the phenol adsorption of resin effectively.

Competing Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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