

ORIGINAL ARTICLE

## Comparison of Phenol Removal between Electrochemical Reaction and Plasma Reaction

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### Abstract

The characteristics of phenol removal and UV<sub>254</sub> matters variance were investigated and compared by the variation of operating factors (NaCl concentration, air flow rate, initial phenol concentration) in electrochemical reaction (ER) and dielectric barrier discharge plasma reaction (DBDPR), respectively. The phenol removal rate was shown as 1<sup>st</sup> order both in ER and DBDPR. Also, the absorbance of UV<sub>254</sub> matters which means aromatic intermediates was analyzed to investigate the complete phenol degradation process. In ER, the phenol degradation and aromatic intermediates production rates increased by the increase of NaCl concentration. However, in DBDPR, the variation of NaCl concentration had no effect on the degradation of phenol and UV<sub>254</sub> matters. Air flow rate had a little effect on the removal of phenol and the variation of UV<sub>254</sub> matters in ER. The phenol removal rate in ER was a little higher than that in DBDPR. The produced H<sub>2</sub>O<sub>2</sub> and O<sub>3</sub> amounts in ER were 2 times and 10 times higher than those in DBDPR. The chlorine intermediates (ClO<sub>2</sub> and free chlorine) were produced in ER, however, they were not produced in DBDPR.

**Key words** : Electrochemical reaction, Dielectric barrier discharge plasma, Phenol removal, UV<sub>254</sub> matter, Oxidants

### 1. Introduction

Industrial processes generate a variety of molecules that may pollute water and air in ecosystem. Phenol is one of the most common pollutants. Wastewater containing phenolic compounds shows a serious discharge problem due to their high toxicity and poor biodegradability (Kennedy et al., 2007). Phenols are considered as priority pollutants because they are very harmful to organisms at low concentrations and many of them haven classified as hazardous pollutants (Huang et al., 2007). Phenols are present in wastewater of various industries, such as refineries, coking operations, coal processing, and manufacture

of petrochemicals. Phenols are also the main organic constituents present in condensate streams in coal gasification and liquefaction processes. Other sources of wastewater containing phenols are pharmaceutical, plastics, wood products, paint, and pulp and paper industries (Busca et al., 2008).

Wastewater containing phenols need careful treatment before discharging into the water environment. Separation (steam distillation, extraction, adsorption, membrane pervaporation, membrane-based solvent extraction) and destruction (wet air oxidation, oxidation with ozone, oxidation with hydrogen peroxide, supercritical water gasification, biochemical oxidation) methods are the most widely used methods for removing phenol

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and phenolic compounds from wastewater (Abdelwahab et al., 2009; Ahmaruzzaman, 2008; Annadurai et al., 2002; Mohan and Chander, 2001).

In recent years, advanced oxidation processes (electrochemical reaction, non-thermal plasma treatment) have been used for removing phenolic compounds from wastewater. Electrochemical oxidation is one of the most common technique for the treatment of phenol from wastewater, which have easy applicability to automation, high efficiency and environmental compatibility (Sun et al., 2012). This technique is based on the direct oxidation and indirect oxidation. In direct oxidation, the organic pollutants are adsorbed on the electrode surface and then they are oxidized through the direct electron transport reaction. Also, in indirect oxidation, organic pollutants are oxidized by the oxidants such as ozone, hydrogen peroxide, hydroxyl radicals which are produced through the electrochemical reaction (Zhi et al., 2003).

Non-thermal plasma treatment, an advanced oxidation process with potential applications in purification of environmental contaminants, has been widely studied for more than 30 years (An et al., 2011). It is based on the production of several oxidants (ozone, hydroxyl radicals), which has been applied for the removal of various contaminants in air and wastewater (Fan et al., 2009; Hao et al., 2007). Much research also has been processed on the removal of phenol in aqueous solutions using plasma reactors (Faungnawakij et al., 2006; Sato et al., 2008).

In this study, the characteristics of phenol removal and  $UV_{254}$  matters (aromatic intermediates) variation were investigated and compared by the variation of operating factors (NaCl concentration, air flow rate, initial phenol concentration) in electrochemical reaction (ER) and dielectric barrier discharge plasma reaction (DBDPR), respectively. In order to compare the applicability of two reactors in economic aspects, the removals of phenol and the variation of  $UV_{254}$  matters were compared by the variation of electric

power. Also, the production of several oxidants ( $ClO_2$ , free chlorine,  $H_2O_2$ ,  $O_3$ , OH radical) on the phenol treatment process compared.

## 2. Experimental

### 2.1. Experimental setup

Two experimental reactors, electrochemical reaction (ER) and DBD plasma reaction (DBDPR), are shown in Fig. 1 ER used in this study comprised JP 202 (anode) and Pt (cathode) coated titanium mesh electrodes ( $11\text{ cm} \times 6.3\text{ cm}$ ). The electrodes were positioned vertically and parallel to each other with an inter electrode gap of 2 mm. The batch experiments were done using 1.0 L of phenol solution with constant stirring at 150~200 rpm using a magnetic stirrer to maintain uniformity throughout the system. The area of each electrode exposed for the electrolysis was fixed at  $69.3\text{ cm}^2$ . The air diffuser was positioned on the bottom of the reactor.

Total volume of DBDPR which was made by circular tempered glass was 1.0 L. The inner diameter and thickness of quartz tube was 7 mm and 1 mm, respectively. And the titanium ground ( $\Phi 1\text{ mm}$ ) and discharge electrode ( $\Phi 2\text{ mm}$ ) were made to coil and straight type, respectively. Air which was produced by an air pump (YP-15 A) was injected into the quartz tube. Oxygen was supplied from the pure oxygen cylinder. The air and oxygen flow rates were controlled by rotameter and DC high-voltage supply was used to supply power.

### 2.2. Analysis

Measurement of phenol concentration was fulfilled by direct photometric method (Standard Method, 1991) using HACH spectrophotometer (DR 2800). In this study, the  $UV_{254}$  matter refers to all matters having absorbance at 254 nm wavelength. The measurement of  $UV_{254}$  matter was performed using an UV-Vis spectrophotometer (Spectronic, Genesis 5)

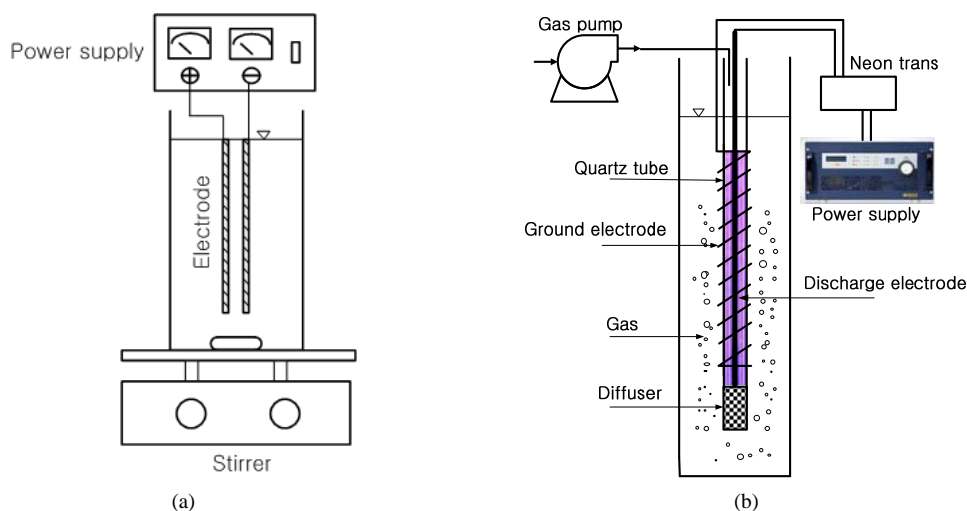


Fig. 1. Schematic diagrams of electrochemical reactor (ER) and dielectric barrier discharge plasma reactor (DBDPR).

at 254 nm wavelength. To measure the production of free radicals formed during phenol treatment, 50 mg/L RNO (*p*-nitrosodimethylaniline) was used because RNO reacts rapidly with hydroxyl radicals selectively (Li et al., 2009). The RNO absorbance was measured by UV-Vis spectrophotometer (Spetronic, Genesis 5) at 440 nm and RNO concentration could be calculated by the calibration curve between RNO absorbance and RNO concentration.  $O_3$  concentration was measured by Indigo methods (Standard Methods, 1991). The measurement of  $H_2O_2$  concentration was done by measuring the absorbance at 596 nm using UV-Vis spectrophotometer after the addition of 1 M NaOH 50  $\mu$ L to sample, and then the  $H_2O_2$  concentration was calculated by the calibration curve between the absorbance and  $H_2O_2$  standard concentration. The concentrations of free chlorine and  $ClO_2$  were measured by DPD (*N,N*-diethyl-*p*-phenylenediamine) method using HACH pocket colorimeter.

Reaction rate constant was calculated using the integral method (Fogler, 1986). The integral method is most commonly used when the reaction order is known and specific reaction rate constants are required. The equation for reaction rate ( $r_A$ ) is in (1).

$$\frac{dC_A}{dt} = r_A = kC_A^n \quad (1)$$

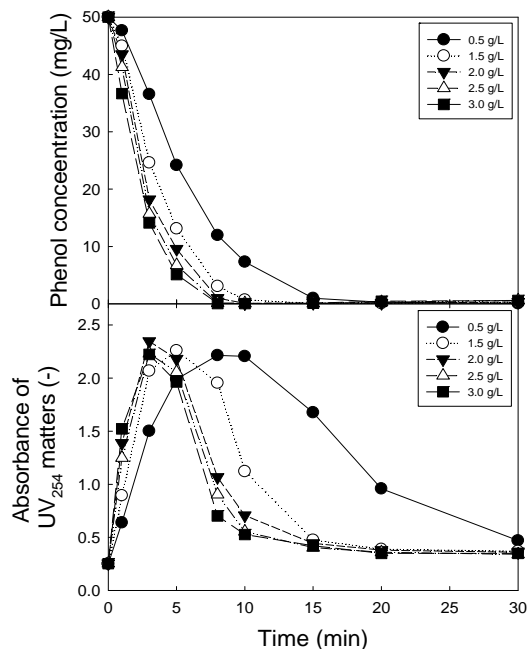
Where,  $t$ ,  $C_A$ ,  $k$ , and  $n$  are time, concentration, the reaction rate constant, and reaction order, respectively.

### 3. Results and discussion

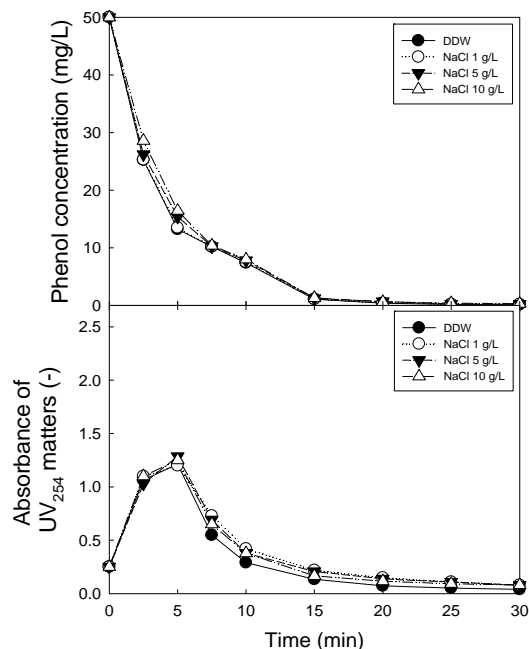
#### 3.1. Effect of NaCl concentration

Effect of NaCl concentration on the removal of phenol and  $UV_{254}$  matters was observed in ER (Fig. 2) and DBDPR (Fig. 3) when the initial phenol concentration was maintained constant as 50 mg/L.

In electrochemical oxidation reactor (Fig. 2), as the progress of electrochemical oxidation, the phenol concentration decreased sharply until 3 min. The phenol removal rate increased according to the increase of NaCl concentration. The phenol removal rate constant and  $UV_{254}$  production rate constant are summarized in Table 1. The phenol removal rate was shown as 1<sup>st</sup> order because the correlation factor ( $r$ ) in 1<sup>st</sup> order reaction was close to 1 comparing that in 2<sup>nd</sup> order. Also,  $UV_{254}$  matters production rate was shown



**Fig. 2.** Effect of NaCl concentration on the removal of phenol and the variation of UV<sub>254</sub> matters in ER.



**Fig. 3.** Effect of NaCl concentration on the removal of phenol and the variation of UV<sub>254</sub> matters in DBDPR.

as 1<sup>st</sup> order reaction. The phenol removal rate constant increased as the increase of NaCl concentration. As NaCl concentration was changed from 0.5 g/L to 1.5 g/L and 2.0 g/L, the phenol removal rate constant increased from 0.2 to 0.42 and 0.64. The phenol removal rate constant did not increase sharply above 2.0 g/L of NaCl concentration. Therefore, the optimum NaCl concentration for phenol removal in this experiment was shown as 2.0 g/L.

During the phenol removal, the absorbance of UV<sub>254</sub> matter increased sharply at the initial time and then declined moderately. It revealed that aromatic intermediates such as quinines were generated by the oxidative degradation of phenol at the initial time and then aromatic intermediates were destructed by the continuous electrochemical oxidation. For oxidation processes, UV<sub>254</sub> absorbance is an easily measured parameter that appears to show consistent correlation

with the degradation of the trace organic compounds (Wert et al., 2009). Therefore, we can see that the degraded phenol was not completely removed but converted to aromatic intermediated at the initial time within 10~15 min. The maximum point of UV<sub>254</sub> absorbance was similar as 2.2~2.3 at all NaCl concentrations because of the same initial phenol concentration of 50 mg/L. However, the time that was required to reach a maximum point was shortened as the increase of NaCl concentration. Therefore, we can see that the phenol degradation rate and aromatic intermediates production rate increased by the increase of NaCl concentration, however, the produced aromatic intermediates amounts was almost constant irrespective of the NaCl concentration because the phenol was completely converted to aromatic intermediate regardless of the variation of NaCl concentration. This result was coincident with

**Table 1.** Effect of NaCl concentration on phenol removal rate constant and UV<sub>254</sub> matters production rate constant in ER

NaCl concentration (g/L)	Phenol				UV <sub>254</sub>			
	1st order		2nd order		1st order		2nd order	
	k (1/min)	r (-)	k (L/mg·min)	r (-)	k (1/min)	r (-)	k (1/min)	r (-)
0.5	0.20	0.99	0.01	0.94	0.25	0.88	0.35	0.77
1.5	0.42	0.98	0.12	0.80	0.42	0.89	0.60	0.78
2.0	0.64	0.96	1.22	0.72	0.67	0.88	1.02	0.80
2.5	0.70	0.98	1.19	0.88	0.66	0.90	1.02	0.81
3.0	0.79	0.97	2.67	0.9	0.65	0.85	1.02	0.79

k: reaction rate constant

r: correlation factor

the produced oxidants (O<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>, ClO<sub>2</sub>, free chlorine) concentration at the peak time using our previous experimental results in electrochemical oxidation. The concentration of oxidants at peak time was similar in all NaCl concentrations (Yoo and Kim, 2011). Also, the UV<sub>254</sub> matter destruction rate after peak time was proportional to the UV<sub>254</sub> matter production rate.

In DBDPR (Fig. 3), the variation of NaCl concentration had no effect on the degradation of phenol and UV<sub>254</sub> matters. The phenol removal rate was shown as 1st order which was equal to the reaction order in electrochemical oxidation reactor. The phenol removal rate constant was almost constant as 0.231~0.237 1/min regardless of the variation of NaCl concentration, however, it was lower than the rate constant of 0.42 1/min at 1.5 g/L NaCl concentration in ER (Fig. 2). Similar to the case of ER, during the phenol removal, the absorbance of UV<sub>254</sub> matter increased sharply at first and then declined. The UV<sub>254</sub> production rate was almost same as 0.314~0.326 1/min irrespective of the NaCl concentration. However, the peak value in absorbance of UV<sub>254</sub> was almost 1.2 which was much lower than 2.2 in ER. This means that phenol could not easily converted to the aromatic intermediates in DBDPR

comparing ER because the produced oxidants amounts in DBDPR was lower than that in ER at this experimental condition (Kim and Park, 2012; Yoo and Kim, 2011).

### 3.2. Effect of air flow rate

Effects of air flow rate on the variation of phenol and UV<sub>254</sub> matters in ER and DBDPR are shown in Fig. 4 and Fig. 5.

At constant current density of 50.5 mA/cm<sup>2</sup>, NaCl concentration 2.0 g/L in ER, phenol concentration and absorbance of UV<sub>254</sub> were observed by the variation of air flow rate from 0 L/min to 4 L/min (Fig. 4). The phenol degradation rate constant at 0 L/min air flow rate was 0.35 1/min, however, it was almost constant as 0.50~0.56 1/min above 0 L/min air flow rate. And the UV<sub>254</sub> matters production rate constant at 0 L/min was 0.60 1/min, and it was almost constant as 0.64~0.65 1/min from 1 L/min to 4 L/min air flow rate. Therefore, we can see that air flow rate had a little effect on the removal of phenol and the variation of UV<sub>254</sub> matters comparing the results between 0 L/min and 1 L/min. However, we also see that air flow rate had no effect above 1 L/min.

The result was consistent with the report from

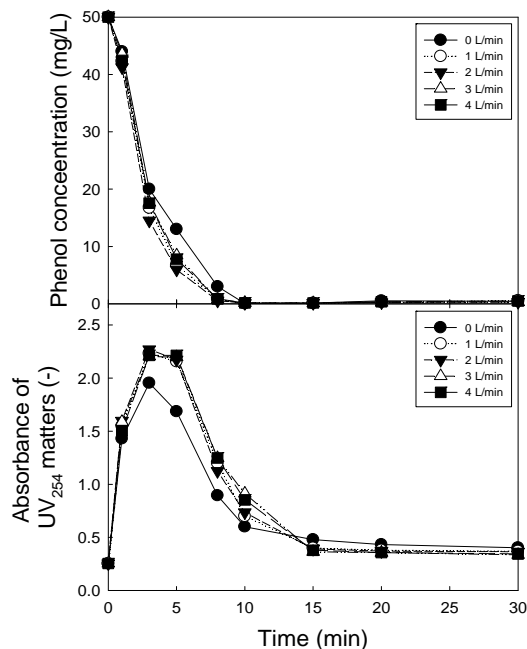


Fig. 4. Effect of air flow rate on the removal of phenol and the variation of UV<sub>254</sub> matters in ER.

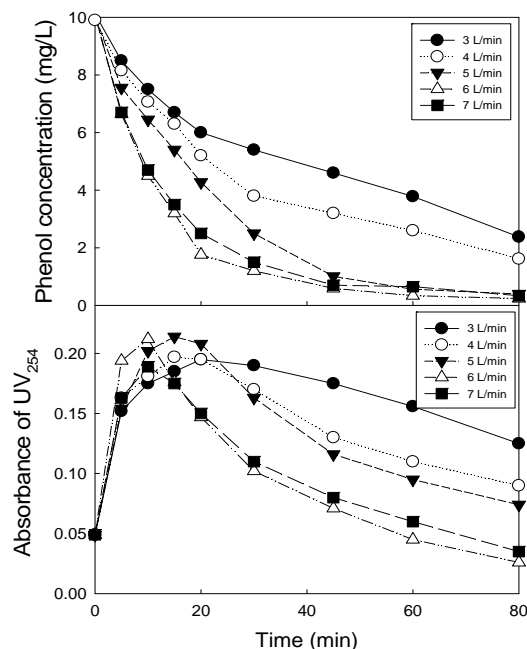


Fig. 5. Effect of air flow rate on the removal of phenol and the variation of UV<sub>254</sub> matters in DBDPR.

Wang et al.(2008). They reported that the color removal by electro-Fenton technology increased for the oxygen flow rates of 0.1~0.4 L/min, however, the removal efficiency remained almost constant at the oxygen flow of 0.3~0.4 L/min.

In DBDPR (Fig. 5), the phenol removal rate increased by the increase of air flow rate. The effect of air flow rate on phenol removal in ER was not great, however, that in DBDPR was not negligible because air has a great effect on the production of plasma by the electric barrier. The phenol removal rate was almost constant above the air flow rate 6 L/min. Therefore, we can see that the optimum air flow rate in this experiment was 6 L/min. In spite of the low initial phenol concentration of 10 mg/L, the phenol removal efficiency in DBDPR was very low as 26%~53% at 10 min comparing 99% at the initial phenol concentration of 50 mg/L in ER.

And the UV<sub>254</sub> matter production rate and removal rate increased as the increase of air flow rate from 3 L/min to 6 L/min. Above 6 L/min, the UV<sub>254</sub> matter production rate and removal rate was almost constant. The peak value of UV<sub>254</sub> absorbance at oxygen flow rate of 3 L/min was lower than that at 5 L/min, which means the produced aromatic intermediates was low because of the low production of oxidant by the low oxygen supply.

In DBDPR, the air was changed to oxygen as electric barrier in order to improve the phenol removal efficiency. Similar to the case of air, the phenol removal rate increased by the increase of oxygen flow rate from 2 L/min to 7 L/min. It was almost constant at oxygen flow rate of 6 L/min and 7 L/min. Therefore, the optimum oxygen flow rate in this experiment was considered as 6 L/min. However, the phenol removal efficiency was high as 63%~87%

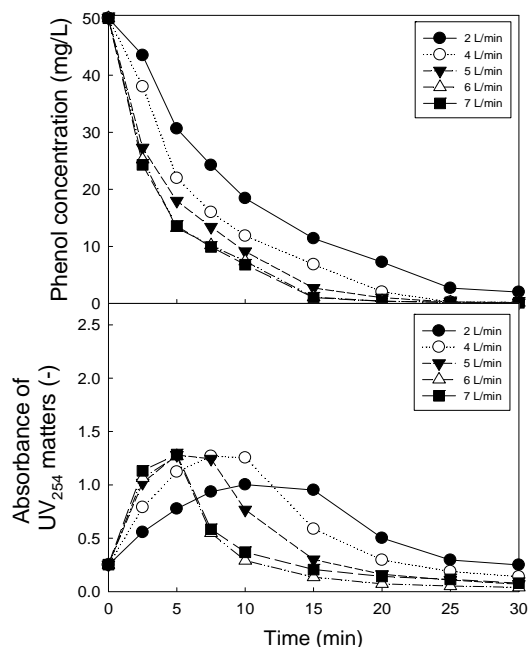


Fig. 6. Effect of oxygen flow rate on the removal of phenol and the variation of  $UV_{254}$  matters in DBDPR.

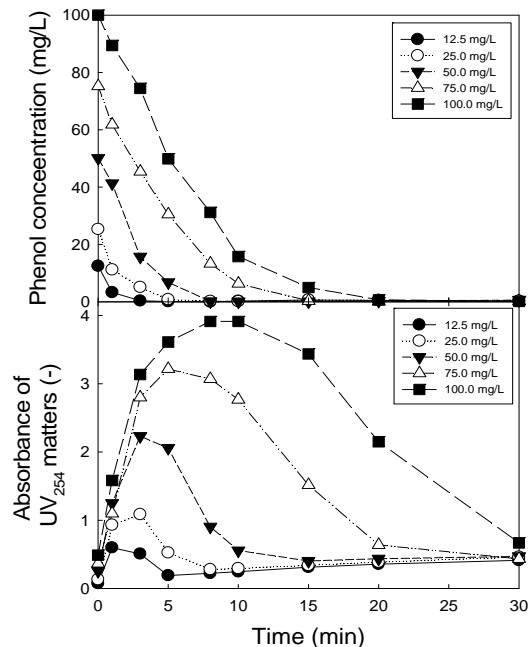


Fig. 7. Effect of initial phenol concentration on the removal of phenol and the variation of  $UV_{254}$  matters in ER.

comparing the case using air. Chen et al.(2004) have compared the removal efficiency of phenol using oxygen and nitrogen bubbling in pulsed high-voltage discharge plasma which was similar to DBDPR, and the removal efficiencies of phenol using oxygen was higher than that using nitrogen. They concluded that ozone and additional active species such as OH, O and H radicals could be produced when oxygen was used. The ozone formed in the discharge region may react with phenol, or it may decompose to form secondary oxidants such as OH radicals. Air is composed of 21% oxygen and 79% nitrogen. Therefore, the removal efficiency of phenol was low using air because of the low composition of oxygen and high composition of nitrogen in air.

The peak value of  $UV_{254}$  absorbance at oxygen flow rate of 2 L/min was lower than that at 4 L/min, which means that the produced aromatic intermediates

was low because of the low production of oxidant by the low oxygen supply. The peak value was almost constant above 4 L/min. However, the  $UV_{254}$  matter production rate and removal rate increased as the increase of oxygen flow rate from 4 L/min to 6 L/min. Above 6 L/min, the  $UV_{254}$  matter production rate and removal rate was almost constant. According to these results (phenol concentration, absorbance of  $UV_{254}$ ) in this DBDPR experiment, we can see that the optimum oxygen flow rate for the complete removal of phenol was 6 L/min. Comparing these results to ER, the phenol removal and  $UV_{254}$  production rates and peak value of  $UV_{254}$  matters in DBDPR were lower than ER.

### 3.3. Effect of initial phenol concentration

The variation of phenol concentration and absorbance of  $UV_{254}$  in ER (Fig. 7) and DBDPR (Fig. 8), respectively, were observed according to the variation

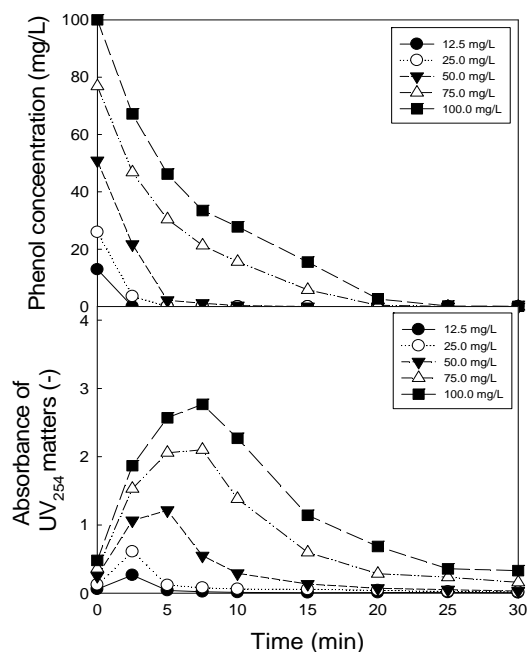


Fig. 8. Effect of initial phenol concentration on the removal of phenol and the variation of  $UV_{254}$  matters in DBDPR.

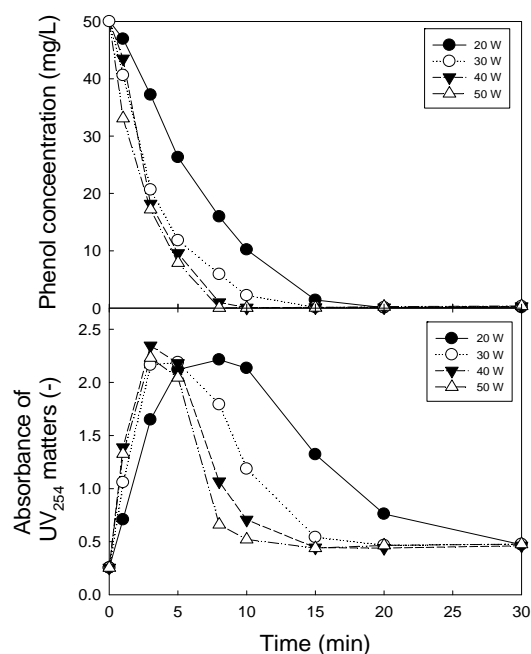


Fig. 9. Effect of electric power on the removal of phenol and the variation of  $UV_{254}$  matters in ER.

of initial phenol concentration from 12.5 mg/L to 100 mg/L in order to know the effect of the initial phenol concentration on the phenol and aromatic intermediates ( $UV_{254}$  matters). In ER, phenol concentration decreased rapidly at all initial phenol concentration; however, the time required to reach 95% removal was lengthened as 3 min, 5 min, 8 min, 15 min, and 20 min as the increase of initial phenol concentration. The peak values of  $UV_{254}$  absorbance and the times required to reach a peak point were increased as the increase of initial phenol concentration. This means that the production of aromatic intermediates increased by the degradation of phenol as the increase of initial phenol concentration.

In DBDPR, the trends of phenol removal and  $UV_{254}$  absorbance were similar to ER. However, the time required to reach the 95% removal efficiency was shown as 3 min, 5 min, 10 min, 20 min, 25 min by the increase of initial phenol concentration from

12.5 mg/L to 100.0 mg/L, which means that the phenol removal rate was a little lower than that of ER. And the peak values of  $UV_{254}$  absorbance were lower than that of ER at the same experimental condition. Therefore, the production of aromatic intermediates in DBDPR was lower than that in ER and the complete degradation of phenol could be easily done in ER than in DBDPR.

#### 3.4. Effect of electric power

Effects of electric power on the variation of phenol concentration and  $UV_{254}$  absorbance in ER and DBDPR are shown in Fig. 9 and Fig. 10.

The electric powers in ER and DBDPR were controlled from 20 W to 50 W. In both reactors, the phenol removal was increased as the increase of electric power. In ER, phenol was almost removed within 20 min. However, it was taken about 30 min in DBDPR. And phenol removal rate constants in ER and DBDPR increased as the increase of electric



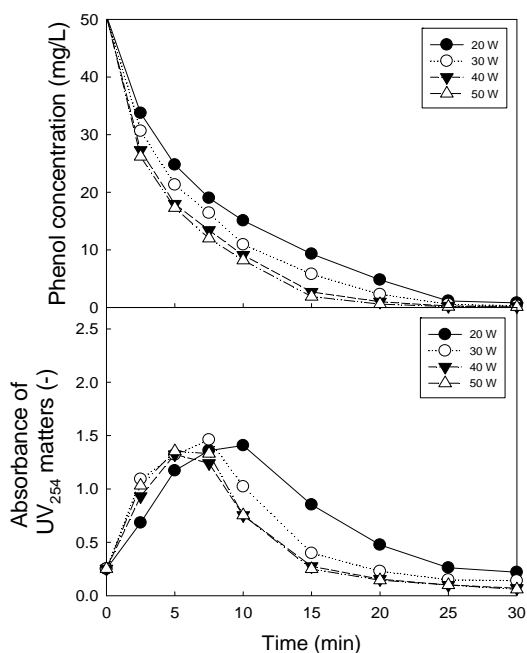


Fig. 10. Effect of electric power on the removal of phenol and the variation of UV<sub>254</sub> matters in ER.

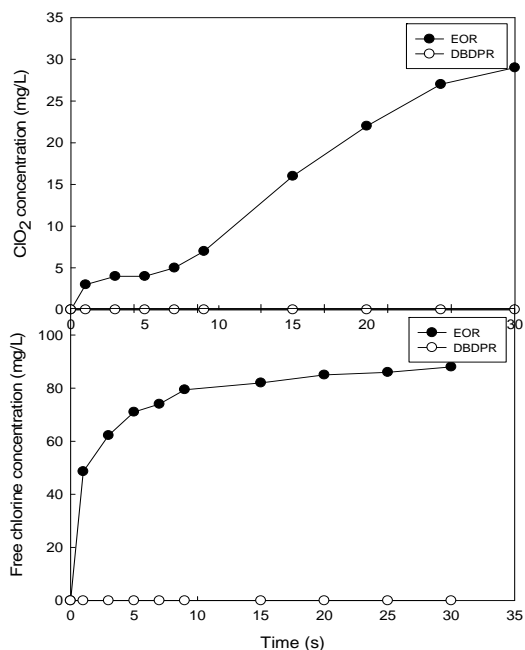


Fig. 11. Production of ClO<sub>2</sub> and free chlorine in ER and DBDPR.

power (Table 2 and 3). Phenol removal rate constants in ER were higher than those in DBDPR at the same electric power condition. Therefore, we can see that the phenol removal in ER is a little more active than that in DBDPR.

Several oxidants which were produced at the same electric power condition in ER and DBDPR were

observed and compared in order to know the effect of electric power on phenol removal. In ER, the chlorine oxidants (free chlorine, ClO<sub>2</sub>) were produced during the electrochemical oxidation reaction and the maximum concentration of free chlorine and ClO<sub>2</sub> at 30 min was 85 mg/L and 30 mg/L, respectively (Fig. 11). However, the chlorine oxidants were not detected

Table 2. Effect of electric power on phenol removal rate constant, UV<sub>254</sub> matters production rate constant and UV<sub>254</sub> matters removal rate constant in ER

Electric power (W)	Phenol removal		UV <sub>254</sub> matters production		UV <sub>254</sub> matters removal	
	k (1/min)	r	k (1/min)	r	k (1/min)	r
20	0.23	0.97	0.25	0.86	0.102	0.99
30	0.30	0.99	0.66	0.93	0.110	0.99
40	0.35	0.99	0.67	0.89	0.140	0.94
50	0.36	0.99	0.66	0.89	0.163	0.94

k: rate constant  
r: correlation factor

**Table 3.** Effect of electric power on phenol removal rate constant, UV<sub>254</sub> matters production rate constant and UV<sub>254</sub> matters removal rate constant in DBDPR

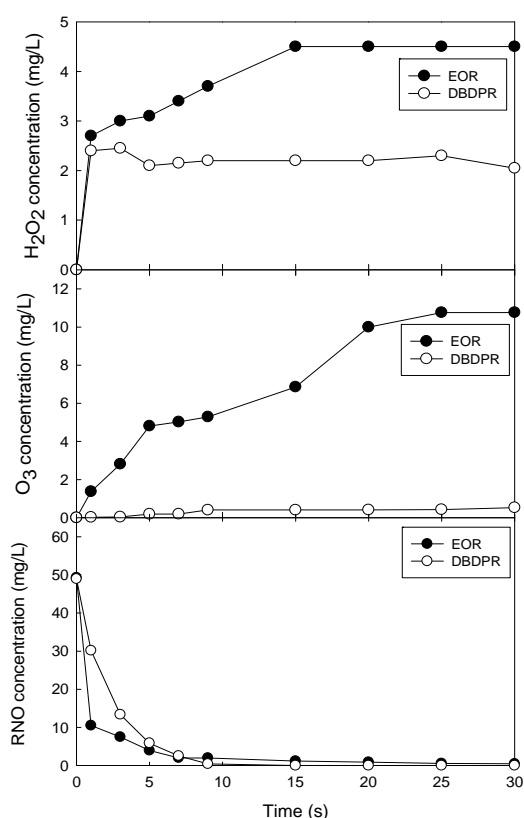
Electric power (W)	Phenol removal		UV <sub>254</sub> matters production		UV <sub>254</sub> matters removal	
	k (1/min)	r	k (1/min)	r	k (1/min)	r
20	0.136	0.98	0.165	0.90	0.097	0.99
30	0.147	0.99	0.218	0.86	0.110	0.97
40	0.184	0.99	0.331	0.91	0.122	0.98
50	0.197	0.99	0.331	0.95	0.127	0.98

k: rate constant

r: correlation factor

in DBDPR.

H<sub>2</sub>O<sub>2</sub> and O<sub>3</sub> concentration increased, and RNO concentration decreased as the progress of reaction

**Fig. 12.** Production of H<sub>2</sub>O<sub>2</sub> and O<sub>3</sub>, and removal of RNO in ER and DBDPR.

both in ER and DBDPR (Fig. 12). H<sub>2</sub>O<sub>2</sub> concentration in ER was two times higher than that in DBDPR at 30 min. O<sub>3</sub> was detected over 10 mg/L at 30 min in ER because of the high O<sub>3</sub> solubility by the micro-bubble size of O<sub>3</sub>, however, O<sub>3</sub> concentration in DBDPR was very low below 1 mg/L because of the low O<sub>3</sub> solubility by the large bubble size of produced O<sub>3</sub>. RNO (*p*-nitrosodimethylaniline) was used to measure the production of free radicals because RNO reacts rapidly with hydroxyl radicals selectively (Li et al., 2009). RNO degraded rapidly both in ER and DBDPR. The RNO degradation rate constants in ER was shown as 0.437 1/min (r = 0.98), which was a little lower than 0.495 1/min (r = 0.99) in DBDPR. This result showed that the production of OH radicals in DBDPR was a little higher than that in ER.

Therefore, we can see that the phenol removal rate in DBDPR was lower than that in ER because of the low production of chlorine oxidants and O<sub>3</sub> in DBDPR.

#### 4. Conclusion

Effects of four operating factors (NaCl concentration, air flow rate, initial phenol concentration, electric power) on the phenol removal in electrochemical reaction (ER) and dielectric barrier discharge plasma reaction (DBDPR) were investigated to compare the

characteristics of two reactors. The phenol removal rate was shown as 1st order both in ER and DBDPR. During the phenol removal, the absorbance of UV<sub>254</sub> matters which means aromatic intermediates increased sharply at the initial time and then declined moderately. In ER, the phenol degradation rate and aromatic intermediates production rate increased by the increase of NaCl concentration. However, in DBDPR, the variation of NaCl concentration had no effect on the degradation of phenol and UV<sub>254</sub> matters. Air flow rate had a little effect on the removal of phenol and the variation of UV<sub>254</sub> matters in ER, however, the phenol removal rate increased by the increase of oxygen flow rate in DBDPR. At the initial phenol concentrations, the phenol removal rate in ER was a little higher than that in DBDPR and the peak values of UV<sub>254</sub> absorbance in ER were also higher than those in ER. The phenol removal rate in ER was a little higher than that in DBDPR. After the investigation of several oxidants, the production amounts in ER were higher than those in DBDPR and the produced O<sub>3</sub> amounts in ER was 10 times higher than that in DBDPR. However, the chlorine intermediates which cause several pollution problems were not produced in DBDPR.

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