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Organic pollutants treatment in a water aerosol reactor by pulsed corona discharge at atmospheric pressure

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Abstract. An atomization reactor with a gas cyclor is designed, and the phenol is treated as wastewater by pulse corona discharge, which is sprayed into the reactor through a pneumatic atomization nozzle. To fully utilize the ozone and enhance organic pollutant removal, we maintained continuous gas circulation in the reactor. The resulting phenol solution was subjected to six passes of decomposition treatment. Approximately 87.4% of 100 mg/L phenol concentration was decomposed in the plasma system gas circulation, whereas only 51.9% was decomposed in the same system but without gas circulation after the sixth pass treatment. The decomposition efficiency increased with applied voltage and pH. In addition, homogeneous catalysts such as hydrogen peroxide (H₂O₂) and iron (II) (Fe²⁺) enhanced the removal of phenol. The novel reactor was also used for the deep treatment of phenolic compounds in coking wastewater. Compared with systems using ozonation, the plasma system working under optimal conditions considerably improved the performance of industrial wastewater treatment, which can be regarded as a promising alternative AOP for wastewater remediation.

1. Introduction

The non-thermal plasma (NTP) method effectively produces UV radiation and active chemicals (•OH, H₂O₂, and O₃), which are important to the removal of organic compounds. The NTP technology is an effective and environment-friendly process for pollutant decomposition [1-5]. An increasing number of investigations have focused on improving the efficiencies of electrode configurations and reactors in NTP processes for water treatment. The reaction system can be divided into three main categories according to the discharge characteristics of the gas and liquid phases: discharge reactors for the continuous liquid and dispersed gas phase, those for the continuous gas and dispersed liquid phase, and those for the continuous liquid and gas phases [6]. Typically, the most efficient pulsed-powered reactors for H₂O₂ formation and chemical oxidation in solutions are the discharges formed in the gas phase, and waste solutions are sprayed into the gas [7-10]. The liquid spray expands the contact surface area with plasma in the discharge zone area. With a large surface-to-volume ratio, the reactive substance can diffuse to the sprayed liquid surface over a short distance. Pollutant molecules directly react with O₃ in the plasma zone and with oxidative radicals.



Minamitani et al. [11, 12] studied dye decomposition by spraying water solution into the pulsed-corona discharge reactor of coaxial electrodes. The results showed that spraying water droplets into the gas-phase discharge system can increase the rate and efficiency of water treatment. Ajo [13, 14] designed a reactor with a perforated bottom plate, which can spray wastewater in the discharge system. The treated solutions were showered in jets, droplets and films between the high voltage wires and the grounded plate electrodes through the plasma zone. Chen et al. [15] established a plasma processing system by using DBD arrays in water mist spray. Within 35 min treatment, phenol concentration decreased from 100 mg/L to 9.8 mg/L. Grabowski et al. [16] found that 50% phenol degradation can be reached in 8 min by using a thin layer of water and in less than 30 s by spraying water in a pulsed batch reactor. However, to the best of our knowledge, only a few works have taken full advantage of the exhaust gas discharged from the plasma reactor. Pokryvailo et al. [17] added a compressor to extract air from the reactor exhaust for liquid treatment. The efficiency of circulating the exhaust air into the pulsed corona discharge on organic compounds degradation has not been investigated.

Based on the device in literature [11], a new type of plasma reactor was proposed. This reactor uses a wire-cylinder configuration with a gas circulation system, thereby increasing O_3 utilization in air exhaust, reducing the pollution of O_3 gas, and enhancing corona discharge efficiency in wastewater treatment. This experiment investigated the effects of parameters such as discharge voltage, initial pH and catalyst addition on the degradation of pollutants. These operating parameters were optimized so that the feasibility of degrading phenol water with the novel reactor can be demonstrated. Furthermore, the novel reactor was also used for the deep treatment of low concentration pollutants in coking wastewater samples. The comparison of the operating costs of the plasma and O_3 oxidation method was studied. This work focuses on the optimization of the corona discharge process, which provides a reference for later practical applications. It is expected to provide a new alternative for coking wastewater and pretreatment method in future commercial applications.

2. Materials and methods

2.1. Experimental device

The reaction system mainly includes an ns-pulsed power supply, a water aerosol reactor and gas lines, as shown in Figure 1. The reactor employed for this study was developed from the one previously described in Ref. [11]. It consists of a stainless steel cylindrical vessel, a stainless steel wire, springs and porcelain insulators. The vessel placed horizontally was 1,525-mm long with an inner diameter of 89 mm, which acts as a grounded electrode. The stainless-steel wire was used as a high voltage electrode with 0.35 mm diameter along the axis of the vessel that was used as grounded electrode. One end of the cylinder was installed with an atomizing nozzle and a vent of 80 mm diameter. The other end was installed with a vent of 50 mm diameter. The two vents were connected to an air fan by the plastic pipes.

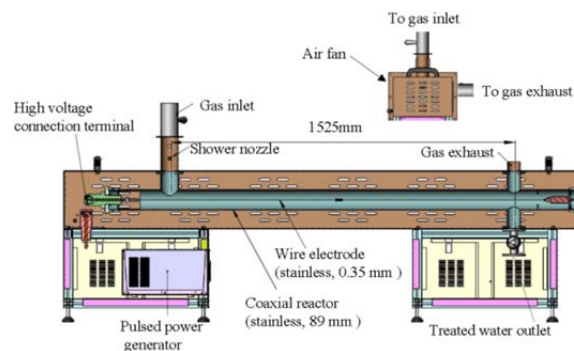


Figure 1. Plasma discharge experimental device diagram.

A positive polarity discharge was generated by increasing the voltage between the high voltage electrode and the ground electrode. Gas-liquid mixture was sprayed directly into the reactor by the

nozzle at a rate of 150 mL/min. The air fan continuously circulated the gas obtained from the air exhaust of the reactor in an almost closed loop to fully use the O₃ produced by NTP. A container located below the reactor collected the treated solutions. After treatment, the collected solutions were sprayed into the reactor and treated by plasma again. Solutions were exposed to the plasma six times in a batch plasma treatment experiment. Voltage probe (Tektronix P6015A) was used to adjust the applied voltage of 20-35kV and digital oscilloscope was used for real-time monitoring (Tektronix TDS3014, 100 MHz, 1.25 GS/s). The discharge was excited using a monopole pulsed power supply with rise time of 150 ns and pulse width of 350 ns.

2.2. Materials and analytical methods

A batch of plasma treatment experiments was conducted using 1 L phenol solution of 100 mg/L initial concentration. The pH and conductivity of the 100 mg/L phenol solution were 6.30 and 15 µs/cm, respectively. The chemical oxygen demand (COD) was approximately 244.6 mg/L. Phenol decomposition was determined by measuring the phenol conversion as a function of passes through the reactor numbers at different experimental parameters. The concentration of phenol in water was monitored by the 4-aminoantipyrene method [18]. The minimum detectable amount of phenol was 0.1 mg/L. The intermediates and final products of phenol were determined by GC/MS (GC-TOF, Micromass). Based on the potassium dichromate oxidation method, the COD value in the solution was determined [18]. The pH value and conductivity of the solution were measured by a pHS-3C pH meter (Shanghai Precision & Scientific Instrument Co.Ltd.) and a DDS-11D conductivity meter (Shanghai Precision & Scientific Instrument Co.Ltd.), respectively. The O₃ content in the solution was determined by the indigo method [19]. The concentration of dissolved H₂O₂ was monitored by the potassium titanium (IV) oxalate method using a UV-vis 2450 spectrophotometer (SHIMADZU Co.Ltd., Japan) [20].

The removal efficiency of phenol is calculated as follows:

$$\eta\% = \frac{C_0 - C}{C_0} \times 100\% \quad (1)$$

where C_0 and C are the initial and final concentration of the phenol in solution, respectively.

Electric energy per mass (E_{EM}) is the electric energy in kilowatt-hours [kWh] required to bring about the degradation of a unit mass of a contaminant C in solution. The E_{EM} value [kWh/kg] was calculated by using the following formula [21, 22]:

$$E_{EM} = \frac{P \cdot t \cdot 10^6}{C_0 \cdot R \cdot V} \quad (2)$$

where C_0 is the initial concentration of the organic compound (mg/L), V is the solution volume (L), R is the decomposition efficiency (%), P is the rated power (kW) and t is the treatment time (h).

3. Results and discussion

3.1. Phenol decomposition in the novel reactor

Figure 2 shows the decomposition rate of the 100 mg/L phenol solution in three modes at an applied voltage of 30 kV with a discharge frequency of 600 Hz. Due to the hydrodynamic cavitation volatility of phenol, approximately 5.0%-10.4% phenol was removed through the nozzle from the 1st-6th passage. In the plasma treatment process of the gas circulation system, the phenol concentration decreased rapidly and gradually with cyclic treatment number in this case. After the 6th plasma treatment with gas circulation, 87.4% phenol was decomposed and 65.2% COD was removed, which is higher compared without gas circulation with 51.9% phenol decomposition and 35.6% COD removal.

Discharge at the gas-liquid interface at atmospheric pressure will cause many chemical reactions, which can increase the production of active free radicals [23]. It is a great source of active free radicals which can degrade most organic pollutants. The rate constants for the reactions of O₃, •OH, and •H with phenol are 1.3×10³ L/(mol·s), 6.6×10⁹ L/(mol·s), and 1.7×10⁹ L/(mol·s), respectively [24,25].

The high-energy electrons, hydroxyl radicals, O_3 and other reactive species formed during discharge can degrade phenol in water. The O_3 from the gas exhaust of the vessel also plays an important role on phenol decomposition. Wen [26] found that the use of pulsed corona discharge combined with O_3 led to the high degradation rate of 4-CP (96% within 30 min) and COD removal. Therefore, the obvious synergistic effect is attributed to the enhanced O_3 decomposition by electron collisions and photolysis in the plasma region.

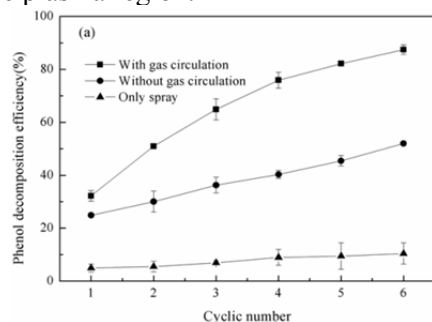


Figure 2(a). Phenol decomposition in three modes.

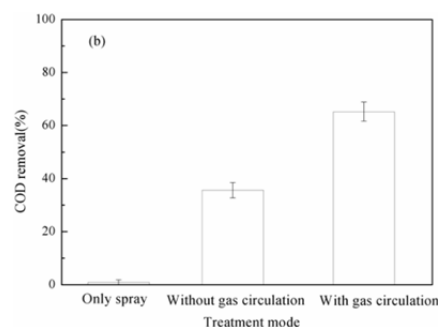


Figure 2(b). COD removal in three modes.

3.2. Dissolved O_3 and H_2O_2 concentration

O_3 ($E^0 = 2.07$ V/SHE) and H_2O_2 ($E^0 = 1.77$ V/SHE) generated during electrical discharge are powerful oxidants and are relatively stable in aqueous solution. To study the importance of oxidizing substances on phenol degradation, the aqueous concentrations of O_3 and H_2O_2 in pure water and phenol solution were analyzed. Figure 3 shows that the O_3 concentrations in the two solutions increase with cyclic numbers following a trend that is almost similar to that of H_2O_2 concentrations. Due to the consumption of O_3 and H_2O_2 by reacting with phenol, the concentrations of those reactive species are lower in phenol solution than in pure water. During the high-voltage pulsed discharge of air, the second positive system of nitrogen in the wavelength range of 290-420 nm emits and high-energy electrons generate [27]. They can decompose O_3 to generate oxygen atoms and O_2 [28]. Then excited oxygen reacts with H_2O to produce $\cdot OH$ radicals [29]. In addition, O_3 generated in the gas phase is hydrolyzed in the aqueous solution, and other reactive oxygen species (such as $\cdot O_2^-$, $\cdot OH$) are also generated [30-31].

The direct reactions of H_2O_2 with organic pollutants are generally slow. O_3 is an essential oxide which plays an important role in the oxidation of phenol. Therefore, with increasing treatment time increasing, O_3 production decreases much more than H_2O_2 production in the phenol solution.

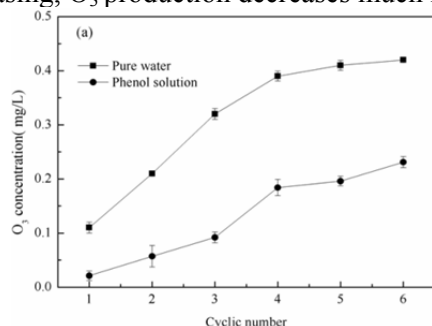


Figure 3(a). Concentrations of O_3 in the pure water and phenol solution during discharge.

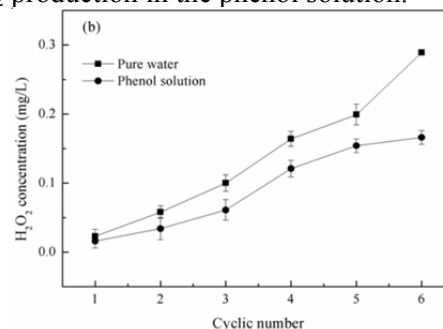


Figure 3(b). Concentrations of H_2O_2 in the pure water and phenol solution during discharge.

3.3. Byproducts and changes in pH, and conductivity of the treated solution

Phenol solution shifted from colorless to faint yellow and then diminished during treatment with gas circulation, indicating the formation of other intermediate products. The pollutants in the original

phenol solution (Figure 4a) and the solution after the second treatments (Figure 4b) were analyzed by GC/MS. The main intermediates during treatment process were benzoquinone, toluene, p-xylene, 3-hydroxyl 1-butyl propionate and other unidentified trace products. The main reaction of phenol decomposition involved $\bullet\text{OH}$ radical attacking its aromatic ring. Subsequently, benzoquinone, hydroquinone and catechol were formed. During further oxidation by active species (such as $\bullet\text{OH}$), and ring cleavage occurred. Finally, carbonic acids were formed. Deep oxidation leads to the mineralization of these substances to CO_2 and H_2O .

Figure 5 shows the pH value and conductivity of the solution monitored during phenol decomposition. The solution pH decreased from 6.27 to 3.15 after the 1st treatment and to 2.32 after the 6th treatment. The variation in pH value indicated that the amount of carbonic and carboxylic acids in the solution increased with increasing treatment time. Conductivity growth is caused by the electrolyzable byproducts of phenol degradation formed from phenol degradation. During discharge, the excitation, dissociation and ionization of N_2 and O_2 in air promote the formation of HNO_3 [32, 33]. It also caused variation in conductivity and pH.

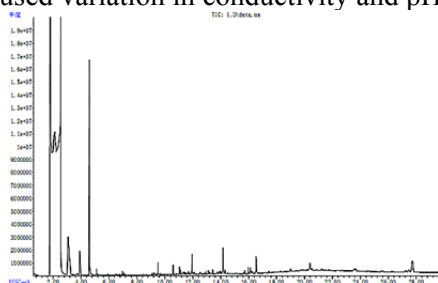


Figure 4(a). GC-MS of the original phenol solution.

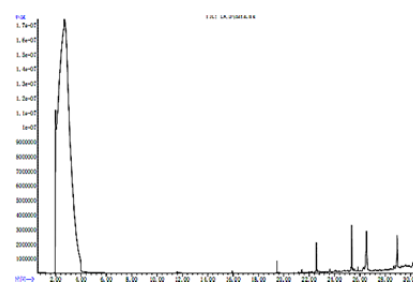


Figure 4(b). GC-MS of phenol after two reactions.

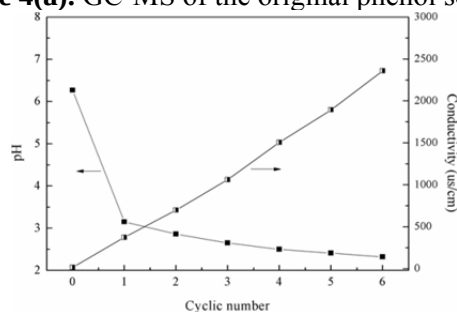


Figure 5. The variation of pH and conductivity of the phenol solutions with gas circulation system.

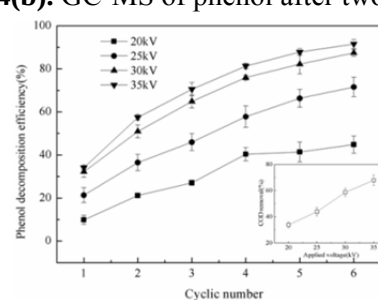


Figure 6. Effect of applied voltage on the phenol decomposition and COD removal.

3.4. Effect of applied voltage

The effects of applied voltage variation on phenol decomposition efficiency were investigated. Figure 6 shows that the decomposition rate of phenol increased with the increase of applied voltage at the same discharge frequency. The maximum phenol degradation rate reached at 35 kV from 34.0% to 91.5% from the 1st to 6th treatment. The COD removal reached 67.9% at a charging voltage of 35 kV after six times of plasma treatment. In fact, the increase of the applied voltage caused the increase of the discharge power, which made the physicochemical reactions in the reaction process more severe and indirectly enhanced the production of active free radicals in the water. The applied voltage played an important role on the oxidation of phenol.

3.5. Effect of initial pH

The initial pH of the solution is an important factor that influences the decontamination of organic compounds. H_2SO_4 (1.0 mmol/L) or NaOH (1.0 mmol/L) was used to adjust the initial pH of the phenol solution to the desired levels. Figure 7 shows that with increasing pH, the removal rates of phenol increase at 30 kV and 600 Hz. The phenol removal efficiencies in alkaline conditions were

higher than that in acidic solutions. At pH 9 and 11.0, more than 90.0% phenol was decomposed after the 2nd treatment, and the overall decontaminations were almost equal. Similar to the results of phenol removal, the COD removal are higher in alkaline conditions than in acidic conditions. COD removals were 69.0% at pH 9 and 82.3% at pH 11, respectively.

The results were similar to the conclusions reported in other literature [34]. As the pH of the solution gradually increased, O_3 would decompose faster, and the yield of $\bullet OH$ would increase [35]. The existence of OH^- species enhanced the decomposition of O_3 into the hydroxyl radical. Although $\bullet OH$ can participate in a side reaction, which transforms OH^- to its less reactive species (e.g. HO_2 , O_2^-) [36]. But the influence of this transformation was eliminated by O_3 decomposition during the treatment stage for high alkaline solution. In addition, many H^+ in the acidic solution may reduce the effective role of high-energy electrons. Few activated species such as HO_2 and O_2 are formed, and the formation of $\bullet OH$, $\bullet H$, and O_3 was reduced. Therefore, the decomposition of phenol in alkaline solutions was higher than that in acidic ones.

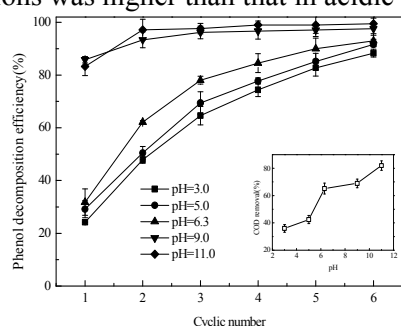


Figure 7. Effect of initial pH on the phenol decomposition and COD removal.

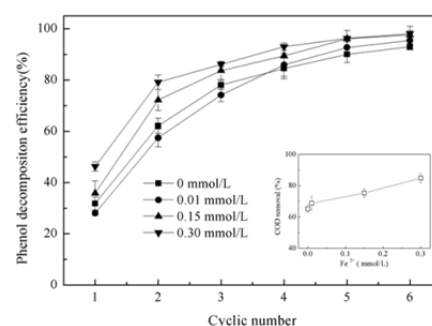


Figure 8. Effect of Fe^{2+} on the phenol decomposition and COD removal.

3.6. Effects of ferrous ions and H_2O_2 addition

In order to make full use of the physicochemical effects of the plasma discharge process, a combination of pulsed discharge and catalysts was proved effective. The combination was applied in several commercialized products [37]. Ferrous salt was added to facilitate the Fenton reaction in plasma gas-liquid reactors as studied in literature. To determine the influence of ferrous ions on decomposition efficiency, different $FeSO_4 \cdot 7H_2O$ dosages (calculated by Fe^{2+} concentration) were added to phenol solutions before passing through the plasma region. Figure 8 shows that the changes in the removal efficiency with treatment times for Fe^{2+} addition at 30 kV and 600 Hz. The degree of decomposition and degradation were improved in the presence of different Fe^{2+} dosages. Treatment with 0.3 mmol/L Fe^{2+} improved the phenol decomposition rate from 93.0% for pure phenol solution to 98.1%, and the COD removal rate from 65.8% to 84.8% at the 6th treatment. Experiments with Fe^{2+} showed the influence on the phenol removal for the concentration of H_2O_2 produced during the plasma discharge process, which implied that the Fenton reaction can occur without adding H_2O_2 .

To determine the influence of H_2O_2 addition on phenol removal, different volume concentrations of 30% *m/m* H_2O_2 (0.1‰, 0.3‰, 0.9‰) were added to the solution. Phenol degradation can be accelerated in the presence of H_2O_2 , as shown in Figure 9. The removal of phenol increased with the addition of H_2O_2 . Phenol removal rate was 64.8% with the addition of 0.9‰ H_2O_2 at the 1st treatment, which was more effective than 31.8% in plasma treatment alone. The results show that H_2O_2 can initiate a variety of free radical reactions and promote the decomposition of phenol. It is attributed to the more efficient $\bullet OH$ generation in the water by adding the proper concentration of H_2O_2 [10, 38].

3.7. Treatment of coking wastewater

Phenolic compounds are the main organic pollutants in coking wastewater, accounting for about 80% of COD [39-41]. To investigate the oxidizing capability of the reactor with gas circulation, a kind of refractory and toxic coking wastewater (phenols 35 mg/L, COD 250 mg/L) was examined.

Figure 10 shows that the composition of phenolic compounds was 92.0%, and COD removal reached 38.6% at the 6th treatment. For refractory organic compounds in the coking wastewater, COD degradation rate was low. With 9‰ H_2O_2 addition, 89.8% of phenolic compounds decomposed at the 2nd treatment and 99.9% at 6th treatment, which was almost equal with the addition of 0.3 mmol/L Fe^{2+} . COD removal with 0.9‰ H_2O_2 addition reached 61.3%, which is 22.7% higher under the addition of NTP during the 6th pass. In the NTP/ Fe^{2+} treatment, COD removal reached 66.1%. The addition of H_2O_2 and Fe^{2+} played an important role in accelerating organic matter degradation and thoroughly oxidizing organic compounds.

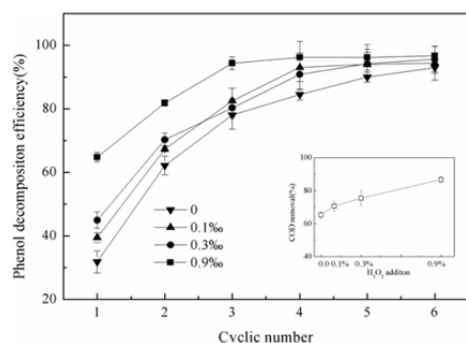


Figure 9. Effect of H_2O_2 addition on the phenol decomposition and COD removal.

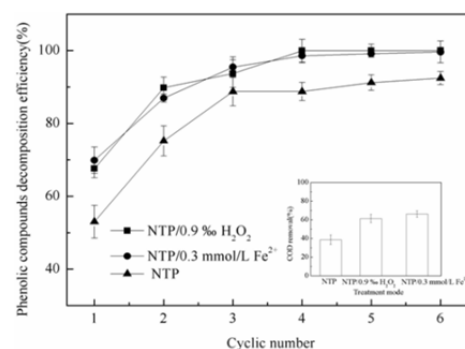


Figure 10. Effect of H_2O_2 and Fe^{2+} on the phenolic compounds decomposition and COD removal.

O_3 oxidation is a common treatment method for coking wastewater [41]. Ozonation experiment was performed in a Plexiglas column ($\text{Ø}50 \times 1,000$ mm). About $16 \text{ mg}/(\text{L} \cdot \text{min})$ of O_3 was added to 1 L of coking wastewater by aeration device, and the concentration of O_3 in the water was $80 \text{ mg}/\text{L}$. After 60 min of reaction, 85.6% phenol decomposition and 40.11% COD removal were obtained, as shown in Figure 11. In the same reaction time, the degradation efficiency of phenolic compounds and COD removal by plasma technology are better, which is mainly due to various physical and chemical effects produced during the discharge process to improve the oxidation of pollutants.

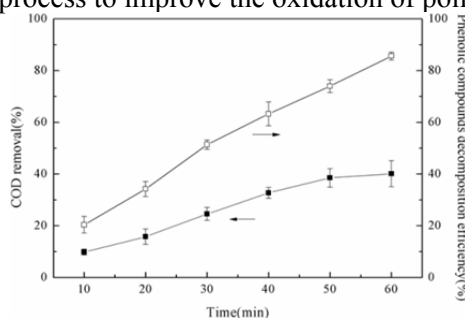


Figure 11. The removal rate of phenolic compounds and COD by ozone oxidation.

3.8. Comparison of processes and cost evaluation

Based on the amount of catalyst and the power consumption of the reaction, the operation cost of different processes was evaluated. For the two processes, the O_3 generator power was 10.2 kW and the NTP system was 2.68 kW (power supply 1.5 kW, air fan 1.1 kW, air compressor 0.08 kW). For COD reduction, the E_{EM} values of NTP and ozonation were 18.5 kWh/g (30 kV, 6th pass) and 101.7 kWh/g , respectively. NTP/ H_2O_2 and NTP/ Fe^{2+} were 11.7 and 10.8 kWh/g , respectively. Considering the oxygen consumption, the cost of ozonation treatment was approximately \$16.65 per gCOD. The cost of plasma treatment was approximately \$1.90 per gCOD. Therefore, for coking wastewater treatment, the plasma system was more economical than the O_3 oxidation. Compared with other wastewater treatment processes such as biological method and adsorption method, plasma discharge system can reduce treatment time, land occupancy rate and cost.

4. Conclusions

The decomposition of organic pollutants was studied experimentally by applying plasma generated by the pulsed discharge in a novel gas-liquid reactor with an exhaust gas circulation system. Two different systems for phenol degradation were examined: 87.4% phenol decomposition and 65.2% COD removal were observed in the 6th treatment by the plasma with gas circulation, which was higher without gas circulation with 51.9% phenol decomposition and 35.6% COD removal. The apparent improvement of treatment efficiency is attributed to the full utilization of O₃ in the exhaust gas of the plasma reactor. The removal efficiency of phenol increased with the increase of applied voltage and pH value. The addition of 0.9‰ H₂O₂ and 0.3 mmol/L Fe²⁺ apparently enhanced the phenol removal. The reactor was also used to treat phenolic compounds in coking wastewater. Compared with other processes, the plasma system has higher treatment efficiency and is suitable for the treatment of industrial wastewater.

Acknowledgments

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