Multi-tube parallel surface discharge plasma reactor for wastewater treatment

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ABSTRACT

A multi-tube parallel surface discharge plasma reactor has been designed to investigate its mineralization potential for organic wastewater. p-Nitrophenol (PNP) was used as the target pollutant. The effects of some operational parameters such as solution pH value, discharge voltage, air flow rate and air moisture on COD removal were investigated. Ozone utilization and biodegradability (BOD_5/COD) of wastewater were evaluated. The results indicated that this discharge system presented a great potential for COD removal. For 40 L of PNP wastewater, complete PNP removal and 97% of COD removal efficiency were achieved after 1 h of discharge treatment with energy yield of 27.4 g_{COD} kW h^{-1}, and correspondingly, the ozone consumption and ozone utilization efficiency were 80% and 1.3 g_{COD} g_{O3}^{-1}. Increasing solution pH value and increasing discharge voltage were both beneficial for COD removal. The optimal air flow rate was observed as 1.5 m^3 h^{-1}. Dry air contributed to a greater COD removal efficiency, due to more O_3 generation. The biodegradability of PNP wastewater increased up to 0.42 after 1 h of treatment.

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1. Introduction

In recent years, the degradation of biologically recalcitrant and toxic organic compounds in water has become the focus of environmental remediation efforts. As an advanced oxidation process, non-thermal discharge plasma technology exhibits a great prospect in wastewater treatment. The main merits of non-thermal discharge plasma may be the in situ generation of strong active species such as O_3 and ·OH radicals, which are highly effective for pollutants removal in water [1,2].

The design of discharge plasma reactor is the core for pollution control, which effects active species generation, pollutant degradation efficiency and energy yield. A great deal of research has been conducted on the structures of discharge reactors and discharge types. Usually the electrode structures of the discharge reactors are mainly classified as needle to plate electrode [3], line to plate electrode [4,5], line to tube electrode [6], line to line electrode [7], ring to tube electrode [8] and dielectric barrier [9,10]. In terms of discharge types, there are liquid phase discharge [11], gas phase discharge with high voltage electrode above the water surface [12] and gas–liquid mixture discharge [2].

The most abundant oxidizing species produced by non-thermal discharge plasma with the addition of air or oxygen is O_3 that is formed in the gas phase, although small amounts of other species such as H_2O_2, ·OH and ·HO_2 radicals are also produced in the water phase. Therefore, to efficiently utilize the O_3 for pollutants removal in water, it is very important to increase the interfacial area between the water phase and the gas phase as much as possible. One way is to disperse the gas containing O_3 in water in the form of fine bubbles. On the one hand, gaseous O_3 and dissolved O_3 can directly react with contaminants at the gas–liquid interface and in the bulk phase, respectively; on the other hand, O_3 dissolved in water can be decomposed into ·OH radicals, which has much stronger oxidative potential than O_3, and reacts quickly with the contaminants [13].

In our previous study, an active species bubbling reactor using gas phase surface discharge has been developed to degrade organic pollutants in water [14]. In that discharge system, active species generated in the gas phase were injected into the water in the form of fine bubbles by gas diffuser. The whole process was little affected by water conductivity and no electrode corrosion. More importantly, the active species could be impregnated into water completely, and thus a great potential was presented for organic pollutants removal in water. However, it was mainly conducted on a laboratory scale and the treatment volume was very limited (1.5 L). At present, little has been reported on the industrial application of non-thermal discharge plasma in wastewater treatment. This may be due to the fact that a discharge reactor with good performances such as great active species propagation, operational stability and high energy efficiency needs to be further studied when used for industrial application.

In the present study, a multi-tube parallel surface discharge plasma reactor (40 L), based on our previous active species bubbling reactor [14], was designed. The aim of this study was to

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evaluate the potential of this discharge system for pollutant removal in wastewater. p-Nitrophenol (PNP) was chosen as the target pollutant, which has been widely used as an important raw material for production of insecticides, herbicides and various synthetic compounds [15], and has been listed as one of the 129 priority toxic pollutants by US Environmental Protection Agency [16]. The effects of several operational parameters, such as solution pH value, discharge voltage, air flow rate and air moisture on COD removal of PNP wastewater were investigated. Ozone utilization and biodegradability of PNP wastewater were evaluated. It is expected to develop an up-scaled, efficient and capable reactor for organic wastewater treatment and promote the industrial application of non-thermal discharge plasma technique.

2. Experimental

2.1. Materials

PNP (purity > 95%) was purchased from Shenyang Chemical Reagent Factory, China. All other organic and inorganic reagents used were analytical grade and were used as purchased without further purification. The solution pH was adjusted by NaOH (0.1 mol L^{-1}) and HCl (0.1 mol L^{-1}) solutions. PNP wastewater was prepared by adding solid PNP into a certain volume of tap water.

2.2. Multi-tube parallel surface discharge plasma reactor

The schematic diagram of the multi-tube parallel surface discharge plasma reactor system is shown in Fig. 1, which is similar to our previous study [14]. Fig. 1a is the schematic diagram of the reactor and Fig. 1b is the photograph of the high voltage electrode system. Alternating Current (50 Hz) power supply was used in the present study, and the voltage was 0–50 kV adjustable. The reactor was a cylindrical stainless steel tube with the inner diameter of 280 mm and the height of 1000 mm. The high voltage electrode was made of spiral stainless steel, which was tightly attached onto the inside wall of a cylindrical quartz glass tube. The diameter of the spiral stainless steel was 1.5 mm and the thread pitch was about 15 mm. The inner diameter of the quartz glass tube was 40 mm and the length was 900 mm. The whole discharge length of quartz glass tube was 700 mm. The high voltage electrode system was up-scaled by combination of four quartz glass tubes in parallel as shown in Fig. 1b. There was a gas diffuser at the bottom

of each quartz glass tube. The outside wall of the cylindrical stainless steel tube was grounded.

In each batch experiment, 40 L wastewater with initial COD concentration of 80 mg L^{-1} was treated. Prior to discharge treatment, air was injected into the four quartz glass tubes, and then the high voltage electrode system was immersed into the wastewater. Herein air moisture (g m^{-3}) means the mass of water vapor in a stere of air.

2.3. Methods and analyses

The discharge voltage and current were measured using a Tektronix TDS2014 digital oscilloscope equipped with a Tektronix P6021 high voltage probe and a Tektronix A6021 current probe. A capacitance (8 μF) was connected into the ground electrode in series to measure the discharge charge, and Lissajous figure was recorded with the digital oscilloscope with the help of two voltage probes and a current probe. The typical discharge voltage and current waveforms are shown in Fig. 2. The Lissajous figures of different discharge voltages are presented in Fig. 3. Based on the Lissajous figures, the input powers were 33.9, 37.7 and 152 W at discharge voltage of 23, 26 and 30 kV, respectively.

PNP concentration was measured by an HPLC (SCL-10ACP, Shimadzu, Japan) equipped with a Hypersil ODS (25 μm, 4.6 mm × 250 mm) reverse phase column, and the mobile phase consisted of a mixture of 1% aqueous acetic acid and methanol (v/v = 3:7) with a total flow rate of 1.0 mL min^{-1} and the wavelength was set at 315 nm. Gas phase O_3 concentration was measured with ozone detector (EG-2001, Japan). COD was measured by the potassium dichromate standard method. BOD_5 was measured with dilution and seeding method after 5 days of culture.

The discharge power was calculated as described in our previous study [14].

The specific energy density (SED) can be calculated by the following equation,

$$SED = \frac{Pt}{V} \tag{1}$$

where V is the total volume of wastewater (L), P is the discharge power, and t is the discharge time (s).

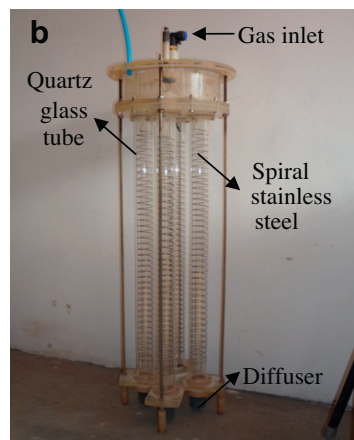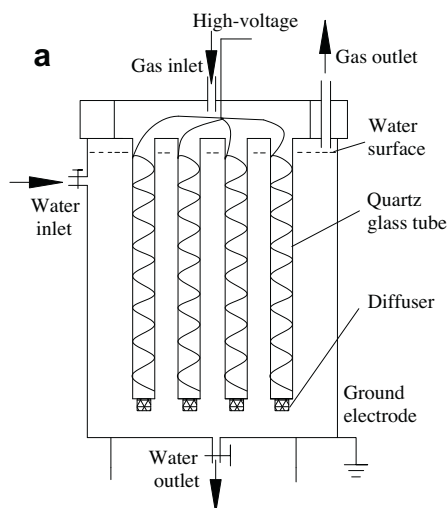The energy yield (G) is defined as the removed COD divided by input energy,

Fig. 1. Schematic diagram of the experimental system ((a) schematic diagram of the reactor; (b) photograph of the high voltage electrode system).
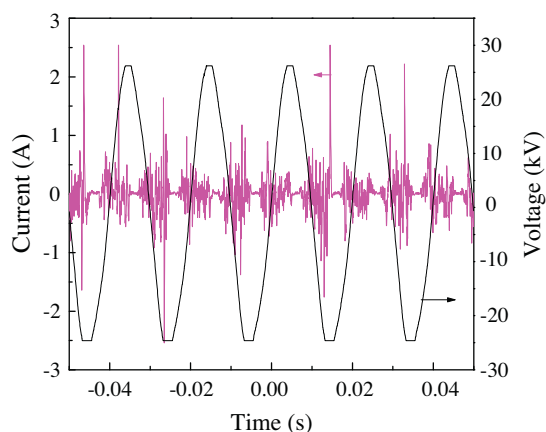
Fig. 2. The typical discharge voltage and current waveforms obtained in this study.
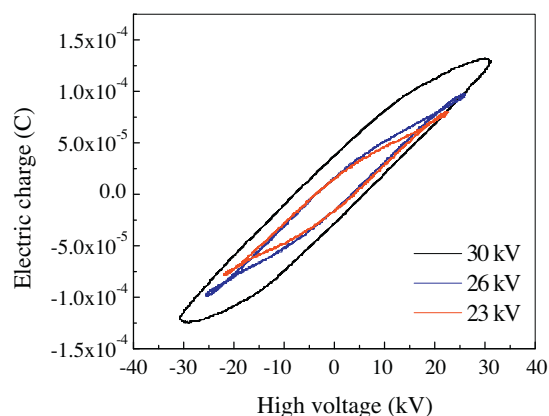

Fig. 3. Lissajous figures under different discharge voltages.

$$G = \frac{\Delta m_{COD}}{P \cdot t} \qquad (2)$$

where Δm_{COD} is the amount of COD removed (g).

COD decomposition rate (υ) and COD removal efficiency (η_{COD}) are calculated as follows,

$$\upsilon = \frac{\Delta m_{COD}}{t} \qquad (3)$$

$$\eta_{COD} = \frac{m_{COD_0} - m_{COD_t}}{m_{COD_0}} \times 100\% \qquad (4)$$

where the m_{COD_0} and m_{COD_t} are the amount of COD at time 0 and t, respectively.

Ozone consumption (φ_{O_3}) and ozone utilization efficiency (U_{O_3}) are calculated as follows,

$$\varphi_{O_3} = \frac{m_{O3,0} - m_{O3,PNP}}{m_{O3,0}} \times 100\% \qquad (5)$$

$$U_{O_3} = \frac{\Delta m_{COD}}{m_{O3,0} - m_{O3,PNP}} \times 100\% \qquad (6)$$

where $m_{O3,0}$ and $m_{O3,PNP}$ are the outlet mass of O_3 in tap water and PNP wastewater at the same discharge time, respectively.

3. Results and discussion

3.1. Effect of solution pH on COD removal

Organic compounds in solutions with diverse acid–base properties distribute in different forms, such as ionic and molecular states, due to their acid–base equilibrium. The ionic form is generally more reactive towards oxidants than the molecular form [17]. Thus, it is of great significance to examine the effect of solution pH on organic wastewater treatment.

The effect of solution pH on COD removal of PNP wastewater is shown in Fig. 4. As could be seen in Fig. 4, solution pH imposed an apparent effect on COD removal. About 58.3% of COD removal efficiency was obtained at pH 12.0 within 3 h of discharge treatment; while only 27.5% was achieved at pH 3.0 within the same time. The energy yield and COD decomposition rates under different pH values are listed in Table 1. The greatest energy yield and the highest COD decomposition rate were achieved at pH 12.0. It is ascribed to the fact that PNP undergoes acid–base equilibrium with pK_a = 7.15. At pH 7.15, both $C_6H_5NO_3$ and $C_6H_5NO_3^-$ are equally abundant; whereas above 7.15, $C_6H_5NO_3^-$ becomes the predominant species. O_3 played an important role in this type of discharge system [14]. For an ionizable organic compound, the reaction rate of O_3 with the charged anion form is typically faster than with the conjugate acid. Furthermore, under alkaline condition, O_3 is unstable and can be decomposed into ·OH radicals [18], which can oxidize the organic compound more efficiently. Therefore, the greatest COD removal efficiency was achieved in alkaline condition. The same conclusion was also presented by Hsu et al. [19], where higher COD removal of phenolic solutions occurred at alkaline condition, due to the reason that O_3 underwent decomposition to generate ·OH radicals.

The following studies were all conducted at solution pH value of 12.0.

3.2. Effect of discharge voltage on COD removal

The effect of discharge voltage on COD removal is shown in Fig. 5. COD removal efficiency increased with the discharge voltage. At discharge voltage of 30 kV, about 83.0% of COD was removed within 3 h of treatment; while approximately 58.3% and 10.0% of COD could be removed at discharge voltages of 26 kV and 23 kV within the same time, respectively. The discharge was just initiated and only a few active species could be generated at 23 kV, and thus COD removal efficiency was very low. When the discharge voltage increased to 26 kV, the discharge became much stronger and more active species were generated, which enhanced
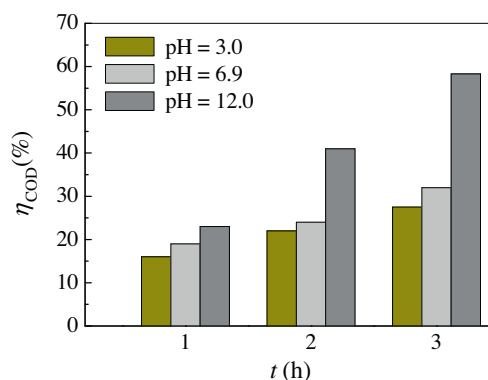

Fig. 4. Effect of solution pH on COD removal (experimental conditions: discharge voltage of 26 kV, air flow rate of 1.5 m³ h⁻¹, and moisture content of 21.3 g m⁻³).

Table 1
Energy yield and COD decomposition rates under different solution pH values.

t (h)	G (g_{COD} kW h^{-1})			υ (mg min^{-1})		
	pH = 3.0	pH = 6.9	pH = 12.0	pH = 3.0	pH = 6.9	pH = 12.0
1	13.5	15.8	19.0	8.5	10.0	12.0
2	8.9	10.3	17.4	5.7	6.5	10.9
3	7.8	8.8	16.6	4.9	5.6	10.4

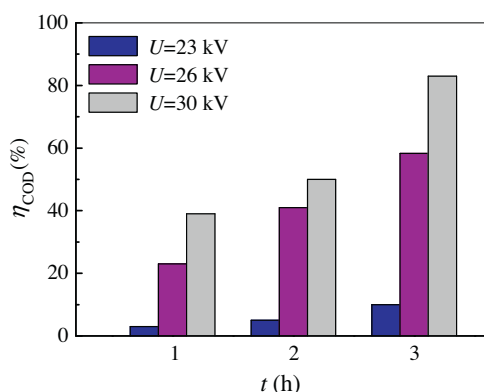**Fig. 5.** Effect of discharge voltage on COD removal (experimental conditions: air flow rate of 1.5 m^3 h^{-1}, moisture content of 21.3 g m^{-3}, and solution pH of 12.0).

COD removal greatly. However, the discharge became unstable and was near spark discharge at 30 kV, and in this case, lots of energy would be consumed by heat although the input energy was very huge, which was not beneficial for COD removal.

The energy yield and COD decomposition rates under different discharge voltages are listed in Table 2. With the increase of discharge voltage, the energy yield increased firstly and then decreased; while the COD decomposition rate increased. The greatest energy yield was obtained at 26 kV, and the fastest COD decomposition rate occurred at 30 kV. Based on comprehensive consideration of the COD removal efficiency, the energy yield and the COD decomposition rate, the optimized discharge voltage was 30 kV in the present study. The following studies were all conducted at discharge voltage of 30 kV.

3.3. Effect of air flow rate on COD removal

Fig. 6 presents the effect of air flow rate on COD removal. With the increase of the air flow rate, the COD removal efficiency firstly increased to a certain point and then decreased gradually. After 3 h of discharge treatment, COD removal efficiency increased from 51.0% to 83.0% when the air flow rate increased from 1.0 to 1.5 m^3 h^{-1}, and further increase presented a negative effect. At the air flow rate of 2.5 m^3 h^{-1}, COD removal efficiency was only 14.0%.

The energy yield and COD decomposition rates under different air flow rates are listed in Table 3. Similar to the trend of COD removal, the energy yield and COD decomposition rate both

Table 2
Energy yield and COD decomposition rates under different discharge voltages.

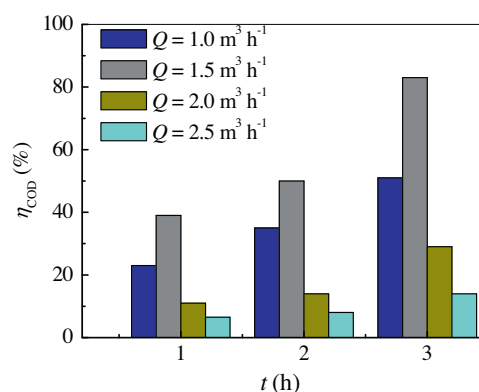| t (h)	G (g_{COD} kW h^{-1})			υ (mg min^{-1})		
	U = 23 kV	U = 26 kV	U = 30 kV	U = 23 kV	U = 26 kV	U = 30 kV
1	2.8	19	8.2	1.6	12	20.8
2	2.4	17.3	5.3	1.3	11	13.3
3	3.1	16.6	5.8	1.8	10	14.8

Fig. 6. Effect of air flow rate on COD removal (experimental conditions: moisture content of 21.3 g m^{-3}, solution pH of 12.0, and discharge voltage of 30 kV).

increased firstly, and then followed by a decline trend. The greatest energy yield and COD decomposition rate both occurred at the air flow rate of 1.5 m^3 h^{-1}.

To a certain extent, the increase of the air flow rate was helpful for the transfer of O$_3$ from gas phase to liquid phase, which was beneficial for organic pollutant removal; however, with its further increase, the retention time of O$_3$ in liquid phase was shortened [19]. Therefore, it could be deduced that when the air flow rate increased from 1.0 to 1.5 m^3 h^{-1} in the present study, more O$_3$ was transferred into PNP wastewater, resulting in the enhancement of COD removal; further increase in air flow rate would shorten the retention time of O$_3$ in wastewater, leading to the decrease of COD removal.

Therefore, the optimized air flow rate was 1.5 m^3 h^{-1} in this study.

3.4. Effect of air moisture on COD removal

Air moisture has a great influence on active species generation when the discharge occurs in gas phase [20]. In order to investigate the effect of air moisture on COD removal, the comparison of COD removal efficiency between moist air and dry air was conducted. Herein the dry air was obtained by passing the moist air through a silica gel column to dehydrate.
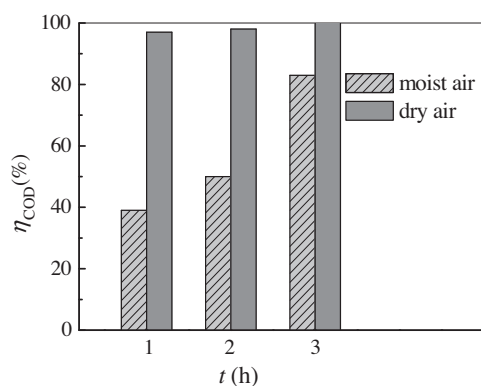
The results of COD removal efficiencies of PNP wastewater under moist and dry air conditions are presented in Fig. 7. It was found that greater COD removal efficiency was obtained when the discharge occurred in dry air. For example, only 40.0% of COD removal efficiency was achieved after 1 h of discharge treatment under moist air (moisture content of 21.3 g m^{-3}, and herein air moisture means the mass of water vapor in a stere of air); while it was enhanced to 97.0% under dry air (moisture content $\approx$0%). The reason might be that some energy was consumed by the excitation of H$_2$O molecule when the discharge occurred in moist air, resulting in the decrease of energy consumed by O$_2$ excitation. Furthermore, due to the property of electron-withdrawing of H$_2$O, the energy of electrons was restricted in moist air, leading to the quenching of active species. Therefore, less active species such as O$_3$ was generated under moist air [21,22].

In addition, greater energy yield and higher COD decomposition rate were both obtained in dry air. For example, the COD decomposition rate was 20.8 mg min^{-1} with energy yield of 8.2 g_{COD} kW h^{-1} after 1 h of discharge treatment in moist air; while it was enhanced to 28 mg min^{-1} with the energy yield of 27.4 g_{COD} kW h^{-1} in dry air. The results suggested that dry air was more beneficial for O$_3$ generation, resulting in higher COD decomposition rate and greater energy yield.

Table 3
Energy yield and COD decomposition rates under different air flow rates.

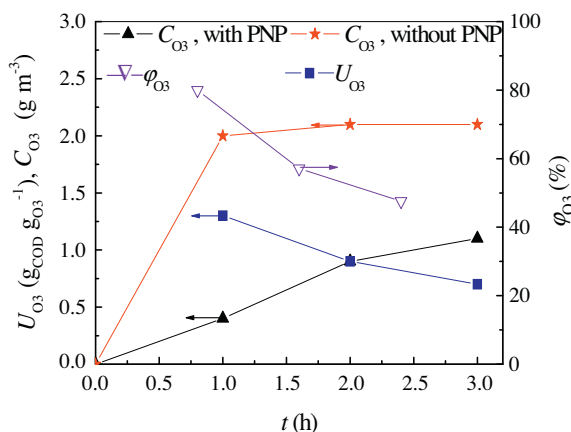| t (h)	G (g_{COD} k Wh^{-1})				v (mg min^{-1})			
	$Q = 1.0$ m^3 h^{-1}	$Q = 1.5$ m^3 h^{-1}	$Q = 2.0$ m^3 h^{-1}	$Q = 2.5$ m^3 h^{-1}	$Q = 1.0$ m^3 h^{-1}	$Q = 1.5$ m^3 h^{-1}	$Q = 2.0$ m^3 h^{-1}	$Q = 2.5$ m^3 h^{-1}
1	4.8	8.2	2.3	1.4	12.3	20.8	5.9	3.5
2	3.7	5.3	1.5	0.8	9.3	13.3	3.7	2.1
3	3.5	5.8	2.0	1.0	9.1	14.8	5.2	2.5

Fig. 7. Effect of air moisture on COD removal (experimental conditions: air flow rate of 1.5 m^3 h^{-1}, discharge voltage of 30 kV, and solution pH of 12.0).

The following studies were all conducted in dry air.

3.5. Ozone consumption

O_3 utilization is an important parameter to evaluate the potential of gas phase discharge plasma for wastewater treatment. In order to evaluate the O_3 utilization efficiency in the present discharge system, it is very important to understand the variations of outlet O_3 concentration during discharge plasma process.

The outlet O_3 concentrations with/without PNP, O_3 utilization efficiency and O_3 consumption are presented in Fig. 8. On the one hand, the outlet O_3 concentrations with/without PNP both increased with treatment time, and the O_3 concentration without PNP was obviously higher than that with PNP. After 1 h of discharge treatment, the outlet O_3 concentration was about 2.0 g in tap water (without PNP); while it was only 0.4 g in the case of PNP wastewater. These results suggested that most of the O_3 was utilized for wastewater treatment in this stage. The gradual increase of the outlet O_3 concentration with PNP after 1 h indicated

that some open-ring intermediates such as small organic acids might be generated, which were difficult to be oxidized, after almost complete removal of COD (97%).

On the other hand, the O_3 utilization efficiency and O_3 consumption both decreased with the increase of treatment time. Within 1 h of discharge treatment, the O_3 utilization efficiency was 1.3 g_{COD} g_{O3}^{-1}, and it decreased to 0.7 g_{COD} g_{O3}^{-1} after 3 h of treatment. 80.0% of O_3 consumption was obtained within 1 h of discharge treatment, which decreased to 47.6% after 3 h of treatment. These results also indicated that some open-ring intermediates were generated. Due to the difficulty of oxidation of these intermediates, the O_3 utilization efficiency and O_3 consumption both decreased.

In order to compare the surface discharge plasma system with pure ozonation system, COD removal by pure ozonation was evaluated, and the ozone was generated by home-made ozone generator. The experimental results on pure ozonation and surface discharge plasma for COD removal are presented in Fig. 9. Herein, dry air with air flow rate of 1.5 m^3 h^{-1} was used as carrier gas, and the discharge voltage in surface discharge system was 26 kV. The ozone concentration in the inlet of reactor was 1.5 g m^{-3} in these two systems. As could be seen in Fig. 9, greater COD removal efficiency was obtained in surface discharge plasma system than in pure ozonation system. Within 1 h of treatment, about 87% of COD was removed in the surface discharge plasma system, while only 55% in pure ozonation system. These results suggested that ozone played a very important role for COD removal in the surface discharge plasma system, and some other chemical and physical effects resulted from discharge plasma also contributed to COD removal.

3.6. Biodegradability of wastewater

In order to understand the toxicity of PNP wastewater after discharge treatment, the biodegradability of PNP wastewater before and after treatment were evaluated and the results are summarized in Table 4. As could be seen in Table 4, the ratio of BOD$_5$/COD increased from 0.07 to 0.42 after 1 h of discharge treatment. This interesting result presented that the biodegradability of PNP wastewater was enhanced greatly after discharge treatment.
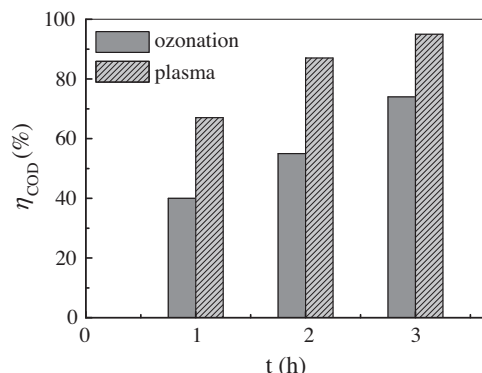

Fig. 8. Changes of outlet O_3 concentration, utilization and consumption with treatment time.

Fig. 9. Comparison of pure ozonation and discharge plasma for COD removal.

Table 4
Comparisons of COD, BOD$_5$ and BOD/COD$_5$ of solutions before and after treatment.

t (h)	η_{PNP} (%)	COD (mg L^{-1})	BOD$_5$ (mg L^{-1})	BOD$_5$/COD
0	0	80	5.6	0.07
1	100	2.4	1.0	0.42

Adams et al. [23] have reported that ozonation could enhance the biodegradability of nitrophenols wastewater. The biodegradability of perchloroethylene and dichlorophenol wastewater was also greatly enhanced after pulsed discharge plasma treatment [24].

Therefore, this type of discharge system can be used as a pretreatment process for bio-refractory organic wastewater treatment in industry. Further research about its characteristics for real organic wastewater treatment is in progress.

4. Conclusions

A multi-tube parallel surface discharge plasma reactor has been designed to investigate its potential for PNP wastewater treatment. Alkaline condition, higher discharge voltage and dry air atmosphere were beneficial for COD removal. The increase in air flow rate to a certain extent could enhance COD removal due to more chemically active species generation; while further increase presented a negative effect. The decrease of O$_3$ consumption and utilization efficiency with treatment time suggested the formation of some open-ring intermediates, which were difficult to be oxidized. By comparing the contributions of pure ozonation and discharge plasma for COD removal, it was found that ozone played a very important role in the present discharge plasma system. The biodegradability of PNP wastewater was enhanced greatly after discharge treatment.

This multi-tube parallel surface discharge plasma reactor is expected to be promising for industrial application.

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