

Low-temperature plasma-induced degradation of aqueous 2,4-dinitrophenol

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Abstract

The degradation behavior of 2,4-dinitrophenol (DNP) by low-temperature plasma was investigated and the effect of some factors that might affect the degradation process was further examined. The results indicated that DNP could be effectively removed from aqueous solution. The degradation value was 83.6% when the input power was 150 W and 60 s was selected as the discharge time. Increasing the input power increased the degradation efficiency. The degradation process fitted first-order dynamics and the reduction was mainly caused by the reaction of DNP with $\cdot\text{OH}$. The degradation efficiency decreased with the increase of initial concentration at the same discharge time. H_2O_2 at the concentration of 0.25% enhanced the degradation process, however, hindered the degradation at 1.00 and 2.00%. The presence of Fe^{2+} could benefit DNP degradation. However, the increment in degradation efficiency might be suppressed to some extent at a high concentration level. Cu^{2+} inhibited the degradation process within 30 s and enhanced the reduction after 30 s. Furthermore, the increment of Cu^{2+} concentration could enhance the effect. A little acid environment was conducive to DNP degradation and the pH value became lower with increasing discharge time by low-temperature plasma.

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Keywords: Degradation; 2,4-Dinitrophenol; Low-temperature plasma

1. Introduction

2,4-Dinitrophenol (DNP) is widely used as raw materials and intermediates in chemical industries for the manufacture of pesticides [1]. Discharge of DNP into the environment poses significant health risks due to its high carcinogenicity [2]. Because of its high stability and solubility in water, the pollution of drinking-water reservoirs and the environment by it is a dramatic problem recently. The U.S. Environmental Protection Agency has listed DNP as “priority Pollutant” [2]. It is noteworthy that the pollution resulting from DNP discharge into the aqueous environment is considered as one of the general problems in the dye industry in China.

Due to DNP's high stability and solubility in water, the conventional technologies used for the purification of wastewaters that contained DNP were not effective [3]. The degradation processes using microorganisms like bacteria were relatively

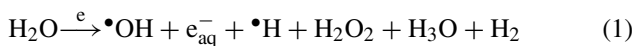
very slow and inefficient [2]. Hence more studies were being concentrated for the development of new methods for DNP degradation. The advanced oxidation process (AOP) was possibly to be an effective technology for treating DNP in wastewater, however, there was some trouble with economics, equipment, or efficiency [4,5]. Contact glowdischarge electrolysis has been employed to destroy DNP in the solution and the results showed that this technology was effective for DNP degradation [6]. Nowadays, low-temperature plasma is widely used in environmental protection field. When the discharge of low-temperature plasma begins, both physical and chemical processes happen [7]. The former could lead to large quantities of UV light and intense shock waves and the later could cause the formations of chemically active species. This technology that integrates light, electronic and chemical oxidation into one process has a collective effect on degrading organic species. The detailed mechanisms are as follows:

- (1) The role of high-energy electron. When the discharge begins, high-energy electrons are formed [8]. High-energy

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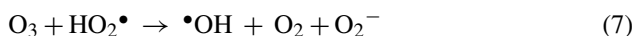
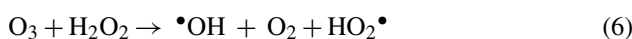
electrons could react with water in 10^{-7} s and then a lot of radicals are produced [9,10].



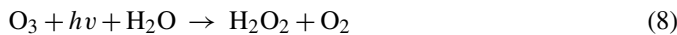
At the same time, the following reactions happen and O^\bullet and O_3 are also formed [8].



- (2) The role of ozone. When the discharge begins, ozone is also formed. Ozone is of highly solubility in water and reported to be an effective oxidant. Furthermore, it could remove pollutants by $\bullet\text{OH}$ formed as follows [11,12]:



- (3) The role of UV light. The efficiency of producing $\bullet\text{OH}$ in water only by UV light is very low. But the efficiency is relatively high in the presence of ozone [12]:



e_{aq}^- and $\bullet\text{H}$ are subject to be scavenged by dissolved oxygen in water [10] and $\bullet\text{OH}$ is the most oxidative radical among these radicals mentioned above. So, $\bullet\text{OH}$ is the main radical that is responsible for the degradation of pollutants.

The degradation behavior of DNP by low-temperature plasma has not yet been studied and it was possibly different from that by contact glowdischarge electrolysis. The purpose of the present study was to investigate the possibility of DNP degradation from aqueous solution by low-temperature plasma. Furthermore, the degradation processes of DNP in the presence of H_2O_2 , Fe^{2+} and Cu^{2+} were also examined.

2. Experimental

2.1. Experimental apparatus

The experimental apparatus was bought from Nanjing Suman Electronics Co., Ltd., PR China and shown in Fig. 1. It consisted of a reaction cell (DBD-50) and a power supplier (CTP-2000 K) that could provide a steady voltage of 100 V. The reaction cell, which was between the high voltage electrode and ground electrode, consisted of two parts. The upper part of the reaction tank was a little bigger than the lower part. The lower part was used to contain the solution, which was 84 mm in inner diameter, 88 mm in outer diameter and 6 mm in height. The reaction tank was put in the center of the two electrodes. The power was supplied by an AC source, which could be operated at an adjustable amplitude voltage. The intensity of discharge in the reaction tank could be denoted by the input power, which was calculated by the average voltage and current of the AC power. The dielectric barrier was made of quartz.

2.2. Chemicals and reagents

DNP was obtained from Shanghai Chemicals Factory and used without further purification. Methanol used in the analysis was HPLC grade. All chemicals were of reagent grade with purity higher than 99%.

2.3. Sample preparation

DNP solution at initial concentration of $0.025 \text{ mmol L}^{-1}$ was used to test the effect of input power, 2-propanol, *tert*-butanol, H_2O_2 , Fe^{2+} , Cu^{2+} and initial pH value on the degradation and to examine the change of pH value by low-temperature plasma. The input power of 150 W was employed to investigate the effect of 2-propanol, *tert*-butanol, DNP initial concentration, H_2O_2 , Fe^{2+} , Cu^{2+} and initial pH value on the degradation and to determine the change of pH value by low-temperature plasma. The pH value of the solutions was adjusted using HCl (0.01 mol L^{-1}) or NaOH (0.01 mol L^{-1}) solution. All the samples were treated at the voltage of 100 V.

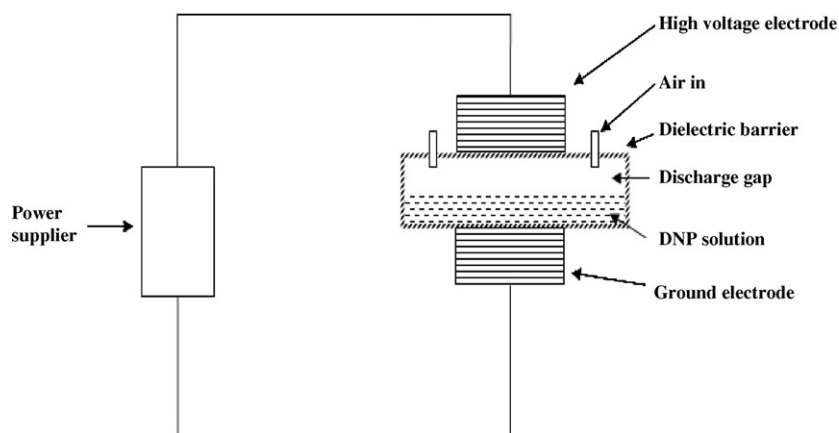


Fig. 1. Scheme of the experimental apparatus.

2.4. Analysis

DNP concentration was determined using a HPLC system (Agilent, USA, 1200 Series high-performance liquid chromatography) equipped with Hypersil ODS HPLC column (250 mm × 4.6 mm i.d., 5 μm, Agilent, USA), a multiple wavelength UV diode array detector and an auto sampler controlling under a Chemstation data acquisition system. 5.0 μL of DNP solution before and after being treated at a given time was injected automatically into the HPLC system. The eluent consisted of 70% methanol and 30% water and the flow rate was 1.0 mL min⁻¹. The determination wavelength was set at 360 nm and the column temperature was kept at 30 °C. The degradation efficiency for each sample was calculated from the following Eq. (10):

$$\eta = \left(\frac{C_0 - C_t}{C_0} \right) \times 100\% \quad (10)$$

where η was DNP degradation efficiency (%); C_t was DNP residual concentration at a given time t (mmol L⁻¹); C_0 was DNP initial concentration (mmol L⁻¹). The pH value was measured by pH monitor (Shanghai Kangyi Instrument Co., Ltd. China, PHS-2C).

3. Results and discussion

3.1. Effect of the input power on DNP degradation by low-temperature plasma

Fig. 2 showed the change of DNP degradation values with different input powers using low-temperature plasma. It was indicated that DNP could be effectively removed from aqueous solution by low-temperature plasma and increasing input power favored the degradation process. When the discharge time was 60 s, 67.7, 80.6 and 83.6% of DNP was removed when the input powers were 90, 120 and 150 W, respectively. As the degradation curves in Fig. 2 appeared to be exponential, an attempt was made to apply the data to the integral rate equation for the first-order

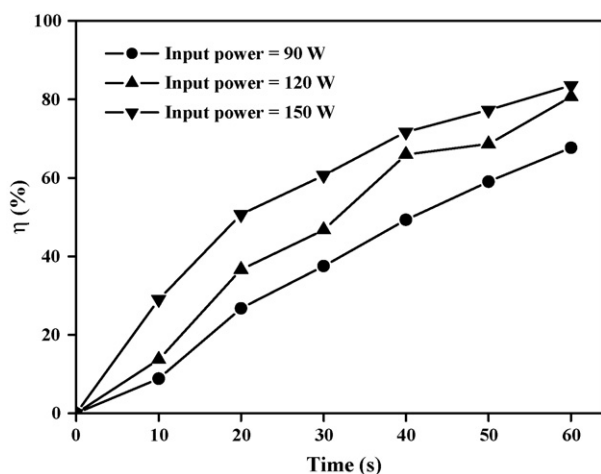


Fig. 2. Change of DNP degradation values with different input powers.

Table 1

Kinetic coefficients of the first-order dynamics for DNP degradation at different input powers

Output power (W)	k (s ⁻¹)	R	S.D.	N	P
90	0.0191	0.9954	0.0436	7	<0.0001
120	0.0271	0.9888	0.0968	7	<0.0001
150	0.0295	0.9980	0.0446	7	<0.0001

reaction Eq. (11).

$$\ln \left(\frac{C_0}{C_t} \right) = kt \quad (11)$$

where k denoted the rate constant (s⁻¹). The parameters of the first-order reaction at different input powers were obtained and shown in Table 1. The results indicated that good correlation was obtained for each set of data and the degradation of DNP by low-temperature plasma fitted first-order kinetics.

Hydroxyl radical, produced in the discharge process, had a strong electrophilic character and was mainly responsible for DNP degradation. Hydroxyl radical firstly attacked one of the carbon atoms of the aromatic ring with highest electron density [3]. When both of -OH substituent and -NO₂ substituent were present, the electrophilic attack would occur preferentially in *ortho* position with respect to phenolic -OH group [3], resulting in dinitro catechol. Dinitro *o*-benzoquinone was the product of the reaction between dinitro catechol and •OH. In turn, dinitro *o*-benzoquinone might be subjected to be further attacked by •OH and the aromatic ring opened. At the same time, a series of aliphatic acids, oxalic acid, formic acid and acetic acid were formed. These acids could further react with •OH and CO₂ and H₂O were produced. The -NO₂ group could be easily removed by direct attack of •OH and NO₃⁻ was further formed in the oxidizing media [3]. The possible mechanism of DNP degradation by low-temperature plasma was proposed and shown in Fig. 3.

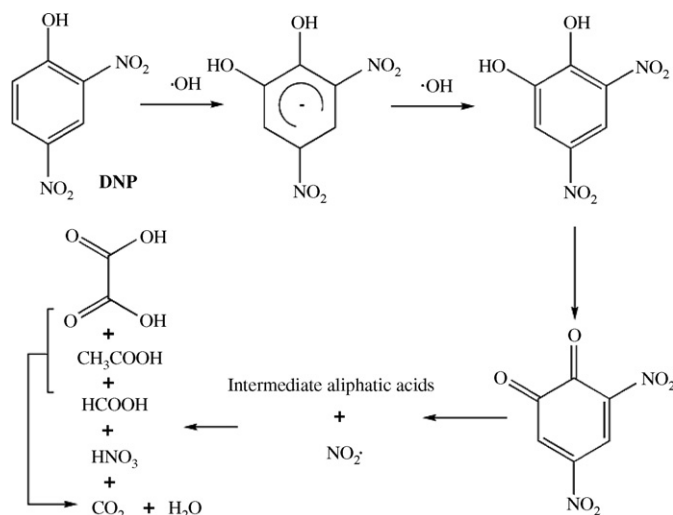


Fig. 3. The proposed mechanism of DNP degradation by low-temperature plasma.

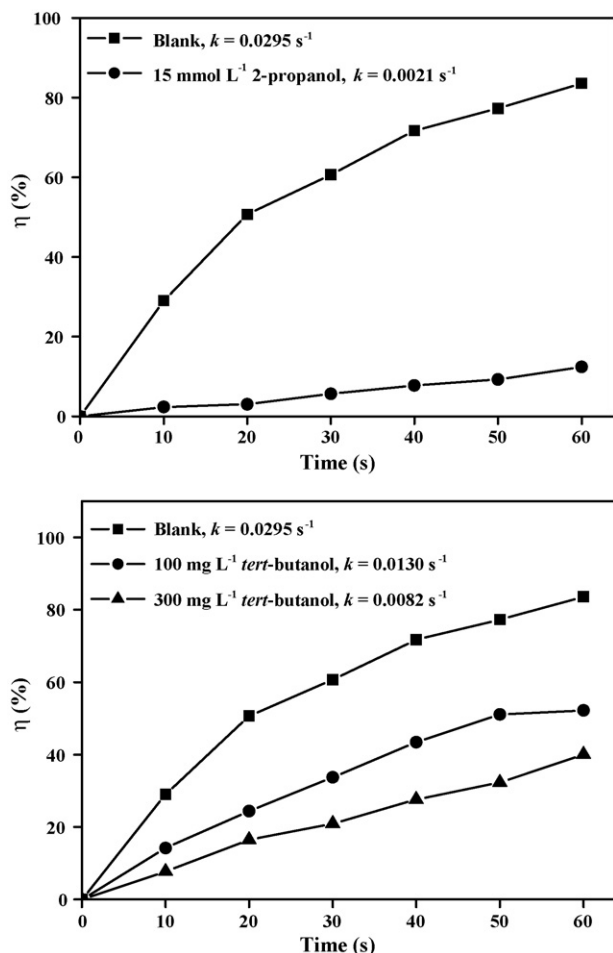


Fig. 4. Effect of 2-propanol and *tert*-butanol as additives on DNP degradation.

3.2. Effect of 2-propanol and *tert*-butanol on DNP degradation by low-temperature plasma

Generally, it was impossible that there was only one pollutant in the wastewater. Therefore, it was necessary to study the effect of other compounds existing in DNP solution. Here, 2-propanol and *tert*-butanol were chosen as the model radical scavengers to study their effect on DNP degradation behavior.

Fig. 4 showed the effect of 2-propanol and *tert*-butanol on DNP degradation by low-temperature plasma. It could be concluded from Fig. 4 that DNP concentration decreased with increase of discharge time in the absence and presence of 2-propanol and *tert*-butanol. At the same discharge time, DNP degradation value in the absence of 2-propanol and *tert*-butanol was higher than that in the presence of them. When 15 mmol L⁻¹ 2-propanol and *tert*-butanol of 100 and 300 mg L⁻¹ were added into DNP solution and 60 s was selected as the discharge time, the removal efficiencies were 12.4, 52.2 and 40.0%, respectively. Furthermore, When 60 mmol L⁻¹ 2-propanol was added, DNP concentration was almost changeless with increase of discharge time. This indicated that the increase of 2-propanol and *tert*-butanol would result in the decrease of the degradation values and they had a strong inhibition effect on DNP degradation. Furthermore, the effect of them on rate constants

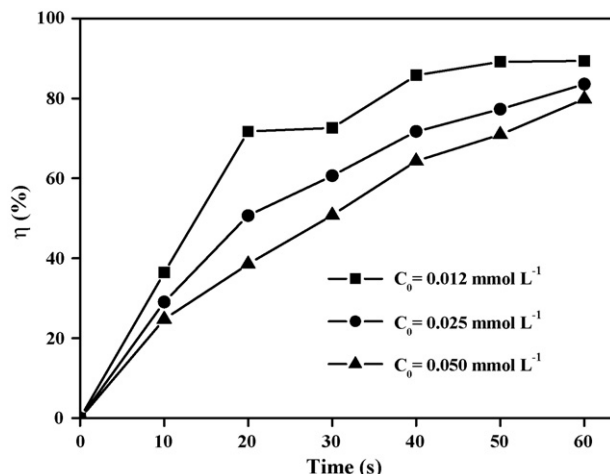


Fig. 5. Effect of the initial concentration on DNP degradation.

(shown in Fig. 4) was similar with the effect on the degradation efficiencies. As we knew, $\bullet\text{OH}$ could be scavenged by 2-propanol quickly: $\bullet\text{OH} + 2\text{-propanol} \rightarrow (\text{CH}_3)_2\bullet\text{COH} + \text{H}_2\text{O}$ (rate constant was $1.9 \times 10^{10} \text{ mol L}^{-1} \text{ s}^{-1}$) [13]. However, *tert*-butanol could scavenge $\bullet\text{OH}$ by the reaction: $\bullet\text{OH} + \text{tert-butanol} \rightarrow \bullet\text{CH}_2\text{C}(\text{CH}_3)_2\text{OH} + \text{H}_2\text{O}$ (rate constant was $6.0 \times 10^8 \text{ mol L}^{-1} \text{ s}^{-1}$) [13]. Both $(\text{CH}_3)_2\bullet\text{COH}$ and $\bullet\text{CH}_2\text{C}(\text{CH}_3)_2\text{OH}$ were inert radicals [14]. The great inhibition effect of 2-propanol and *tert*-butanol also suggested that $\bullet\text{OH}$ was the most responsible oxidant for DNP degradation by low-temperature plasma.

3.3. Effect of the initial concentration on DNP degradation by low-temperature plasma

Fig. 5 showed the effect of the initial concentration on DNP degradation by low-temperature plasma. As shown in Fig. 5, the degradation value decreased with the increasing of initial concentration at the same discharge time. It was indicated that the initial concentration greatly affected DNP degradation behavior. When DNP initial concentration was 0.012 mmol L⁻¹, the rate constant was 0.0391 s⁻¹ and DNP degradation efficiency was 89.4% when 60 s was chosen as the discharge time. However, when the initial concentrations were 0.025 and 0.050 mmol L⁻¹, the rate constants were 0.0295 and 0.0259 s⁻¹, respectively. When 60 s was the discharge time, removal efficiencies of 83.6 and 79.9% were achieved at the initial concentrations of 0.025 and 0.050 mmol L⁻¹, respectively. It might be the reason that the degradation amount of DNP was affected by input power of low-temperature plasma. Therefore, under invariable input power, the degradation rate decreased when the initial concentration increased. Furthermore, the degradation rate decreased largely after the discharge time of 20 s.

3.4. Effect of H₂O₂ as the additive on DNP degradation by low-temperature plasma

H₂O₂ could enhance the degradation of diphenylamine by increasing the concentration of $\bullet\text{OH}$ [15]. In order to test the

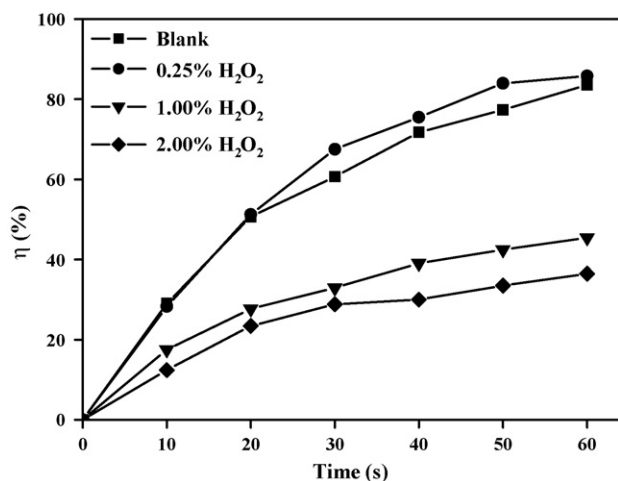


Fig. 6. Effect of H₂O₂ as the additive on DNP degradation.

effect of H₂O₂ on DNP degradation by low-temperature plasma, H₂O₂ was added at different concentrations of 0.25, 1.00 and 2.00% and the results were shown in Fig. 6. It was indicated that the degradation value in the presence or absence of H₂O₂ improved with increasing discharge time and H₂O₂ enhanced DNP degradation at the concentration of 0.25%, however, hindered the degradation at 1.00 and 2.00%. When 60 s was the discharge time and H₂O₂ was added at the concentration of 0, 0.25, 1.00 and 2.00%, the degradation values were 83.6, 85.8, 45.4 and 36.4%, respectively. The same conclusion could be obtained from the change of rate constant when H₂O₂ was added at different concentrations. The rate constants in the presence of H₂O₂ at the concentrations of 0, 0.25, 1.00 and 2.00% were 0.0295, 0.0344, 0.0097 and 0.0071 s⁻¹, respectively. This result indicated that DNP degradation by low-temperature plasma could be enhanced when H₂O₂ was added at the appropriate concentration of 0.25%. It might be the reason that when H₂O₂ was added at the concentration of 0.25%, •OH was formed by the decomposition of H₂O₂ (shown in Eq. (9)) [16] and then •OH concentration increased. So, the degradation process was enhanced. But, when H₂O₂ was added at 1.00 and 2.00%, the hydroxyl radicals generated produced hydroperoxyl radicals (HO₂•) in the presence of a local excess of H₂O₂ [17,18].

3.5. Effect of Fe²⁺ as the additive on DNP degradation by low-temperature plasma

Fe²⁺ had an evidently catalytic effect and the effect was more remarkable when the concentration of Fe²⁺ was higher [6]. The effect of Fe²⁺ as the additive on DNP degradation by low-temperature plasma was also tested and shown in Fig. 7. It was indicated that the degradation value in the presence or absence of Fe²⁺ improved with increasing the discharge time and Fe²⁺ benefited the degradation at the concentration of 30 mg L⁻¹, however, the enhancement effect was reduced when Fe²⁺ was added at 120 mg L⁻¹. Total disappearance of DNP was achieved at 10 s when Fe²⁺ was added at the concentration of 30 mg L⁻¹. But only 39.7% of DNP disappeared at the same discharge time when Fe²⁺ was added at 120 mg L⁻¹. The rate constant changed from

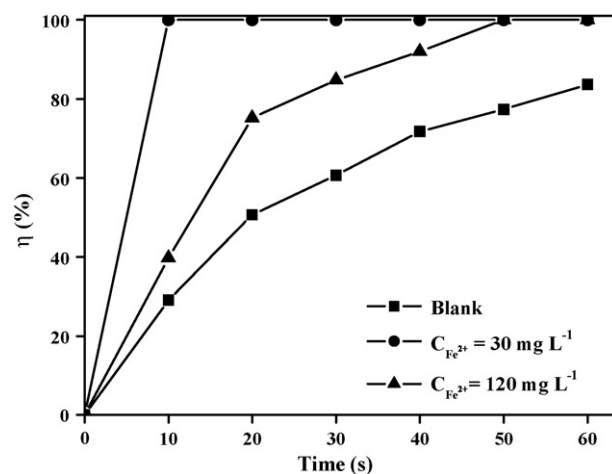
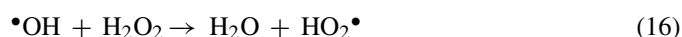
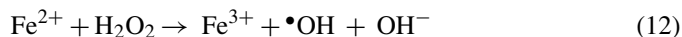


Fig. 7. Effect of Fe²⁺ as the additive on DNP degradation.

0.0295 to 0.0643 s⁻¹ when Fe²⁺ was in the absence and in the presence at a concentration of 120 mg L⁻¹. These results indicated that DNP degradation by low-temperature plasma could be largely enhanced when Fe²⁺ was added at the appropriate concentration of 30 mg L⁻¹. In any case, Fe²⁺ enhanced DNP degradation process by low-temperature plasma. The reason was as follows [19,20]:



The addition of Fe²⁺ as catalyst enhanced the oxidizing power of H₂O₂ because of the production of •OH in the solution (shown in Eq. (12)). Furthermore, the regeneration of Fe²⁺ from additional reduction of Fe(OH)²⁺ benefited the production of •OH (shown in Eq. (15)) and, hence, DNP degradation process was enhanced. When the concentration of Fe²⁺ was high, the reactions (shown in Eq. (13) and (16)) were enhanced and the enhancement effect was accordingly weakened.

3.6. Effect of Cu²⁺ as the additive on DNP degradation by low-temperature plasma

The degradation curves of DNP by low-temperature plasma in the absence and presence of Cu²⁺ were plotted in Fig. 8. It was revealed that Cu²⁺ inhibited the degradation process within 30 s and enhanced the reduction after 30 s. Furthermore, the increment of Cu²⁺ concentration could enhance the effect. When 20 s was the discharge time and Cu²⁺ was in the absence and in the presence at 30 and 120 mg L⁻¹, DNP degradation values were 50.7, 44.6 and 39.8%, respectively. But when 50 s was selected as the discharge time and Cu²⁺ was in the absence and in the presence at 30 and 120 mg L⁻¹, the degradation values were 77.3, 83.2 and 88.1%, respectively. The most appropriate explanation was that within 30 s, the octahedron complex containing Cu²⁺

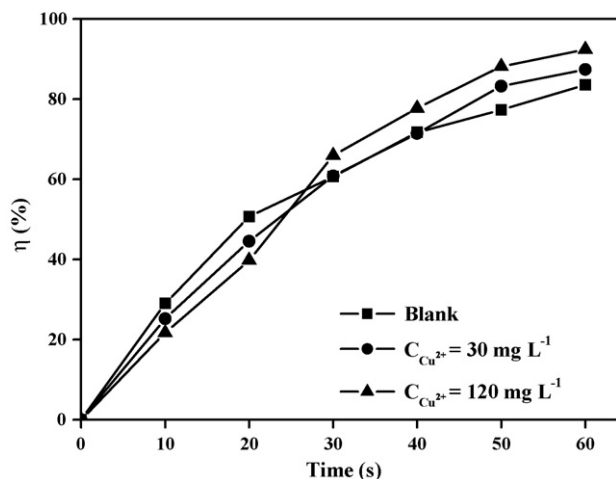
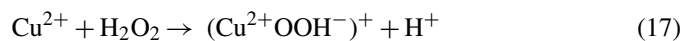


Fig. 8. Effect of Cu²⁺ as the additive on DNP degradation.

and DNP was produced and the reaction probability of DNP and •OH was reduced [21]. So, the degradation process was hindered. The concentrations of the complex and DNP decreased with increment of the discharge time and Cu²⁺ was released from the complex. Cu²⁺ accelerated DNP degradation process after 30 s by low-temperature plasma (shown in Eq. (17) and (18)) [22].



3.7. Effect of initial pH value on DNP degradation by low-temperature plasma

The pH values of the different wastewaters are different, and it influences the oxidation processes for the removal of pollutants. Similarly, the pH value plays an important role in DNP degradation. Fig. 9 showed the effect of initial pH value on DNP degradation by low-temperature plasma. The results showed that the degradation process was enhanced at initial pH value of 4.57. At initial pH value of 4.57, a reduction of 83.6% was achieved at

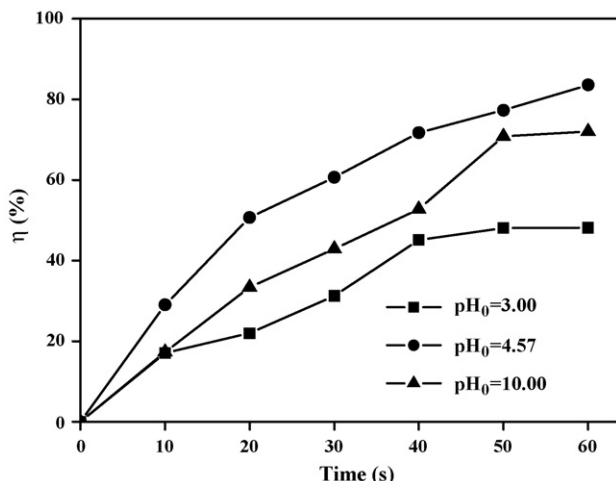


Fig. 9. Effect of initial pH value on DNP degradation.

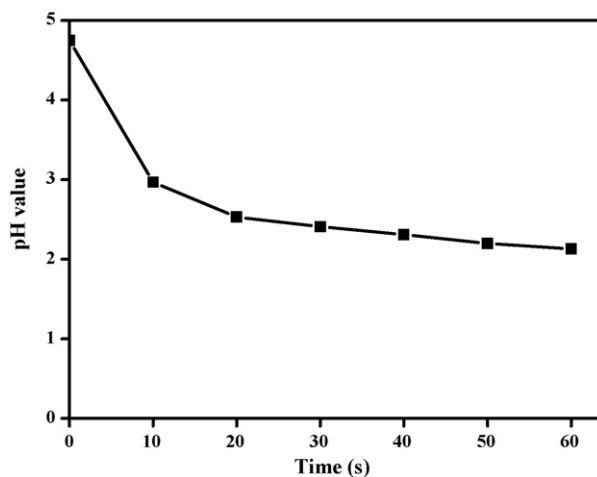


Fig. 10. Change of pH value of DNP solution by low-temperature plasma.

60 s. While at the same discharge time and at initial pH values of 3.00 and 10.00, the degradation efficiencies were only 48.2 and 72.0%, respectively. So, the change of initial pH value resulted in a marked change of the degradation efficiency. When the initial pH value was 4.57, the rate constant was 0.0295 s⁻¹, while initial pH values were 3.00 and 10.00, the rate constants were only 0.0116 and 0.0223 s⁻¹, respectively. This may be presumed that more •OH was produced from low-temperature plasma than H₂O₂ in a little acid condition, and the former was more active than H₂O₂. However, as a result of forming of a hydrogen bond inside DNP molecule, DNP degradation rate was slower under more acid conditions [6]. While in an alkaline solution, HO₂• ion would be decomposed by the discharge, which scavenged •OH [15]. An innovative finding that DNP degradation process fitted first-order kinetics irrespective of change of the initial pH values, which indicated that the change of initial pH value did not lead to the change in the degradation kinetics.

3.8. Change of pH value of DNP solution by low-temperature plasma

Fig. 10 showed the change of pH value of DNP solution by low-temperature plasma. The results showed that the pH value decreased with increasing discharge time due to lots of H₃O⁺ and acid substances produced during the degradation process.

4. Conclusions

DNP could be effectively removed from aqueous solution by low-temperature plasma. The degradation efficiency was 83.6% when the input power was 150 W and 60 s was selected as the discharge time. DNP degradation process by low-temperature plasma fitted first-order dynamics. The great inhibition effect of *tert*-butanol and 2-propanol on DNP degradation was achieved, demonstrating that the degradation was mainly caused by the reaction of DNP with •OH.

DNP degradation efficiency decreased with the increase of initial concentration at the same discharge time. H₂O₂ as the additive enhanced the degradation process at the concentration

of 0.25%, however, hindered the degradation at 1.00 and 2.00%. Fe^{2+} benefited the degradation at 30 mg L^{-1} , however, the enhancement effect was weakened when Fe^{2+} was added at 120 mg L^{-1} . Cu^{2+} inhibited the degradation process within 30 s and enhanced the reduction after 30 s. Furthermore, the increment of Cu^{2+} concentration could enhance the effect.

The pH value could affect the removal efficiency and the degradation process was enhanced in a little acid condition. The pH value became lower with increasing discharge time by low-temperature plasma. Based on the experimental results above, low-temperature plasma was an effective method to decompose DNP.

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