Effect of electrical discharge in water on concentration of nitrate solution

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In this work, the effect of electrical discharge on nitrate concentration is considered in aqueous solution. The atmospheric pressure plasma was produced by a high-voltage power supply at 27 kHz using pin-to-pin configuration. Air, argon, and argon/methane mixture were used to study the working gas effect. UV-VIS spectroscopy and ion chromatography were used to analyze the effect of the electrical discharge on nitrate concentration in deionized water. Optical emission spectroscopy (OES) was applied to diagnose active species inside and on the surface of the deionized water solution. The results of the present work showed that the atmospheric pressure electric discharge with air increases nitrate concentration while it remains constant using argon and argon/methane electrical discharges. It was also revealed that in the presence of air, the electrical discharge reduces pH, acidifying the solution and increasing solution conductivity due to production of extra nitrate ions. On the other hand, argon electrical discharge increases pH and conductivity due to production of OH^- ion in water.

Keywords: electric discharge, nitrate solution, optical spectroscopy

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1. Introduction

Atmospheric-pressure plasma processing has drawn great interest for applications such as surface cleaning, deposition, etching, sterilization, and pollution decontamination due to its low cost, high processing speed, simple system, and without any vacuum equipment.[1–9] Recently, much attention has been focused on electrical discharge process in liquid phase and many experimental investigations have been carried out to recognize production mechanism and chemical reactions between non-thermal plasma and liquid phases.[10–16] In the last few years, the use of non-thermal atmospheric pressure plasma in water has been investigated because of its importance in electrical transmission processes and its practical applications in biology, chemistry, and electrochemistry. Atmospheric pressure plasma in air contains active species such as positive and negative ions, free electrons, free radicals, ozone, and ultraviolet radiation that can be dissolved in liquid to react and form other different species. During discharge in water, hydroxyl (OH) and hydroperoxyl (HO_2) radicals form in the plasma phase, dissolve in water to react and form H_2O_2. Generally, strong electric fields applied to water (electrohydraulic discharge) initiate both chemical and physical processes.[11,12,17–20]

In this work, we report the effects of atmospheric pressure, non-thermal plasma produced with argon, argon/methane, and air as working gases, on an aqueous solution of nitrate. The components and characteristics of solution, before and after plasma treatment, were analyzed with, UV-VIS spectroscopy (UV-2100), pH meter (Metrohm 780), conductivity meter (Jenway), and ion chromatography (Metrohm Model 850). Meanwhile optical emission spectroscopy IR-UV (SOLAR LAZER SYSTEM, S100) with scanning range from 190 nm to 1100 nm and the resolution 0.1 nm, was used to find reactive plasma species applied to water. The diffraction grating and slit width are 300 lines/mm and 20, respectively.

This paper is organized as follows: In Section 2, we present the experimental setup, in Section 3, we discuss the results and discussion. Finally, conclusions will be given in Section 4.

2. Experimental setup

Figure 1 shows the schematic picture of the experimental setup, pin-to-pin configuration, used in this study. The setup contains two powered electrodes; both electrodes are made of copper, the upper electrode diameter is 7-mm hold close to the solution surface, the lower electrode diameter is 10 mm and has four orifices to produce bubbles in water by gas injection. These produced bubbles lead to a decrease in the breakdown voltage. The gap between two electrodes is 17 mm. The atmospheric pressure plasma was driven with high-voltage power supply (0 kV–15 kV at 27 kHz).

The argon (99.999%), methane (99.99%), and air were used as working gases for generating non-thermal plasma passed through the space between two powered electrodes. The atmospheric pressure plasma was generated between the two electrodes both on the water surface and inside of it close to the second powered electrode. For argon and argon/methane electrical discharges, the chamber was initially filled with the gases at atmospheric pressure and then the plasma ignited. The flow rates of argon, methane, and air were controlled with digital mass flow meter. The flow rates of argon and air gases were 600 sccm. In the case of argon/methane mixture, the flow rates of argon and methane were 300 sccm.

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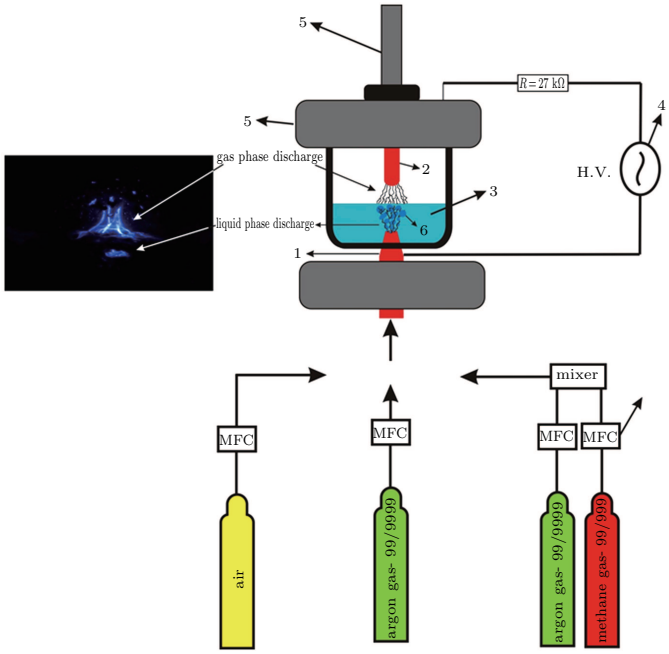


Fig. 1. (color online) Experimental setup for plasma treatment of nitrate solution. 1: powered electrode, 2: powered electrode, 3: aqueous solution of nitrate, 4: power supply, 5: holder, 6: bubbles, 7: digital mass flow meter.

The effect of electrical discharge on aqueous solution of potassium nitrate was investigated in two wavelength ranges, visible and ultraviolet, with UV-VIS spectroscopy. Atmospheric pressure plasma was applied to 25 ml of KNO_3 solution with different concentrations, for 13-minutes treatment. In order to find the treatment time effect, we treated the solution with constant concentration of KNO_3 (80 ppm) in different times, too.

In order to investigate the plasma effect on nitrate concentration by UV-VIS spectroscopy, one pack of nitrate reagent[21] was added to 25-milliliter solution of potassium nitrate before and after applying plasma. Nitrate reagent contains Cd^{+2} ion and sulfonic acid. The reaction between nitrate and Cd^{+2} ions produces nitrites. The nitrites then react with sulfalinic acid forming a red color solution whose absorption of about 543 nm indicates the original concentration of nitrate in the solution.

3. Results and discussion

The results obtained in this research are divided into three parts, air, argon, and argon/methane plasmas. Finally the results will be compared. A photograph of air, argon and argon/methane plasmas in which they were ignited on the aqueous nitrate solution is shown in Fig. 2. The electrical discharge produces very powerful oxidizing species such as radical hydroxyl (OH), H, O_3, and H_2O_2.[12]

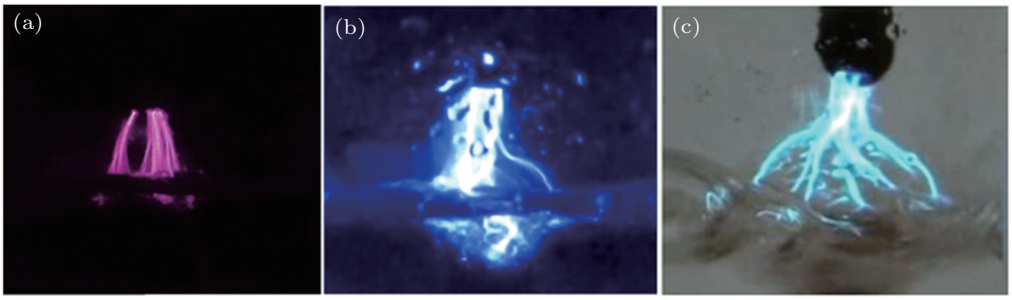


Fig. 2. (color online) Plasma treatment of aqueous solution of nitrate by (a) air, (b) argon, (c) argon/methane plasmas. The upper electrode and water surface are seen in each discharge.

3.1. Air plasma

In this case, we prepared solutions of nitrate with concentration of 40, 80, and 100 ppm. These solutions were treated for 13 minutes, continuously. Figure 3 shows that the absorption intensities of each concentration were increased. In the UV spectral range, the absorption band of nitrate is about 301 nm. The results indicate that the nitrate concentration increases by electrical discharge when air is used as a working gas.

Figure 4 shows the results obtained in the visible range in the presence of nitrate reagent. As shown by applying air plasma to nitrate solution, absorption intensity of each concentration of 50, 60, 70, 80, and 90 ppm was increased, which

confirmed the previous results. It should be noted that the spectrum of the nitrate solution presents an absorption band in the visible region at 543 nm.

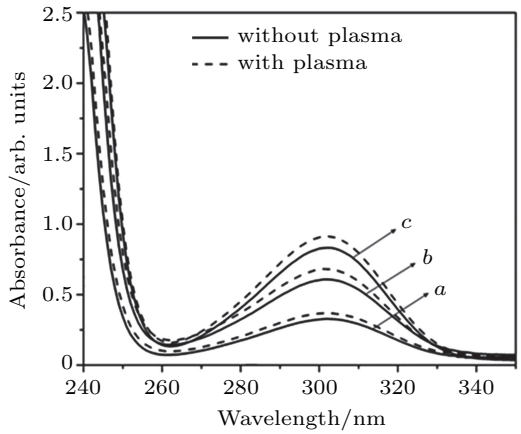


Fig. 3. Comparison between UV-VIS spectra of nitrate solutions, curve *a*: 40, curve *b*: 80, and curve *c*: 100 ppm before and after applying air plasma.

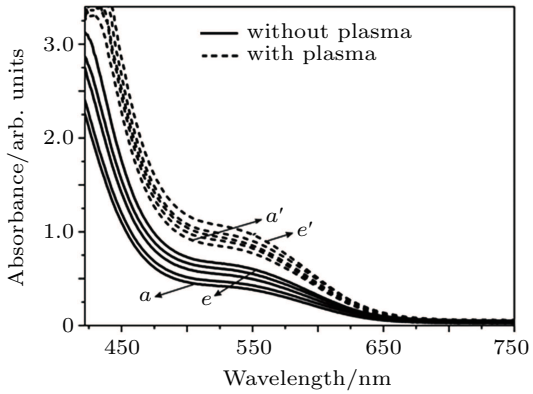


Fig. 4. Comparison between absorption intensity, before and after applying air plasma to aqueous solution of nitrate at visible range: (curve *a*) 50, (curve *e*) 60, (curve *a*′) 70, (curve *e*′) 80, and (curve *e*) 90 ppm.

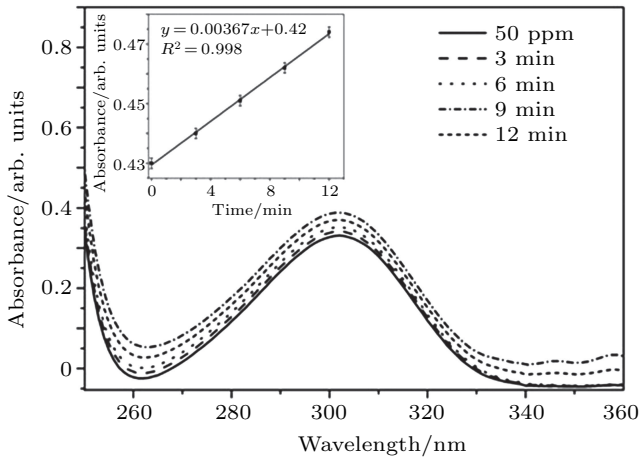


Fig. 5. UV-visible spectra of the nitrate solution (initial concentration 50 mg/L) before and after air plasma treatment.

The nitrate reagent was purchased from Hach company. The reaction equations of nitrate reagent with KNO_3 salt based on its specification.

$$H_2O + KNO_3 \rightarrow K^+(aq) + NO_3^-(aq), \quad (1)$$

$$KNO_3 \xrightarrow{Cd^{2+}} KNO_2, \quad (2)$$

$$KNO_2 \xrightarrow[\text{acid}]{\text{sulfalinic}} (\text{diazonioum}) \text{ salt}. \quad (3)$$

Figure 5 shows the effect of duration of applying air plasma to nitrate solution with the initial concentration of 50 ppm, at ultraviolet range at wavelength of 300 nm. As indicated, by increasing treatment duration, nitrate concentration increases.

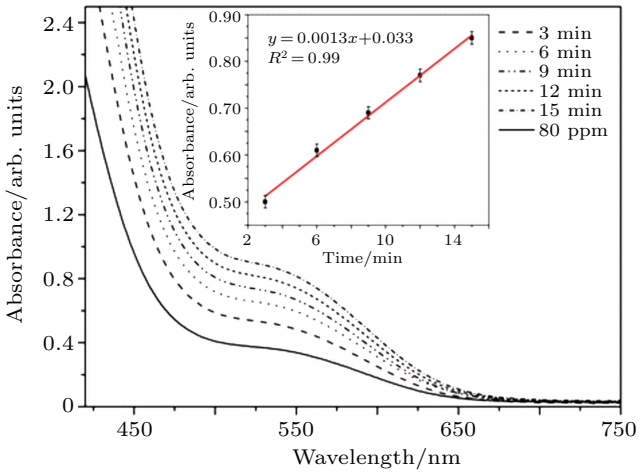


Fig. 6. (color online) Comparison between absorption intensity, before and after applying plasma to aqueous solution of nitrate at visible range with initial concentration of 80 ppm.

Effect of duration of applying air plasma with the initial concentration of 80 ppm has been shown in Fig. 6 at visible range. As shown, by increasing treatment time, nitrate concentration increases.

The following reactions seem to be responsible for increasing nitrate concentration in air plasma treatment.[12] First, we write the main chemical reactions in the discharge channels that happened over the liquid surface in air:[12,22–30]

$$e + N_2 \rightarrow N + N + e, \quad (4)$$

$$e + O_2 \rightarrow O + O + e, \quad (5)$$

$$O + H_2O \rightarrow 2OH, \quad (6)$$

$$e + 2O_2 \rightarrow O_2^- + O_2, \quad (7)$$

$$O + N \rightarrow NO, \quad (8)$$

$$O + N + O_2 \rightarrow NO + O_2, \quad (9)$$

$$N_2 + O_2 + e \rightarrow 2NO + e, \quad (10)$$

$$O + N + N_2 \rightarrow NO + N_2 \quad (11)$$

$$O + O_2 + M \rightarrow O_3 + M \ \ (M = N_2, O_2), \quad (12)$$

$$NO + O_3 \rightarrow NO_2 + O_2, \quad (13)$$

$$NO + O \rightarrow NO_2, \quad (14)$$

$$2NO + O_2 \rightarrow 2NO_2, \quad (15)$$

$$N + O_2^- \rightarrow NO_2 + e, \quad (16)$$

$$O + NO_2 \rightarrow NO_3, \quad (17)$$

$$NO_3 + NO_2 \rightarrow N_2O_5, \quad (18)$$

$$O + H_2O \rightarrow H_2O_2, \quad (19)$$

$$H_2O_2 + NO \rightarrow H_2O + NO_2, \quad (20)$$

$$O + H_2O_2 \rightarrow OH + HO_2, \quad (21)$$

$$NO + OH \rightarrow HNO_2, \quad (22)$$

$$N_2O_5 + H_2O \rightarrow 2HNO_3, \quad (23)$$

$$N_2O_5 + 2H_2O \rightarrow HNO_3 + H_2O. \quad (24)$$

NO_2 molecules can be solved into the nitrate solution and initiate several chemical reactions:

$$2NO_2 + H_2O \rightarrow HNO_2 + HNO_3, \quad (25)$$

$$3NO_2 + H_2O \rightarrow HNO_3 + NO, \quad (26)$$

$$NO_2 + H_2O \rightarrow HNO_3 + H^+ + e, \quad (27)$$

$$NO_2 + OH \rightarrow HNO_3. \quad (28)$$

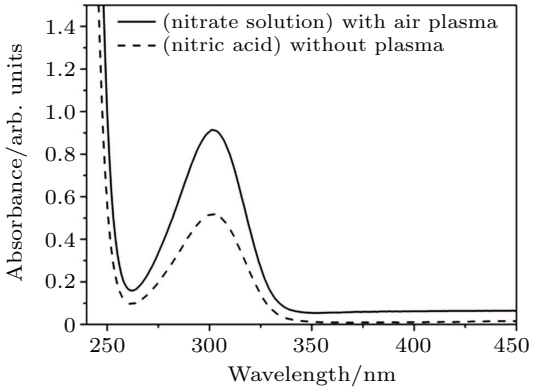


Fig. 7. Absorption spectra measured by UV-VIS spectrometer of nitric acid (without plasma) and potassium nitrate solution treated by air plasma.

In order to confirm the bi-products, we prepared nitric acid solution using deionized water. Then, we took the absorption curve of the sample (nitric acid solution) by UV-VIS spectroscopy about 300 nm, a well-known absorption band of the nitric acid solution. It was revealed that the absorption curve of the nitric acid solution coincides with that of the treated nitrate solution samples by air plasma. This was depicted in Fig. 7. It shows the absorption spectra of potassium nitrate solutions treated by air plasma with pin-to-pin configuration at 7-kV high voltage together with that of nitric acid solution without treatment. The results are in agreement with those obtained by Ref. [11].

3.2. Argon plasma

In Subsection 3.1, we showed that synthetic NO_2 by air plasma change the nitrate solution samples to be acidified by nitric acid yield. The main channels for production of NO_2 in our setup can be seen in equations which initiated by dissociation of nitrogen and oxygen molecules. We changed air for argon gas to prevent NO and NO_2 products over the samples. The flow rate of argon was 600 sccm. The argon plasma prevented acidification of the nitrate solution. Figure 8 indicates the effect of applying argon plasma at different nitrate concentrations. As shown in Fig. 8, argon plasma neither produces extra nitrate nor reduces it. The concentration of nitrate remains almost constant.

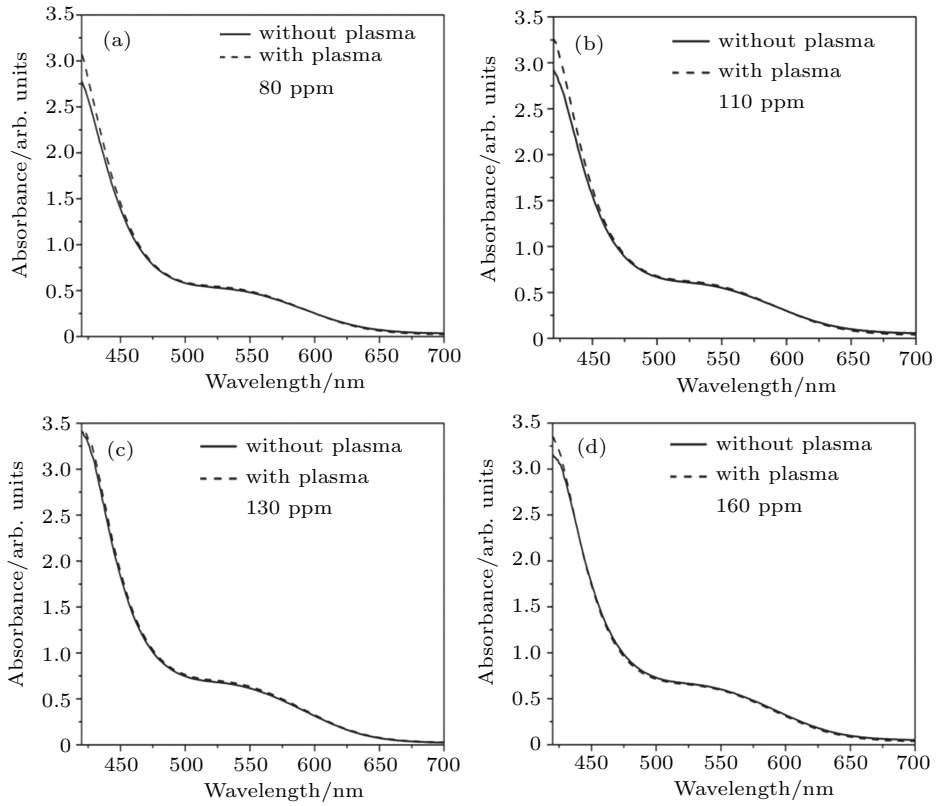


Fig. 8. Comparison between absorption intensity, before and after applying argon plasma to aqueous solution of nitrate at visible range at different concentrations.

The ion chromatography was used for measuring the nitrate concentration before and after plasma processing. The initial concentration of the nitrate solution was 31.66 ppm. Argon and air plasmas were applied 12 min and 3 min, respectively. The results of the ion chromatography are shown in Fig. 9 and listed in Table 1. After applying the air plasma for 3 min, the nitrate concentration increased from 31.66 ppm to 55.097 ppm while in the case of the argon plasma for 12 min treatment, it remained almost unchanged, i.e., 33.29 ppm. Therefore, the air plasma led to an increase in concentration of the nitrate solution via the production of the extra nitrate by synthesizing NO, NO_2, and NO_3, significantly.

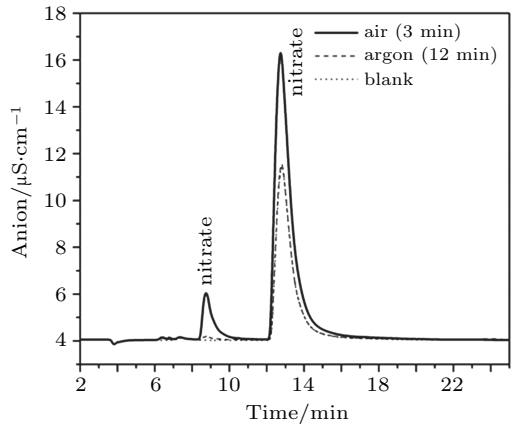


Fig. 9. Ion chromatography results of nitrate solution treated by air and argon plasmas.

Table 1. Comparison between concentration of nitrate solution, after applying argon and air plasmas.

	Component name	Concentration /ppm	Argon plasma (12 min)/ppm	Air plasma (3 min)/ppm
1	Nitrate	31.66	33.29	55.097
2	Nitrite	0	0	2.168

The reactions in aqueous solution of nitrate in the presence of argon plasma can be written as[10,15,22,23,26,30,31]

$$\mathrm{Ar} + \mathrm{e} \rightarrow 2\mathrm{e} + \mathrm{Ar}^*, \quad (29)$$

$$\mathrm{Ar} + \mathrm{e} \rightarrow \mathrm{e} + \mathrm{Ar}^*, \quad (30)$$

$$\mathrm{Ar}^* + \mathrm{e} \rightarrow \mathrm{e} + \mathrm{Ar}, \quad (31)$$

$$\mathrm{Ar}^* + 2\mathrm{Ar} \rightarrow \mathrm{Ar}_2^* + \mathrm{Ar}, \quad (32)$$

$$2\mathrm{e} + 2\mathrm{H_2O} \rightarrow 2\mathrm{OH}^- + \mathrm{H_2}, \quad (33)$$

$$\mathrm{O} + \mathrm{H_2O} \rightarrow 2\mathrm{OH}, \quad (34)$$

$$\mathrm{e} + \mathrm{H_2O} \rightarrow \mathrm{H} + \mathrm{OH} + \mathrm{e}, \quad (35)$$

$$\mathrm{H_2O} + \mathrm{H_2O} \rightarrow \mathrm{H_2O_2} + \mathrm{O_2}, \quad (36)$$

$$\mathrm{H} + \mathrm{O_2} \rightarrow \mathrm{HO_2}, \quad (37)$$

$$\mathrm{HO_2} + \mathrm{OH} \rightarrow \mathrm{H_2O} + \mathrm{O_2}. \quad (38)$$

3.3. Argon/methane plasma

Figure 10 and Table 2 show the comparison between concentration of nitrate solution after applying argon and argon/methane plasmas by ion chromatography measurement. As can be seen, treatment of the nitrate solution by Ar and Ar/CH_4 mixture does not change the nitrate concentration. In the presence of argon/methane plasma, the reactions that occur in aqueous solution of nitrate can be written as[32]

$$\mathrm{CH_4} + \mathrm{e} \rightarrow \mathrm{CH_3} + \mathrm{H} + \mathrm{e}, \quad (39)$$

$$\mathrm{CH_3} + \mathrm{e} \rightarrow \mathrm{CH_2} + \mathrm{H} + \mathrm{e}, \quad (40)$$

$$\mathrm{CH_2} + \mathrm{e} \rightarrow \mathrm{CH} + \mathrm{H} + \mathrm{e}, \quad (41)$$

$$\mathrm{CH_4} + \mathrm{e} \rightarrow \mathrm{CH_2} + \mathrm{H_2} + \mathrm{e}, \quad (42)$$

$$\mathrm{CH_3} + \mathrm{e} \rightarrow \mathrm{CH} + \mathrm{H_2} + \mathrm{e}. \quad (43)$$

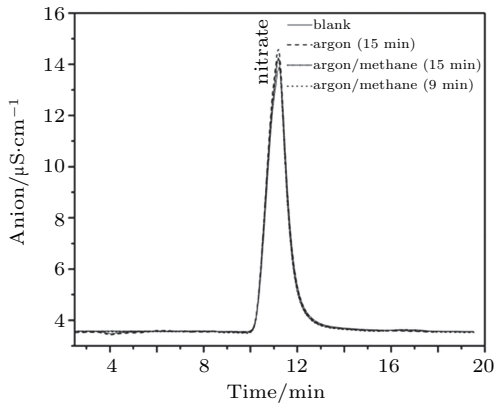


Fig. 10. Ion chromatography of nitrate solution before and after treatment by Ar and Ar/CH_4 plasmas.

The pH of the samples was measured before and after plasma treatment which is indicated in Fig. 11. There was a rapid change in the acidity of the nitrate solution from the initial pH of 5.3 to 2.2 after 15-min running air plasma. The nitrogen oxides change the pH and conductivity as shown in Figs. 11 and 12 through production of ion and acids in water. This can be attributed to HNO_2, HNO_3, and H^+ yields.[10,12,25] In the case of argon plasma, OH^- ions can be produced by treating the samples leading to an increase in pH of the solution.[10]

As shown in Fig. 12, the plasma processing of the nitrate solution changes its conductivity. The change depends on the working gas type, strongly. In order to study this effect, we prepared a nitrate solution with initial conductivity of 0.190 (mili-siemens/cm). After 3-minutes treatment by air discharge, the conductivity of the solution increased to about 50 mS/cm, two orders of magnitudes increase. The argon discharge effect on the solution conductivity was minor, it increases from 0.190 mS/cm to 0.225 mS/cm after 15-minutes treatment. Ar/CH_4 discharge effect on the conductivity was also considerable. The conductivity of the nitrate solution changed about twice of its initial value after 15-minutes treatment by Ar/CH_4 discharge. Most of the change took place up to 3 minutes and there was a gradual increase between 3 minutes to 15 minutes treatment times. These results are in agreement with the pH change as indicated in Fig. 11.

Table 2. Comparison between concentration of nitrate solution, after applying argon and argon/methane plasmas.

	Component name	Concentration /ppm	Argon plasma (15 min)/ppm	Argon/methane plasma (9 min)/ppm	Argon/methane plasma (9 min)/ppm
1	Nitrate	42.96	43.92	43.367	43.34
2	Nitrite	0	0	0	0

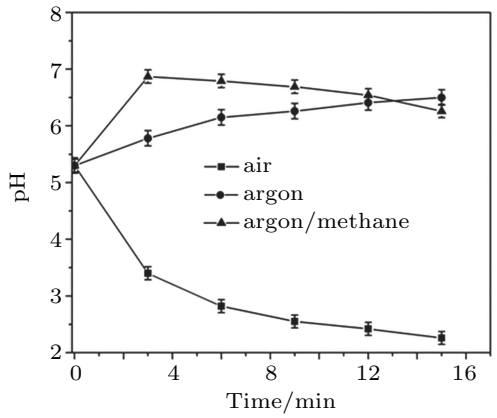


Fig. 11. Comparison between pH of solution, after applying argon/methane, argon, and air plasmas to aqueous solution at different times.

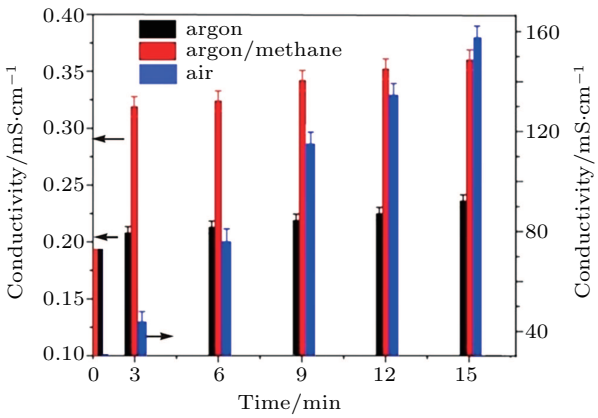


Fig. 12. (color online) Comparison between conductivity of solution, after applying air, argon, and argon/methane plasmas to aqueous solution at different times.

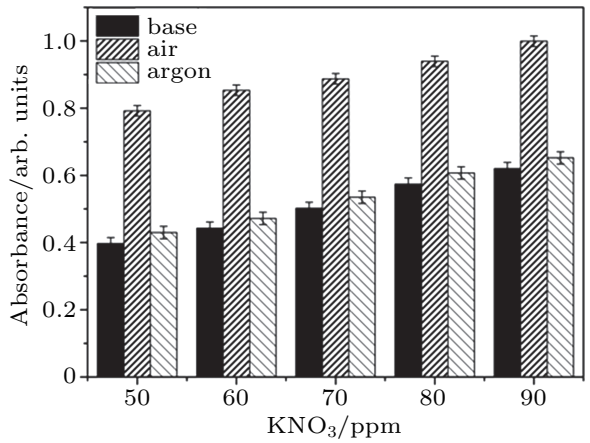


Fig. 13. Comparison between absorbance of nitrate solution, after applying argon and air plasmas at different concentrations.

The results obtained by UV-VIS spectroscopy for air and argon plasmas processing on nitrate solution have been compared in Fig. 13. This comparison shows that an increase in absorption by nitrate solution after applying air plasma is much more than that of Ar discharge which justifies increasing nitrate concentration by air discharge.

Typical V–I characteristics of our setup in the absence and presence of discharge are shown in Figs. 14(a) and 14(b), respectively.

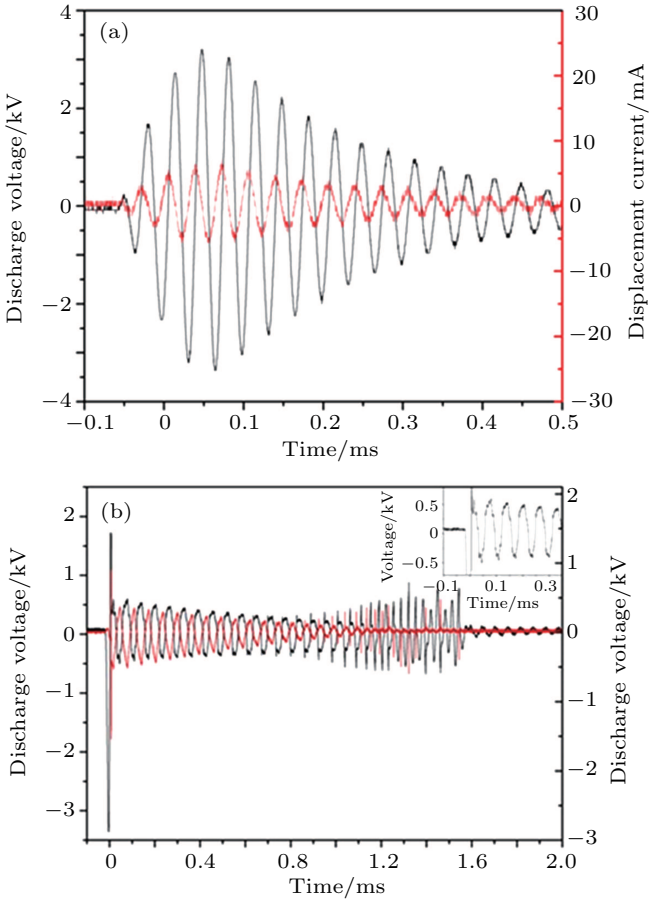


Fig. 14. (color online) Time dependence of discharge voltage and current (a) in the absence of plasma (b) in the presence of plasma.

Figure 15 shows the optical emission spectroscopy (OES) spectra from 200 nm to 1000 nm, for detecting active species over the solution surface with air discharge. To record the emission of plasma irradiation, we employed UV-IR compact wide range 190 nm–1100 nm spectrometer (S-100, solar-laser system). It is seen that NO, N_2^+, and N are synthesized during air discharge running over the nitrate solution. These synthetic gases are responsible for increasing nitrate concentration and reducing the pH of the solution, drastically.

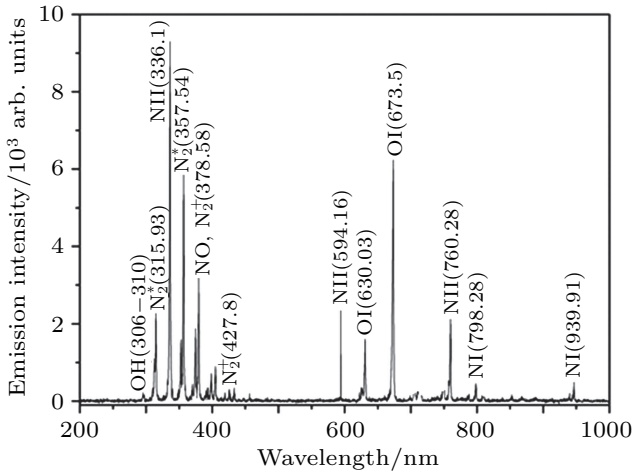


Fig. 15. Optical emission spectra of air plasma formed in pin-to-pin configuration, between upper electrode and the solution surface.

Figure 16 indicates the OES spectra of argon plasma, between upper electrode and the solution surface. As can be seen, nitrogen compounds do not contribute in plasma species over the solution.

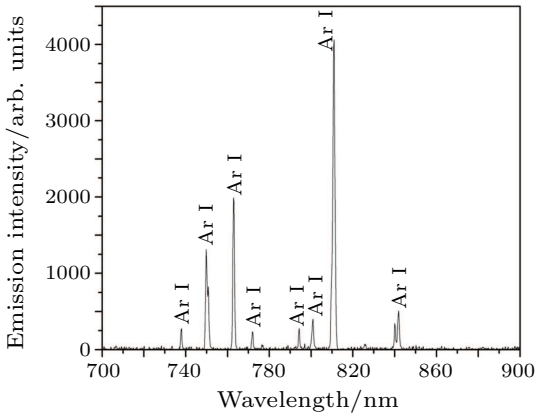


Fig. 16. Optical emission spectra of argon plasma formed in pin-to-pin configuration, between upper electrode and the surface of solution

Optical emission intensity of argon and argon/methane plasmas in the nitrate solution were indicated in Figs. 17 and 18, respectively.

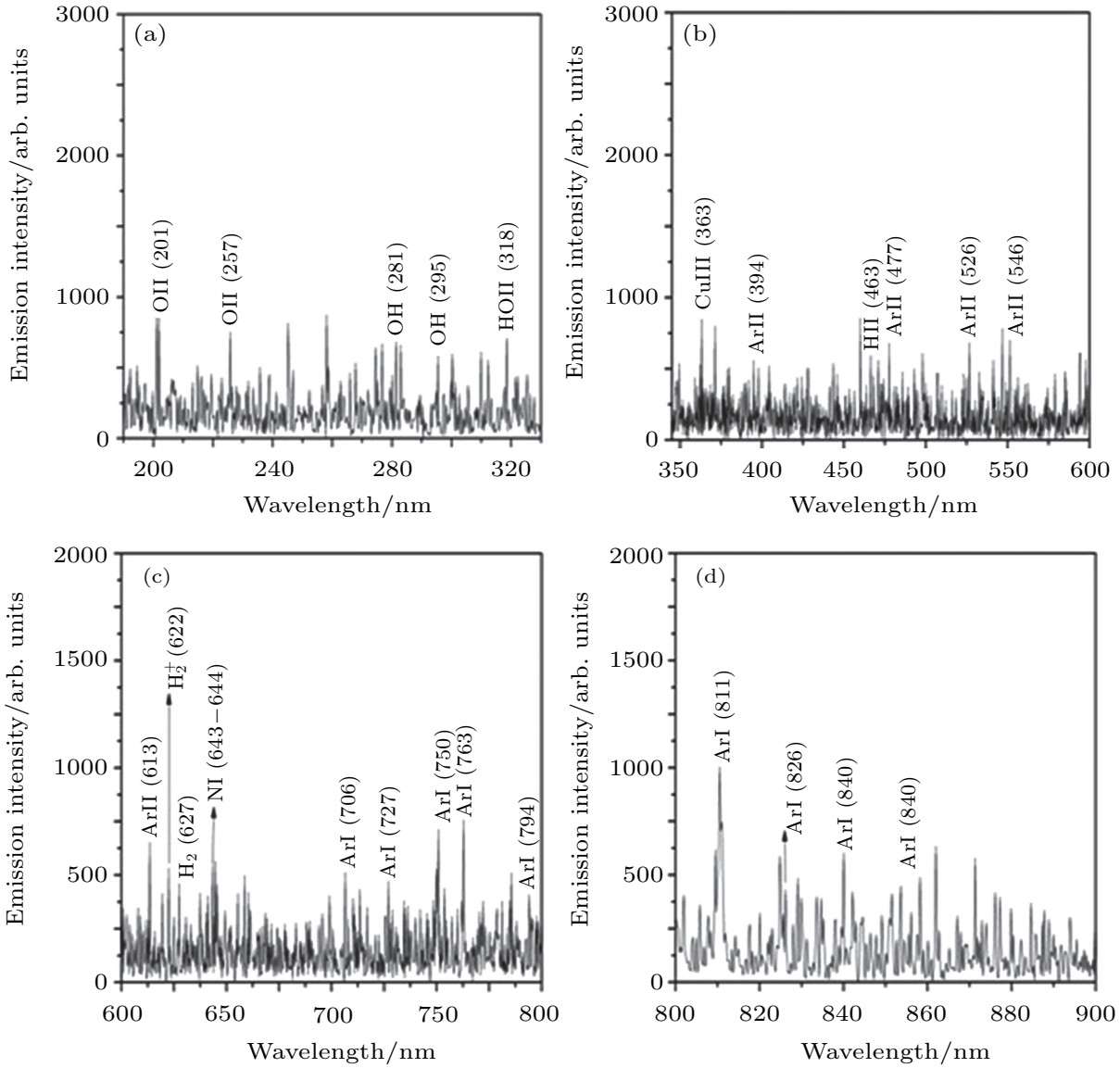


Fig. 17. Optical emission spectra of argon plasma formed in pin-to-pin configuration in the nitrate solution.

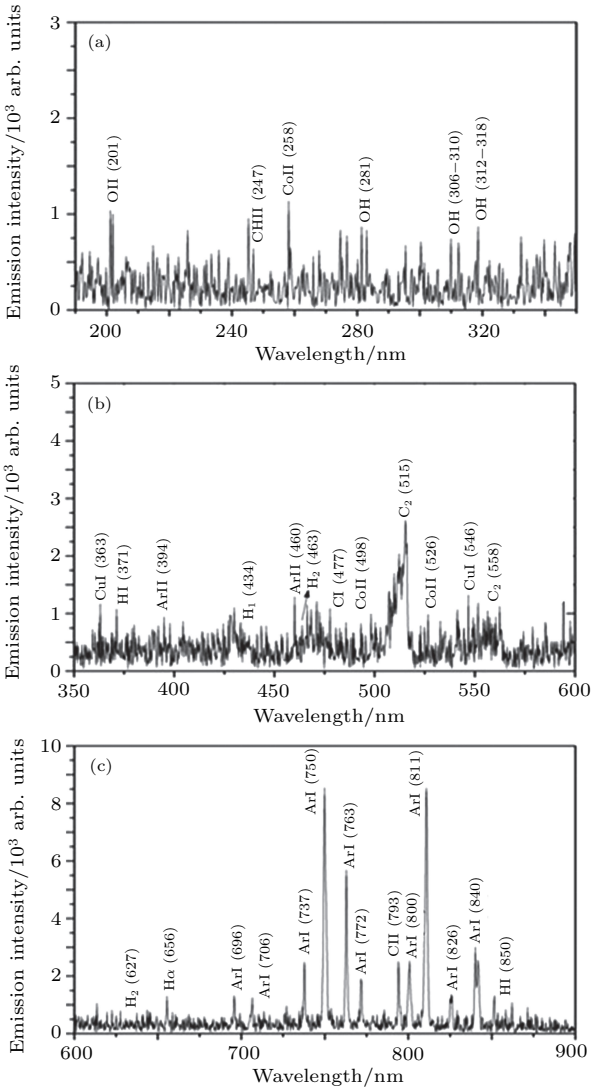


Fig. 18. Optical emission spectra of argon/methane plasma formed in pin-to-pin configuration in the nitrate solution.

4. Conclusion

In this paper, the effect of non-thermal plasma on nitrate solution was investigated. The atmospheric pressure plasma was produced in a pin-to-pin configuration of the electrodes, where one of the electrodes was immersed in the nitrate solution and the other was set over the water surface. The applied voltage was 0 kV–15 kV at 27 kHz. Ion chromatography measurements revealed that the air discharge on nitrate solution increases nitrate concentration by 74%, while Ar and Ar/CH4 discharges, change nitrate concentration by less than 5%. The former was attributed to the extra nitrate production via chemical reactions initiated in the discharge channels over the nitrate solution. On the other hand, spectroscopic measurements showed that air discharge on the nitrate solution yield nitric acid in the solution. This effect could be a great challenge in water purification processes by such plasmas. Solution pH decreased from 5.3 to 2.2 after 15-min running air plasma while it was increased from 5.3 to 6.5 by Ar and Ar/CH4 discharges. The former can be attributed to HNO_2, HNO_3, and H^+ products and the latter to the OH^- ions. The conductivity of the solution was increased for all discharges that we used. In the case of air discharge, the change in conductivity was three orders of magnitudes while it increased to twice its initial value for the Ar and Ar/CH4 discharges. The results of this study can be useful in water purification by plasmas, and nitrate remediation in drinking water by such discharges.

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