Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2022.28429

258 (2022) 143–152
May

Experimental study on the treatment of polyacrylamide wastewater by non-equilibrium plasma

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Received 17 June 2021; Accepted 24 March 2022

ABSTRACT

While polymer flooding improves oilfield recovery, it also produces a large amount of polyacrylamide wastewater (PAM). Polyacrylamide wastewater has the characteristics of a high degree of emulsification, high viscosity, and difficulty degrading. The treatment of polymer-containing sewage has become a research hotspot. In this paper, based on the dielectric barrier discharge (DBD) experiment, the effect of non-equilibrium plasma on the degradation rate and viscosity of PAM solution was studied by changing the discharge time, discharge voltage, and PAM solution concentration. At the same time, the environmental scanning electron microscopy was used to observe the PAM before and after discharge. As the morphology of the solution changed, the degradation products of PAM solution after discharge were detected by Fourier-transform infrared absorption spectrometer. The results show that under the conditions of a discharge voltage of 20 kV and a frequency of 10 kHz, the degradation rate of DBD to a PAM solution with a concentration of 1,000 mg/L can reach up to 89%. DBD can cause the PAM molecular chain to curl up and reduce its viscosity rapidly. DBD is produced as the active substance first broke the PAM macromolecular chain into small molecules. Further treatment can produce acrylamide monomer and acrylic acid.

Keywords: Non-equilibrium plasma; Dielectric barrier discharge; Polyacrylamide; Degradation rate; Viscosity; Environmental scanning electron microscopy; Fourier-transform infrared spectroscopy

1. Introduction

When polymer flooding improves oil recovery, lots of polyacrylamide (PAM) wastewater will be produced [1,2]. Direct discharge of untreated polyacrylamide wastewater will cause significant damage to the ecological environment [3,4]. Therefore, the treatment of polyacrylamide wastewater has become a research hotspot. Traditional treatment methods mainly include physical, chemical, and biological methods. Gao [5] successfully managed to achieve degradation of PAM by changing the type of peroxide and increasing the degradation temperature. Yen and Yang [6] studied the degradation of PAM using a ultrasound-assisted method under different irradiation time, reaction temperatures, and solution concentrations by using the viscosity method. Wen et al. [7] isolated two strains of PAM-degrading bacteria in the soil of an oil field for green degradation of polyacrylamide. However, with the increasing molecular weight of PAM used for

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polymer driving [8], traditional degradation methods can no longer meet degradation requirements. Therefore, many scholars have improved the degradation rate of polymer-containing wastewater by multi-stage treatment. Pi et al. [9] pretreated polymer-containing wastewater using the Fenton method, and then poured the pretreated polymer-containing wastewater into the anaerobic reactor (ABR) with the degrading strain, in which way degradation rate could reach up to 91.08%. Wang et al. [10] found that CFAc/Fenton/ultrasonic process could efficiently improve degradation of polymer-containing wastewater with a rate of 93%.

Dielectric barrier discharge (DBD), as an advanced oxidation method, has the advantage of being green and efficient compared with traditional degradation methods, so it is widely used in pollutant treatment [11,12]. Its principle is to use a high voltage electric field to push electrons to collide with background gas particles and generate active substances with high chemical activity, such as free radicals, high-energy electrons, excited state particles, and photons [13,14]. These active substances further degrade pollutants through chain-chemical reactions and eventually generate H_2O, CO_2, and other products [15,16]. Kinandana et al. [17] studied the changes in solution temperature, PH, O_3, and H_2O_2 concentration after DBD degradation of PAM through varied DBD air gap widths. Rong et al. [18] investigated the effects of non-equilibrium plasma on PAM degradation rates by varying initial pH, solution concentration, discharge time, and discharge voltage of PAM wastewater. Song et al. [19] rapidly decomposed PAM by a novel advanced oxidation process based on gas–liquid interface plasma and proved that $^{\bullet}$OH plays an important role in PAM degradation by adding $^{\bullet}$OH scavengers.

The above studies show that the degradation efficiency of physical and biological methods is relatively low, while the chemical method centered with Fenton has a high degradation rate of PAM. However, the degradation process was easy to form iron sludge [20], causing secondary pollution. Though environmental friendly and of high efficiency, degradation of PAM via DBD lack further investigation on the relationship between the degradation rate, viscosity, and its microscopic morphology.

In this paper, based on the DBD experiment, the influence of non-equilibrium plasma on the degradation rate of PAM solution was studied by changing the concentration, discharge time, and discharge voltage of the PAM solution. The viscosity of PAM solution before and after discharge was measured by rotary viscometer, and the morphology of PAM solution before and after discharge was observed through environmental scanning electron microscopy (ESEM). The composition of PAM solution after discharge was detected using Fourier-transform infrared absorption spectrometer (FTIR), along with the degradation products of PAM solution, which provided a basis for the application of PAM solution treated by DBD.

2. Experimental equipment and methods for DBD treatment of PAM solution

2.1. Experiment apparatus

The schematic diagram of the experimental equipment for DBD treatment of the PAM solution is shown in Fig. 1. The quartz reactor was designed to equip a double-layered trough structure, in which the wall was 3 mm thick, the diameter of the quartz reactor was 126 mm, the width of the cooling water tank was 15 mm, the diameter of the inlet and outlet for air and cooling water was 5 mm, and the stainless steel electrode was 2 mm. The air flow rate was set at 0.02 m^3/s, the cooling water flow rate was set at about 8.5×10^{-6} m^3/s, and the magnetic stirrer speed was set at 30 rpm.

2.2. Experimental steps

To study the effect of non-equilibrium plasma treated by DBD on PAM solution, the experimental procedure was divided into the following 6 steps:

(1) A balance was used to measure PAM with masses of 1,000; 2,000; 3,000; 4,000, and 5,000 mg, with 1 L of deionized water poured into a beaker. When the stirrer speed

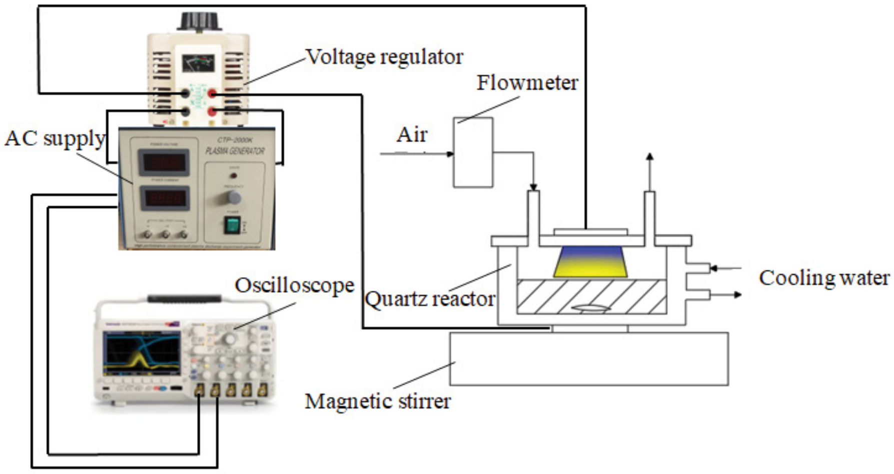


Fig. 1. Diagram of experimental equipment for DBD degradation of PAM solution.

was adjusted to 200 rpm, PAM was slowly poured into deionized water, and then stirred for 2 h at a speed of 800 rpm. After the stirring, the solution was stored at room temperature for 24 h;

(2) The absorbance of PAM solution was measured according to China National Petroleum Corporation Standard Q/SY DQ0928-2017, and a standard curve of absorbance and solution concentration was depicted, as shown in Fig. 2. When the concentration of the PAM solution exceeded 300 mg/L, the PAM solution was diluted N times with deionized water until the concentration of the solution was less than 300 mg/L, and then measured according to the standard. The fitting equation of the standard curve was:

$$y = \left(1{,}112.4x + 9.1\right)N \tag{1}$$

where y was the concentration of the PAM solution, x was the absorbance, and N was the dilution times of the solution.

(3) The DBD device was filled with 150 mL configured PAM solution with the stirring speed of 30 rpm. The AC power supply voltage was adjusted to 16, 17, 18, 19, and 20 kV with 10 kHz. The discharge time was set as 0, 60, 120, 180, 240, and 300 min;

(4) The solution was poured out after the discharge was completed and stood to cool to room temperature. The absorbance of the PAM solution after the reaction was measured according to the standard. Eqs. (2) and (1) was substituted to obtain the concentration of the PAM solution after discharge and calculate its degradation rate:

$$\omega_{\text{COD}} = \frac{\omega_1 - \omega_2}{\omega_1} \times 100\% \tag{2}$$

where ω_1 was the concentration of PAM solution before discharge, and ω_2 was the concentration of PAM solution after discharge;

(5) The relationship between shear rate and viscosity of PAM solution was obtained by measuring PAM solution before and after discharge with a rotary viscometer.

(6) The prepared PAM hydrogel was coated thin and fixed with 2% glutaraldehyde for more than 4 h. After the samples were washed, dehydrated, dried, pasted, and sprayed with gold, they were placed under an environmental scanning electron microscope to photograph the morphology of the PAM solution before and after the discharge reaction.

(7) The PAM solution before and after discharge was evenly smeared on the surface of the prepared KBr sheet and placed in an infrared spectrometer for detection.

2.3. Principle of the experiment

DBD can produce a variety of active substances in the process of non-equilibrium plasma, including hydroxyl radical ($^{\bullet}OH$), active oxygen (O), and hydrogen peroxide (H_2O_2).

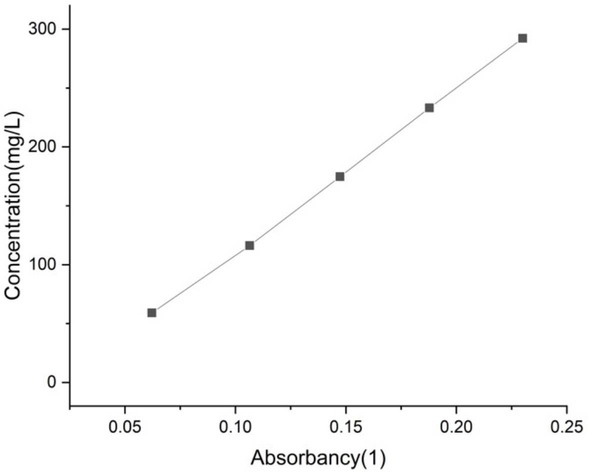


Fig. 2. Standard curve of polyacrylamide concentration.

$^{\bullet}OH$ redox, as a highly efficient and strong oxidation particle, can charge up to 2.8 eV, and ignore water molecules and directly react with pollutants. In the DBD process, the collision between electrons and water molecules will lead to dissociation, ionization, and vibration/rotational excitation of water molecules, resulting in $^{\bullet}OH$ production [21]. The specific reactions are as follows:

$$H_2O + e \rightarrow {}^{\bullet}OH + {}^{\bullet}H + e \tag{3}$$

$$H_2O + e \rightarrow H_2O^{+} + 2e \tag{4}$$

$$H_2O + e \rightarrow H_2O^{\bullet} + e \tag{5}$$

In the DBD process, the joint action of electrons, oxygen molecules, and oxygen atoms will produce O_3. The reaction of high-energy oxygen atoms with water molecules will also generate H_2O_2 [22,23], and the specific reaction is as follows:

$$e + O_2 \rightarrow O + O + e \tag{6}$$

$$O + O_2 \rightarrow O_3 \tag{7}$$

$$e + O_2 \rightarrow O_2^{\bullet} + e \tag{8}$$

$$O_2^{\bullet} + O_2 \rightarrow O + O_3 \tag{9}$$

$$e + O_3 \rightarrow O + O_2 + e \tag{10}$$

$$e + H_2O \rightarrow {}^{\bullet}OH + {}^{\bullet}H \tag{11}$$

$$^{\bullet}OH + {}^{\bullet}OH \rightarrow H_2O_2 \tag{12}$$

$$O + H_2O \rightarrow {}^{\bullet}OH + {}^{\bullet}OH \tag{13}$$

$$^{\bullet}OH + H_2O \rightarrow H_2O + {}^{\bullet}HO_2 \tag{14}$$

3. Results and discussion

3.1. Study on the influence of different PAM solution concentrations on the degradation rate of PAM solution

Under the condition of an electric voltage of 18 kV and a frequency of 10 kHz, the PAM solution with concentrations of 1,000; 2,000; 3,000; 4,000 and 5,000 mg/L were all discharged for 0, 60, 120, 180, 240 and 300 min. The degradation rates of PAM solution before and after discharge were obtained from Eqs. (1) and (2) in which the absorbances were measured under standard conditions. The software Origin was used to depict the relationship between the concentration of different PAM solutions and the degradation rate of PAM solution, as shown in Fig. 3.

It can be seen from Fig. 3 that as the discharge time increases, the degradation rate of PAM solution gradually increased and then tended to be flat. For a 1,000 mg/L PAM solution, the degradation rate can rise up to 83% after 300 min of discharge, and decreases gradually with further increase in concentration of PAM solution. This is because active substances such as free radicals generated by DBD reacted with PAM to break the molecular chain, and finally generated water and other inorganic substances, which speeded up the degradation rate of the solution. In this case, the number of PAM molecules per unit volume increased but the content of free radicals remained unchanged, and the reduction of the two reactions resulted in a decrease in the degradation rate of PAM.

3.2. Research on the influence of different discharge voltages on the degradation rate of PAM solution

Under the condition of an electric voltage of 16, 17, 18, 19, and 20 kV and a frequency of 10 kHz, the PAM solutions with concentrations of 1,000 mg/L were discharged for 0, 60, 120, 180, 240 and 300 min. The degradation rates of PAM solution before and after discharge were obtained from Eqs. (1) and (2) in which the absorbances were measured under standard conditions, The software origin was used to illustrate the relationship between the DBD discharge voltage and the degradation rate of the PAM solution, as shown in Fig. 4.

Based on the DBD equipment in this experiment with the discharge air gap of 6mm and the airflow rate of 0.02 m^3/s, the initial voltage was set at 16 kV since the air cannot be stably broken down to form a non-equilibrium plasma when the discharge voltage was less than 16 kV. Only a small amount of purple arc would be generated when the voltage intensity breaks through the air under the discharge voltage of 16 kV. The relatively low non-equilibrium plasma intensity led to the overall low degradation rate of the PAM solution. As the discharge voltage rose, the width of streamer channels in the discharge air gap increases significantly, and the electrons reacted actively with air and water, which can produce more active substances, thereby improving the degradation rate of the PAM solution.

3.3. Research on the influence of different PAM solution concentration and discharge voltage on the viscosity of PAM solution

When the PAM solutions with concentrations of 1,000; 2,000; 3,000; 4,000 and 5,000 mg/L were discharged at 0, 5, 10, 15, 20, 25 and 30 min, and shear rates were measured respectively with the rotational viscometer. Under the condition of an electric voltage of 16, 17, 18, 19, and 20 kV and a frequency of 10 kHz, the PAM solution with concentrations of 1,000 mg/L was discharged for 0, 5, 10, 15, 20, 25, and 30 min. The corresponding shear rate was measured with a rotary viscometer. The relationship between shear rate and PAM solution viscosity was obtained, as shown in Fig. 6.

It can be seen from Figs. 5 and 6 that the viscosity of PAM solution decreases with increased discharge time. Combining with Figs. 3 and 4, it can be seen that the degradation rate of PAM solution was very low at this time, which can rule out the viscosity reduction caused by PAM degradation. Therefore, the main reason for the viscosity decrease of PAM solution was the change of PAM

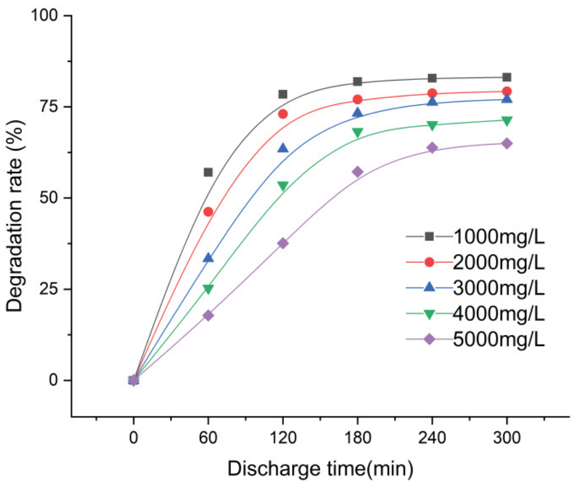


Fig. 3. The relationship between the concentration of different PAM solutions and the degradation rate of PAM solutions.

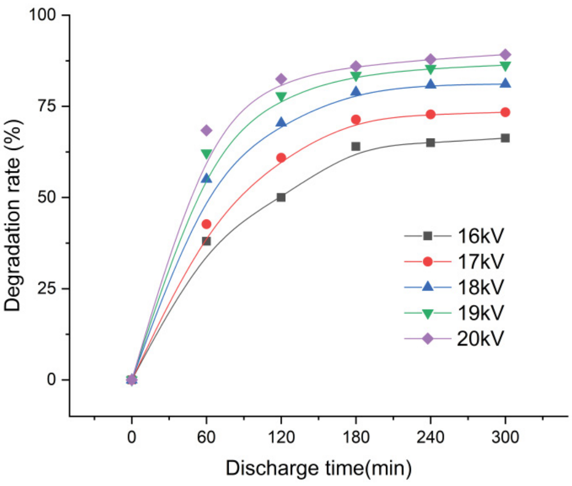


Fig. 4. Relationship between different discharge voltage and degradation rate of PAM solution.

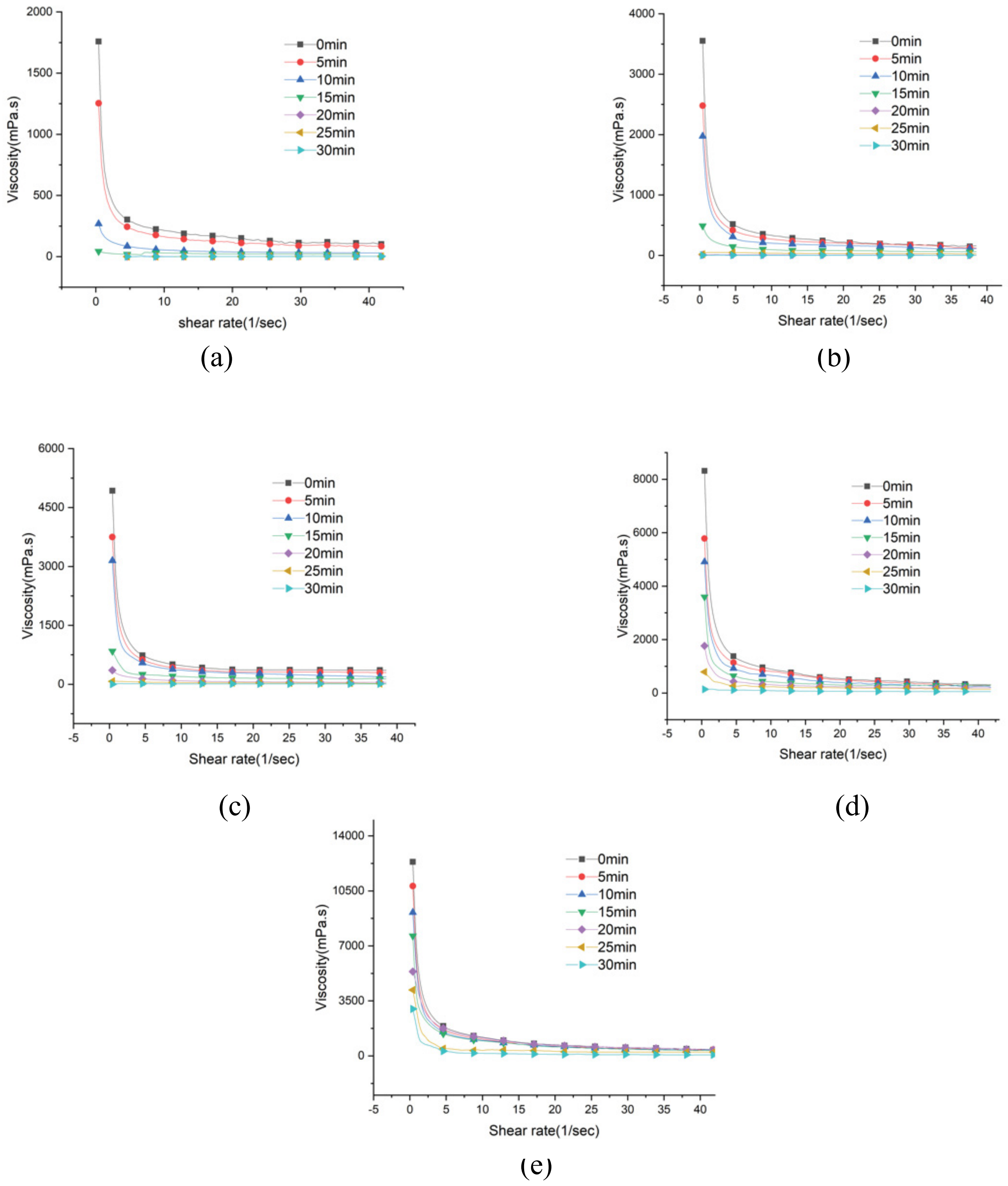
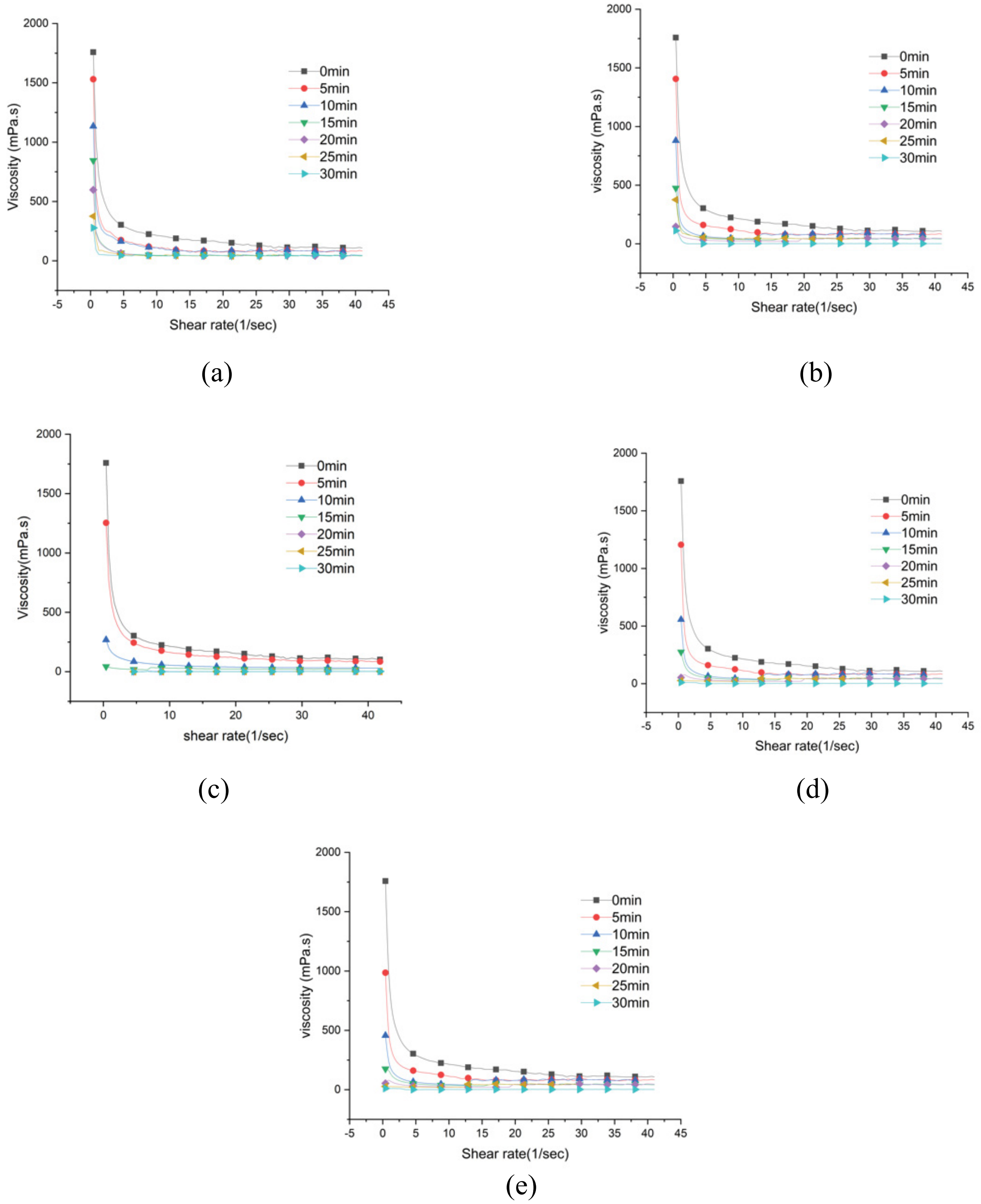


Fig. 5. The relationship between the concentration of different PAM solutions and the viscosity of PAM solutions: (a) 1,000 mg/L, (b) 2,000 mg/L, (c) 3,000 mg/L, (d) 4,000 mg/L, and (e) 5,000 mg/L.

molecular chain shape: The long-chain stretching from the line became the ball shrinking and curling ones [24]. Because PAM contained a large number of negative groups, they repelled each other and resulted in a extended state of the macromolecules which were longer and the viscosity were higher. However, the DBD process produced a large number of positively charged particles, so that PAM molecule around negative bases would form a double electric layer, weakening the repulsion between the negative base, turning PAM long chain into the crimped state, resulting



(a) (b) (c) (d) (e)

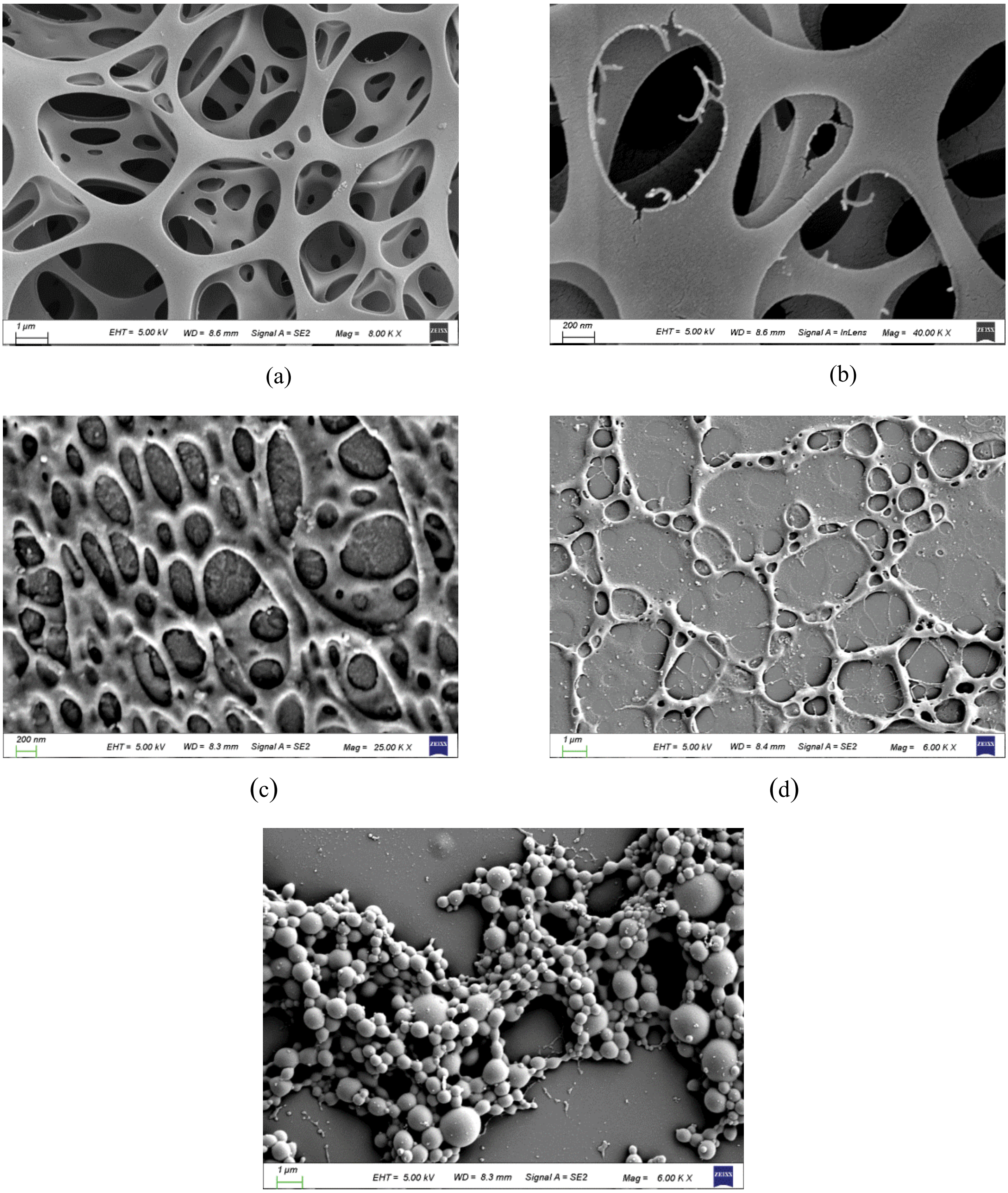
Fig. 6. Relationship between different discharge voltage and PAM solution viscosity: (a) 16 kV, (b) 17 kV, (c) 18 kV, (d) 19 kV, and (e) 20 kV.

in declined PAM solution viscosity, and diluting PAM solutions.

3.4. Study on the effect of DBD on the morphology of PAM in PAM solution

To study the changes in the morphology of the PAM solution before and after DBD, the prepared PAM hydrogel was spread into a thin layer, fixed with 2% glutaraldehyde for more than 4 h, and then washed with PBS with a concentration of 0.1 mol/L and a pH of 7.2 After dehydration with different concentration gradients of ethanol, the samples were dried, glued and sprayed in the LEICA CPD300 automatic critical point dryer, and finally placed under the Germini SEM300 field emission scanning electron microscope for observation and shooting. The resulting picture is shown in Fig. 7.

As can be seen from Fig. 7a, water molecules entered into the interspace of the crimped PAM molecular chain, resulting in stretched PAM molecular chain and assembling a network structure. Fig. 7b shows viscosity of PAM solution was very low with slightly damaged PAM structure due to DBD-induced oxides. Hence the viscosity of PAM solution decreased not because of PAM macromolecular chain fracture, but because of



(a) (b)

(c) (d)

(e)

Fig. 7. The morphology of the polyacrylamide solution after the discharge of different time: (a) 0 min, (b) 30 min, (c) 60 min, (d) 180 min, and (e) 300 min.

PAM molecular chain curls. Fig. 7c and d show that prolonged discharge time can effectively destroy the PAM network structure, and a large number of small molecules of PAM are shown to be free near the network structure, and further treatment can produce acrylamide monomer [25]. Fig. 7e shows that most of PAM completely degrades into water and other inorganic substances, but a small amount of PAM remains and curls into balls.

3.5. Analysis of degradation products of PAM solution before and after DBD

To study the degradation products of the PAM solution before and after DBD, FTIR was used to analyze the PAM solution at 0 and 300 min. The results are shown in Fig. 8.

In Fig. 8, Fig. 8a shows the FTIR of PAM solution at 0 min of discharge, in which 3,336.72 and 3,187.76 cm^{-1} are the symmetric and antisymmetric stretching vibration absorption peaks of $–NH_2$, and 2,930 cm^{-1} is the stretching vibration peak of saturated C–H, 1,651 cm^{-1} is presumed to be the C=O symmetrical stretching vibration absorption peak in the amide group, 1,448 cm^{-1} is the stretching vibration absorption peak of free $–NH_2$ and 1,120 cm^{-1} is the stretching vibration absorption peak of C–N. Fig. 8b shows the FTIR of PAM solution at 300 min discharge, in which 3,465.31 cm^{-1} is the stretching vibration absorption peak of $–NH_2$, 1,637.09 cm^{-1} is the C=O symmetrical stretching vibration absorption peak, and 1,410.20 cm^{-1} is the stretching vibration absorption peak of free $–NH_2$.

By comparing Fig. 8a and b, it can be seen that after 300 min of discharge, the vibration absorption peak of $–NH_2$ changes from double peaks to single peaks, the antisymmetric stretching vibration of $–NH_2$ disappears, and the absorption peak of $–NH_2$ contraction vibration in Fig. 8b becomes wider, inferred to be the amide group degradation and its degradation product O–H interference. The peak at 1,651 cm^{-1} was inferred from the C=O in the amide group, while in Fig. 8b 1,637.09 cm^{-1} was the C=O symmetrical stretching vibration absorption peak, which was inferred from the free carbonyl C=O generated by the degradation of $–NH_2$ in the amide group and the formation of carboxylic acid by O–H in the solution. From 1,500 to

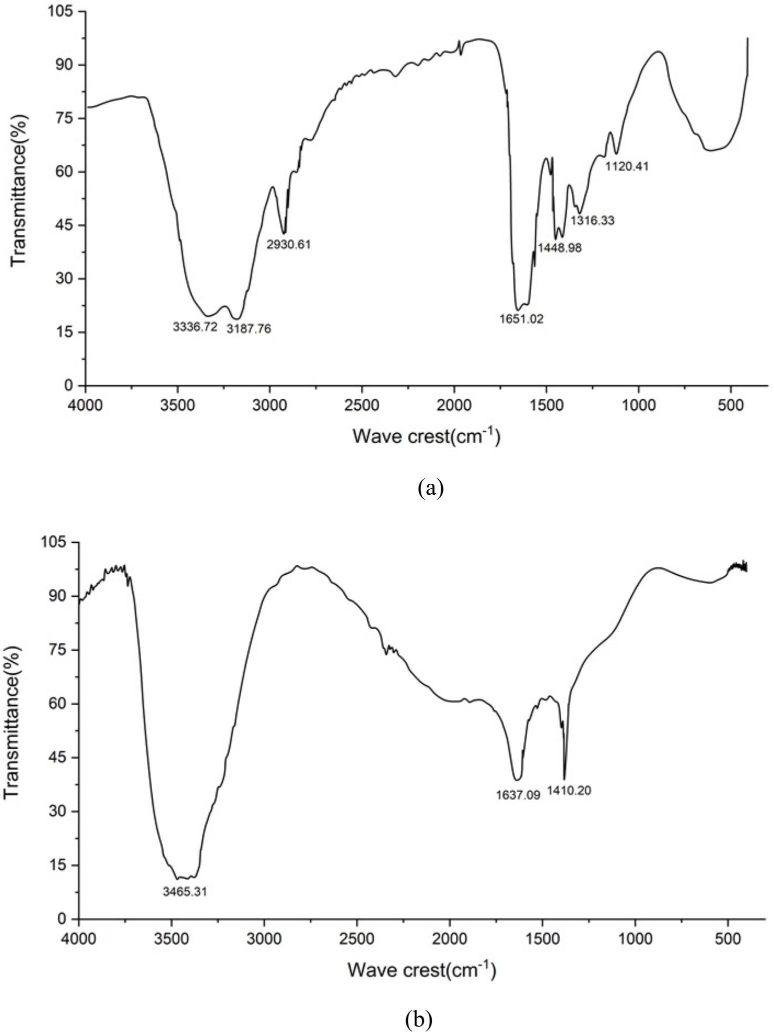


Fig. 8. FTIR diagram of PAM solution before and after DBD: (a) 0 min and (b) 300 min.

1,000 cm^{-1}, the types of peaks shown in Fig. 8b were significantly reduced. It was inferred that most of the PAM were completely degraded to form inorganic substances apart from only a small part of PAM and its degradation products remained.

4. Conclusion

This paper used DBD to generate plasma to degrade PAM solution to study the effects of different PAM solution concentrations, different discharge voltages, and different times on the degradation rate and viscosity of PAM solution. At the same time, the PAM morphology before and after the DBD reaction was obtained by ESEM. After the DBD reaction was obtained by FTIR and the degradation products of PAM solution, and the following conclusions was obtained:

(1) The higher the concentration of PAM solution, the more PAM molecules per unit volume, resulting decreased the degradation efficiency of non-equilibrium plasma.
(2) Increasing the discharge voltage can increase the electron collision rate and promote the production of strong oxidizing actives, thereby increasing the degradation rate of PAM solution.
(3) When DBD for 30 min, the reason for the rapid decrease in PAM solution viscosity was not the degradation of PAM solution, but the large number of positively charged particles generated during the DBD process, which caused the formation of an electric double layer around the negatively charged base of the PAM molecule, weakening the negatively charged base. The repulsive force between the PAM transformed the long chain of PAM into a curled state, which contributed to decreased viscosity of the PAM.
(4) Combining ESEM and FTIR, it can be inferred that the active components produced by DBD first attacked the carbon double bonds, hydroxyl, and amino groups of PAM, breaking long-chain PAM to form short-chain PAM, and gradually degraded to acrylamide and acrylic acid. Extending the discharge time can further degrade small-molecule organic matter into water and other inorganic matters.

Acknowledgements

This work was jointly supported by the Open Project of the Key Laboratory of Enhanced Oil and Gas Recovery of the Ministry of Education (NEPU-EOR-2021-01), the National Natural Science Foundation of China "Research on the Mechanism of Condensation Process Induced by Supersaturated Steam Non-equilibrium Plasma" (No.51706023), and Postdoctoral Research Fund of Heilongjiang Province (No. LBH-Z20118).

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