

DBD plasma reactor for the treatment of crystalline violet simulated dye wastewater

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Received 7 June 2023; Accepted 15 September 2023

ABSTRACT

In this research paper, the degradation performance of crystalline violet (CV) solution was investigated by DBD plasma reactor under different initial conditions. Through change of the different solution concentrations, different discharge powers, different initial pH of the solution and different atmospheric conditions. The best single-factor data processing conditions affected the degradation of the solution were determined by measuring the absorbance of CV at 584 nm. The degradation mechanism of CV was proposed. The results showed that DBD plasma can effectively remove the chromaticity of CV solution, and this technology provided a reference for the treatment of triphenylmethane type dye wastewater.

Keywords: Dielectric blocking discharge; Plasma; Dye wastewater; Crystalline violet

1. Introduction

Recent years, water, like rare metals, is becoming a precious and limited resource. According to the United Nations, 3 billion people worldwide will face severe water shortage by the middle of this century [1]. According to statistics, the top four wastewater discharge industries were the design, development and processing of chemical-based products, the paper industry, the textile industry, and the coal mining industry [2]. Dyestuffs as a chemical raw material are widely used in the textile industry, and a large percentage of wastewater is generated during their processing. The composition of wastewater is highly complex, containing dyes, auxiliaries, oil agents, surfactants, fiber impurities, acids and bases, various textile slurries and inorganic salts, etc [3]. Most of these dyes are toxic to human health and the environment,

and release in environment can cause environmental problems [4]. It is difficult to degrade by conventional physical (adsorption [5], extraction and membrane separation techniques [6], etc.), chemical (acid–base neutralization, ion exchange [7], electrochemical and advanced oxidation [8], etc.) and biological water treatment methods. If the undegraded dye wastewater discharged directly into environment, it will cause irreparable pollution and harm to the ecological environment.

Triphenylmethane dyes are the third largest class of dyes after azo and anthraquinone. It has specific color, complex structure and difficult to break the bond degradation. It has potential toxic; but it has low price, high color and good dyeing effect, so it was widely used in the textile industry. Especially, crystalline violet (CV) is the most widely used triphenylmethane dye, which produces toxic metabolites

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due to chemical reactions and may have toxic or poisonous effects on the ecological environment and human health [9]. So, the degradation treatment of CV dye wastewater was studied in this research.

With the rapid development of science and technology, advanced oxidation processes [10] (AOPs), characterized by the generation of hydroxyl radicals ($\cdot\text{OH}$) with strong oxidizing power, have attracted much attention. And DBD low-temperature plasma technology (NTP) [11,12], as an emerging advanced oxidation technology, which can generate strong oxidizing substances such as high-energy electron groups, hydroxyl radicals ($\cdot\text{OH}$), and ozone (O_3) [13]. All those oxidizing substances can cause ring-opening and bond-breaking of substances in CV dye wastewater. Through DBD low temperature plasma technology (NTP), it can degrade large complex organic compounds into small molecules, thus improving the biochemical degradability of wastewater and even making it directly mineralized [14]. Four methods are commonly used to generate low-temperature plasma: glow discharge [15–17], corona discharge [18,19], sliding arc discharge [20,21] and dielectric barrier discharge [22–25].

In this study, DBD plasma reactor was used to treat CV solution as a simulated dye wastewater. It obtained the best single factor data treatment effect affecting the solution degradation, explored the degradation mechanism of CV and provided reference for the treatment of triphenylmethane type dye wastewater.

This research was conducted on a common triphenylmethane-based dye, C.I. Basic Violet 3 (also known as CV). CV is the most typical triphenylmethane type of dye, which has toxic or poisonous effects on ecology and human health due to chemical reactions such as oxidation and weathering. The CV molecule has a carbon atom connected to three benzene ring structures at its centre, which makes the degradation of this type of wastewater is difficult. The structural formula of CV is shown in Fig. 1.

2. Materials and methods

2.1. Materials

High purity of CV reagent was purchased from Sinopharm Chemical Reagent Co. High purity of HCl

reagent was purchased from Beijing Chemical Plant, China. High purity of NaOH reagent was purchased from Chengdu Kelong Chemical Reagent Plant. High purity of N_2 and O_2 were purchased from Lishui Industrial Gas Manufacturing Co.

2.2. Methods

2.2.1. Experiment methods

In this experiment, the DBD reactor is a concentric tube structure (Fig. 2), the inner and outer round tubes of the reactor are made of quartz glass, and the reaction is carried out at room temperature and pressure. The outer tube has an outer diameter of 25 mm and an inner diameter of 20 mm, with a wall thickness of 2.5 mm. The inner tube has an outer diameter of 8 mm and an inner diameter of 5.5 mm, with a metal bar in the inner tube. At the same time, the air gap between the inner and outer tubes is 6 mm. The concentric tube is fixed vertically by the external iron frame, the two iron plates above and below the iron frame are used to conduct electricity. The external tube is wrapped with a layer of stainless-steel mesh, and the discharge interval is 200 mm. During the research, high pressure is applied to the metal mesh electrode on the outer tube and low pressure is applied to the metal rod of the inner tube. During the discharge, it is passed through a laboratory peristaltic pump to simulate dye wastewater into the inner and outer tube gap of the reactor. And the liquid can react with the plasma when it flows through the discharge interval. The strong oxidizing substances such as high-energy electron groups, hydroxyl radicals ($\cdot\text{OH}$), and ozone (O_3) is generated, which can degradation the CV solution. The pulse frequency set for the power supply in this research was 200 Hz, and the peristaltic pump was set at a flow rate of 120 mL/min.

Modula table pulsed low temperature plasma experimental power supply from Nanjing Suman Plasma Technology Co., Nanjing, China. Its voltage and current can be adjusted and detected in real time.

The instruments and equipment used in this research, as well as the specific models and manufacturers of the relevant instruments, are shown in Table 1.

2.2.2. Analysis method

UV-Vis Spectrophotometer was calibrated in intensity and wavelength before used. The intensity was calibrated by the standard guaranteed reagent 0.1 mol/L NaCl. Place the NaCl solution into the sample cell, scan the absorbance within the spectral range, and record the absorbance value. Calculate the concentration of the standard substance based on its molar absorption coefficient. Adjust the instrument until the absorbance of the standard substance matches its known concentration. Wavelength calibrated used 486.02 nm of deuterium lamp.

The CV solution was scanned and measured in the full waveband from 200 to 650 nm by UV-Vis Spectrophotometer, as shown in Fig. 3. The characteristic absorption peak of CV solution was 584 nm according to the scanned measurement data. So, the standard curve of CV solution at the absorption wavelength of 584 nm was plotted in this research.

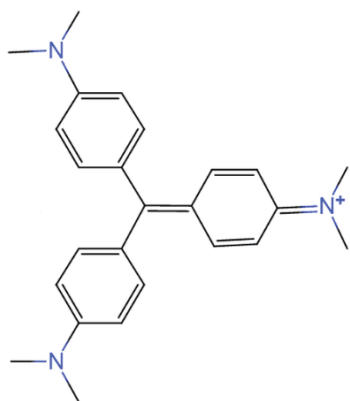


Fig. 1. Chemical formula for crystalline violet.

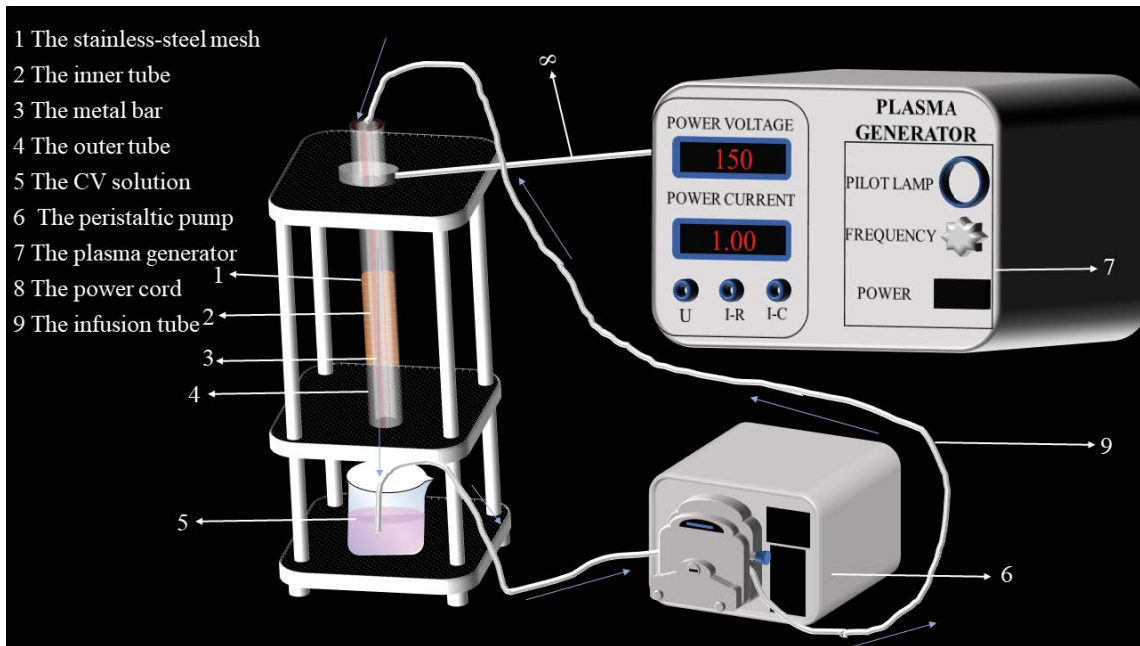


Fig. 2. Dielectric barrier discharge concentric tubular plasma processing device, plasma reactors, peristaltic pumps.

Table 1
Experimental instruments

Equipment	Model	Manufacturer
Mortar	70 mm	Shanghai Lichen Bonsey Instrument Technology Co., Shanghai, China
Analytical scales	EX2242H	Osho, USA
Ultrasonic cleaning machine	JP-010T	Shenzhen Clean Union Cleaning Equipment Co., Shenzhen, China
Laboratory ultrapure water system	Plus-E3 TS	Nanjing Puyida Technology Development Co., Nanjing, China
Ultraviolet spectrophotometer	UV754N	Shanghai Aoan Scientific Instruments Co., Shanghai, China
Modulated pulsed low-temperature plasma experimental power supply	CTP-2000K/P	Nanjing Suman Plasma Technology Co., Nanjing, China
Peristaltic pumps	LLS Plus Kamoer	Fluid Technology Co.
pH meter	Ray Magnet PHS-3C	Shanghai Yidian Scientific Instruments Co., Shanghai, China
Dissolved oxygen meter	HQ30D	Hach, USA

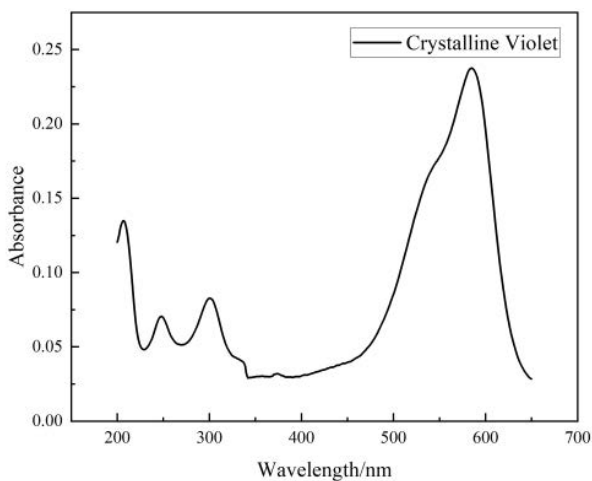


Fig. 3. Crystalline violet absorption spectrum (5 mg/L).

Different concentrations of CV solution were measured by UV-Vis Spectrophotometer at 584 nm, then plotted the standard curve, as shown in Fig. 4.

The standard curve equation was obtained from the standard curve of CV solution at 584 nm.

The absorbance value measured in the experiment was applied to the standard curve equation to obtain the concentration of CV solution, c_v and the removal rate of CV, η , was calculated.

3. Results and discussion

3.1. Effect of different initial concentrations on the degradation rate of CV solution

Under the conditions of 150 W discharge power, pH = 7 and 500 mL the initial concentrations of CV solution were 5, 9, 13 and 17 mg/L. Under different initial concentrations, the absorbance of the treated CV solution at 584 nm

with the time variation is shown in Fig. 5a. The degradation rate curves at 584 nm with time is shown in Fig. 5b. The color change of different initial concentrations of CV solution with the increase of discharge treatment time is shown in Fig. 6.

From Fig. 5a, it can be seen that the higher initial concentration of CV solution the longer times it needed to attain the same degradation rate under the same discharge power and the same initial pH. From Fig. 5b, it can be seen that the degradation rates of CV solutions with different concentrations of 5, 9, 13, and 17 mg/L were 99.64%, 98.36%, 97.36%, and 97.18% after 15 min of discharge treatment, respectively.

From Fig. 6 it can be seen that under the same discharge power and the same initial pH value, the color of 5 mg/L of CV solution changed from dark violet to transparent only after 3~5 min of discharge treatment, while the CV solution of 9 mg/L required 6~7 min and the solution of 17 mg/L required more than 10 min. The conclusion was that the higher l concentration, the longer the time required for the

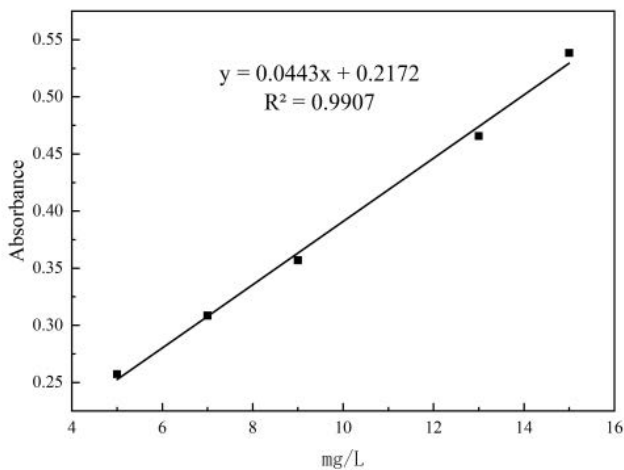


Fig. 4. Crystalline violet standard curve (584 nm).

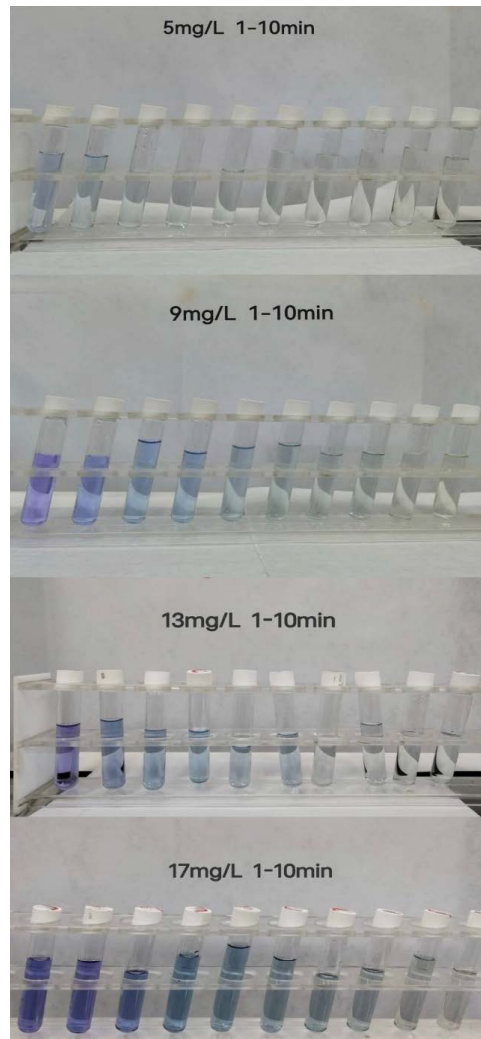


Fig. 6. Color change of crystalline violet solution after degradation under different initial concentration conditions at pH = 7 discharge power 150 W.

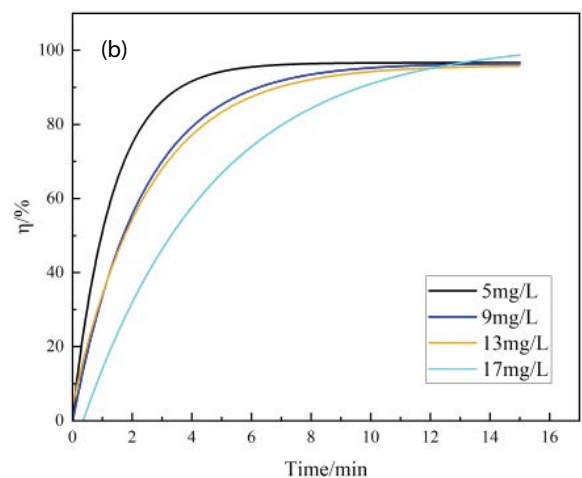
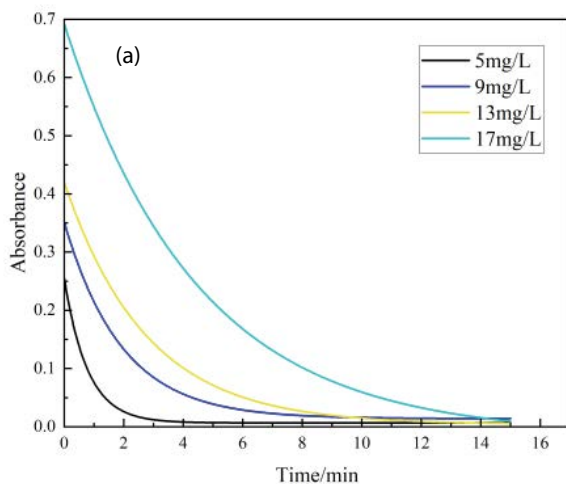


Fig. 5. Variation curve at 584 nm after degradation of crystalline violet solution at pH = 7 with different initial concentrations of discharge power 150 W. (a) The absorbance change curve and (b) the degradation rate change curve.

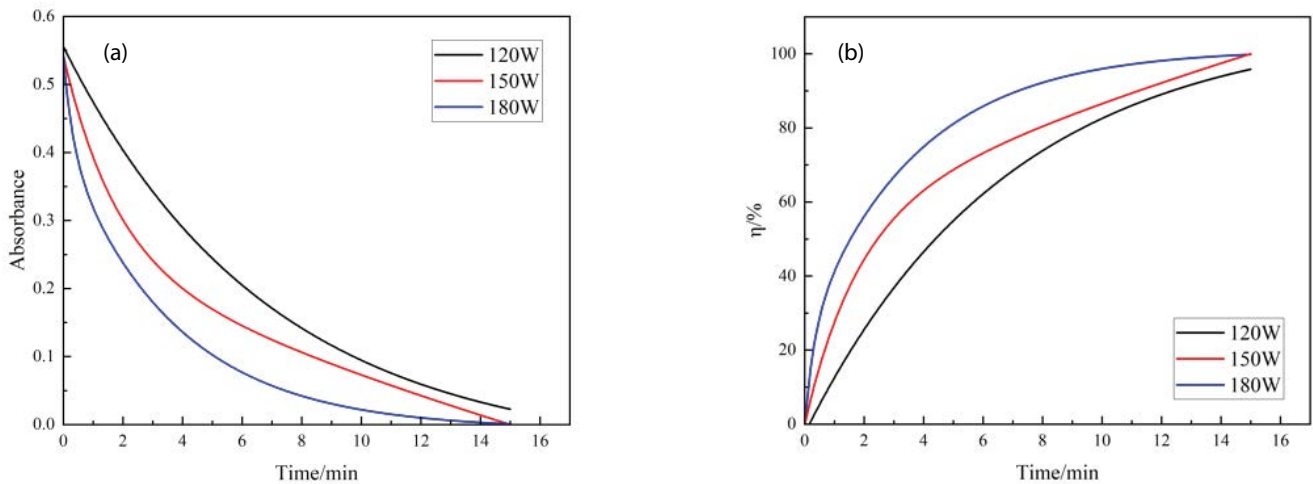


Fig. 7. Variation curve at 584 nm after degradation of crystalline violet solution at pH = 7 with different discharge power conditions of initial concentration of 10 mg/L. (a) The absorbance change curve and (b) the degradation rate change curve.

color change. And the higher concentration of the CV solution, the lower the degradation effect under the same treatment time.

The conclusion was that the DBD plasma reactor was limited in its ability to treat different concentrations of CV solutions.

3.2. Effect of different discharge power on the degradation rate of CV solution

Under the conditions of 10 mg/L, pH = 7 and 500 mL CV solution, the initial discharge power were 120, 150 and 180 W, respectively. Under the different initial discharge power, the absorbance of the treated CV solution at 584 nm with the time variation is shown in Fig. 7a. The degradation rate curves at 584 nm with time is shown in Fig. 7b. The color change of the CV solution with increasing degradation time under different discharge power conditions is shown in Fig. 8.

From Fig. 7 the degradation rate of CV solution after 15 min of discharge treatment at different discharge powers was almost colorless. It can be seen that the degradation rate reached the highest at about 13 min of discharge treatment and then started to level off. The results showed that the degradation rate of CV solutions was 93.54%, 98.15% and 99.69% after 15 min of discharge treatment at different discharge powers of 120, 150 and 180 W, respectively.

From Fig. 8 it can be seen that the color change of the CV solution under the condition of discharge power of 180 W was more rapid, and the conclusion was that the discharge power with the highest degradation rate is 180 W.

The conclusion was that the optimal discharge power condition is 180 W. But in the actual research process, it obeyed the theory only in the early stage of discharge. Because when the reactor processing time is longer, the temperature in the reactor is higher, so the CV solution was easily evaporated to form steam, which affected the generation and distribution of micro-discharge filaments in the

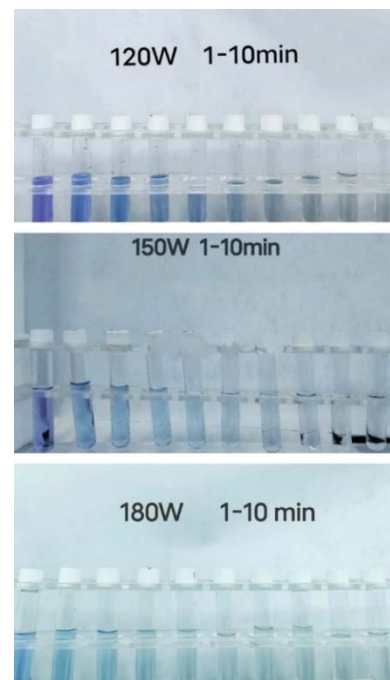


Fig. 8. Color change of crystalline violet solution after degradation under different discharge power conditions at pH = 7 initial concentration of 10 mg/L.

discharge interval. Thus, it resulted part of the CV solution flowing through the discharge interval without suffering from electron bombardment. In summary, under the high power the reactor discharge is unstable. When the reactor is processing CV solution, the degradation rate is not significantly improved and the damage to the equipment is great. So, in this research, considering the economy and the protection of the research equipment in the research process, the discharge power of 150 W was used for the wastewater treatment in this research subsequently.

3.3. Effect of different initial pH on the degradation rate of CV solution

This single-factor research CV solution was prepared freshly with buffer solution to adjust the initial pH value. The pH values of the three groups of CV solutions were adjusted to 4.00, 6.86 and 9.18, respectively. The absorption spectra of CV solutions at 350–700 nm is shown in Fig. 9. The CV solutions were at different initial pH with the same discharge power of 150 W and the same initial concentration of 10 mg/L. Under the different initial pH, the absorbance of the treated CV solution at 584 nm with the time variation is shown in Fig. 10a. The degradation rate curves at 584 nm with time is shown in Fig. 10b. The color change of the CV solution with increasing degradation time under different discharge power conditions is shown in Fig. 11.

As shown in Fig. 9, the maximum absorption peak of CV at different initial pH values was located at 584 nm

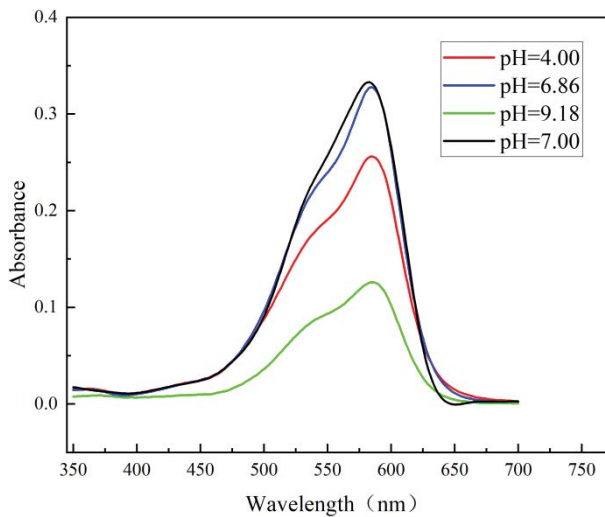


Fig. 9. Absorption spectra of crystalline violet solutions at 350–700 nm at different initial pH values with a discharge power of 150 W and an initial concentration of 10 mg/L.

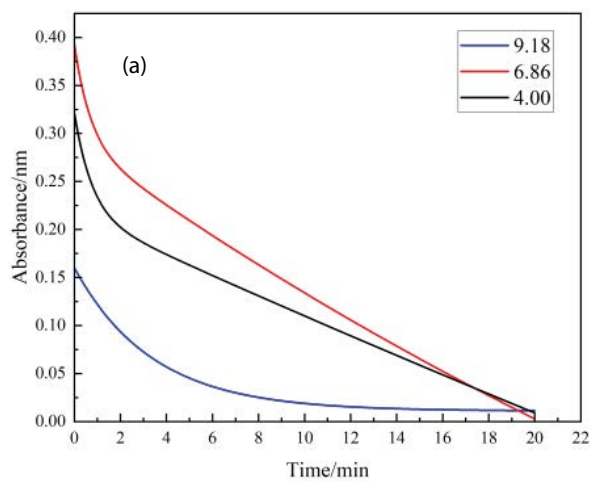


Fig. 10. Variation curves of crystalline violet solution at 584 nm after degradation at different initial pH values with a discharge power of 150 W and an initial concentration of 10 mg/L. (a) The absorbance change curve and (b) the degradation rate change curve.

without peak shift. Thus, in this experiment, the change in pH value of the CV solution did not lead to a change in its maximum absorption peak. The experimental data obtained by measuring the absorbance of the solutions treated with different discharge times at 584 nm under different initial pH conditions were reliable.

From Fig. 10 the different pH conditions may mask the chromogenic groups of the CV solution. After discharge treatment of CV solutions with the same initial conditions

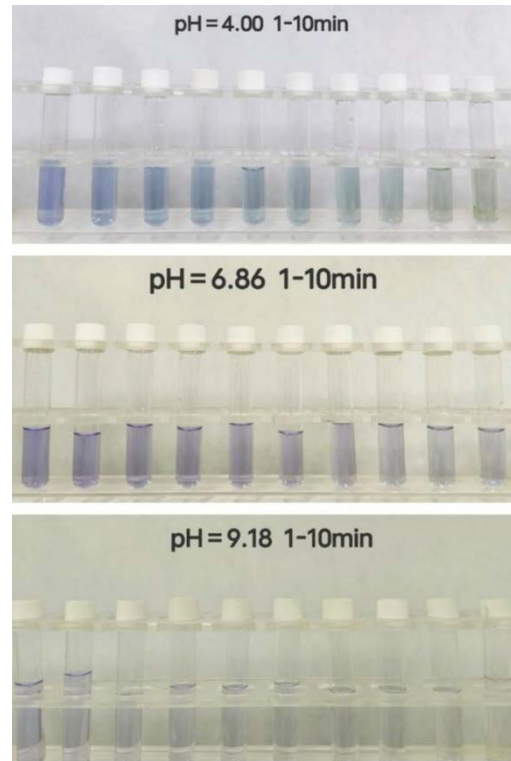


Fig. 11. Color change of crystalline violet solution after degradation under different initial pH conditions at an initial concentration of 10 mg/L with a discharge power of 150 W.

at different pH conditions, the pH value of the test samples was measured every minute, and it was found that the pH value of the CV solutions did not change during the treatment. It showed that the degradation of CV was caused by the bombardment of high-energy particles generated from DBD plasma during the experiment. The degradation effect of CV solution at pH = 9.18 was more obvious than the other two groups of pH values, and the degradation rate was up to 89.03% after discharge treatment for 15 min. And the degradation rates at pH = 4.00 and pH = 6.86 were 77.01% and 80.66%. In comparison with the degradation rate of CV solution prepared with ultrapure water under the same conditions, it can be concluded that the addition of buffer solution will mask the chromogenic group of CV solution and slow down its degradation.

From Fig. 11 it can be seen that for the same treatment time, the CV solution color of dye wastewater with pH = 9.18 is significantly lighter compared with the other two pH groups, which means that the removal of CV was more effective under this pH condition.

In combination with the preliminary tests, it can be seen that the pH = 7.00 CV solution prepared in the experimental ultrapure water by the DBD plasma reactor discharge treatment for 1–15 min and sampling every minute, the pH value was found to be unchanged before and after comparison. Thus, when no buffer solution was added, the CV degradation was due to the bombardment of energetic particles generated by the DBD plasma. So, the absorption peak of CV solution at 584 nm was affected by the pH value, but it was not related to the type of buffer solution. From Fig. 11 it can be seen that the decrease in absorbance strength corresponds to a diminishing color. So, it can be tentatively judged that the maximum absorption peak was the absorption peak of the chromogenic group, which indicated that the acidic and alkaline conditions masked part of the chromogenic group of the CV solution.

The conclusion was that the absorbance at 584 nm of the CV solutions in the three pH test conditions tends to decrease from the discharge treatment, but the degradation

time required was longer in the different pH conditions contrast with CV solution prepared with ultrapure water. Meanwhile, during the discharge process, under different pH conditions, the pH value did not change during the reactor treatment. So, the decrease in absorbance was due to plasma bombardment of the CV chromophore rather than masking of the chromophore.

3.4. Effect of different atmospheric conditions on the degradation rate of CV solution

Different atmospheres were ventilated to the bottom for 30 min in 500 mL of CV solution with an initial concentration of 10 mg/L and initial pH = 7. Under the same initial conditions, the CV solutions were discharged in different atmospheres, the color changes were observed and the absorbance was measured. Under the different initial atmospheres, the absorbance of the treated CV solution at 584 nm with the time variation is shown in Fig. 12a. The degradation rate curves at 584 nm with time is shown in Fig. 12b. The color change of the CV solution with increasing degradation time under different discharge power conditions is shown in Fig. 13.

From Fig. 12a it can be seen that the CV solution treated with different atmospheric conditions under the same initial conditions showed a decreasing trend in absorbance at 584 nm. In air and oxygen atmosphere conditions discharge treated CV solutions produce new material at the same rate, while nitrogen atmosphere condition was different. From Fig. 12b, under the same initial conditions, the degradation rate of CV solution after 15 min of discharge treatment at different atmospheres was: oxygen > air > nitrogen. The degradation rate of CV solution first 10 min of discharge treatment at different atmospheres was: nitrogen > oxygen > air. The degradation rates of the three groups of solutions were basically similar. Under the conditions of air, oxygen and nitrogen, the degradation rate of CV solution with initial concentration of 10 mg/L was 94.06%, 95.08% and 93.27% after 15 min of discharge treatment, respectively.

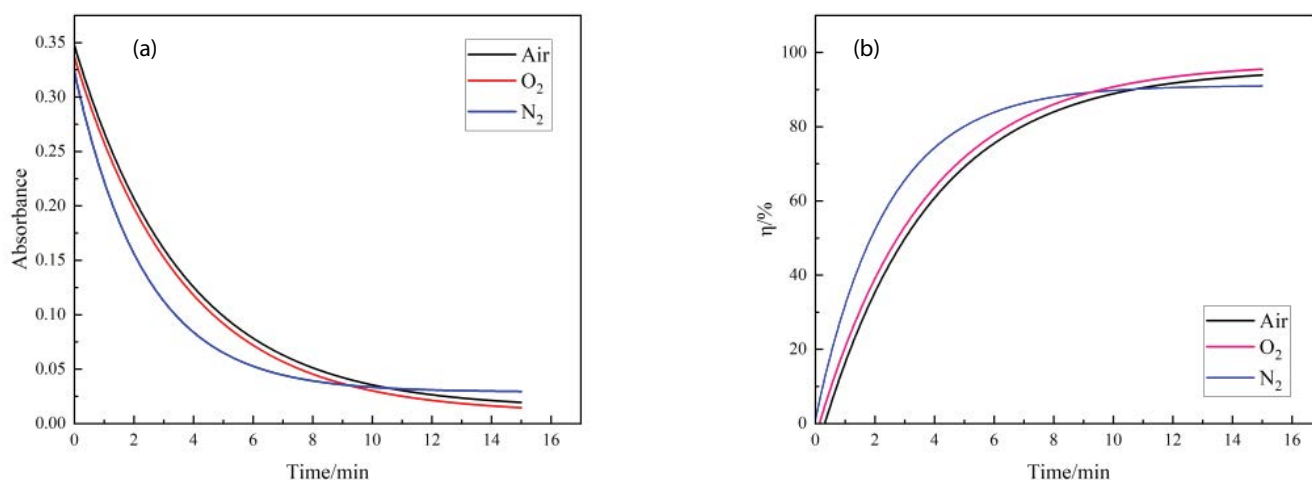


Fig. 12. Degradation of crystalline violet solution at 584 nm under different atmospheric conditions with pH = 7 discharge power of 150 W and initial concentration of 10 mg/L. (a) The absorbance change curve and (b) the degradation rate change curve.

From Fig. 13 it can be seen that visually the color change of the CV solution corresponds exactly to the curve changes in Fig. 14.

This phenomenon may be due to that under nitrogen atmosphere discharge treatment at the beginning of the action of the CV solution results that the high-energy electrons generated by the DBD plasma reactor collide with the solution N_2 molecules causing dissociation, excitation and ionization of N_2 molecules resulting in N , N^+ , N_2^+ and excited N_2 molecules [26], which in turn degrade the CV

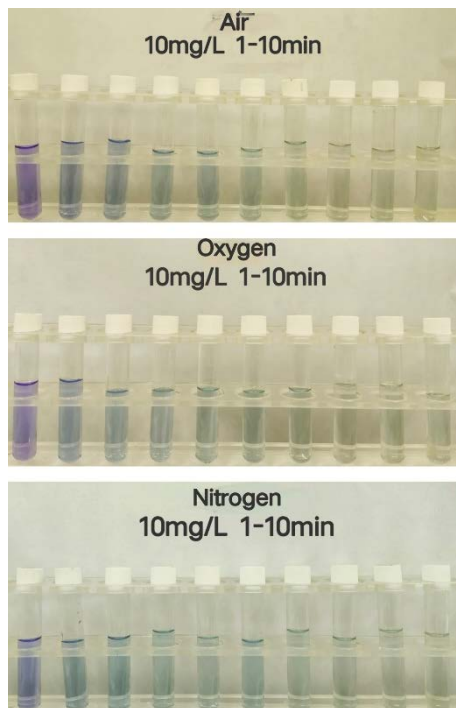


Fig. 13. Color change of crystalline violet solution after degradation under different atmospheric conditions with pH = 7 discharge power of 150 W and initial concentration of 10 mg/L.

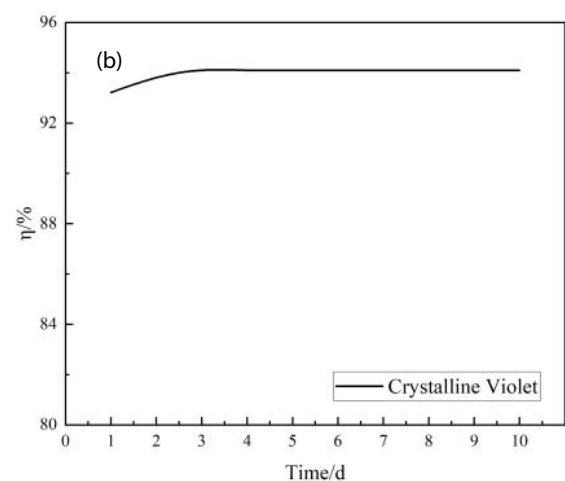
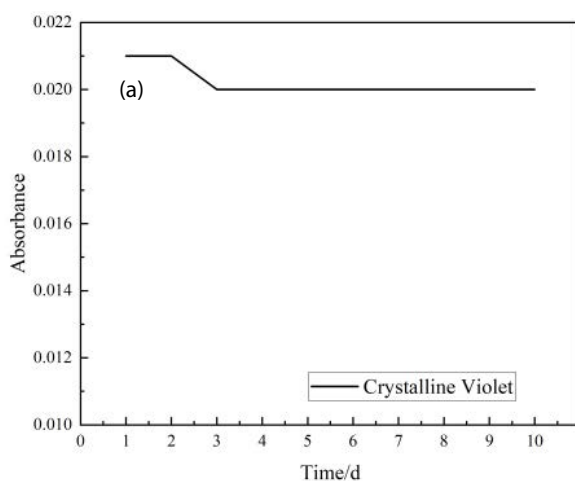


Fig. 14. Variation curve at 584 nm of the crystalline violet solution after discharge treatment at pH = 7 with a discharge power of 150 W and an initial concentration of 10 mg/L for 10 d. (a) The absorbance change curve and (b) the degradation rate change curve.

solution by breaking the bonds through the excited N_2 molecules and other substances. By the late stage of treatment discharge gap could not form a large number of micro-discharge filaments degradation rate gradually became slower [27]. Under oxygen atmosphere discharge treatment at the beginning of the action of the CV solution results that the high-energy electrons generated by the DBD plasma interact with the solution O_2 molecules at a slower rate compared to nitrogen. But in the later stages of the discharge may produce a large number of positive oxygen molecular ions O_2^+ [28], etc. The degradation rate of CV by strong oxidizing substances is gradually accelerated.

3.5. Effect of standing time on the degradation rate of CV solution

Under the conditions of 150 W, 10 mg/L, pH = 7, the CV solution was discharged for 15 min. After treatment, the CV solution was left for 10 d, and the effect of the leaving time on its color change was observed and the absorbance was measured. Under the standing time, the absorbance of the treated CV solution at 584 nm with the time variation is shown in Fig. 14a. The degradation rate curves at 584 nm with time is shown in Fig. 14b. The color change of different initial concentrations of CV solution with the increase of discharge treatment time is shown in Fig. 15.

From Figs. 14 and 15, the absorbance and degradation rate of CV changed very little. From Fig. 15, the color change of the CV solution was difficult to be observed by the naked eye, and the solution remained in a light-yellow state after 10 d. It is shown that the DBD plasma reactor is effective for the treatment of CV solution and the stability of the CV solution after treatment is excellent.

3.6. Effect of different reactors on the degradation rate of CV solution

Under the conditions of 150 W, 10 mg/L, pH = 7, the CV solution was treated with DBD plasma reactor, ozone generator and ozone-DBD plasma coupled reactor, respectively. Under the different reactors, the absorbance of the treated

CV solution at 584 nm with the time variation is shown in Fig. 16a. The degradation rate curves at 584 nm with time is shown in Fig. 16b. The color change of the CV solution with increasing degradation time under different discharge power conditions is shown in Fig. 17.

From Fig. 16 it can be seen that use of different reactors to treat the CV solution has different effects. It can be seen that the CV solution was completely degraded after 15 min of discharge treatment under different reactor conditions. The conclusion showed that the degradation rate of the CV solution treated with DBD plasma reactor, ozone generator and ozone-DBD plasma coupled reactor were 90.88%, 99.09% and 99.70% after 15 min of discharge treatment, respectively.



Fig. 15. Color change of crystalline violet solution after discharge at pH = 7 with a discharge power of 150 W and an initial concentration of 10 mg/L for 10 d.



Fig. 17. Color change after degradation of crystalline violet solution treated with different reactors at pH = 7 with a discharge power of 150 W and an initial concentration of 10 mg/L.

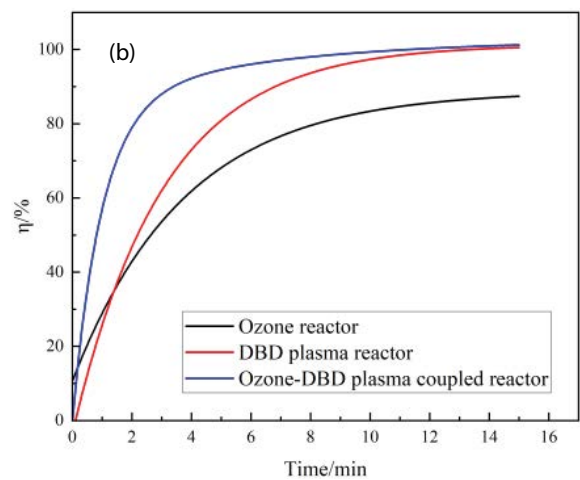
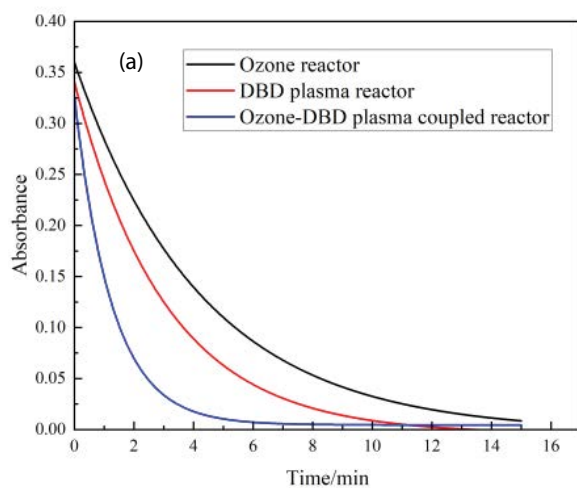


Fig. 16. Variation curve at 584 nm after degradation of the crystalline violet solution treated with different reactors at pH = 7 with a discharge power of 150 W and an initial concentration of 10 mg/L. (a) The absorbance change curve and (b) the degradation rate change curve.

From Fig. 17, the color changed of different reactors showed the ozone reactor was much less effective than DBD plasma reactor. It can be obtained that the CV solution treated by the ozone reactor was still lavender after 15 min of treatment, while as well as the CV solution after treatment was close to transparent.

The conclusion was that the effect of the ozone DBD coupled plasma reactor in treating the CV solution was optimal compared to the other reactors. The DBD plasma reactor treatment effect was better than the ozone generator.

3.7. Mechanism of CV degradation process

Under DBD discharge treatment plasma water molecules are bombarded by high-energy electrons to produce a series of highly reactive particles, free radicals, ozone and hydrogen ions, etc. At the moment of the discharge treatment, the color of CV solution starts to change. The discharge reaction caused these substances produced in the reaction system to continuously bombard the chemical bonds of the CV, resulting in the breakage of the chemical bonds of the CV. And combine to produce new substances under the combined action of free radicals, and the newly produced substances are also degraded by the non-selective bombardment of the plasma. From the reference DBD discharge degradation is caused by the bombardment of pollutants by $\cdot\text{OH}$ generated in the plasma. $\cdot\text{OH}$ is chemically active and is the main acting substance in the process of plasma degradation of CV. With the extension of the treatment time, most of the CV in the solution will eventually be decomposed into small molecules, some of which remain in the solution, and some evaporate to the atmosphere with other.

During the degradation of CV, a colorless alcoholic structure may be produced, which may indicate that the $\cdot\text{OH}$ produced during the discharge process played a major role in the decolorization and degradation of CV. The degradation intermediate products of CV should be mainly small molecular weight carboxylic acids, and the final products are CO_2 , H_2O and NO .

4. Conclusion

Through the experiment of degrade CV solution by DBD plasma reactor. Six conclusions were drawn: (1) when treating different initial concentrations of CV solution under the same discharge power and initial pH conditions, the lower the initial concentration of the solution, the higher degradation rate obtained after the discharge treatment, and the degradation rate of 5 mg/L CV solution can reach 99.64% after the treatment, the better degradation and removal effect, and the more rapid the color change of CV. (2) When CV solution was treated with different discharge power under the same initial concentration and initial pH value, the removal effect of CV was better when the discharge power was 180 W, and the degradation rate could reach 99.69%. (3) During the discharge process, the absorbance at 584 nm of the CV solutions tends to decrease in the three pH conditions, but the degradation time required was longer in the different pH conditions contrast with CV solution prepared with ultrapure water. Meanwhile, the decrease in absorbance was due to plasma bombardment of the CV chromophore

rather than pH change masked the chromogenic group. The degradation effect of CV solution at $\text{pH} = 9.18$ was more obvious than the other two groups of pH values, and the degradation rate was up to 89.03% after discharge treatment for 15 min. (4) Aeration of the CV solution will have a certain degradation effect. The best degradation effect is achieved by passing oxygen. The CV solution treated under air, oxygen, nitrogen atmosphere, the degradation rate was up to 94.06%, 95.08%, 93.27%. (5) The degradation rate of CV solution could reach more than 99% after 15 min of discharge treatment, and the solution remained in light yellow state after 10 d, which indicates that the DBD plasma reactor has obvious effect on the treatment of CV solution simulating dye wastewater and the stability of the treated solution is good. (6) When CV solution was treated with different reactors under the same initial conditions, the ozone-DBD coupled reactor treated CV solution with better removal effect and degradation rate up to 99.70%. Therefore, DBD plasma reactor water treatment technology can be considered more economical with other technology coupling.

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