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Experimental Study on Ultrasonic Cavitation Intensity Based on Fluorescence Analysis



Linzheng Ye^{1*}, Shida Chuai¹, Xijing Zhu¹ and Dong Wang^{2*}

Abstract

The Ultrasonic cavitation effect has been widely used in mechanical engineering, chemical engineering, biomedicine, and many other fields. The quantitative characterization of ultrasonic cavitation intensity has always been a difficulty. Based on this, a fluorescence analysis method has been adopted to explore ultrasonic cavitation intensity in this paper. In the experiment of fluorescence intensity measurement, terephthalic acid (TA) was used as the fluorescent probe, ultrasonic power, ultrasonic frequency, and irradiation time were independent variables, and fluorescence intensity and fluorescence peak area were used as experimental results. The collapse of cavitation bubble will cause molecular bond breakage and release $\cdot\text{OH}$, and the non-fluorescent substance TA will form the strong fluorescent substance TAOH with $\cdot\text{OH}$. The spectra of the treated samples were measured by a F-7000 fluorescence spectrophotometer. The results showed that the fluorescence intensity and fluorescence peak area increased rapidly after ultrasonic cavitation treatment, and then increased slowly with the increase of ultrasonic power, which gradually increased with the increase of irradiation time. They first decreased and then increased with the increase of ultrasonic frequency from 20 kHz to 40 kHz. The irradiation time was the most influential factor, and the cavitation intensity of low frequency was higher overall. The fluorescence intensity and fluorescence peak area of the samples increased by 2–20 times after ultrasonic treatment, which could increase from 69 and 5238 to 1387 and 95451, respectively. After the irradiation time exceeded 25 min, the growth rate of fluorescence intensity slowed down, which was caused by the decrease of gas content and TA concentration in the solution. The study quantitatively characterized the cavitation intensity, reflecting the advantages of fluorescence analysis, and provided a basis for the further study of ultrasonic cavitation.

Keywords Ultrasonic cavitation, Cavitation intensity, Fluorescence intensity, Fluorescence peak area

1 Introduction

Practical applications of ultrasonic cavitation have been found for ultrasonic cleaning, atomization, emulsification, crystallization and so on since 1927, when Loomis

first observed the chemical effects of ultrasound. It is quite popular and essential to do ultrasonic cavitation studies for a detailed understating of sonochemistry. The quantitative characterization of ultrasonic cavitation intensity has always been a difficulty. Hydrophones have been normally used to measure sound pressure, which is used to calculate the ultrasonic cavitation intensity. Although various hydrophones, such as Polyvinylidene fluoride (PVDF) and fiber optic hydrophones, are available for ultrasonic field mapping, many hydrophones suffer from a lack of uniform response over a wide range of frequencies while still maintaining sensitivity, particularly below 200 kHz where power ultrasonic applications operate. Furthermore, any measurement device must be

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robust enough to withstand the vibration associated with high power ultrasound measurement. To attain increased levels of sensitivity, spatial resolution, and non-invasiveness, hydrophone devices are generally quite delicate and fragile, which can be easily damaged by power ultrasound. Therefore, it is necessary to conduct the measurements using non-invasive techniques.

Ultrasonic cavitation phenomenon refers to a high-intensity nonlinear dynamic process in which the cavitation nucleus undergoes various stages in the liquid medium, such as primary growth, expansion, shock, compression, and collapse, accompanied by secondary physicochemical phenomena such as strong shock wave, high-speed micro-jet, acoustically induced free radical, sonoluminescence, and acoustically induced heating [1, 2]. The main ways to induce cavitation include ultrasound, hydraulic, particle, laser, etc. Among them, ultrasonic-induced cavitation has become the main method because it is convenient to adjust the location and intensity of cavitation, and it has a stronger cavitation effect than hydraulic cavitation [3] and will lead to the generation of hydroxyl radical $\cdot\text{OH}$ [4]. With the progress of modern science and technology, ultrasonic cavitation technology has been applied in many fields such as mechanical processing, biomedicine, surface cleaning, chemical engineering, and so on [5–8], and its research is of great significance.

Scholars had done a series of studies on the effect and function of ultrasonic cavitation [9, 10]. In 2017, Ye et al. [11] established a three-dimensional fluid-structure coupling model to discuss the impact effect of micro-jet produced by cavitation collapse near the wall on a 1060 aluminum plate. In 2021, Chowdhury et al. [12] connected 6063 aluminum alloy and C26000 copper alloy through friction stir welding and ultrasound-assisted friction stir welding and analyzed the mechanical properties. They observed that when ultrasonic vibration of 10–14 kHz was added to the welding, the mechanical properties increased from 10% to 25%. This indicated that more scholars had been using the cavitation effect to make the processing effect better. In 2021, Liang et al. [13] deduced a new theoretical model to describe the translation, pulsation, and deformation of double bubbles in the ultrasonic field. Du et al. [14] used the numerical simulation method to predict the volume and pressure pulsation of the cavitation cavity, and the consistency between the results and the experimental data was about 90%. In 2020, Kalmár et al. [15] studied the chemical generation of bubbles excited by harmonics. Based on considering a total of 44 different chemical reactions and the evaporation, condensation, heat transfer, and diffusion of 9 different chemical substances, a mathematical model was established. Without considering the chemical kinetics,

the chemical activity could be accurately predicted. Researchers had studied cavitation bubbles and chemical products through theoretical models or numerical simulations, but they made a lot of assumptions, which may lead to deviation from reality. In 2020, Ye et al. [5] used the ultrasonic cavitation effect to improve the surface properties of AZ31B magnesium alloy, and studied the effects of ultrasonic amplitude, treatment time, and distance from the sample on the Vickers hardness and grain size. The Vickers hardness was increased by 1.5–3 times and the grain size was refined to 2–10 μm . In addition, ultrasonic cavitation can produce residual compressive stress on the metal surface. Hitoshi Soyama et al. [16] measured that the surface residual stress after cavitation shot peening is about -300 MPa by using two-dimensional X-ray diffraction method. After 100 times of cyclic fatigue tests with bending stress of 400 MPa, the residual stress is still about -168 MPa. Gu et al. [17] studied the influence of ultrasonic cavitation on the surface strengthening of Q235 steel by combining simulation and experiment. The research showed that the surface had plastic deformation after cavitation shot peening, and the residual stress could reach -126 MPa. Soyama et al. [18] introduced residual stress of about -220 MPa into Ti6Al4V material by using cavitating micro abrasive jet, reducing surface roughness and improving fatigue life strength. These researches not only showed that ultrasonic cavitation would produce residual compressive stress on the metal surface, but also expounded the gain effect of ultrasonic cavitation on materials. The above showed that the study of ultrasonic cavitation intensity had very important practical significance, and the quantitative analysis and measurement of ultrasonic cavitation intensity had always been a difficulty. Some scholars directly measured the degree of cavitation reaction through high-speed photography and aluminum foil corrosion [19–21], but they could not quantitatively describe cavitation.

In 2008, Hu et al. [22] studied the ultrasonic field by detecting hydroxyl radicals through the chemiluminescence method, and pointed out that the content of hydroxyl radicals increased with the increase of ultrasonic power. In 2012, Capocelli et al. [23] used the consumption of nitrophenol to estimate the production of hydroxyl radicals, and indicated that the content of hydroxyl radicals gradually increased and there was an extreme point between 40–1000 kHz. Ding et al. [24] quantified the fluorescence peak area in the emission wavelength range of 220–600 nm to characterize the generation of $\cdot\text{OH}$. In 2006, Sahni et al. [25] used dimethyl sulfoxide (DMSO) and disodium terephthalate (NaTA) as chemical fluorescence probes, set the excitation wavelength to 315 nm, collected the fluorescence spectrum

of the sample in the range of 340 to 625 nm, and quantitatively determined the hydroxyl radical through the fluorescence peak area. The essence of fluorescence peak area is to select the appropriate excitation wavelength and calculate the fluorescence intensity integrally in a certain range. In 2015, Jimenez-Relinque et al. [26] used terephthalic acid as a fluorescent probe to quantitatively analyze the hydroxyl radical produced by several photocatalytic building materials. The standard TAOH solution had a linear relationship between fluorescence spectrometry intensity and concentration, which is independent of specific pollutants. Moreover, this method has the advantages of fast, simple, cheap, and has a wide application range. In 2010, Hasanzadeh et al. [27] proved that the fluorescence analysis method had the advantages of low cost, high repeatability, and strong real-time performance compared with the subharmonic spectral analysis method to characterize the intensity of cavitation.

In the engineering application of ultrasonic cavitation, the quantitative characterization of cavitation intensity is an important problem. Some scholars use polyvinylidene fluoride (PVDF) needle hydrophone [28, 29] to measure the pressure in the cavitation field and then characterize the cavitation intensity. However, the essence of this method is to measure the pressure at the point through the pressure sensor, which can only characterize the point cavitation intensity and cannot characterize the global cavitation intensity. Fluorescence analysis can characterize the overall cavitation intensity. In addition, it has the advantage of being able to describe steady-state and transient cavitation directly and quantitatively. Using TA as a fluorescent probe has been widely used in sonochemical research to detect $\cdot\text{OH}$ [26, 30]. In a liquid medium, ultrasound causes cavitation collapse to release energy, causing instantaneous high temperature and high pressure (5000 K and 100 MPa) locally. In this case, the molecular bond in the medium to break and recombine alternately. The reactions caused by chemical bond breaks, such as the opening of water molecular bonds to form hydroxyl radicals [9, 22, 31], the combination of oxygen ions to generate oxygen, and the combination of hydrogen ions and oxygen ions to generate water, continue to occur [32, 33]. The increase in fluorescence intensity is caused by the strong fluorescence substance TAOH generated by $\cdot\text{OH}$ and TA, as shown in Eq. (1). Capocelli et al. [23] gave the concentration of TAOH in cavitation, as shown in Eq. (2).



$$C_{\text{TAOH}} = Va_{\text{TAOH}}v', \quad (2)$$

where C_{TAOH} represents the concentration of TAOH in the bubble, V is the bubble volume, a_{TAOH} is the stoichiometric weight, and v' represents the reaction rate.

In addition, the reaction of $\cdot\text{OH}$ with terephthalic acid is almost not affected by other cavitation products or active substances [32]. When detecting fluorescence, the excitation wavelength was selected as 315 nm and the emission wavelength was selected as 425 nm. The concentration of TAOH within the range of PH 6–11 could be characterized by fluorescence intensity, which had a linear relationship [26]. Therefore, fluorescence intensity and fluorescence peak area could be used to characterize the concentration of TAOH to reflect the production of $\cdot\text{OH}$, and then the ultrasonic cavitation intensity was characterized.

In conclusion, the ultrasonic cavitation effect has been applied in many fields, and the quantitative characterization of cavitation intensity is the research focus. Based on this, in this paper, we took TA as the fluorescence probe, used F-7000 to measure the fluorescence intensity and fluorescence peak area of the treated samples to characterize the cavitation intensity, and then analyzed the effects of ultrasonic power, irradiation time, and ultrasonic frequency, to provide a basis for the quantitative characterization of cavitation intensity in theory and experiment.

2 Fluorescence Analysis Experiments

The principle of fluorescence spectrometry is that the non-fluorescent substance TA reacts with $\cdot\text{OH}$ formed by the molecular bond fracture in the solution after ultrasonic treatment to form a strong fluorescent substance TAOH. TAOH receives external energy (i.e., exciting light in the experiment) and jumps from the ground state to the excited state. When it returns to the ground state, the energy is emitted as electromagnetic radiation (luminescence). The output of TAOH can be characterized by the change of fluorescence intensity, and the amount of $\cdot\text{OH}$ can be determined to characterize the ultrasonic cavitation intensity. After ultrasonic treatment, the sample was left standing at room temperature for 1 h, and the fluorescence intensity and fluorescence peak area of the sample were measured by the Hitachi F-7000 fluorescence spectrophotometer shown in Figure 1. The amount of $\cdot\text{OH}$ generated is proportional to the fluorescence intensity and fluorescence peak area, and the yield of $\cdot\text{OH}$ in TA solution can be used to characterize ultrasonic cavitation intensity. The basic properties of fluorescent probe TA are shown in Table 1. The pH value of the sample was measured before and after the experiment, which fluctuated in the range of 10.6 to 10.8, reaching the technical indicators of using fluorescence analysis.

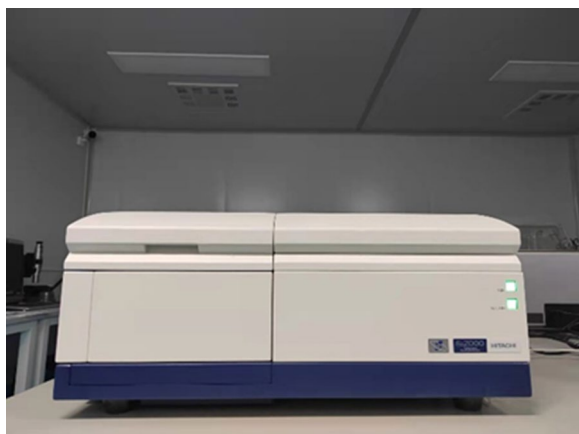


Figure 1 Hitachi F-7000 fluorescence spectrophotometer

Table 1 Basic properties of fluorescent probe TA

Chemical formula	Molecular weight	Density (g/cm ³)	Water solubility
C ₈ H ₆ O ₄	166.131	1.51	Slight soluble



Figure 2 Ultrasonic cavitation experiment device

The ultrasonic cavitation experiment device is shown in Figure 2. It is composed of an ultrasonic generator, an ultrasonic transducer, an ultrasonic vibration device, a loading support, a working platform, a lifting mechanism, and a beaker. The alternating current is converted into a high-frequency current signal through the ultrasonic generator, matched with the transducer, and converted

into mechanical vibration with the same vibration frequency as the tool head by the transducer. The preparation method for TA solution is as follows. The TA used in the experiment is almost insoluble in water but soluble in strong alkali. Accurately measure 25.0 mL NaOH solution (1.0 mol/L) to dissolve 0.25 g TA, and then add distilled water to prepare 2 L terephthalic acid solution. Three variables were selected in the experiment, namely ultrasonic power (200 W, 400 W, 600 W, and 800 W), irradiation time (4 min, 6 min, 8 min, 12 min, 16 min, 25 min, and 30 min), and ultrasonic frequency (20 kHz, 30 kHz, and 40 kHz). In this paper, the fluorescence intensity and fluorescence peak area of the treated samples were quantified, and the effects of the above three factors on them were analyzed respectively.

Fluorescence intensity and fluorescence peak area are important indexes to evaluate ultrasonic cavitation intensity, and directly proportional to it, which means that the larger the value, the stronger the ultrasonic cavitation intensity, and ultrasonic cavitation intensity is an important reference index to evaluate the effect of ultrasonic cavitation. The spectrum of TA solution under ultrasonic irradiation was measured by the Hitachi F-7000 fluorescence spectrophotometer. Before that, the instrument parameters were adjusted. The PMT voltage was 400 V, the slit width was 10 nm, and the scan speed was set to 2400 nm/min. When the fluorescence intensity was measured, the excitation wavelength and the emission wavelength were 315 nm and 425 nm, respectively. When the fluorescence peak area was measured, the wavelength scan mode was selected, the excitation wavelength was 315 nm, and the fluorescence peak area was quantified in the range of 365–600 nm. The fluorescence intensity and fluorescence peak area of each group experiment were measured three times, and their average value was taken as the final experimental results.

For spectral analysis, the TA solution samples after ultrasonic treatment were sub-packed with a 50 mL transparent screw glass reagent bottle. After 1 h at room temperature, a 2 mL disposable Pasteur pipette was used to transfer the appropriate solution to a four-side transparent glass dish as a fluorescence observation target, and the fluorescence spectrophotometer was used for sample detection and analysis.

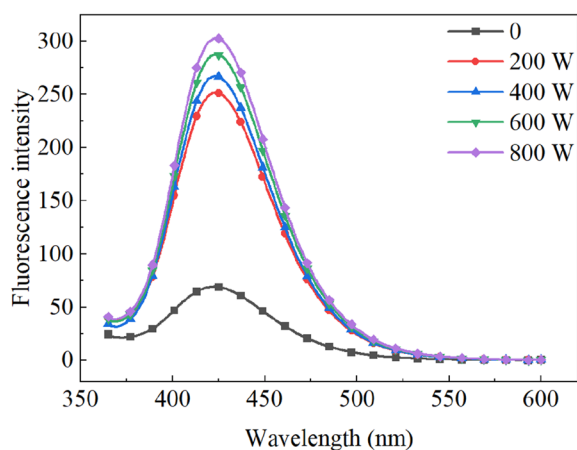
3 Results and Discussions

3.1 Effect of Ultrasonic Power on Ultrasonic Cavitation Intensity

The ultrasonic frequency was 30 kHz, the irradiation time was 6 min, and the ultrasonic power is 0, 200, 400, 600, and 800 W respectively. The TA solution was subjected to ultrasonic treatment, and then the treated solution was detected and analyzed by the Hitachi F-7000

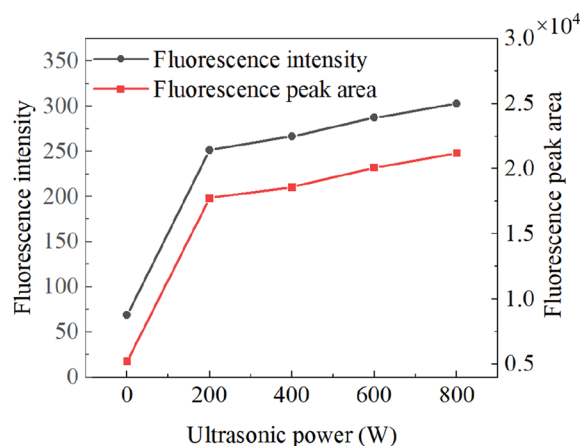
Table 2 Experiment results under different ultrasonic powers

Ultrasonic power (W)	Fluorescence intensity	Fluorescence peak area
0	69.0	5238
200	251.4	17751
400	266.6	18574
600	287.3	20098
800	302.7	21198

**Figure 3** Fluorescence spectra under different ultrasonic powers

fluorescence spectrophotometer. The experiment results were shown in Table 2. The fluorescence intensity and fluorescence peak area reached the maximum when the ultrasonic power was 800 W, which were 302.7 and 21198, respectively. The spectral curve was shown in Figure 3, where “0” represented the untreated TA solution. It could be seen that the fluorescence intensity and fluorescence peak area of the sample after ultrasonic treatment increased significantly.

Figure 4 showed the changes in fluorescence intensity and fluorescence peak area with ultrasonic power. It could be seen that the fluorescence intensity and fluorescence peak area increased rapidly after ultrasonic cavitation treatment, and then increased slowly with the increase of ultrasonic power. Both of them increased by 3.3–4.5 times after ultrasonic treatment. This is because with the increase of ultrasonic power, the growth rate of cavitation will increase, and the cavitation reaction will be more intense. The energy released by cavitation collapse will increase, and more molecular bonds will break, so that more $\cdot\text{OH}$ will be released to react with TA, increasing TAOH content. In addition, although the ultrasonic power increased, the irradiation time remained unchanged, and there was not enough time to

**Figure 4** Fluorescence intensity and fluorescence peak area under different ultrasonic powers**Table 3** Experiment results under different irradiation times

Process time (min)	Fluorescence intensity	Fluorescence peak area
0	69.0	5238
4	193.3	13838
6	287.3	20098
8	374.2	26485
12	481.8	32925
16	690.1	46727
25	1218	81692
30	1387	95451

fully react to generate TAOH, so the fluorescence intensity and fluorescence peak area increased slowly with the ultrasonic power from 200 W to 600 W. Therefore, the influence of irradiation time was discussed next.

3.2 Effect of Irradiation Time on Ultrasonic Cavitation Intensity

The ultrasonic frequency was 30 kHz and the ultrasonic power was 600 W. Different irradiation times (0, 4, 6, 8, 12, 16, 25, and 30 min) were selected for ultrasonic treatment of TA solution. The experiment results were shown in Table 3. After 30 min, the fluorescence intensity and fluorescence peak area reached the maxima, which were 1387 and 95451 respectively.

The fluorescence spectra of samples under different irradiation times were shown in Figure 5. It could be seen that the fluorescence intensity and fluorescence area were gradually increased with the extension of irradiation time. The yield of $\cdot\text{OH}$ in each oscillation period of the cavitation bubble could be given by Eq. (3) [23]. Therefore, the yield of $\cdot\text{OH}$ within a certain

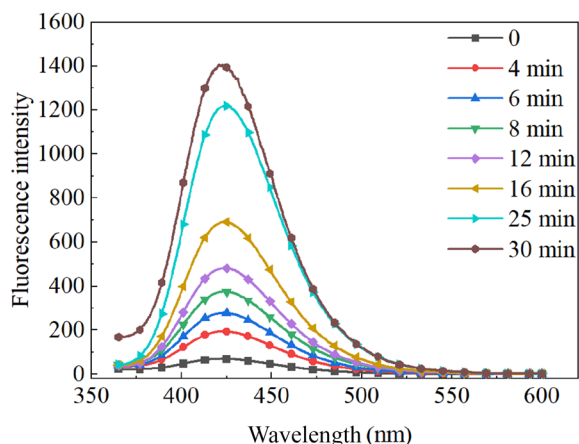


Figure 5 Fluorescence spectra under different irradiation times

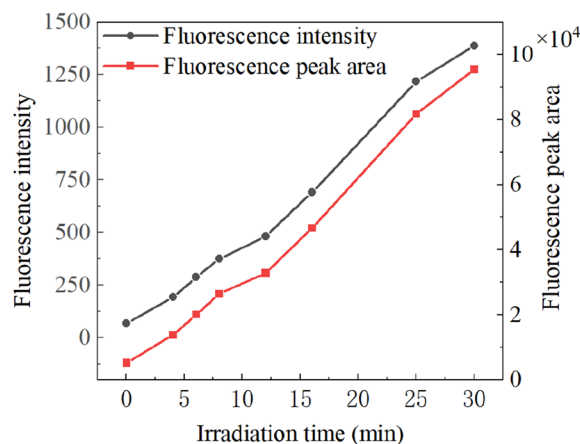


Figure 6 Fluorescence intensity and fluorescence peak area under different irradiation times

time t was given by Eq. (4), which could explain the increase in fluorescence intensity and fluorescence peak area.

$$P_{(OH)} = \int_{R_1}^{R_2} P_{OH} N_{R_0} dR_0, \tag{3}$$

$$W = \int P_{(OH)} dt, \tag{4}$$

where N_{R_0} is the distribution of nuclei in water in the initial state, R_0 , R_1 , and R_2 are the initial radius, minimum radius, and maximum radius of the bubble, and the latter two depend on the ultrasonic frequency. P_{OH} is the specific hydroxyl radical production, and $P_{(OH)}$ is the amount of $\cdot OH$ produced by the bubble.

Although the concentration of $\cdot OH$ increased and the fluorescence intensity increased with the extension of irradiation time, the growth rate of fluorescence intensity gradually slowed down after 25 min, which could be calculated by Eq. (5). The calculation showed that $v_{16-25} = 58.7 > v_{25-30} = 33.8$. This was also clearly shown in Figure 6. On the one hand, the gas content in TA solution decreased with the increase of irradiation time, resulting in the reduction of cavitation gas core and the weakening of cavitation effect. On the other hand, the content of TA in the solution decreased, and $\cdot OH$ was difficult to collide with TA in an effective time due to its extremely short life of 10^{-8} s. In addition, we also found that the irradiation time had a greater impact on the fluorescence intensity and fluorescence peak area than the ultrasonic power.

$$v = \frac{FL_{t_2} - FL_{t_1}}{t_2 - t_1}, \tag{5}$$

where t is the irradiation time, and FL_t is the corresponding fluorescence intensity.

3.3 Effect of Ultrasonic Frequency on Ultrasonic Cavitation Intensity

The experiment results with ultrasonic power of 600 W, irradiation time of 5 min, and ultrasonic frequency of 20, 30, and 40 kHz were shown in Table 4. The maximum fluorescence intensity and fluorescence peak area appeared at 20 kHz, which were 355.4 and 24895 respectively. The fluorescence spectra of samples under different ultrasonic frequencies were shown in Figure 7. The fluorescence peak of each fluorescence spectrum curve was 425 nm, which also indicated that the selection of emission wavelength was appropriate.

Figure 8 showed the changes in fluorescence intensity and fluorescence peak area with ultrasonic frequency. It could be seen that the fluorescence intensity and fluorescence peak area first increased and then decreased with the increase of ultrasonic frequency. When the ultrasonic frequency changed from 20 kHz to 30 kHz, the fluorescence intensity and fluorescence peak area were decreased. This was because the ultrasonic frequency was negatively correlated with the oscillation period. Due to the increase of ultrasonic frequency, the oscillation

Table 4 Experiment results under different ultrasonic frequencies

Ultrasonic frequency (kHz)	Fluorescence intensity	Fluorescence peak area
0	69.0	5238
20	355.4	24895
30	225.2	15871
40	274.0	19130

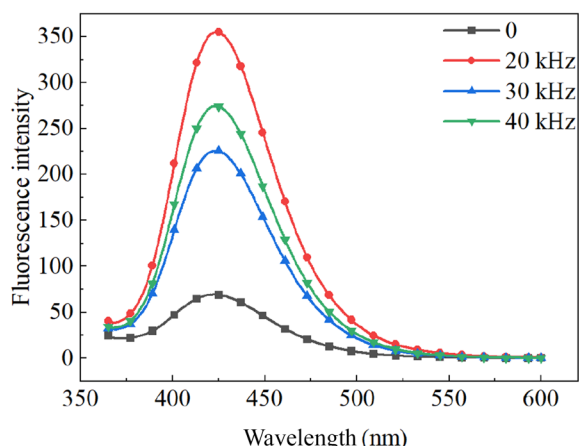


Figure 7 Fluorescence spectra under different ultrasonic frequencies

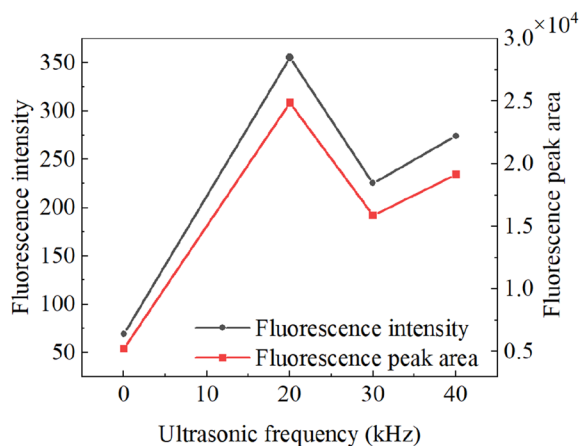


Figure 8 Fluorescence intensity and fluorescence peak area under different ultrasonic frequencies

period of a single cavitation bubble decreased, and a single cavitation bubble had not enough time to experience the whole process of growth and expansion. The process of cavitation bubble growth was essentially a process of energy storage [34]. Due to the decrease of oscillation period, the cavitation could not store enough energy, resulting in the reduction of energy released by bubble collapse, so that it could not cause enough molecular bond breaks and the reduction of ·OH yield.

With the increase of ultrasonic frequency from 30 kHz to 40 kHz, the fluorescence intensity and fluorescence peak area were increased. Although the increase of ultrasonic frequency would reduce the oscillation period of a single cavitation bubble, it would increase the total amount of cavitation bubbles and cause more cavitation collapse. In addition, the wavelength was longer when the ultrasonic frequency was lower, and cavitation events were unevenly distributed in the solution, which was also

the reason for the increase of fluorescence intensity and fluorescence peak area from 30 kHz to 40 kHz. However, the increase range is much smaller than the decrease range of ultrasonic frequency from 20 kHz to 30 kHz. Generally speaking, the cavitation intensity of low frequency is higher than that of high frequency.

4 Conclusions

In this paper, the ultrasonic cavitation experiment platform was equipped, and ultrasonic treatment experiments were carried out in TA solution. The effects of ultrasonic power, irradiation time, and ultrasonic frequency on ultrasonic cavitation intensity were studied by measuring fluorescence intensity and fluorescence peak area with the F-7000 fluorescence spectrophotometer. The fluorescence intensity and fluorescence peak area of the samples increased by 2–20 times after ultrasonic treatment. The research conclusions were as follows:

- (1) The fluorescence intensity and fluorescence peak area could characterize the cavitation intensity, which increased rapidly after ultrasonic cavitation treatment, and then increased slowly with the increase of ultrasonic power, and could reach the maximum values of 302.7 and 21198. With the increase of ultrasonic power, the growth rate of cavitation would increase, and the cavitation reaction would be more intense. The energy released by cavitation collapse would increase, and more molecular bonds would break, so that more ·OH would be released to react with TA, increasing TAOH content, then the fluorescence intensity and fluorescence peak area would be enhanced.
- (2) The fluorescence intensity and fluorescence area were gradually increased with the extension of irradiation time and could reach the maximum values of 1387 and 95451, which was the most influential factor. This was because the fluorescence was determined by the content of fluorescent substance TAOH, which needed enough time for reaction. When the irradiation time was more than 25 min, the growth rate of fluorescence intensity slowed down, which was caused by the decrease of gas content and TA concentration in the solution.
- (3) The fluorescence intensity and fluorescence peak area first decreased and then increased with the increase of ultrasonic frequency from 20 kHz to 40 kHz, and they could reach the maximum values of 274 and 19130. This was due to the comprehensive influence of the changes of cavitation oscillation period, cavitation energy, and the number of cavitation bubbles caused by the variation of ultrasonic frequency. In a word, the cavitation inten-

sity of low frequency was higher overall. The study quantitatively characterized the cavitation intensity, reflecting the advantages of fluorescence analysis, and provided a means of the measurement of ultrasonic cavitation intensity and a basis for the further application of ultrasonic cavitation.

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Authors' Contributions

LY contributed significantly to the conception of the study and wrote the manuscript, and provided the funding assistance. SC carried out the experiments and analyzed the experiment results. XZ performed the analysis, and revised the manuscript. DW helped perform the analysis with constructive discussions. All authors read and approved the final manuscript.

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Data availability

The data are available from the corresponding author on reasonable request.

Declarations

Competing Interests

The authors declare no competing financial interests.

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