

Effects of Ultrasound Working Parameters on the Ultrasonic Power Density Some Neglected Problems in the Application of Ultrasound Bath

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ABSTRACT: *Ultrasound has been widely employed in food industry, while some of its working parameters are usually neglected to be described in its application, which caused the difficulty of reproducing and comparing the results obtained by different groups. So it is definitely not sufficient to simply state the ultrasound conditions such as ultrasound power, frequency and temperature, additionally, other parameters including the geometry of the reaction vessel, vessel distance, initial temperature of water, ultrasonic power density and positions used for fixing vessel are also very important for the actual/practical ultrasound power delivered to the targeted solution. Considering these facts, the above-mentioned factors were systematically conducted to study their effects on the actual ultrasound power, i.e. ultrasound power density for achieving maximum benefit, as a result, to highlight the importance of fully describing the ultrasound working parameters about its application. As Expected, the results confirmed our assumption that nearly all the studied parameters did have a great influence on the actual ultrasound power density dissipated into the targeted solution, excluding vessel position. In a word, this research would make this paper valuable to the scientific community, underline the importance of these neglected or rejected problems in the application of ultrasound bath, and persuade the readers to give some more thoughts to these issues.*

KEYWORDS: *Ultrasonic power density; Frequency; Initial temperature; Vessel; Ultrasound application.*

INTRODUCTION

Recently, ultrasound has been widely applied in many fields, especially in food industry, such as extraction, emulsifying, drying, filtration, separation, cleaning [1-5], promoting alcohol fermentation and controlling

crystallization [6, 7]. However, the ultrasonic equipment and practical working parameters used in the experimental section are very poorly described, since these are the key factors affecting the ultrasound

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efficiency, and the reproducibility and comparability of the results obtained. Perhaps anyone who is not familiar with these problems related to the application of ultrasound bath might consider this subject insignificant, or of only minor importance. For example, many researchers did not give the ultrasonic power of input in the reaction or extraction system in articles, and only gave the electrical power of the ultrasonic transducers (namely theoretical ultrasonic power), but which supplied by the manufacturer has a great gap with the actual power employed. *Vinatoru* [8] definitely pointed out that it is not sufficient to state simply the ultrasonic equipment used in the experimental part and it is also necessary to mention the ultrasonic frequency of the machine and the actual power used for the extraction. On the other hand, the actual power is related to many factors such as initial reaction temperature, the volume of water in the bath, container shape and so on. If the electrical power is directly used in the experiment, it is difficult to repeat the experimental data due to the ambiguity of the ultrasound conditions. *Mason* [9] has reported that the ultrasonic energy input to a chemical reaction was affected by the following important reaction parameters: theoretical power, the presence of bubbled gas, temperature, solvent composition and reaction volume. *Vinayak* [10] pointed out that the rate of sonochemical reactions were influenced by the shape of reactor, operating power density, fraction of dissolved gases, physicochemical properties of liquid medium, surrounding pressure field in the sonochemical reactor and operating temperature. In addition, *Weber* [11] also reported the tube distance above the transducer affected measurements of the intensity of ultrasonic cavitation. However, we believe that the factors affecting the actual ultrasound power density are more than the above-mentioned, i.e. theoretical ultrasonic power, frequency, volume of water in the ultrasound bath and vessel position in the ultrasonic bath may also have a great influence on the actual power. So it is necessary to systematically discuss the influences of different factors on the actual power in order to provide more comprehensive information about the utilization of ultrasound in food industry, that is to say, what conditions or ultrasound working parameters should be pointed out. In this way, the results obtained by different groups can be more comparative and repeatable. This study is a very important supplement to the above literatures.

The expression of the ultrasound power included the ultrasound electrical power/theoretical power (W, namely the power displayed by the instrument), acoustic energy and power density (expressed with W/cm^2 for ultrasound probe or W/L for ultrasound bath) [12]. Therefore, it is very important to use the definite ultrasound power to reflect the actual power dissipated into the reaction/extraction system. In our opinion, the expression "W/L" is the preferable expression of ultrasound power shown in the materials and methods section of a research work. Generally, the indirect methods are operable to measure the acoustic energy [13, 14], namely the Radiation Force Balance (RFB), the Weisler reaction and the calorimetric methods [13, 15-18]. RFB can be the most accurate method of measuring acoustic energy below approximate 20 W. The Weisler reaction is a chemical dosimeter method for measuring the acoustic energy [19, 20]. Measurement of cavitation intensity using the thermocouple probe has been shown to offer an efficacious correlating parameter for extraction in the ultrasonic field [21].

Considering the above-reviewed studies, seven factors (theoretical power, ultrasonic frequency, initial temperature, volume of water in the ultrasound bath, vessel shape, position in the ultrasonic bath and vessel distance) were systematically studied to understand their effects on the ultrasonic power density calculated by the calorimetric method so as to highlight these neglected problems in the application of ultrasound bath.

EXPERIMENTAL SECTION

Experimental designs

The following variables were examined by using one factor experimental design to investigate the effects of different ultrasound conditions on the ultrasonic power density (W/L) including theoretical ultrasonic power, ultrasonic frequency, initial temperature, volume of water in the ultrasound bath, vessel shape, vessel position in the ultrasonic bath and vessel distance (the distance between the bottom of vessel and the bottom of the ultrasonic bath), respectively. Ultrasonic treatments were carried out in an ultrasonic bath (KQ-300VDE, KunShan Ultrasonic Instruments Co. Ltd., Jiangsu Province, China) which can work at the frequencies of 45, 80 and 100 kHz with a variable theoretical power output of 120 to 300 W. Ultrasonic energy was delivered from the bottom to the

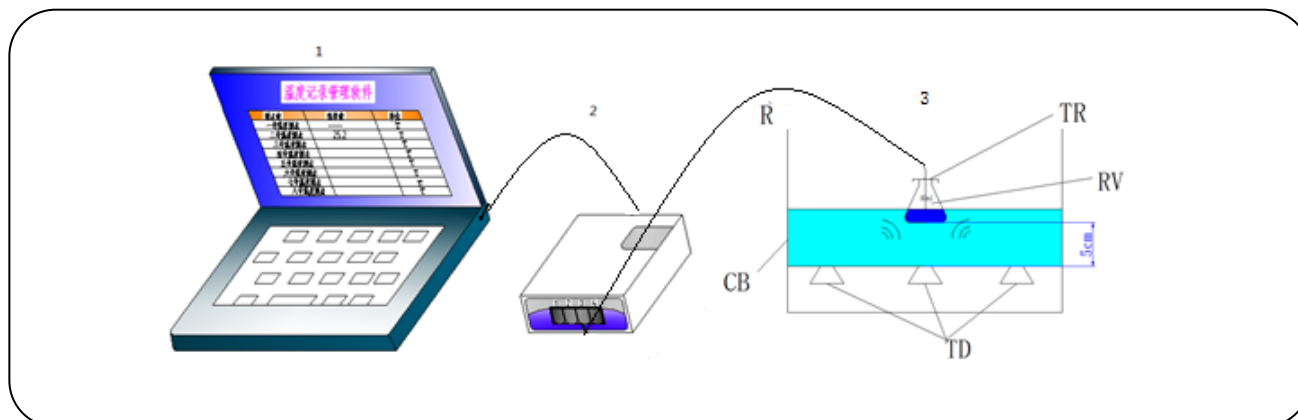


Fig.1. Experimental configurations for measuring, 1=Computer, 2=M-5000 temperature collector, 3=KQ-300VDE ultrasonic cleaning bath (Ultrasonic cleaning bath KQ-300VDE. CB= cleaning bath, TC= thermocouple, R= recorder, RV= reaction vessels, TD= transducer).

water in the tank by 6 annealed transducers, and their rated maximum power output was 300 W. The experimental setup is shown in Fig. 1. The ultrasound device was wrapped with foam to prevent heat loss in all the experiments. M-5000 temperature collector (Beijing Anfu Electronic Co. Ltd., China) placed in the ultrasound bath or vessel was used to record the temperature changes, respectively. And the distilled water was employed all through the experiments as it has excellent and constant properties as a standard solvent.

Calculation of ultrasonic power density

The ultrasonic power density was calculated by the method of *Kimura* [15] and *Mason* [9] with slight modifications. The temperature (T) increase measured by M-5000 temperature collector was recorded against time (t), with the interval of 1 min. From the T versus t data, the temperature rise at zero time, dT/dt , could be estimated by curve-fitting the data to a polynomial equation in t. The ultrasonic power actually entering the system might then be obtained by substituting the value of dT/dt , at time zero, thus obtained into Equation (1):

$$\text{Power} = (dT/dt)c_p M \quad (1)$$

Where c_p is heat capacity of the solvent (J/kg·K) and M is the mass of solvent used (kg).

If this power (W) is dissipated from ultrasound bath or vessel of volume V (dm^3), then the density of power (W/dm^3) produced by the source of ultrasound is given by Equation (2):

$$\text{ultrasonic power density} = \text{ultrasonic power}/\text{volume} \quad (2)$$

All measurements for ultrasonic power density were repeated for three times and average values were reported.

Single factor experiment design

Seven sets of experiments were performed to discuss the effects of seven factors on the ultrasonic power density.

Effects of the theoretical ultrasonic power on the actual ultrasonic power density

The effect of different theoretical ultrasonic power levels (120, 150, 180, 210, 240, 270 and 300 W) was investigated, being the ultrasonic treatments performed at the initial temperature of 20 °C for 30 min with a constant ultrasonic frequency, a 100% water level (4.29 L) of the bath and a constant position C (Fig.2). Then the contact terminal of the M-5000 temperature collector was immersed in the ultrasound bath to record the temperature changes. After that, the ultrasonic power density was calculated by Equation (2).

Effect of ultrasound frequency

The effect of ultrasound frequency was assessed with 45, 80 and 100 kHz, respectively, at the theoretical ultrasound power level of 120 W for 30 min with the initial temperature of 20 °C, 100% water level and a constant position C (Fig.2).

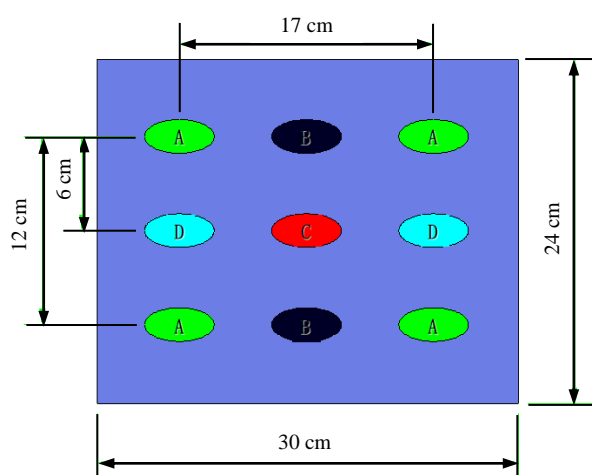


Fig. 2: Ultrasonic location in the bath divided into zones of A, B, C and D.

Effect of initial temperature

A set of experiments were carried out with the frequency of 45 kHz and theoretical ultrasound power of 120 W at the different initial temperatures of 10, 15, 20, 25, 30, 35, 40, 45 and 50 °C which were regulated by circulating pump of cryogenic coolant (XiangYa Instrument and Equipment Co. Ltd., Shanghai, China) for 30 min with a 100 % water level and a constant position C (Fig. 2).

Effect of water volume in ultrasound bath

In order to investigate the effect of water volume in ultrasound bath on the ultrasonic power density, different volumes of water were assayed including 1.5, 2.5, 3.5, 4.5, 5.5, 6.5 and 7.5 L. For this set of experiments, the selected ultrasound power, frequency, initial temperature and position in ultrasonic bath were 120 W, 45 kHz, 20 °C and position C (Fig.2), respectively.

Effect of vessel shape

The effects of vessel shape on the delivered ultrasonic power were investigated. The vessels used included beaker (50, 100 and 200 mL), paper cup (50 and 150 mL), conical flask (50,100 and 150 mL), distillation flask (250 mL round bottom and 250 mL flat bottom) and centrifugal tube (50 mL tip bottom and 50mL spherical bottom). For these experiments, the selected theoretical ultrasound power, frequency, initial temperature, position in ultrasonic bath and vessel distance were 120 W,

80 kHz, 20 °C, position C (Fig.2) and 5 cm, respectively. 50 mL vessel was filled with 20 mL of water, while other vessels were filled in proportion to the corresponding water, i.e. 100, 150, 200 and 250 mL vessels were filled with 40, 60, 80, 100 mL water. After that, the vessels were fixed in the ultrasonic bath.

Effect of position of the vessels in the ultrasonic bath

The vessel position in the ultrasonic bath is also a very important parameter, which can affect the ultrasonic power density significantly. In order to study the influence of different positions on the power density, the ultrasonic location in the bottom of bath wall was divided into several zones marked with A, B, C and D, respectively (Fig.2), and the vessel height above the transducer was essentially kept constant. The contact terminal was fixed at a designated height within the 50 mL conical flask to measure temperature change rate at the four positions of A, B, C and D, respectively. The theoretical ultrasound power, frequency, initial temperature and vessel distance were fixed with 120 W, 80 kHz, 20 °C and 5 cm, respectively.

Effect of vessel distance

The effect of vessel distance on the ultrasonic power density was assayed including 1, 2, 3, 4, 5 and 6 cm. For this set of experiments, a 50 mL conical flask was used as the vessel, and the working parameters of ultrasound were with the frequency of 80 kHz, theoretical power of 120 W, time of 30 min, initial temperature of 20 °C, a 100% water level and a constant position C (Fig.2), respectively.

RESULTS AND DISCUSSION

Effects of the theoretical ultrasonic power on the actual ultrasonic power density

As shown in Fig. 3, the ultrasonic power density calculated was significantly lower than the theoretical ultrasonic power displayed by the instrument (namely electrical power of ultrasonic transducers), and the maximum multiple was up to 10 times. This phenomenon can be boiled down to two reasons. On the one hand, the deviation between the actual power and the theoretical power provided by the equipment might come from the equipment fatigue and breakdown of long working time. On the other hand, part of the ultrasonic energy did not

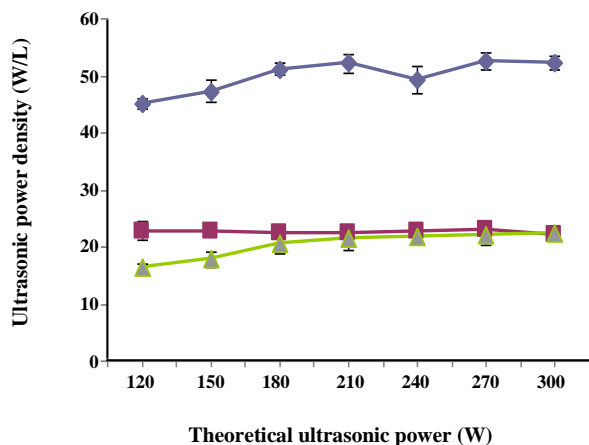


Fig. 3: Effects of the theoretical ultrasonic power on the actual ultrasonic power density.

contribute to the rise of the water temperature but contributed to a rise in the temperature of the bath wall which was not included in the calculation of ultrasonic power density. Consequently, the ultrasonic power calculated from the water temperature was smaller than the displayed value of the ultrasound [13]. For example, when the maximum ultrasonic power density was about 52 W/L, it was only 74 % of the theoretical power density of 70 W/L (theoretical power of 300 W).

Fig. 3 also demonstrates that the ultrasonic power density increased correspondingly with the increase of the theoretical ultrasonic power, which can be obviously observed from the results that, when the theoretical ultrasonic power ranged from 120 to 300 W, the power density with the frequency of 45 and 100 kHz correspondingly increased from 44.96 to 52.27 W/L, 16.36 to 22.3 W/L, respectively. But unexpectedly, when the frequency was fixed at 80 kHz, the ultrasonic power density did not have obvious variation ($P > 0.05$) with the changes of theoretical ultrasonic power, which may explain the reason why some articles described that the change of theoretical power had little effect on the response [22]. It is well-known that the ultrasonic wave shall be strong enough to ensure the formation of vibratory cavitation in reaction liquid after it goes through the bottom wall of vessel. From this sense, not all the ultrasonic cleaning bath has enough power to serve as a chemical reactor. So it is very important to confirm whether the actual power is suitable for controlling chemical reactions or not. This has also proved that

the use of theoretical power is unreasonable and it is necessary to calculate the ultrasonic power density actually delivered into the mixture regarding the application of ultrasound.

Effect of changes in ultrasonic frequency

As mentioned-above, the ultrasonic frequency is also a very important parameter of ultrasound in any studies of sonochemistry. In Fig. 3, it can be observed that the ultrasonic power density descended significantly with the increase of frequency ($P < 0.01$). To be specific, when the theoretical ultrasonic power was fixed at 120 W, the power density induced by ultrasonic frequency was 44.96, 22.78 and 16.36 W/L at the employed three frequencies of 45, 80 and 100 kHz, respectively. These are in accordance with the results reported by Vinatoru [23], who conducted a similar research on the extraction of alkaloids, and the results showed that the degradation of the alkaloids was very obvious at the lower frequency.

Generally, the maximum temperature obtained from the bubble collapse decreases with the increase of ultrasonic frequency according to the bubble dynamics, while the cavitation threshold increases with the frequency increasing, which leads to a more narrow active field to grow the bubbles in an uneven sound field [24, 25]. Besides, the shearing force generated by ultrasonic waves is one of the reasons causing the degradation reaction. When the frequency is reduced, the shearing force will increase with the enhancement of cavitation effect [26]. As a result, the ultrasonic power density goes down with the increase of frequency on the whole. Based on these facts, the frequency should be pointed out and lower frequency is suggested regarding the application of ultrasound bath in extraction of active compounds.

Influence of initial temperature of water in bath on the ultrasonic power density

Kimura [15] has mentioned that the temperature of the water inside the reactor/bath should be thermostated at an appropriate value before the irradiation of ultrasound. Therefore, in order to confirm whether initial temperature influences the rise of water temperature, experiments were conducted to investigate the effect of initial water temperature on the ultrasonic power density. As shown in Fig. 4, the power density did not indefinitely

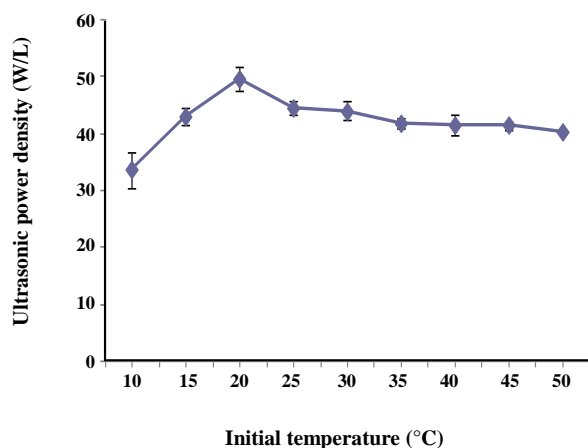


Fig. 4: Influence of initial temperature of water on the ultrasonic power density.

increase with the increasing of the initial water temperature, but reached a certain optimum value, after which any further increase in initial water temperature would decrease the power density.

In general, the ultrasound experimental results under different temperatures may be attributed to the combination of the cavitation effect and thermal effect, for instance, from the perspective of cavitation effect, temperature rising has a negative effect because of the decreased cavitation intensity. To be specific, cavitation bubbles can be generated at lower ultrasound intensity with the increasing of vapor pressure of the liquid media. Thus, the bursting strength of the cavitation bubbles is lower, and the ultrasonic chemical effect caused by the collapse of the bubble is correspondingly reduced [14]. Nevertheless, from the viewpoint of thermal effect, the increase in temperature has a positive influence because it can increase many chemical reaction rates. *Entezari & Kruus* [27] have reported that the iodine liberated (Weissler's reaction) at the ultrasonic frequency of 900 kHz increased with the increase of temperature within a maximum temperature, and followed by a reduction of iodine production. Therefore, it should be clearly noted here that, where chemical reactions are occurring, an optimum operating temperature might exist. This may be attributed to the fact that the higher concentration of chemical species is present in the cavitation bubbles due to the higher vapor pressure at a higher operating temperature, which results in formation of much higher amounts of free radicals in the liquid leading to higher reaction rate [28].

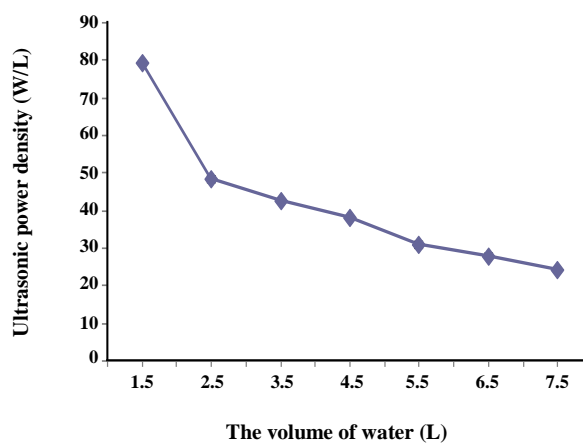


Fig. 5: Effect of the water volume in ultrasound bath on the ultrasonic power density.

As also can be seen in Fig. 4, the initial water temperature of 20 °C owned the maximum ultrasonic power density among all the temperatures employed, and the possible cause of this phenomenon may come from the surrounding environment impact on the temperature measurement during ultrasound irradiation. To be specific, if the initial water temperature in bath is much lower (below the room temperature), more heat energy will be dissipated during ultrasonication, while if the temperature is much higher, the water temperature will be very difficult to be increased. Generally, only at temperature close to the room temperature, the heat loss would be minimized and the dT/dt would be increased. Therefore, in case of without consideration of the requirements of chemical reactions, the water temperature in the ultrasound bath would be better consistent with the room temperature in the ultrasonic processing, and the initial temperature of about 20 °C is suggested in the application of ultrasound.

Effect of the water volume in the ultrasound bath

With other variables controlled, the effect of water volume in the ultrasound bath on ultrasonic power density was studied, The obtained results in Fig. 5 illustrated that the value of ultrasonic power density decreased when the water volume in the bath increased. That is to say when the theoretical ultrasonic power and frequency were fixed, the smaller the volume of water is, the higher the temperature rises. Form shown in Fig. 5, the highest ultrasonic power density was about 79.45 W/L

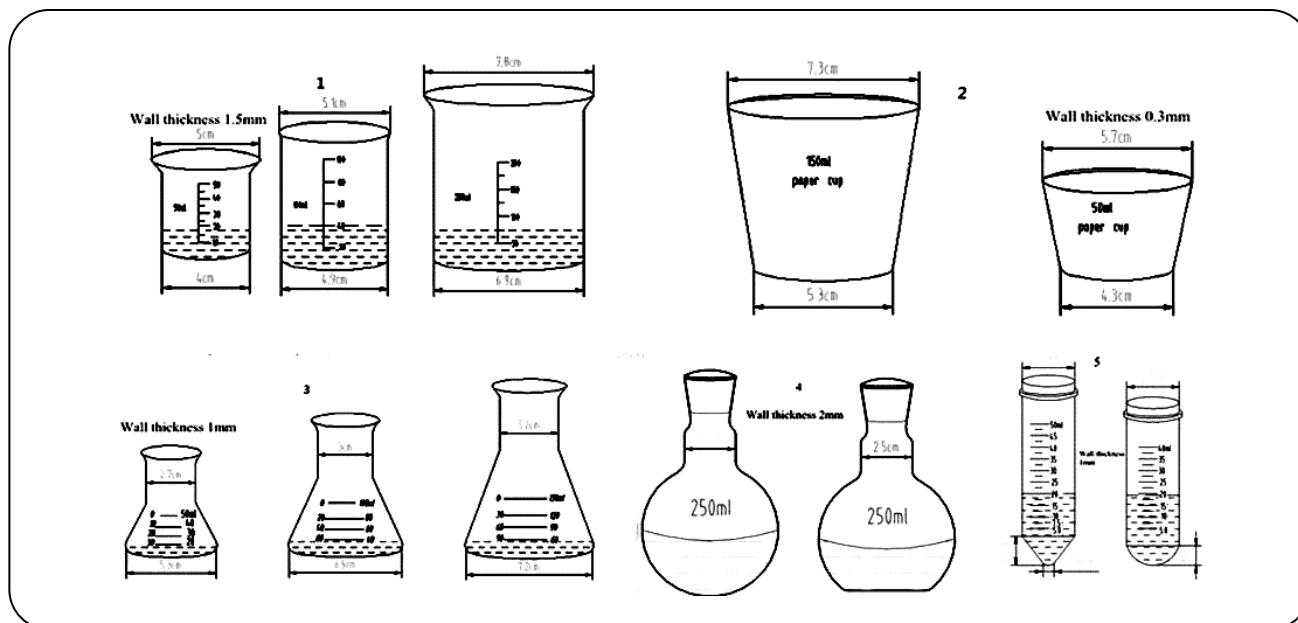


Fig. 6: Some commonly used experiment vessels in laboratory. 1=beaker (50, 100 and 200 mL), 2=paper cup (50 and 150 mL), 3=conical flask (50,100 and 150 mL), 4=distillation flask (250 mL round bottom and flat bottom distillation flask).

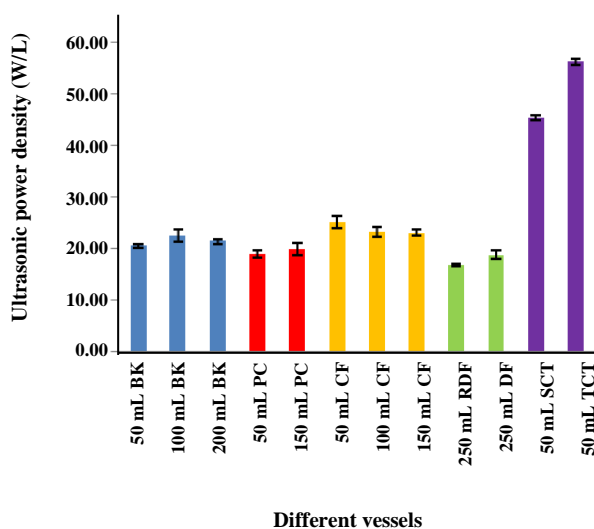


Fig. 7: Effect of different vessels on the ultrasonic power density. TCT=tip bottom centrifugal tube, SCT= spherical bottom centrifugal tube, BK=beaker, PC=paper cup, CF=conical flask, RDF=round bottom distillation flask, DF=distillation flask.

when the water volume was 1.5 L, and it is the minimum amount allowed for the ultrasonic cleaning bath of KQ-300VDE. Based on this result, water volume between 2.5-4.5 L (about 58 % to 100% water level of the ultrasound bath capacity) is suggested in the application of ultrasound.

Effect of different vessels

As suggested, in order to properly describe the experimental work conducted with ultrasound, the geometrical feature of vessels employed (round or flat bottomed and the diameter of the base of the flask) should be well stated [8]. In this study, some experiment vessels commonly used in laboratory were shown in Fig. 6 and the influence of these different experimental vessels immersed in water bath on the ultrasonic power density was also discussed. As shown in Fig. 7, the vessel shape did have significant ($P < 0.01$) effect on the ultrasonic power density and the maximum powder density value was 39.43 W/L higher than the minimum value. The highest ultrasonic power density was about 56.13 W/L (the mean value of three measurements) and obtained by using the 50 mL centrifugal tube of tip bottom. Interestingly, the change of water temperature in centrifugal tube was different with the other vessels as shown in Fig. 8. The calculation of ultrasonic power density was divided into two parts, and the results were 45.6 W/L and 56.9 W/L by using centrifugal tubes with spherical bottom and tip bottoms, respectively. These results may be attributed to the different heat transfer coefficients and acoustic impedance of materials used for vessels, leading to a difference in the absorption and reflection of ultrasonic wave, such as paper cup, glass and

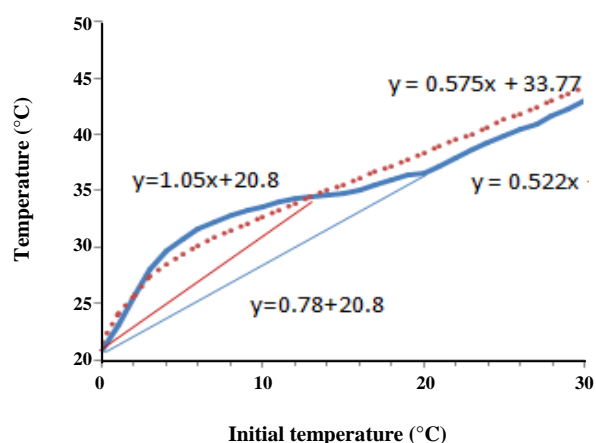


Fig. 8: Change of water temperature in the centrifugal tube.

plastic container. The acoustic impedance between the water in the ultrasound bath and the vessel is very higher. A large portion of the ultrasonic wave is reflected on the surface of the vessel, that is, the energy loss of the ultrasonic wave is very large. For example, when the employed vessel is glass, the reflectivity is as high as 70 %, that is, only 30 % of the ultrasound efficiency can be utilized [29]. In the meantime, the larger external diameter of the vessel mouth the more heat dissipated, the smaller external diameter of the vessel mouth the higher ultrasonic power density obtained, such as beaker and conical flask. In addition, the thickness of the vessel is also important for the propagation of the heat, such as the distillation flask and the conical flask. Furthermore, the measured datas from round bottom flask and flat bottom flask were also different, and the ultrasonic power density obtained with the flat bottom of distillation flask was generally higher than that of the round bottom distillation flask. Regarding the reason, when the bottom of flask is flat, sound waves will be vertical to incident, and when the bottom of flask is spherical, sound waves will be oblique incidence. The acoustic energy of vertical incident in the reaction solution is more effective than that of an oblique incidence. This reduction in power density is believed to be due to the partial cancellation of waves generated by and reflected from the hemispherical bottom of the flask [11, 30]. The same result was found by the fact of the power density of tip bottom centrifugal tube greater than that of the spherical bottom centrifugal tube. This further demonstrated that the spherical bottom

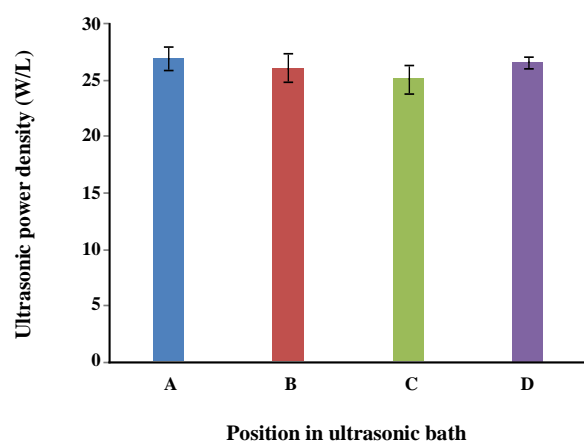


Fig. 9: Effect of position in ultrasonic bath on the ultrasonic power density.

container reflected the greater partial cancellation of waves than that of other shape bottom containers. Hence, attentions on the vessels employed (flat bottom flask or tip bottom centrifugal tube is suitable) should be focused in order to get higher power density during ultrasound application.

Effect of the position in ultrasonic bath

Kanthale [31] has pointed out that the erratic behavior of cavitation activity exhibited in a sonochemical reactor pose a serious problem in the efficient design and scale-up. Thus it becomes important to identify the active and passive zones existing in the reactor so as to enable proper place of the reaction mixtures for achieving maximum benefits. In this study, the ultrasonic energy was delivered from the bottom to the water in the tank by the 6 annealed transducers, which were namely the positions of A and B in Fig.2. As shown in Fig. 9, no significant changes ($P > 0.05$) were observed in the power density among the four kinds of positions (A, B, C and D). To be specific, the power density at the same height above the bath bottom was not greatly affected by the different ultrasound irradiation zones. The possible reason for the inconsistency with the results of *Kanthale* [31] is that the ultrasonic vibration might make the water temperature in the ultrasonic bath more uniform.

In brief, the position of the vessel placed in the bath has no significant influence on the ultrasonic power density due to the good thermal conductivity of water.

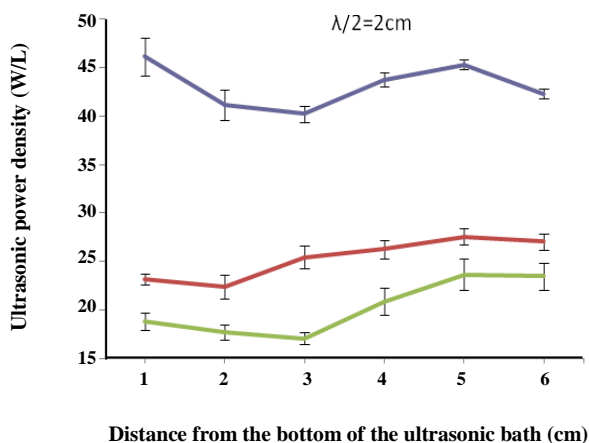


Fig. 10: Effect of distance from the bottom of the ultrasonic bath on the ultrasonic power density.

Effect of vessel distance

The influence of the vessel distance on the power density was examined in this paper, since many authors have reported the influence of the vessel distance on the chemical yield [11, 15, 18]. Theoretically, when the vessel distance is equal to the half wavelength of the ultrasound wave, the cavitation would be more intense [32, 33].

The vessel distance was shown in Fig.1 and the effect of different vessel distance on the ultrasonic power density was studied at three frequencies. The results in Fig.10 indicate that the power density varied with the vessel distance changing at the same position of C, and the variation took on a similar changing trend on the whole. More specifically, with the frequency of 45 kHz as an example, the higher power densities were obtained at the vessel distance of 1 cm and 5 cm above the transducer, respectively. With the ultrasound frequency of 45 kHz, its one-half wavelength is about 2 cm, thus the highest power density should be theoretically obtained at the one-half wavelength position. This result is consistent with the reports of Kimura [15] and Niemczewska [32], which is expected since resonance occurs in a non-cavitating system when a rigid wall is placed at an integral half wavelength from the source of sound [11]. But when the frequency was fixed at 80 kHz and 100 kHz, the ultrasonic power density was not obviously influenced. The possible reason is that the wavelength of 80 kHz and 100 kHz is shorter, and ultrasonic vibration can produce a certain distance error. In addition, the thickness of the vessel wall and the bath wall might also have a certain effect on the measurement of the vessel distance to the power density.

CONCLUSIONS

As discussed above, the ultrasonic power density was definitely influenced by the ultrasonic parameters employed including theoretical ultrasonic power, ultrasonic frequency, initial temperature, volume of water in ultrasound bath, vessels and the vessel distance. These results suggest that the attention should not only be focused on experiment process, but also on the ultrasound equipment and its optimum working conditions. Based on the results obtained in this paper, the conditions are suggested as follows in future application of ultrasonic bath. The water volume in ultrasound bath can be controlled between 58 % to 100 % water level of the ultrasound bath capacity. The vessel should also be considered, and a flat bottom, glass material and small caliber vessel such as conical flask or tip bottom centrifugal tube is preferred according to our results. In case of no consideration of the reaction requirements, the initial water temperature should be about 20 °C in accordance with the room temperature. Additionally, the vessel distance should be controlled with the integer times of 1/2 wavelength at the corresponding frequency in order to receive the maximum ultrasound power dissipated. In a word, researchers should give more serious consideration to these neglected or rejected paths.

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