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A phenomenological investigation into the opposing effects of fluid flow on sonochemical activity at different frequency and power settings.

1. Overhead stirring

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ABSTRACT

The effect of flow in an ultrasonic reactor is an important consideration for practical applications and for the scale-up of ultrasonic processing. Previous literature on the influence of flow on sonochemical activity has reported conflicting results. Therefore, this work examined the effect of overhead stirring at four different frequencies, 40, 376, 995 and 1179 kHz, in two different reactor configurations. Comparable power settings were utilised to elucidate the underlying mechanisms of interactions between the flow and sonochemical activity. The sonochemical activity was determined by the yield of hydrogen peroxide, measured by iodide dosimetry, and the active region was visualised with sonochemiluminescence imaging. The overhead stirring in the low frequency reactor altered the yield of hydrogen peroxide so it produced the maximum yield out of the four frequencies. The increase in hydrogen peroxide yield was attributed to a reduction in coalescence at 40 kHz. However at the higher frequencies, coalescence was not found to be the main reason behind the observed reductions in sonochemical yield. Rather the prevention of wave propagation and the reduction of the standing wave portion of the field were considered.

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1. Introduction

Ultrasonic processing is a potential alternative for enhancing industrial processes in applications such as waste water treatment [1] and biomass treatment [2]. Ultrasound causes violent collapse of bubbles in liquids, producing radicals from oxidation reactions inside the collapsing bubble [3]. These radicals are able to react with components of the solution and can enhance radically driven processes. For the industrial potential of ultrasound to be realised the effect of different reactor parameters on the radical yield in solution such as stirring, frequency and power need to be elucidated.

At lower frequencies (under 100 kHz) the bubble has more time to grow and therefore the cavitational collapse is more violent. Consequently, studies looking for mass transfer improvement generally opt for the lower frequency range. At higher frequencies more bubbles are produced which collapse, producing more radicals. Mason et al. [4] demonstrated the inverse dependence of mechanical and chemical effects on frequency in their treatment of a polyphenylene ether [4]. Additionally, increased sonochemical activity at high frequencies was shown in a comparative study by Entezari and Kruus [5]. Hence at higher frequencies a higher number of radicals means processes such as oxidations are faster at higher frequencies [6]. At even higher frequencies, as in the megahertz range, the rarefaction of the wave becomes too short for maximal sonochemistry effects [6]. The exact maximum frequency for cavitation to occur is also dependent on the geometry, temperature, ambient pressure, viscosity and the gas composition of the reactor solution. Although, there is general agreement radically driven processes are maximised at high frequencies and mechanical effects are maximised at low frequencies.

Previous literature reports involving of sonochemical activity measurements under the influence of flow have found conflicting results. At 23, 44, 99 and 141 kHz, the introduction of flow was able to enhance sonochemical activity [7,8]. Additionally, a cyclic flow study on the degradation rate constant of chlorobenzene found the degradation rate increased with an increase in flow rate at 94 kHz [9]. However at 133.2 kHz, with flow across the face of a multi-transducer array, the rate constant was decreased with the introduction of flow, when there was no reflection of the ultrasonic wave [10]. Furthermore, at 500 kHz the sonochemical degradation rate of pentachlorophenol was decreased by the introduction of flow [11]. Lastly, at 490 kHz an overhead stirrer increased the sonochemical efficiency and at 486 kHz stirring was able to enhance sonochemical yield in some cases [12,13]. The current literature presents evidence for an increase in sonochemical activity from the introduction of flow in an ultrasonic reactor at high and low frequencies. However it is difficult to compare the effects of

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flow in reactors of varied geometry since reactor geometries are also known to influence sonochemical activity [14–16].

At low frequencies coalescence of active bubbles leads to large degas bubbles, which are inactive, whereas at high frequencies small, inactive bubbles rely on coalescence to become larger active bubbles Therefore, the observed increases of sonochemical activity at low frequencies was theorised to be caused by a reduction of bubble coalescence and aggregation. Then, at high frequencies, flow was hypothesised to also reduce coalescence but to decrease the active bubble population [7]. The observed increases in sonochemical activity at higher frequencies were attributed to increased mixing and the provision of additional nucleation sites [12,13]. Since the literature produced conflicting results, the present work was aimed to test these hypotheses at different frequencies. This was done via the introduction of an overhead stirrer at 40, 376, 995 and 1179 kHz at two different power settings. The overhead stirrer was positioned so that the increase of nucleation sites would be minimised. In order to fully evaluate the effect of flow we will also characterise the ultrasonic field using sonochemiluminescence imagery and surface stabilisation.

2. Methodology and experimental details

2.1. Reactors

Experiments were conducted with a low frequency reactor (Meinhart Ultraschalltechnik, 90 mm diameter, 40 kHz) and a multi-frequency reactor (Meinhart Ultraschalltechnik, 57 mm diameter, 376, 995 and 1179 kHz). For experiments with overhead stirring a propeller overhead stirrer was used (Caframo), with two propeller blades of total diameter of 55 mm, positioned 67 mm and 138 mm from the surface of the transducer in the low- and multi-frequency reactors, respectively. The volume of liquid was 500 ml, which was liquid height of 87 and 155 mm from the transducer base in the multi-frequency and the 40 kHz reactors, respectively. Fig. 1 shows the schematic representations of the 40 kHz (Fig. 1(i)) and the multi-frequency (Fig. 1(ii)) reactors. Surface stabilisation was achieved with a lid made of foam and coated in parafilm, shaped to float on the surface of the reactor solution. The volumetric power densities from the dissipation of ultrasound in each reactor at each power setting were measured for comparative values via calorimetric technique [17] and are shown in Table 1. The maximum input power was 250 W and 240 W for the multi-frequency and the 40 kHz reactors, respectively. It should be noted that the input power settings were the same at each frequency for the multi-frequency reactor.

Table 1
Ultrasonic power at each setting as determined by standard calorimetric techniques.

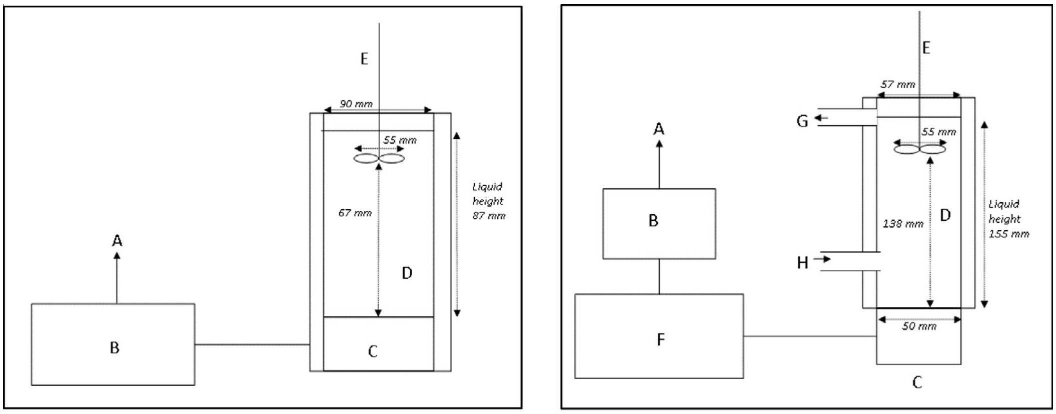
Frequency (kHz)	A ($W L^{-1}$)	B ($W L^{-1}$)
40	72 ± 1	92 ± 1
376	58 ± 1	76 ± 4
995	52 ± 1	72 ± 7
1179	50 ± 4	72 ± 7

2.2. Iodide dosimetry

The yield of hydrogen peroxide was determined via iodide dosimetric techniques [17]. Briefly, 500 ml of 0.1 M potassium iodide (Ajax Finechem) was sonicated at room temperature under various experimental conditions for 30 min, and the yield of the triiodide ion was measured spectrophotometrically. The absorption of triiodide was measured on a Hewlett Packard 8453 Ultraviolet–Visible Spectrophotometer at the wavelength 351 nm. Calibration curves were constructed each day of measurement using solutions of triiodide standardised against sodium thiosulphate (Ajax Finechem). Dosimetry experiments were repeated four to seven times for each experimental setting. The yield of triiodide represented the yield of hydrogen peroxide produced ultrasonically.

2.3. Sonochemiluminescence images

Sonochemiluminescence images were taken of the multi-frequency reactor, the low-frequency reactor was made of stainless steel and so imaging was not possible. The sonochemiluminescent solutions were made of stock solution (2.5 mmol L^{-1} luminol and 2.5 mol L^{-1} sodium carbonate, Sigma–Aldrich) in air-saturated deionised water (1/100 v/v) [7,18]. The sonochemiluminescence images were taken with a Digital Single-Lens Reflex (DSLR) camera (Lumix) with a 60 s exposure time. The images were all taken in the first minute of the introduction of ultrasound after an initial waiting period of 10 s. Each image was taken using a fresh solution, in a dark box and at least two images were captured at each setting. The colour temperature of the images was decreased to identify the blue regions of the images due to sonochemiluminescence. Images were processed with identical colour treatment in Picassa version 3.



(i) (ii)

Fig. 1. Schematics of (i) the 40 kHz reactor. (LHS) A – power source, B – sinus wave generator, C – ultrasonic transducer, D – reaction vessel, E – overhead stirrer. (ii) Schematic of the multi-frequency reactor (RHS). A – power source, B – sinus wave generator, C – ultrasonic transducer, D – reaction vessel, E – overhead stirrer, F – power amplifier, G – flow outlet, H – flow inlet.

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