A novel particle separation technique using 20-kHz-order ultrasound irradiation in water

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Abstract. Ultrasound techniques such as washing, fine-particle manipulation and mixing have been investigated. MHz-band ultrasound was usually used in the previous work, and studies of kHz-order ultrasound are very rare. In the usual manipulation technique, \(\mu\)m-order particles are targeted due to wavelength limitations. We discovered an interesting phenomenon that holds promise for a novel particle separation technique using kHz-order ultrasound. Here, particles with sub-mm- or mm-order diameters were flocculated into a swarm in water irradiated by 20-kHz ultrasound. To develop a practical separation process, we investigated the stationary position and dia. of the particle swarms and the sound-pressure profiles in a vessel, as well as the flocculation mechanism, by varying the irradiation frequency, water level, particle diameter and particle amount. The primary stationary position corresponded to the wavelength calculated from the resonant frequency regardless of the particle diameter. Subtle changes in the frequency and water level resulted in a significant change in the stationary position. Based on these results, we propose a new separation process based on the particle diameter for sub-mm- or mm-order particles.

1. Introduction

Many research groups have been devoted to the investigation of MHz-band ultrasound techniques such as ultrasonic cleaning \[1\], fine-particle manipulation \[2\] and ultrasonic atomization \[3\]. In contrast, studies of kHz-order ultrasound techniques are very rare. In addition, regarding fine-particle manipulation, only \(\mu\)m-order particles are targeted due to limitations of the wavelength.

We discovered an interesting phenomenon when particles with diameters ranging from sub-mm to mm were flocculated into a spherical swarm in water irradiated by 20-kHz-order ultrasound. The present study’s authors Mizushima and Saito \[4\] reported that the particle flocculation of a spherical swarm was caused by acoustic cavitation-oriented bubbles adhering to particles. The stationary position of the swarm shifted with a change in the particle concentration and particle diameter. Higher particle concentrations and larger diameters resulted in a lower stationary position.

In the present study, as part of our attempt to develop a new separation process, we investigated the stationary position and diameter of the particle swarm and the sound-pressure profile in a vessel, as well as the flocculation mechanism. Based on the experimental results obtained from careful control of the...
irradiation frequency, water level, particle diameter and particle amount, we propose a novel, practical and diametrical particle separation process against equivalent density and different-diameter particles, using kHz-order ultrasound.

2. Experimental Setup

Figure 1 is a schematic of the experimental setup used in this study. A sine wave voltage signal (frequency 19.7–20.6 kHz) was supplied to a bolt-clamped Langevin-type transducer (HEC-45254M, Honda Electronics, Tokyo) through a power amplifier circuit. Ultrasound was irradiated upwardly/vertically from the bottom of an acrylic water vessel (inner size: 54 mm × 54 mm). The coordinate origin was defined as the intersection point at the bottom of the vessel and the centre of the transducer. The $z$-axis was defined as the vertical direction, and the $x$-$y$ plane was horizontal. The water temperature in the vessel was controlled at approx. 20ºC.

2.1. Experiment 1: Measurement of the sound-pressure profile

Sound-pressure profiles in the vessel were measured through a hydrophone (HPM1/1 and HP1, tip dia. 1.5 mm, sensor dia. 1.0 mm; Precision Acoustics, Dorchester, UK) under several irradiation frequencies and water levels. The vessel was filled with degassed ultrapure water. The degassed water was made with a vacuum pump at −0.1 MPa for 24 hours. The water level was set at 65, 90 or 120 mm. The measurement range of the $z$-direction was set from 2 mm of $z$ axis to water surface. The hydrophone was moved at a rate of 4 mm/s in the $z$-direction by the servo actuator. The sound pressure was stored in a digital recorder (8861-50, Hioki, Nagano, Japan).

2.2. Experiment 2: Visualization of particle flocculation

To examine the new flocculation phenomenon in a practical separation process using high-speed visualization, we measured the stationary position and diameter of the spherical particle swarm under various parameters: water level 65 mm, 90 mm and 120 mm; irradiation frequency 19.7–20.6 kHz; particle dia. 400 µm and 800 µm, and the load amount of the particles. The density of the particles was 1.05 g/cm$^3$. The particle flocculation was filmed by a high-speed video camera (Phantom V9, Vision Research, Bedford, UK). The image size was 720 × 1200 pixels (resolution 81.8 µm/pixel), and the frame rate was 500 fps (exposure time 400 µs).

| Condition  | Dia. [µm] | Mass [mg] | Dia. [µm] | Mass [mg] |
|------------|-----------|-----------|-----------|-----------|
| Single-400 | 400       | 400       | –         | –         |
| Single-800 | –         | –         | 800       | 400       |
| Mix        | 400       | 400       | 800       | 400       |

Table 1. Particle diameters and load amounts of particles

Snapshots: Upper (C): bubble adhering to the surface of a particle. Lower (B): particle flocculation in the vessel.
3. Results and discussion

3.1. Results of Experiment 1: Measurement of the sound-pressure profile

Figure 2 shows the sound-pressure profile results for the central axis of the vessel under the irradiation frequency of 20.0 kHz and three water levels. With the increase in the water level, a standing wave formed from a traveling wave from the vessel bottom, and a reflected wave from the water surface grew to a clearer pattern with nodes and anti-nodes. The sound-pressure profile gradually became a suitable profile for trapping the particle swarm. Figure 3 shows the sound-pressure profile for the central axis under the water level of 120 mm and irradiation frequencies of 19.7–20.6 kHz. With the increase in irradiation frequency, the positions of the anti-nodes gradually shifted upward. Based on these results, we discuss the relationship between stationary positions of the particle swarm and the pressure profile of the standing wave below in Section 3.2.

3.2. Results of Experiment 2: Visualization of particle flocculation

The stationary positions and diameters of the spherical particle swarm in the examined water levels are plotted in Figures 4 and 5, respectively. The swarm was trapped in the node and anti-node of the standing wave mentioned above. Under the condition of Single-400 and 19.7 kHz, the swarms were flocculated at two positions: the upper anti-node and the lower node. At the water levels of 65 and 90 mm, the Single-800 results were similar to the results obtained with the other conditions. Interestingly, the Single-800 results at the water level of 120 mm were significantly different from the Single-400 results and Mix results. The Single-800 swarm was shrinking in size under the frequencies of 19.7 and 20.2 kHz (black dots plotted in Figs. 4c and 5c). These phenomena occurred in case of transitional conditions between the cases particle flocculation trapped in the upper position or lower position. With the increase in the irradiation frequency to 20.2 kHz, the stationary position shifted toward the anti-node, and the Single-800 swarm flocculated at the lower anti-node was growing in size again. Although we observed the partial composition of the same-diameter particles, we speculate that this difference in the stationary position between Mix and Single-800 is the result of influences of the smaller particles on the behaviour of the larger particles. These phenomena must be related to the bubbles adhering to the particle surface (Fig. 1c) [4] and the fine structure of the sound-pressure profile; however, the mechanism of this particle flocculation is still unknown. In order to develop a precise separation process for sub-mm- or mm-order particles, further investigations to reveal as-yet unidentified phenomena are needed.

![Fig. 2. Normalized sound pressure (NSP) on the central axis under several water levels, frequency 20.0 kHz.](image1)

![Fig. 3. NSP on the central axis under several irradiation frequencies at a water level of 120 mm.](image2)

![Fig. 4. Stationary position under several water levels and frequencies. The resonant frequency measured through a lock-in amplifier.](image3)
3.3. A proposed novel particle separation process, and a demonstration

Based on the above results, we propose a novel particle separation process based on particle diameter, and we demonstrate its performance herein. Figure 6 shows the process flow and typical behaviour of the particle swarms during processing. Mixture particles were flocculated in the upper region in the initial phase (Fig. 6a). With the slight reduction of the irradiation frequency, the particles were immediately sinking (Fig. 6b–d). After that, with the return of the irradiation frequency to the initial value, the smaller particles re-flocculated in the upper region (Fig. 6e,f). The larger particles re-flocculated in the lower region. The entire process took approx. 10 sec. This separation mechanism is closely related to the sound-pressure profile; therefore under a water level of 120 mm (at which a clear sound-pressure profile was formed), a small transition in the sound-pressure profile played an important role against the particle separation. The stationary position of the particle swarm differed by particle diameter (see [4], Fig. 6b). Based on these results, the minimum differential diameter in this separation technique might be 200 µm when the particle mixture condition is set at 400 µm or 600 µm.

4. Conclusion

Dispersed particles with a sub-mm diameter in water were flocculated into a spherical swarm through irradiation by kHz-order ultrasound. The stationary position and diameter of the particle swarm were investigated under various irradiation frequencies, water levels and particle conditions. We observed that the stationary position and the swarm diameter were well controlled through subtle changes in the frequency and water level. We also discovered that the stationary position for each particle diameter was significantly shifted during the transition state brought about by the changes in the frequency and water level. Based on these results, we propose a novel particle separation process based on the particle diameter, and we have demonstrated its performance.

References
[1] Li J and Sanderson R D 2002 J. Membr. Sci. 205 247–257
[2] Kozuka T 2005 J. ASJ 61 154–159
[3] Julianna C S et al 2012 Phys. Med. Biol. 57 8061
[4] Mizushima Y, Nagami Y, Nakamura Y and Saito T 2013 Chem. Eng. Sci. 98 395–400