

# High-Frequency Ultrasound Technology for Biofuel Production

Subjects: [Engineering](#), [Chemical](#)

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High-frequency ultrasound (HFU) is an ultrasound technology with a frequency higher than 1000 kHz. It has become increasingly recognized as an emerging process intensification technology in various fields, such as biofuel production, carbon dioxide absorption, and wastewater treatment. HFU is seen as a potential intensifier technology for biofuel production, as its mechanisms, such as cavitation phenomena, microstreaming, and fountain formation, can benefit biofuel production.

biodiesel

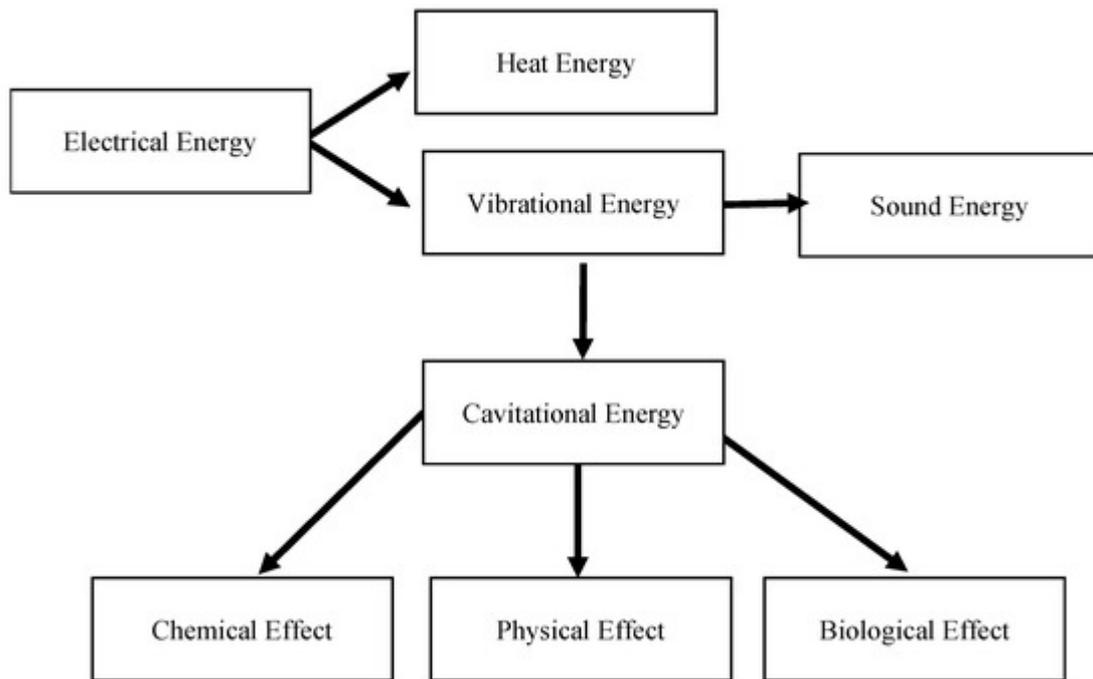
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## 1. Introduction

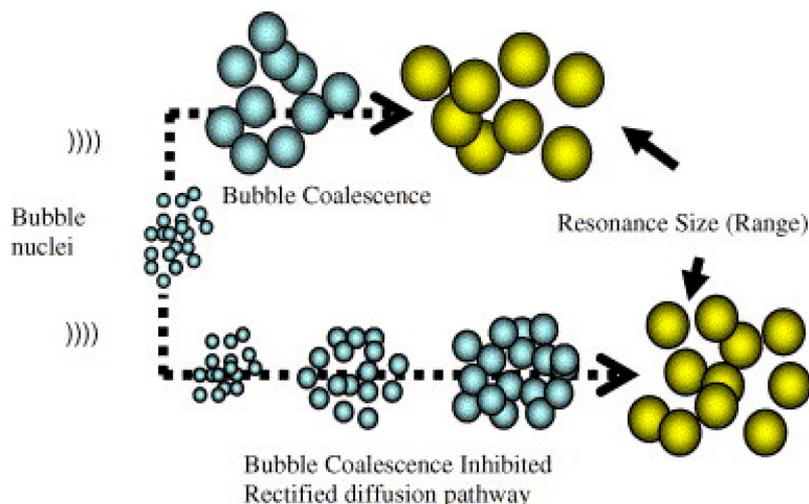
Ultrasound is a type of mechanically oscillating sound wave that can be sustained by an elastic medium, such as air or water. Its frequency ranges from 20 kHz to 10 MHz, which is higher than the limit of human auditory perception (i.e., between 16 Hz and 20 kHz) <sup>[1]</sup>. According to Chuah et al. <sup>[2]</sup>, ultrasound can be classified into two categories based on its frequency: high-frequency ultrasound (HFU), which has a frequency range of 1000–10,000 kHz, and low-frequency ultrasound (LFU), which has a frequency range of 20–100 kHz. While HFU is more commonly used in medical applications, its use in non-medical fields has been increasing due to its unique characteristics and mechanisms. The application of ultrasound technology has extended to different fields, such as carbon dioxide (CO<sub>2</sub>) absorption <sup>[3][4][5]</sup>, chemical synthesis <sup>[6]</sup>, wastewater treatment <sup>[6][7]</sup>, medical imaging <sup>[8]</sup>, algae biomass disruption <sup>[9]</sup>, biodiesel production <sup>[10]</sup>, and the food industry <sup>[11][12]</sup>.

Ultrasonic applicators can produce different forms of energy by converting electrical energy into heat and vibrational energy using an ultrasonic probe <sup>[13]</sup> or discs <sup>[14]</sup>. **Figure 1** illustrates the transformation of electrical energy into various forms of energy using an ultrasonic applicator.



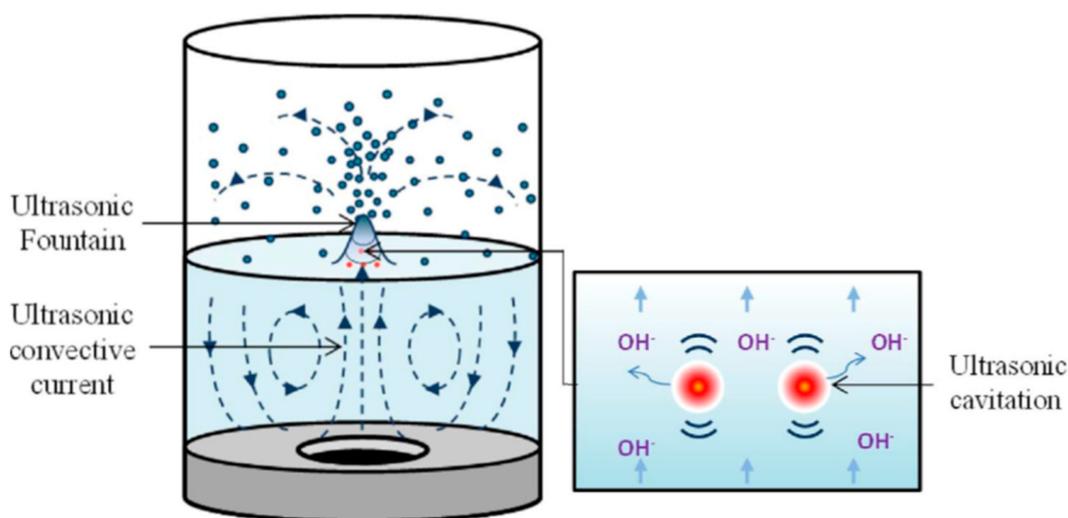
**Figure 1.** Energy transformation in ultrasonic applications.

Ultrasound technology can produce different types of effects, including biological effects, such as the break down of algal cell membranes for extraction [15]. Its physical effects, such as ultrasonic cleaning, emulsification, and atomization, have been extensively utilized in commercial products [16]. Atomization emits micro-droplets when the acoustic intensity exceeds a liquid-dependent threshold [17]. On the other hand, the implosion of bubbles during ultrasound irradiation can lead to the formation of highly reactive species, such as  $\text{OH}^{\bullet}$ ,  $\text{HO}_2^{\bullet}$ , and  $\text{H}^{\bullet}$ , which contributes to the chemical effects of ultrasound technology [18]. The benefits of ultrasound technology extend to various fields, including wastewater treatment, where the vibrational energy from ultrasonic cavitation can help to break down pollutants. The cavitation phenomenon is the formation of bubbles that grow and collapse in a liquid that has been irradiated with ultrasound [19]. **Figure 2** illustrates how bubble nuclei in water grow to reach resonance size through a rectified diffusion pathway and a coalescence pathway under the influence of an ultrasonic field. Resonance bubble size is important in ultrasound applications because it affects the effectiveness of the cavitation process. The size of the cavitation bubbles is influenced by the ultrasonic waves, with low-frequency ultrasound producing larger-diameter bubbles, resulting in stronger shear forces, while higher-frequency ultrasound generates smaller and more stable bubbles (stable cavitation) [20][21]. At lower frequencies, the implosion of cavitation bubbles is more intense compared to that at higher frequencies. At higher ultrasonic frequencies, a larger number of cavitation bubbles are formed, but the collapse intensity is lesser, and higher powers are needed for active bubble formation [22].



**Figure 2.** Schematic illustration of bubble nuclei growth in water through a pathway of rectified diffusion and coalescence under the influence of an ultrasonic field (adapted from Ashokkumar et al. [23] with permission from the publisher).

The frequency and cavitation intensity of ultrasound are inversely proportional to each other, as the formation of cavitation bubbles becomes more difficult with shorter rarefaction phases at higher frequencies [1]. At higher ultrasonic frequencies (>1 MHz), jet-like fountain formation and ultrasonic streaming force are enhanced [24]. Shokrollahi et al. [5] found that the fountain formation created under high-frequency ultrasound contributes to the enhancement of mass transfer process. **Table 1** summarizes the differences between low-frequency ultrasound and high-frequency ultrasound, while **Figure 3** shows the schematic drawing of the behavior of liquid under high-frequency ultrasound conditions.



**Figure 3.** Schematic drawing of the formation of an acoustic fountain (adapted from Tay et al. [3] with permission from the publisher).

Various parameters, such as power, frequency, reaction condition, operation mode, and mechanical vibrations, can affect the occurrence and intensity of ultrasonic effects [25]. Therefore, understanding the interaction between these parameters is crucial in enhancing the use of high-frequency ultrasound for different purposes.

**Table 1.** Comparison between high- and low-frequency ultrasound.

Low-Frequency Ultrasound	Characteristics	High-Frequency Ultrasound
20–100 kHz [2]	Frequency Range	1000–10,000 kHz [2]
Production of larger bubbles [20]	Cavitation Bubbles Size	Production of smaller bubbles [21]
Lesser [22]	Quantity of Bubbles	Higher [22]
Higher intensity [20]	Bubble Collapse Intensity	Lower intensity [20]
Lesser free radical formed [26]	Amount of Free Radical Formation	Higher free radical formed [26]
None or weak fountain formation [27]	Formation of Fountain (Figure 3)	Yes [28]

Schueller and Yang [29] observed that cavitation phenomena formed under lower frequencies aid in the desorption process more than absorption and adsorption processes. At higher frequencies, the cavitation phenomena are less intense, and fountain formation increases mass transfer coefficients for CO<sub>2</sub> absorption. The role of high-frequency ultrasound is slowly gaining attention across different fields, particularly in biofuel production. The mechanism of HFU (e.g., cavitation phenomena, jet-like fountain formation, and ultrasonic streaming force) plays a major role in the formation of eddies, turbulence, and shear forces that enhance the transesterification process [30][31], directly influencing the production of biodiesel. Meanwhile, cavitation phenomena aid in the breakdown of algal cell walls, improving the extraction process of products such as biofuel.

According to Hasan et al. [32], research conducted on biofuel production increased from the year 2005 onwards, with an increase in the number of published articles on the subject being evident over the last 16 years, and 2019 marked the highest rate of publications. Climate change and increasing energy demands are major contributors to the expansion of biofuel economics, and biofuel plays a vital role in addressing the increasing energy demand as a potential energy source [33]. However, the production of biofuel is still not economically viable, and more research and development for technological enhancement is required [34].

Various intensifying technologies have been applied to the production of green biofuels. Still, each technology possesses different advantages and disadvantages. LFU is well-established for biodiesel production [35], but there is still a lack of reports on HFU as an intensification technology for this application [31]. Therefore, a great deal of uncertainty regarding the relationship between HFU technology and biofuel production exists.

## 2. High-Frequency Ultrasound Technology for Biofuel Production

Reliance on fossil fuels for energy and transportation is having detrimental effects on the environment. The combustion of fossil fuels releases greenhouse gases, such as CO<sub>2</sub>, sulfur dioxide (SO<sub>2</sub>), and nitrogen oxide (NO<sub>x</sub>), which contribute to global warming. This situation is alarming as society and industries continue to rely heavily on the use of fossil fuels in their power plants and as a primary source of energy for transportation fuel. Therefore, clean, sustainable, and renewable energy sources are critically needed to reduce the negative environmental impact and ensure a continuous supply of energy for the future. Biofuel, a new potential energy source derived from different renewable feedstocks, may reduce the reliance on the usage of non-renewable fossil fuels. Demirbas [36] denoted biofuel as liquid, gas, and solid fuels produced from biomass. Meanwhile, according to Raboni et al. [37], biofuel comprises any product obtained from biomass and this includes biodiesel, biofuels, biogas, bioethanol, and bio-methanol. Biofuel production is highly supported by various countries, including Brazil, Germany, Switzerland, and Sweden, as efforts have been intensified in order to reduce the reliance on fossil fuels as an energy source.

Biodiesel, or fatty acid methyl esters (FAME), is regarded as a potential source to replace the use of fossil fuels and has been studied globally [38]. Biodiesel can be potentially utilized as an energy resource in the coming future, as it can be produced from numerous potential feedstocks, such as microalgae [38][39][40][41], waste cooking oil [42][43], animal fat [44][45], by products such as rice bran [46], and various vegetable seeds oil [47][48][49][50]. According to Mahbub et al. [51], the usage of biodiesel has several benefits, including a reduction in CO<sub>2</sub> and carbon monoxide (CO) emissions by 8–41%, based on life cycle assessment studies. Previous research has proposed the use of biodiesel as a complete or partially mixed alternative for diesel engines, since biodiesel usage reduces exhaust emissions as the composition of biodiesel contains less carbon, water, and sulfur with a higher amount of oxygen than conventional petroleum [52]. Biodiesel can be introduced into diesel engines without modifications because the current diesel engines are compatible with biodiesel [53].

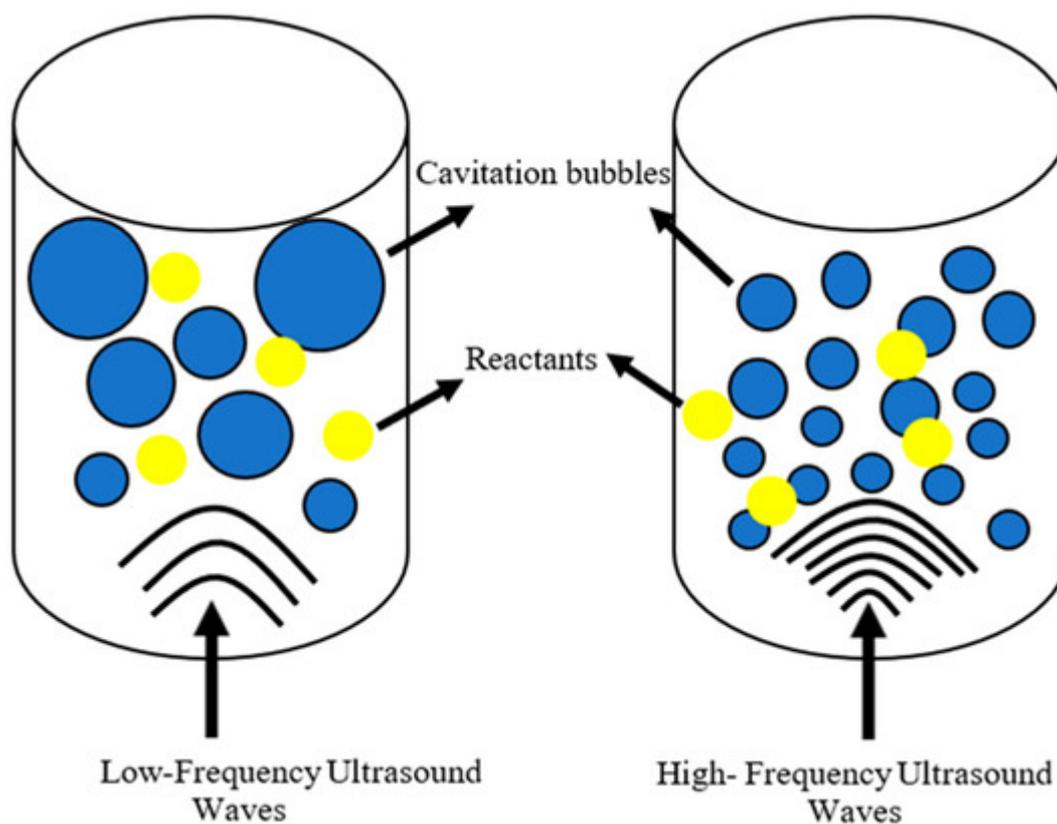
### 2.1. High-Frequency Ultrasound-Assisted Transesterification Process for Biodiesel Production

Transesterification is a more popular method for producing biodiesel compared to other methods, such as the direct use of blended oils, micro-emulsion of oils, and thermal cracking (pyrolysis) of oils [30][54]. This process converts feedstock (i.e., oil) into methyl or ethyl esters using an alcohol source, such as methanol or ethanol, and a catalyst. Glycerol is produced as a side product during the reaction [55]. Transesterification is carried out at a mild temperature [56] and requires simpler conditions in comparison to other methods. For example, micro-emulsification requires different alcohol solvents with colloidal microstructures [57], while pyrolysis requires high temperatures ranging from 300 °C to 1300 °C, which may lead to changes in the chemical structure of the compounds [58]. Therefore, transesterification is more economically feasible and simpler, making it the preferred method for producing biodiesel compared to other methods [59].

However, the production of biodiesel via the transesterification process still faces some challenges. Firstly, the heterogenous nature of the reactants (alcohol and vegetable oil) does not form a homogenous mixture [43][60], which requires intensive mixing processes to increase the mass transfer rate, resulting in a higher power consumption. Additionally, the viscosity of the different feedstocks used may differ, leading to higher or lower agitation requirements. Moreover, the two-way reaction of the transesterification process requires a higher alcohol-to-oil molar ratio. An excess of alcohol aids in product formation instead of reactant formation, resulting in higher conversion rates that directly lead to increasing biodiesel expenditure [61].

Ultrasound-assisted transesterification is considered an approach to enhance biodiesel production. The characteristics of ultrasound technology, such as cavitation bubbles, microstreaming, and fountain formation, enhance mixing between heterogenous mixtures, reducing the reliance on additional mixing processes. The application of ultrasound technology allows for higher reaction rates and lower alcohol-to-oil molar ratios [62][63]. LFU has been widely researched for transesterification processes [15][30][64][65] due to the high-intensity collapse of cavitation bubbles, which aids in overcoming the mass transfer limitation. However, there is still a lack of reports on the use of HFU in the transesterification process [31], which requires further exploration.

Recent studies have shown that high-frequency ultrasound technology is beneficial for biofuel production. The phenomena of cavitation, jet-like fountain formation, and ultrasonic streaming force are prominent characteristics that facilitate the mixing of immiscible liquids, such as alcohol and oil. However, it remains unclear which of these characteristics is more advantageous for mixing immiscible liquids. Oliveira et al. [31] studied the influence of low power (3–9 W) and high frequency (1 MHz and 3 MHz) without an external heating source on the transesterification of soybean oil. The results showed that ultrasound-assisted transesterification increased soybean oil conversion from 48.7% to 79.5% when the alcohol-to-oil ratio was 6:1 at 1 MHz and 3 MHz, respectively, while a molar ratio of 8:1 at 1 MHz and 3 MHz achieved a conversion of 59.5% to 84.6%, kept at the same reaction time of 40 min. Within 10 min, the HFU of 3 MHz achieved a conversion of 79.8% compared to the HFU of 1 MHz, at a conversion rate of 53.6% after 20 min at a 8:1 M ratio. This shows that HFU along with excess methanol can result in a higher reaction rate within a shorter time period, as the excess methanol shifts. This is because the diameter of the cavitation bubbles produced by ultrasonic frequency is dependent on the frequency, causing variations in bubble size [31]. Since the size of the cavitation bubbles is inversely proportional to ultrasonic frequency [66][67], this may, to a certain extent, facilitate higher collision rates between reactants, leading to a greater conversion rate (**Figure 4**).



**Figure 4.** Differences between the amount of cavitation bubbles between LFU and HFU.

Another study conducted by Mahamuni and Adewuyi <sup>[18]</sup> focused on the synthesis of biodiesel via the ultrasound-enhanced base-catalyzed transesterification of soybean oil using a multifrequency ultrasonic reactor. The study revealed that high-frequency ultrasound at 1.3 MHz resulted in more than 90% conversion within 30 min. Aghbashlo et al. <sup>[35]</sup> conducted a separate study on the development and evaluation of a high-frequency piezoelectric-based ultrasonic reactor to intensify the transesterification reaction. The study noted that the transesterification of waste cooking oil (WCO) achieved the highest conversion efficiency of 99.3% at 10 min of ultrasonication with an elevated alcohol/oil molar ratio of 8:1. According to the authors, increasing the ultrasonication time and liquid media temperature improved the conversion efficiency. The longer exposure time allowed for an enhanced emulsification between the alcohol and oil phases due to the increased number of bubbles produced using HFU. The study suggested that HFU may outperform LFU in terms of conversion efficiency, energy requirement, and processing time. **Table 2** provides a summary of the reaction conditions for the HFU-assisted transesterification studies conducted to date.

**Table 2.** Reaction conditions for high-frequency ultrasound-assisted transesterification.

	Authors		
	Oliveira et al. <sup>[31]</sup>	Mahamuni and Adewuyi <sup>[18]</sup>	Aghbashlo et al. <sup>[35]</sup>
<b>Feed</b>		Soybean oil	Waste cooking oil (WCO)

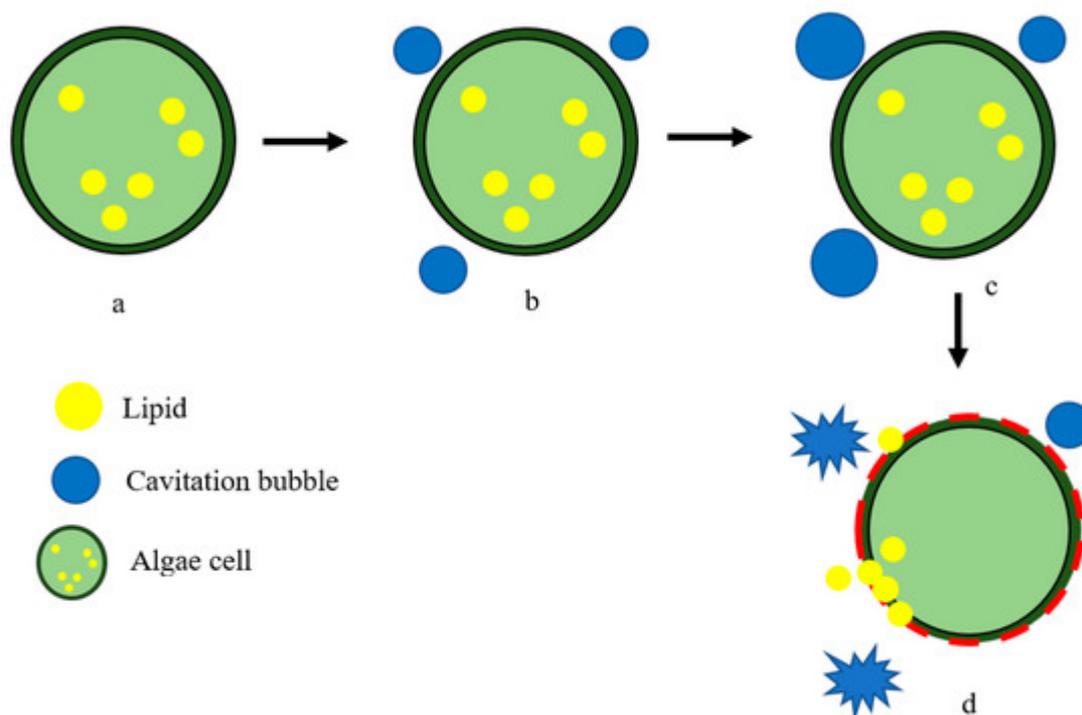
Authors			
Ultrasonic Reactor Type	Transducer		Piezoelectric-based ultrasonic reactor
Catalyst	Potassium hydroxide (KOH)		
Alcohol	Methanol		
Conditions	Study was conducted at the frequencies of 1 MHz and 3 MHz with alcohol-to-oil molar ratios of 6:1 and 8:1, respectively, at a reaction time of 40 min (without external heating or mechanical stirring).	Study was conducted at the frequencies of 323 kHz, 581 kHz, 611 kHz, and 1.3 MHz with an alcohol-to-oil molar ratio of 6:1 and a power between 12 and 223 W, with a reaction time ranging from 60 min to 180 min.	Study was conducted at the frequency of 1.7 MHz with alcohol-to-oil molar ratios of 4:1, 6:1, and 8:1 and a power of 31 W, with the reaction times of 6, 8, and 10 min.

HFU has the potential to aid in biodiesel synthesis, particularly in enzyme-catalyzed biodiesel production. However, most studies have focused on acid- and base-catalyzed transesterification processes [22][68]. The lack of research on enzyme-catalyzed biodiesel production was highlighted by Veljković et al. [69], where it was noted that enzyme-catalyzed ultrasound-assisted transesterification could be beneficial due to its advantages of higher selectivity, better energy efficiency, and lesser generation of by-products. Nonetheless, the impact of HFU on enzyme-catalyzed biodiesel production needs to be investigated further.

Additionally, different feedstocks, such as soybean oil and waste cooking oil, may affect the HFU mechanism due to their viscosity, which can hinder the cavitation process and require higher power supplies, leading to additional operating costs. It is expected that, as viscosity increases, the interactions between molecules become stronger, further hindering cavitation [70], which may reduce the efficiency of HFU during transesterification, necessitating the use of alternative solutions.

## 2.2. High-Frequency Ultrasound on Microalgal Cell Disruption for Biofuel Production

Microalgae, which are photosynthetic microorganisms that require minimal growth requirements, are considered a promising source of lipids, proteins, and carbohydrates. They can produce large quantities of bioproducts in a short time and, thus, are an ideal feedstock for biodiesel production. Microalgae are environmentally and economically advantageous due to their high growth rate and biomass productivity, and their ability to accumulate bioproducts, such as carbohydrates and lipids, under nutrient-limited conditions [71]. Unlike crops, which require growing cycles ranging from three months to three years [72], microalgae have a short growth cycle, and bioproducts such as lipids can be harvested in just 3–5 days. Once the microalgal biomass is cultivated, harvesting and dewatering are carried out, followed by lipid extraction from microalgae [73]. The use of ultrasound pre-treatment is known to increase the lipid yield from the microalgal biomass, as it aids in the disruption of biomass for lipid extraction [74]. Ultrasonic irradiation breaks down the microalgal cell wall and reduces microalgal particle size, leading to a better release of chemical content, thus enhancing extraction efficiency [75]. **Figure 5** illustrates the use of HFU technology to aid microalgal cell lysis.



**Figure 5.** Disruption of algal cell using ultrasound: (a) condition of algal cell prior to disruption; (b) formation of cavitation bubbles; (c) growth of cavitation bubbles; and (d) breakdown of algal cell wall and release of lipids.

The incorporation of high-power, low-frequency (i.e., 100 W and 33 kHz) ultrasound as pre-treatment in the transesterification of microalgal oil showed an increase in biodiesel yield by almost 40% compared to the mechanical stirring method [76]. While previous studies have focused on low-frequency ultrasound for algal cell disruption [77][78][79][80][81][82], high-frequency ultrasound for cell lysis has only recently been explored [9]. Several studies have been conducted to investigate the use of high-frequency ultrasound for the disruption of microalgal cells to aid in lipid extraction [9][78][83]. Wang et al. [9] evaluated the effectiveness of high-frequency focused ultrasound (HFFU) on the cell lysis of the microalgae *Scenedesmus dimorphus* and *Nannochloropsis oculata*. The effects of HFU at 3.2 MHz and 40 W input power on microalgal cell were evaluated in the study. The cell size for both *S. dimorphus* and *N. oculata* was found to decrease after HFFU treatment, and the lipid extraction for *S. dimorphus* increased, ranging between 49% and 113%, as the processing time increased from 1-min HFFU to 5-min HFFU. For *N. oculata*, the relative lipid extraction increased from 25% to 49%, which was lower compared to the increase observed for *S. dimorphus*. Hence, this indicated that the effectiveness depends on the cell (biomass) being treated. The results from the study revealed that using HFFU for cell disruption to assist lipid extraction in microalgae is a feasible method. On the other hand, Yamamoto et al. [78] investigated the effect of ultrasonic frequency ranging from 20 to 1146 kHz and different powers on the disruption of microalgal cells. HFU was found to have a more prominent effect on microalgal cell disruption compared to LFU, and the frequency required for disruption depends on the specific characteristics of the cell. The effect of HFU on *Chlamydomonas concordia* and *Dunaliella salina* was investigated at the different frequencies of 20, 580, 864, and 1146 kHz, as well as at the different power inputs of low (~3 W), medium (~20 W), and high (~60 W). The results showed that the highest frequency for *C. concordia* cell disruption was 1146 kHz, while there was an insignificant difference for *D. salina* at

580 kHz, 864 kHz, and 1146 kHz. However, the higher ultrasound frequency of 580 kHz showed better results than the ultrasound frequency of 20 kHz. The authors explained that the bubble radius ( $\mu\text{m}$ ) was significantly reduced from 160  $\mu\text{m}$ , at a frequency of 20 kHz, to 3.3  $\mu\text{m}$  at a high-frequency 1146 kHz. The resonance radius of 3.3  $\mu\text{m}$  was almost similar to the size of *C. concordia*, ranging between 3 and 6  $\mu\text{m}$ . Hence, HFU aids in the breakdown of vacuoles inside the cell as the size of the vacuole is similar to the resonance radius of the cavitation bubbles.

Varied frequencies for cell disruption are required for different species of algae, which can be explained by the distinct cell characteristics, as some species have thicker cell walls. Kurokawa et al. [83] conducted research on the effectiveness of ultrasonic waves with frequencies ranging from 0.02 MHz to 4.3 MHz on the disruption of algae cells using *Chaetoceros gracilis*, *Chaetoceros calcitrans*, and *Nannochloropsis* sp. The results showed that higher frequencies led to a higher cell disruption for all three microalga species. The most prominent cell disruption was reported at 2.2, 3.3, and 4.3 MHz for *C. gracilis*, *C. calcitrans*, and *Nannochloropsis* sp., respectively. *C. gracilis* showed approximately 100% of cell disruption in 2 min at 2.2 MHz, while at the lower frequency of 0.4 MHz, more than 60% of the cell remain undisrupted after 10 min. *C. calcitrans* showed, at the higher frequency of 3.3 MHz, almost 100% of cell disruption was achieved after 2 min, while at the lower frequency of 0.4 MHz, 60% of the cell remained undisrupted after 10 min. The same pattern was observed for *Nannochloropsis* sp., for which, at 4.3 MHz, nearly 90% of the cell was ruptured, while at lower frequency of 0.4 MHz, only 10% of the cells were ruptured. The research results suggest that HFU is more efficient than LFU at cell disruption, rupturing nearly 90% of cells in all three species studied. The ideal frequency for cell disruption differs among species and is dependent on frequency. This is due to the fact that the ideal frequency is determined by the mechanical properties of the cell, such as the thickness of the algal cell wall.

HFU technology can aid in the extraction of bioproducts from microalgal cells by disrupting their outer cell wall layer. The use of ultrasound is considered more environmentally friendly than using toxic solvents, such as chloroform, which can be harmful to the environment and human health. Since the bioproducts are synthesized inside the cell, the disruption of the cell wall (outer layer of the cell) is essential. However, the efficacy of this technology depends on the unique biological characteristics of the microalgal species, as some species have thicker cell walls that require higher frequency HFU for effective disruption, while others have thinner cell walls that may be susceptible to excessive disruption. An excessive disruption of the cell membrane may cause the bioproducts to disintegrate. Therefore, optimizing the conditions for HFU will require further research and investigation, particularly in the context of microalgae-based biofuel production.

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