

MICROALGAL OIL EXTRACTION AND TRANSESTERIFICATION



BACKGROUND

Biodiesel is a renewable, domestic and environmentally friendly fuel with a potential of becoming a broadly accepted substitute for petrodiesel. Its widespread implementation is, however, currently hindered by excessive costs of production and limited availability of feedstock oils.

Microalgae

Microalgae is one of the few sources of feedstock oil that can meet the existing demand. The major challenge, however, has been the high cost of recovering the oil from the microalgae prior to converting it into biodiesel. The most commonly used method involves mechanical cell disruption followed by hexane extraction. This approach has significant drawbacks when applied on a commercial scale because it involves a complicated and energy-intensive hexane distillation step. Furthermore, large quantities of hexane escape into the atmosphere, which contributes to air pollution and has significant replacement costs.

A promising method called "in-situ transesterification" allows to completely eliminate the oil extraction and refining steps from the biodiesel production process. Instead, the oil is extracted directly into methanol (or ethanol) premixed with a catalyst, where it simultaneously undergoes transesterification and becomes converted to biodiesel. In-situ transesterification has been tried on various oil-bearing materials, including microalgae, yielding promising results. Furthermore, laboratory studies have shown that in-situ transesterification of microalgal oil benefits from exposing the reaction mixture to ultrasound.

ULTRASONIC EXTRACTION AND TRANSESTERIFICATION

Biodiesel production involves two processes: oil extraction and transesterification, both of which are greatly accelerated by high-amplitude ultrasound. Acoustic cavitation created by ultrasound tremendously facilitates solvent access through cell walls and promotes oil extraction. It also provides very efficient mixing of oil and alcohol, which speeds up the phase transfer-limited transesterification reaction. These effects occur due to the mechanical action of ultrasonic cavitation, which produces violently and asymmetrically imploding bubbles and causes micro-jets that pierce cell walls.

WHY ISM ULTRASONIC TECHNOLOGY?

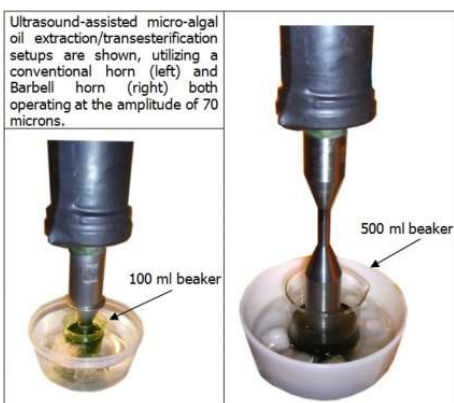
Laboratory studies show that high ultrasonic amplitudes, on the order of 70 - 120 microns peak-to-peak (below, microns), are necessary to maximize the efficiency of ultrasound-mediated extraction and transesterification. Conventional high-power ultrasonic technology, however, inherently forces all processes to run either at a small scale and high amplitude or a large scale and low amplitude, not allowing for the possibility of implementing high amplitudes on an industrial scale. Consequentially, scaling up a traditional ultrasonic system is always associated with a reduction in ultrasonic amplitudes, decreasing the resulting shear forces and sacrificing process efficiencies.

Industrial Sonomechanics, LLC, (ISM) has successfully overcome the aforementioned limitation by developing Barbell Horn™ Ultrasonic Technology ([BHUT](#)), which permits constructing industrial ultrasonic systems able to operate at extremely high ultrasonic

amplitudes (up to about 200 microns). The output tip areas of the incorporated Barbell horns and the resulting productivity rates of the systems are more than 10 times higher than those of any conventional ultrasonic device operating at high amplitudes. Any laboratory study can, therefore, be directly implemented on the industrial scale, without lowering ultrasonic amplitudes or changing any other optimized process parameters.

EVALUATION EXPERIMENTS CONDUCTED BY ISM

In order to assess the feasibility of industrial-scale ultrasonic microalgal oil extraction and biodiesel production, ISM conducted a set of evaluation experiments. All experiments were run in a batch mode and utilized ISM's 1200 W bench-scale ultrasonic system, [BSP-1200](#).



Conventional and Barbell Horn™ Setups

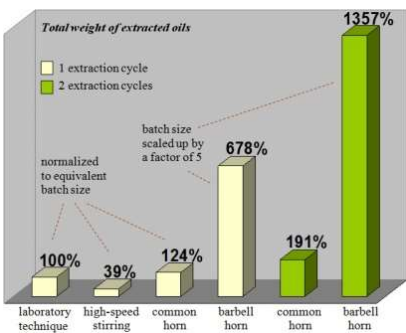
The system comprised an ultrasonic generator, a piezoelectric transducer and either a conventional converging ultrasonic horn with an output tip diameter of 15.7 mm or a Barbell horn with a tip diameter of 35 mm. The ultrasound exposure cycles were 3 minutes for both setups. The ultrasonic amplitudes provided by the converging horn and the Barbell horn were the same - 70 microns. However, since the diameter of the Barbell horn was 2.23 times larger, its output area was 5 times larger, and, therefore, this horn could process 5 times more material during the same amount of time.

This comparison was done to demonstrate the ability of Barbell horns to directly scale up laboratory processes. Dry microalgae was used as the feedstock. The extraction and transesterification medium was dry ethanol.

Other Extraction Methods

In addition, the experiments were repeated using mechanical homogenization with a high-speed laboratory stirrer. Dry microalgae was used as the feedstock. The extraction and transesterification medium was dry ethanol. The exposure cycle for this experiment was also 3 minutes.

Yield baseline (shown below as 100 %) was established by standard laboratory extraction and transesterification techniques (automatic soxhlet extraction/transesterification). These techniques, although very lengthy, provide close to maximum possible yields, and are, therefore, useful for making comparisons with other methods.

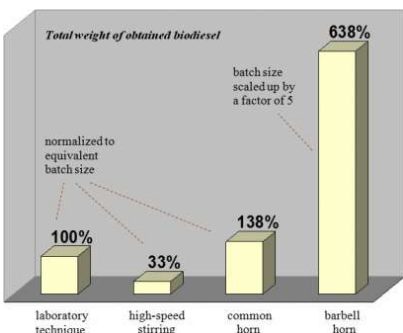


Results of Extraction Experiments

As shown in the figure on the left, high-amplitude ultrasound-assisted extraction provided much higher yields than high-speed stirring and was even more efficient than the lengthy automatic soxhlet extraction (laboratory technique).

Two extraction cycles into the same solvent were possible, without losing the process efficiency (data shown in green).

The use of the Barbell horn permitted a factor-of-five scale up of the ultrasonic extraction process also without any loss in the efficiency.



Results of In-Situ Transesterification Experiments

High-amplitude ultrasound-assisted in-situ transesterification was also more efficient than that assisted by high-speed stirring and the laboratory technique.

The use of the Barbell horn permitted a factor-of-five scale up of the ultrasonic in-situ transesterification process without any loss in the efficiency.

DISCUSSION

The data presented above show that high-intensity ultrasonic exposure affords very high extraction and in-situ transesterification efficiency, which greatly exceeds mechanical homogenization and is even more efficient than lengthy standard laboratory techniques. The data also suggest that it is possible to reuse the alcohol practically without any loss of extraction efficiency by running multiple extraction cycles prior to transesterification, where at the end of each cycle the used-up algae is filtered out and replaced with fresh material. During the last extraction cycle, when sufficient concentration of the oils is built up in the mix, a catalyst may be added to initiate the transesterification reaction.

In addition, our ability to use [BHUT](#) to directly scale up ultrasonic processes has been demonstrated, achieving, in this case, approximately a factor of 5 increase in the extraction and in-situ transesterification rates compared with traditional converging horn ultrasonic technology.

No systematic process conditions optimization (ultrasonic amplitude, pressure, exposure time, etc.) was attempted in the described evaluation experiments. Optimization is necessary in order to be able to calculate the method's energy balance and evaluate its commercial potential.

ENERGY EFFICIENCY ESTIMATION

Industrial implementation of microalgal oil in-situ transesterification should be preceded by a thorough flow-through laboratory study aimed at optimizing all process parameters and evaluating the energy balance. The key ultrasonic exposure parameters are ultrasonic amplitude, static pressure, and exposure time. Once these conditions are established, [BHUT](#) will permit a direct scale up from the laboratory to industrial size without changing any of the identified parameters, with the exception of the ultrasonic reactor volume and the associated total liquid processing capacity.

Based on our previous experience with similar processes, for in-situ transesterification we estimate the optimum flow rate through our 3000 W industrial ultrasonic system, [ISP-3000](#), to be 10 liters per minute (l/min). If the working liquid mixture has about 20 % of microalgae, of which approximately 50% by weight is extracted and converted into biodiesel, the total productivity rate of the system with respect to the final product will be approximately 1 l/min.

HAVE QUESTIONS?

Request a free initial consultation
with a process specialist

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