



Microwave and ultrasound enhanced extractive-transesterification of algal lipids



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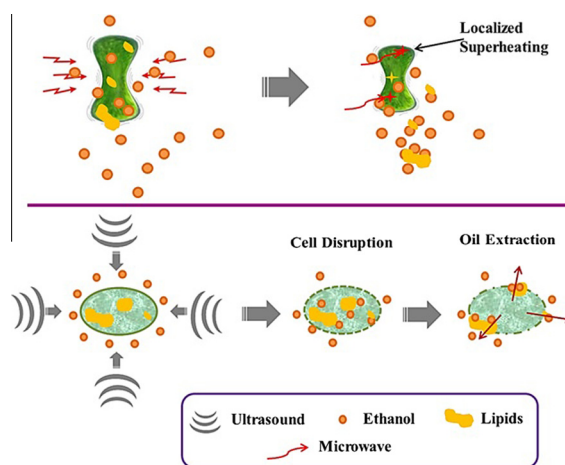
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HIGHLIGHTS

- Microwave/ultrasound based extractive-transesterification of algal lipids was studied.
- Microwave and ultrasound show higher yields/conversions than Bligh and Dyer (BD) Method.
- Lipid yields were (MW-18.8%; US-18.5%) and FAEE conversions were (MW-96.2%; US-95.0%).
- Specific energy consumption for MW was 26 MJ/kg and US was 44 MJ/kg biodiesel.
- The MW or US methods may result in chemical, energy, and economic savings.

GRAPHICAL ABSTRACT



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ABSTRACT

The use of non-conventional methods namely microwaves and ultrasound for extractive-transesterification of algal lipids (*Chlorella*, sp.) using ethanol as a solvent was investigated. Microwaves and ultrasound possess unique enhancing (thermal and non-thermal) mechanisms that can assist in successful and simultaneous extraction and transesterification of algal lipids in a very short reaction time. This paper presents a comparative study of microwave and ultrasound effects on the algal biodiesel production. The following conditions were determined as optimum through experimental studies: (1) microwaves – 1:12 algae to ethanol (wt./vol.) or 1:500 (molar) ratio; 2 wt.% catalyst; 5–6 min reaction time at 350 W microwave power; and (2) ultrasound – 1:6–9 algae to ethanol (wt./vol.) or 1:250–375 (molar) ratio; 2 wt.% catalyst; 6 min reaction time at 490 W ultrasound power. The highest fatty acid ethyl ester (FAEE) yields and conversions for microwave and ultrasound methods were 18.8%; 18.5% (yields) and 96.2%; 95.0% (conversions) respectively. In comparison, ultrasound method resulted in higher FAEE yield and conversion at low solvent ratios while microwaves were able to produce better results at lower power levels compared to ultrasound. The two methods performed better than the conventional bench-top Bligh and Dyer method which followed a two-step extraction and transesterification method with FAEE yields and conversions of 13.9% and 78.1% respectively.

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1. Introduction

Considering current world finite fossil fuel resources and escalating demands for the same, quickly replenishing renewable fuel resources such as oils from microalgae are being actively sought. Algae represent a renewable energy resource which helps capture atmospheric carbon dioxide (CO₂) photosynthetically and produce lipids that can be transesterified to biodiesel. Some algae, like *Nannochloropsis* and others, can be grown in saline waters and do not compete with food crops or consume fresh water resources. However, algal biodiesel production poses significant economic and bioprocessing challenges, including cost effective harvesting of the algae and the recovery of the lipids, which are intracellularly located. Effective means to recover microalgae and extract their intracellular lipids remains a practical and economic bottleneck in algal biodiesel production [1].

The intracellular location of the lipids requires algal cell wall breakage for oil recovery. Extraction of intracellular lipids from intact algae is difficult as the lipids are bound within cell membranes and cell disruption is required to maximize lipid recovery. For this reason, conventional oil extraction methods such as physical straining, mechanical pressing, and solvent extraction are not suitable for algae oil extraction. Mechanical pressing involves high specific energy consumption due to high mechanical strength of algal cell walls which may exceed the extractable energy in algae lipids. Supercritical fluid extraction using dry CO₂ is another well-known technique. This technique requires high temperatures and high pressures but relatively lower chemical solvents and reduced reaction times. But high capital costs and process safety are the major concerns with this technique. Novel techniques with ability to enhance both the diffusive (diffusion of solvent into the cells) and disruptive (disrupt the cell walls to force-release the lipids) mechanisms are essential for efficient algal oil extraction.

The key to algal lipid extraction is an effective solvent which can firstly penetrate the solid matrix enclosing the lipid, secondly physically contact the lipid and thirdly solvate the lipid [2]. Since the microalgae are protected by a cell wall that limits the solvents' access to the lipid, non-conventional extraction techniques such as microwaves and ultrasound may play an important role to enhance both disruptive and diffusive mechanisms. Microwaves and ultrasound have been utilized in the extraction of various valuable bio-products from fruits, herbals, leaves and crop seeds [3–12]. Some recent works have studied the microwave and ultrasound effects on transesterification of various fresh and used oils. Transesterification of waste cooking oil using microwaves [13–14], *Camelina Sativa* oil [15], palm oil [16], hazelnut oil [17], palm oil transesterification using ultrasound [18], vegetable oil [19] and waste cooking oil without and with temperature control [20–22,56] were studied. Apart from that microalgae biodiesel has been produced from extracted lipids via a traditional extraction–conversion approach [23–25]. However, the microalgae biodiesel production via the extraction and transesterification (conversion) route heavily relies on organic solvent extraction efficiency, which has been identified as a major drawback in several recent reports due to incomplete extraction of oils by this method [26,27]. One of the alternatives to overcome this limitation is to conduct “in situ transesterification” of algal lipids. In this method, algal lipids are simultaneously extracted and converted to biodiesel (Fatty Acid Alkyl Esters, FAAEs). Since the in situ approach integrates the extraction and conversion in one step, it eliminates the need to first isolate and refine the lipid before converting it into biodiesel which could lead to a reduction in product cost. Moreover, besides serving as a reactant in the in situ process, the alcohol may weaken the cellular and lipid body membranes to facilitate the FAME conversion [28]. Recently, the in situ process has been applied to

prepare biodiesel from various microalgal species (biomass) with H₂SO₄ [26], [28–30], KOH [31,32,60], and SrO [33,57] as catalyst using conventional, microwave and supercritical methods [34].

In this research, we studied the effects of microwaves and ultrasound on extractive–transesterification of algal lipids from *Chlorella* sp. using ethanol as a solvent (for lipid extraction) and reactant (for transesterification reaction). The premise for the proposed microwave enhanced extractive–transesterification lies in the fact that ionic conduction and dipole moment caused by microwaves increase the oil extractive ability and simultaneously convert the oils to FAEE due to localized superheating. On the other hand, ultrasound induces intense mixing due to continuous compression and rarefaction cycles which cause the cavitation bubbles to generate with super high local temperatures and pressures. This phenomenon automatically increases the temperature of the bulk of the sample medium with disruption of the micro-bubbles and promotes the desired chemical reactions [22]. By use of ultrasonics, chemical synthesis limitations attributed to mass transfer limitations of heterogeneous conditions existing during the transesterification reaction are eliminated. This paper presents a comparative study of microwave and ultrasound enhanced extractive–transesterification of algal biomass using ethanol (process optimization studies) with a discussion on principle mechanisms and process improvements. Energy analysis and a comparison with other previous studies are also presented.

2. Reaction mechanism

2.1. Base catalysis mechanism

The base catalysis mechanism for triglycerides using alkali hydroxide as catalyst is shown in Fig. 1 [35,36]. The mechanism can be assumed to follow the commonly reported route since microwaves or ultrasounds do not have the capability to cause chemical bond alteration at molecular levels. The microwaves may cause electron excitation but do not possess the energy required to break the chemical bonds. However, localized superheating created by microwaves (due to dipole moment and ionic conduction) will allow for expedited reactions. Similarly, ultrasonic waves cause continuous compression and rarefaction cycles (with expansion and contraction) which lead to intense mixing of the reaction compounds increasing the mass and heat transfer rates enormously, and finally promoting expedited chemical reactions. Therefore, the reactions enhanced by these novel techniques would be similar to the route followed by the conventional method except that the reaction speed increased tremendously due to the aforementioned special effects. The base-catalyzed transesterification mechanism follows essentially four important steps; first step (1) is a catalytic reaction with alcohol, producing an alkoxide. The nucleophilic attack of the alkoxide to the carbonyl group of the triglyceride generates a tetrahedral intermediate compound (2) from which the alkyl ester is formed and the corresponding anion of triglyceride (3). Finally, the catalyst is deionized to regenerate the active compound (4), which allows for it to react with a new molecule of alcohol, beginning a new catalytic cycle. In the notation used, B is the base catalyst, R₁, R₂ and R₃ are the carbonyl groups of fatty acids and R is the functional group of alcohol [35,36]. Currently, methanol is a commonly used reagent for transesterification, but ethanol is attractive for long term sustainability since it can be derived from renewable sources, and is less toxic than methanol [37]. Fatty acid ethyl esters (FAEE) typically demonstrate slightly higher cetane numbers, improved low temperature operability, and greater oxidative stability when compared to fatty acid methyl esters (FAME) [38,37]. Apart from this, the majority of the world methanol production is based on natural gas. This is one of

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