



## Optimization of the ionic liquid-microwave assisted one-step biodiesel production process from wet microalgal biomass

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### ABSTRACT

This study illustrates the influence of ionic liquid (IL)-microwave heating on the direct transesterification (in-situ transesterification) of wet *Nannochloropsis* sp. biomass to biodiesel. The ionic liquid used was 1-ethyl-3-methylimidazolium methyl sulphate [EMIM][MeSO<sub>4</sub>]. Direct transesterification process variables such as the wet algae to methanol ratio, reaction time and methanol to IL ratio influencing the biodiesel production were optimised using response surface methodology (RSM). The results revealed that the maximum biodiesel yield (40.9%) was achieved when the ratio of wet algae to methanol (wt/vol) was kept at 1:4, methanol:IL ratio maintained at 1:0.5 at reaction time of 25 min. The FAMES composition depicted a high content of unsaturated FAMES suitable for biodiesel production. The synergetic effect of combining IL-methanol solvent and microwave heating at optimised reaction conditions enabled the production of an incredibly high biodiesel yield per dry biomass of 42.22%.

### 1. Introduction

The first generation biodiesel is derived from agricultural edible crops such as corn, soybean, sugar cane and grains. This has resulted in a great impact on food security making biodiesel production more expensive [1]. Second generation biodiesel such as jatropha oil, waste cooking oil and animal fats is not favourable because it gives rise to problems such as poor cold flow properties. Moreover, the saturated fatty acids contained in animal fats give rise to production difficulties [2]. Microalgae are regarded as the third generation biodiesel source as they have faster growth rates than plants and their productivities can be twenty times that of oilseed crops on a per hectare basis.

Biodiesel can be produced via the transesterification process from animal fats, vegetable oil, waste cooking oil and microalgal oil. The conventional method of lipids extraction and biodiesel synthesis is known to be expensive. Thus the drive for an alternative approach such as direct transesterification technique for biodiesel production is used. In addition, most of the traditional conventional processes usually necessitate the use of environmentally harmful chemicals such as organic solvents, alkali or acidic catalysts. Therefore, the search and application

of green solvents, which includes ionic liquid (ILs), supercritical CO<sub>2</sub>, microwave assisted techniques and aqueous biphasic systems have become topics of great interest [3].

Ionic liquids (ILs) are described as salts consisting of relatively large asymmetric organic cations which are coupled with smaller inorganic or organic anions where they remained as liquids at temperatures (0–140 °C). The attractiveness of IL is its ability to accommodate different structure of size groups, thus varying its polarity [4,5]. ILs are used as substitute solvents to common organic compounds for liquid-liquid extraction and separation analysis because they represented a viable class of environmentally friendly replacements for the currently used organic solvents [6]. They are composed of an organic cation and an inorganic or organic anion in which the ions can be tailored for specific applications. The overwhelming attention to these compounds is due to the uniqueness of their properties such as variable viscosity, low or negligible vapor pressure, and high thermal stability. The tunability of cations and anions has resulted in the variable viscosity of the solvent. Moreover, due to the tailoring of the cation and anion they are capable of achieving high thermal stability, (> 400 °C). They have very low or negligible vapor pressure [7]. With this, they are often

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categorized as green solvents [8,9]. Furthermore, the relative hydrophobic and hydrophilic nature of the resulting ionic liquids can be modified by altering the anions and cations. Halide-containing ILs such as 1-(2-cyanoethyl)-3-methylimidazolium bromide [Cyno-mim][Br] and 1-propyl-3-methylimidazolium bromide [Propy-mim][Br] have been used to recover fat from food-stuffs [7,10]. Mixtures of [Emim][MeSO<sub>4</sub>] and polar organic solvents are also used for lipid extraction of canola oil seed and *Dunaliella* biomass [7].

Besides being readily modified, ILs can also be used as catalysts and thus have gained significant importance in various chemical industries due to their significant role in controlling reactions for various green chemistry processes and applications. Among the ILs applications include the volatile organic solvents replacement, new materials production, effective heat dissipation, enzyme-catalyzed reactions support, polymerization reactions, host for a variety of catalysts, purification of gases, homogenous and heterogeneous catalysis, biological reactions media and metal ions removal [9,11,12].

The interest in the application of microwave heating for lipid extraction and biodiesel production from microalgae biomass has significantly increased. Various researchers successfully utilised microwave assisted heating to extract the oils from the biomass [13–16] and convert the oil to biodiesel [17–19]. The rapid heating in microwave irradiation has led to localized high temperature and pressure gradients which enhanced mass transfer rates and promotes the destruction of cell walls [20]. Direct transesterification combined lipid extraction and transesterification in a single step, thereby simplifying the downstream pathway required for biodiesel production from microalgal biomass. Direct transesterification (DT) is a process blending the microalgae with an alcohol and a catalyst without prior extraction. The methanol extracts the lipids from the microalgal biomass and the reaction is catalyzed by the acid or base. Simultaneously transesterification occurred where the extracted lipids are converted into fatty acid methyl esters. Thus, DT is an alternative technique which facilitates the conversion of lipids to their alkyl esters directly without biomass harvesting and drying thereby eliminating the solvent extraction step in a bid to reduce processing cost [21,22].

Recently Wahidin et al. [23] combined the benefits of microwave technique and several ILs; 1-butyl-3-methylimidazolium chloride ([BMIM][Cl]), 1-ethyl-3-methylimidazolium methyl sulphate [EMIM][MeSO<sub>4</sub>] and 1-butyl-3-methylimidazolium trifluoromethane sulfonate [BMIM][CF<sub>3</sub>SO<sub>3</sub>) in the direct transesterification of algal oils for biodiesel production. The findings revealed that 1-ethyl-3-methylimidazolium methyl sulphate [EMIM][MeSO<sub>4</sub>] was the best IL exhibiting highest cell disruption (99.73%) and biodiesel yield (36.79%) per dried biomass.

To the best of our knowledge besides the work of Wahidin et al., who used a combination of microwave and ILs in production of biodiesel via DT not much work has been reported regarding this novel method. Hence in this study, the significant parameters influencing DT process performed under microwave using ILs were determined via response surface methodology (RSM). The process factors in DT evaluated were reaction temperature, reaction time, ratio of wet algae to methanol and ratio of methanol to ionic liquid and the response variable is the yield of biodiesel obtained from the DT process. Upon identifying the significant variables and curvature, a central composite design (CCD) was used to obtain the second order model thus enabling the optimum conditions for the DT process to be determined.

## 2. Materials and methods

### 2.1. Microalgal cultures

*Nannochloropsis* sp. was provided by the culture collection of Borneo Marine Research Institute (BMRI), Universiti Malaysia Sabah, Malaysia. *Nannochloropsis* sp. was cultivated in a 5 L photobioreactor at 23 ± 0.5 °C, pH 8 ± 0.2 under a light intensity of 100 μmol m<sup>-2</sup> s<sup>-1</sup>

**Table 1**

Lower and upper limits of the main effects for the 2 level full factorial design.

Factor	Units	Low level (–1)	High level (+1)
Reaction temperature (A)	°C	65	95
Algae:methanol ratio (B)	wt/v	1:4	1:12
Reaction time (C)	min	5	25
Methanol:IL ratio (D)	v/v	1:0.5	1:1

with an 18:06 h light/dark cycle [17]. The cells were grown in sterilized Walne's medium for 8 days. The wet microalgae biomass (80 wt% water content) was stored in freezer at –25 °C until use.

### 2.2. Direct transesterification (DT)

The DT was performed in a MAS-II microwave synthesis workstation, 1000 W (Sineo Microwave Chemistry Technology, Co. Ltd., Shanghai, China), operating at a frequency of 2450 MHz and atmospheric pressure [23]. The extraction vessel used during the process was a three-neck round bottom flask which was microwave-transparent. The DT method was carried out with 1 g of wet microalgae biomass (water content, 80 wt%) charged into a 3-necked mini reactor. The output of microwave power was set at 700 W during the heating process. The pressure of the extraction vessel during the process was atmospheric pressure. The variables used in this study and their variation limits were tabulated in Table 1. The experiments were then performed accordingly to 2 level full factorial method and then extended to CCD experiments. A direct infrared temperature sensor was used to monitor the reaction temperature. In order to maintain the solvent volume in the reaction mixture throughout the experiments a reflux condenser was used to condense, return and maintain the solvent to the vessel. After reaction was completed, the mixture was centrifuged at 4000 rpm for 5 min to separate the biodiesel and methanol-IL phases. A rotary evaporator was used to evaporate the solution containing the biodiesel and its contents were analysed using the gas chromatography.

### 2.3. Two-step transesterification process

The performance of DT mentioned in Section 2.2 was compared to the two-step solvent extraction and transesterification: (1) lipid extraction using methanol:IL, and (1) transesterification using sulfuric acid. The experiment was performed using 1 g of wet microalgae biomass. In this two-step transesterification experiment, the corresponding methanol:IL ratio was added to the 1 g of wet microalgae biomass and then subsequently heated to 65 °C. The lipids were extracted and then cell residue was removed to prevent further extraction. The transesterification process was then performed by mixing the extracted lipid with methanol- sulfuric acid and then heated at 65 °C for 25 min [24].

### 2.4. Analysis of fatty acid methyl esters (FAMES)

A gas chromatography (GC-7820A, Agilent, USA) system with a flame ionization detector (FID) was used to analyze FAMES content obtained from direct transesterification process. The GC operating conditions are specified as follows: carrier gas: nitrogen (99.9% purity) at flow rate of 1 ml/min and 21 psi; initial oven temperature was 80 °C held for 3 min and then increased to 220 °C at the rate of 5 °C/min, and held at 220 °C for 5 min. A capillary column (HP-88 Agilent) was used with a dimension of length: 60 m, ID: 0.25 mm, film: 0.20 mm. One microliter of the sample was injected to the GC with split ratio of 1:50 and the data was collected using MSD ChemStation Software, USA [25].

### 2.5. Determination of dielectric constant

Dielectric constant value reflects the ability of the solvents to absorb

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