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Rapid biodiesel production using wet microalgae via microwave irradiation

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ABSTRACT

The major challenges for industrial commercialized biodiesel production from microalgae are the high cost of downstream processing such as dewatering and drying, utilization of large volumes of solvent and laborious extraction processes. In order to address these issues the microwave irradiation method was used to produce biodiesel directly from wet microalgae biomass. This alternative method of biodiesel production from wet microalgae biomass is compared with the conventional water bath-assisted solvent extraction. The microwave irradiation extracted more lipids and high biodiesel conversion was obtained compared to the water bath-assisted extraction method due to the high cell disruption achieved and rapid transesterification. The total content of lipid extracted from microwave irradiation and water bath-assisted extraction were 38.31% and 23.01% respectively. The biodiesel produced using microwave irradiation was higher (86.41%) compared to the conventional method. Thus microwave irradiation is an attractive and promising technology to be used in the extraction and transesterification process for efficient biodiesel production.

1. Introduction

The concerns for greenhouse gas emissions (GHGs), accelerated decrease in fossil fuels, steep increase in crude oil prices and energy insecurity have attracted worldwide interest in sustainable and renewable energy sources [1–5]. In order to meet petro-diesel replacement, renewable energy sources should not only be environmental beneficial but it should also be producible, economical and cost competitive so as to give a meaningful impact on sustainable energy demands [6–8]. Biodiesel fuel is one type of alternative renewable fuel, which is highly biodegradable, has minimal toxicity or non-toxic and environmental friendly. Recently, biodiesel has been considered as one of the best sustainable sources to replace petroleum-based fuels [9–10].

However, algal biodiesel production still faces hurdles due to the high cost associated with microalgae mass productivity, harvesting, dewatering and preparation for conventional lipid extraction and transesterification into biodiesel. The harvesting technology for microalgae biomass requires a universal separation such as universal centrifuge and flocculant agents to recover microalgae mass. The major challenge for industrial commercialized biodiesel production from microalgae is the high cost of recovering the oil from the microalgae prior to converting it into biodiesel. Lardon et al. [11] analyzed and indicated that 90% of energy consumed for biodiesel conversion from microalgae biomass is in the lipid extraction process. Thus, improvements in the extraction process will contribute to a major impact on the microalgae oil productivity and costs. Numerous methods of lipid extraction from microalgae have been applied such as solvent extraction (liquid–liquid extraction), supercritical fluid extraction (SFE), ultrasonic extraction and mechanical pressing. Most of the extraction methods mentioned require long extraction time, large volumes of solvent except when using SFE which is however energy-cost intensive [12–14].

Interest in lipid extraction and transesterification, and the lack of efficient process, in some cases have led to further studies on alternative extraction techniques. Lewis et al. [15] reported that most modifications and adaptations of the procedure involved the change in the solvents and their ratios. For example, methylene chloride was used as an alternative to chloroform and factors such as extraction time, temperature and order of solvent addition were investigated. Another emerging practice is the use of sonication which has the capability to disrupt the cells and encourage better

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lipid solubilization but this method adds significant mechanical costs.

Demirbas [16] reported that supercritical transesterification was one of the alternative processes considered for production of biodiesel from microalgae lipid conversion. In supercritical transesterification, catalyst was not required but high reaction temperature (>240 °C) and pressure (>8 MPa) were essential. Supercritical fluid extraction (SFE) has gained interest because it significantly reduced the requirement for organic solvents and thus considered the concerns for waste disposal after reaction. Past studies, have reported that SFE demonstrated the shortest reaction time (120–240 s) to complete the reaction from lipid to biodiesel [17]. SFE method requires a simple purification step and also produces higher biodiesel yields compared to solvent extraction and soxhlet extraction processes. However, this method has several disadvantages due to the adverse process economics as well as safety concerns that are related to the reaction conditions. Furthermore, the sample size and water content condition influenced the SFE process and must be improved to achieve efficient extractions. Thus, new extraction technologies which are efficient and cost effective on a large scale must be explored and developed for microalgae systems.

Microwave irradiation have been used in the past and is a constructive method to extract oils from biomass, animal fats and vegetable feedstock [18–20] and can be easily scaled-up [21]. Microwave irradiation is known to be more efficient; on the average the cost of microwave heating is two thirds less than that of conventional heating. In addition, the possibility of improved reaction rates due to microwaves can improve micro-biodiesel production rates. Microwave technology has resulted in the development of rapid, safe and cost-effective method for micro-biodiesel production and does not require samples to be devoid of water. Performance of microwave lipid extraction was qualitatively and quantitatively comparable for all lipid classes with that of the conventional Folch's method for various biological samples. However, there are limited studies that investigate the micro-biodiesel production of lipid extracted from microalgae by microwave irradiation and most of these studies use dry biomass. The application of radio frequency microwave energy offered a fast and easy route to this valuable biofuel with the advantages of enhancing the reaction rate (2 min instead of a 2 h process reaction) and thus improved the separation [22]. The advantages of using microwave energy as a non-contact heat source for the extraction of analytes from plant materials include: more effective heating, faster energy transfer, reduce thermal gradients, selective heating, reduce equipment size, faster respond to process heating control, faster start-up, improves production and eliminates process steps [23].

Thus in this study microwave irradiation technique was used for micro-biodiesel production directly from wet *Nannochloropsis* sp. and the fatty acid methyl esters (FAMEs) yields were compared with the conventional water bath-assisted transesterification method. The development of this technique can reduce cost in terms of both energy and unit operation. Therefore it can be a suitable method to decrease the cost of biodiesel and also promote the commercialization of biodiesel.

2. Materials and methods

2.1. Microalgal cultures

The strain of *Nannochloropsis* sp. was originally obtained from the culture collection of Borneo Marine Research institute (BMRI), Universiti Malaysia Sabah, Malaysia. The *Nannochloropsis* sp. cells were cultured in sterilized seawater enriched with Walne's medium which contains: 0.1 g $NaNO_3$, 0.0013 g $FeCl_3{\cdot}6H_2O$; 0.00036 g $MnCl_2.4H_2O$; 0.0336 g H_3BO_3; 0.045 g $Na_2{\cdot}EDTA$; 0.02 g $NaH_2PO_4{\cdot}2H_2O$; 0.021 g $ZnCl_2$; 0.02 g $CoCl_2{\cdot}6H_2O$; 0.009 g $(NH_4)6Mo_7O_{24}{\cdot}4H_2O$; 0.02 g $CuSO_4{\cdot}5H_2O$; 0.01 mg Vitamin B_{12}; 0.01 mg Vitamin B_1 and 0.0002 mg Biotin per litre. *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^{-2} s^{-1} with an 18:06 h light-dark cycle for 8 days as maximum yield was achieved at these conditions [24]. The cultures were sampled at every 24 h interval and microalgal growth was monitored by counting the cell number. Sampling was performed in triplicates so as to ensure reproducibility of data. The cell concentration was determined by a direct microscopic count with a 0.1-mm-deep Neubauer haemocytometer (BOECO, Hamburg, Germany) and a light microscope (Olympus CX21, Japan).

2.2. Measurement of neutral lipid by fluorescent spectrophotometer

Neutral lipids were determined rapidly using improved Nile red staining method and analyzed using Perkin Elmer LS-55 fluorescence spectrophotometer. For the lipid staining, 1 ml of sample was added to 50 μL of Nile red (9-diethylamino-5H-benzo[α]phenoxazine-5-one; Sigma, USA) in acetone representing a concentration of 0.1 mg ml^{-1}. The mixture was pretreated using a microwave oven for 1 min. The excitation and emission wavelengths for the fluorescence determination were selected to be 490 nm and 585 nm, respectively [25].

2.3. Direct lipid extraction

The wet microalgae biomass (10 ml with 80 wt% water content,) which were stored at −25 °C were thawed at room temperature and mixed with methanol–hexane in the ratio of 1:2 (% v/v). The lipid was extracted using two extraction methods listed as follows:

(1) In the conventional method the water bath-assisted solvent extraction protocol was applied. The mixture of sample with solvents was homogenized using vortex mixer for 1 min. The sample was then heated and agitated in a water bath at 150 rpm for 40 min at 65 °C and then left to cool to room temperature. The samples were then transferred into tubes and then filled with deionized water, vortexed for 5 s and centrifuged at 4000 rpm for 5 min so as to separate the aqueous from organic phase. The organic phase was transferred to the mini reactor and the original sample was extracted two more times so as to maximize the lipid product.

(2) The microwave extraction technique was performed using MAS-II microwave synthesis oven (Sineo Microwave Chemistry Technology, Co., Ltd., 1000 W) with an operational frequency of 2450 MHz. The mixture in the 3-necked mini reactor was then subjected to the microwave irradiation with 70% of power and was operated for 5 min at 65 °C under normal pressure. During the extraction process, a reflux condenser was used in order to prevent loss of solvent due to vaporization thus maintaining the solvent volume in the reaction mixture throughout the experiments. The sample was then left to cool to room temperature. After the lipid extraction the methanol–chloroform phase that contained the extracted lipids was centrifuged at 4000 rpm for 5 min and separated using a separating funnel. In both cases, the solvent containing lipid extracted was evaporated by a rotary evaporator. The mass of the lipid obtained from each sample was determined gravimetrically [26].