



Biodiesel production from wet microalgae feedstock using sequential wet extraction/transesterification and direct transesterification processes



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HIGHLIGHTS

- Over 97% biodiesel conversion was obtained using wet microalgae as feedstock.
- Wet extraction was more efficient after microwave and methanol pretreatments.
- Chlorophyll removal was achieved during the process of biodiesel production.
- Direct transesterification with algae cake was operated at a low temperature (45 °C).
- Nearly 100% biodiesel conversion was achieved with the direct transesterification.

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ABSTRACT

Although producing biodiesel from microalgae seems promising, there is still a lack of technology for the quick and cost-effective conversion of biodiesel from wet microalgae. This study was aimed to develop a novel microalgal biodiesel producing method, consisting of an open system of microwave disruption, partial dewatering (via combination of methanol treatment and low-speed centrifugation), oil extraction, and transesterification without the pre-removal of the co-solvent, using *Chlamydomonas* sp. JSC4 with 68.7 wt% water content as the feedstock. Direct transesterification with the disrupted wet microalgae was also conducted. The biomass content of the wet microalgae increased to 56.6 and 60.5 wt%, respectively, after microwave disruption and partial dewatering. About 96.2% oil recovery was achieved under the conditions of: extraction temperature, 45 °C; hexane/methanol ratio, 3:1; extraction time, 80 min. Transesterification of the extracted oil reached 97.2% conversion within 15 min at 45 °C and 6:1 solvent/methanol ratio with simultaneous Chlorophyll removal during the process. Nearly 100% biodiesel conversion was also obtained while conducting direct transesterification of the disrupted oil-bearing microalgal biomass.

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1. Introduction

Biodiesel has many advantages, such as high flash point, high lubricity, and high biodegradability, and can be used in conventional diesel engines without any modification, making it one of the most promising alternatives to fossil fuels (Zabeti et al., 2009). However, the use of plant-based oil feedstock for biodiesel

production has attracted considerable international scrutiny and criticism (Williams and Laurens, 2010). Therefore, increasing attention is being paid to using microalgae as an alternative feedstock for biodiesel production, because cultivation of microalgae does not require arable land and has the advantages of a high growth rate, short maturity, high biomass productivity, and low environmental impact (Goncalves et al., 2013). However, developing the technology for the conversion of microalgae-based oil to biodiesel is more challenging when compared with plant oil (such as palm oil), due mainly to the high water content of microalgae and the difficulty in extracting the oil from the microalgal biomass,

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leading to the higher production cost of microalgae-based biodiesel (Milledge and Heaven, 2013). There is thus an urgent need to, develop a more economically feasible method for producing biodiesel from microalgae.

The common process for obtaining oil from microalgae consists of microalgae harvesting, biomass drying and oil extraction (Cooney et al., 2009), of which the drying step consumes a large amount of energy. Therefore, recent studies have focused on using “wet microalgae” as the raw materials to produce biodiesel (Olmstead et al., 2013; Tran et al., 2013). In addition, some of the downstream technologies still suffer the drawbacks of high energy consumption and low treatment capacity (as seen, for example, with centrifugation and supercritical fluid extraction), limiting the commercialization of these methods. This study thus presents a new process for producing microalgae-based biodiesel from the wet biomass of microalgae, consisting of microwave disruption, partial water removal, wet oil extraction, transesterification, and direct transesterification, skipping the wet extraction step. Moreover, a homogeneous base catalyst (i.e., NaOH) and heterogeneous base catalyst (i.e., Sr_2SiO_4) (Chen et al., 2012) were used to convert microalgae oil to biodiesel, and their catalytic effects on transesterification were compared. The aim of this work was to develop a low cost and effective method for biodiesel production from wet microalgal biomass, with the capacity to be scaled up for commercial applications.

2. Methods

2.1. Materials

The microalgae strain used in this study was *Chlamydomonas* sp. JSC4, which was provided by the Center for Bioscience and Biotechnology, National Cheng Kung University, Tainan, Taiwan and was shown to possess high lipid content (Ho et al., 2015; Nakanishi et al., 2014). The culture conditions were based on the optimal conditions reported by Nakanishi et al. (2014). After cultivation, the microalgae biomass was collected with a centrifuge at $22,400\times g$, and the resulting microalgae slurry had a dry biomass content of 31.3% and an oil content of 26.3% per dry weight of biomass.

Hexane (ACS) and sodium hydroxide (NaOH, ACS) were obtained from Macron Fine Chemicals (Pennsylvania, USA). Strontium nitrate ($\text{Sr}(\text{NO}_3)_2$, 98%) and tetraethyl orthosilicate (TEOS, 99.9%) were obtained from Showa Co. (Tokyo, Japan). Ammonia hydroxide ($\sim 25\%$ NH_3 basis) was purchased from Sigma-Aldrich (Missouri, USA). The methanol (>99%) was purchased from Uni Ward (Miaoli, Taiwan).

2.2. Preparation of the solid base catalyst

The solid base catalyst was prepared using a modified method as reported by Chen et al. (2012). In summary, 48.5 g strontium nitrate and 25.1 mL TEOS were dissolved in 100 mL ammonia hydroxide and 200 mL methanol, respectively. The two solutions were then mixed with each other, and the resulting mixture was heated in an oil bath to generate the solid precursor. Finally, by calcining the precursor at 1100°C , the solid catalyst was obtained and was analyzed with X-ray diffraction (XRD; Rigaku Ultima IV). The catalyst was identified as Sr_2SiO_4 by comparing the XRD pattern with the JCPDS file 39-1256 (Chen et al., 2012).

2.3. Procedures of biodiesel production from wet microalgae

The flowchart of biodiesel production from wet microalgae carried out in this study is shown in Fig. 1. Wet microalgae first went

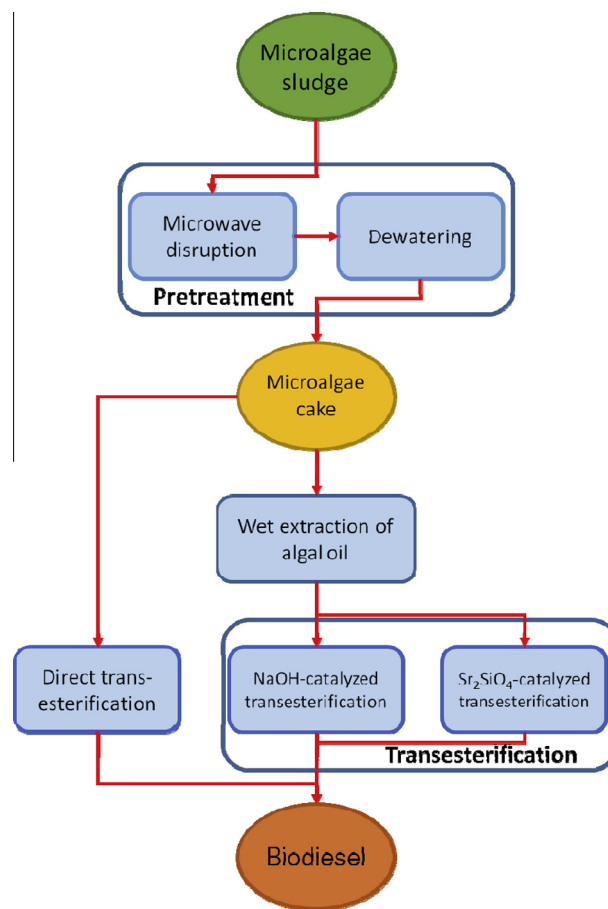


Fig. 1. The flowchart of biodiesel production from wet microalgae.

through the pretreatment process consisting of microwave disruption and concentration via methanol-driven flocculation, which consisted of flocculation with methanol and dehydration with a spin dryer. The resulting microalgae cake (with a solid content of ca. 60 wt%) was then used to produce biodiesel. Two processes were conducted for the production of biodiesel from the wet microalgae cake, as follows (Fig. 1). Process 1: wet oil extraction was first conducted and the extracted microalgal oil was subjected to transesterification using a homogeneous base catalyst (i.e., NaOH) or heterogeneous base catalyst (i.e., Sr_2SiO_4). Process 2: the wet microalgae cake was directly used to carry out transesterification without the oil extraction step.

2.3.1. Pretreatment

One hundred grams of microalgae sludge was placed into a 500 mL serum bottle, and then 100 mL methanol was added to the bottle and mixed for 20 minutes at 400 rpm to increase the fluidity of the sludge. The microalgae-methanol mixture was then input into an open microwave cell disruption system, consisting of a microwave oven (Samsung MW630WA), an open reactor, a cooling system (Fig. 2) with underwent heating at 350 W for 10 min to achieve cell wall disruption. After that, another 200 mL methanol was added to the bottle and the mixture was stirred at 100 rpm for 20 min. The final mixture was poured into a commercial filtration bag, and the bag was centrifuged with a spin dryer to yield the microalgae cake.

2.3.2. Wet oil extraction

The 2 g microalgae cake was placed into a 100 mL serum bottle, and then 4 mL methanol and various amounts of hexane (i.e., 8, 12,

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