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Short Communication

Using wet microalgae for direct biodiesel production via microwave irradiation

Jun Cheng *, Tao Yu, Tao Li, Junhu Zhou, Kefa Cen

State Key Laboratory of Clean Energy Utilization, Zhejiang University, Hangzhou 310027, China

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ABSTRACT

To address the large energy consumption of microalgae dewatering and to simplify the conventional two-step method (cellular lipid extraction and lipid transesterification) for biodiesel production, a novel process for the direct conversion of wet microalgae biomass into biodiesel by microwave irradiation is proposed. The influences of conventional thermal heating and microwave irradiation on biodiesel production from wet microalgae biomass were investigated. The effects of using the one-step (simultaneous lipid extraction and transesterification) and two-step methods were also studied. Approximately 77.5% of the wet microalgal cell walls were disrupted under microwave irradiation. The biodiesel production rate and yield from wet microalgae biomass obtained through the one-step process using microwave irradiation were 6-fold and 1.3-fold higher than those from wet microalgae obtained through the two-step process using conventional heating.

1. Introduction

Algae have a vast potential for biomass energy production. Approximately 1.46×10^{11} tons of biomass are annually produced worldwide through photosynthesis, among which 40% is attributed to algal photosynthesis. The quantum efficiency of microalgae in converting solar energy into biomass ranges from 2% to 10%, whereas that of terrestrial plants is less than 1%. Furthermore, the microalgae growth rate is very fast and can reach as high as 1–3 times per day (Gennady et al., 2008; Damian et al., 2008). Therefore, converting microalgae biomass into biodiesel has outstanding advantages. The heating value of biodiesel is 41 MJ/kg, which is equal to that of traditional petroleum diesel and about 1.4 times higher than that of ethanol. Given their high heating value, biodiesels may be used to replace the fuels of vehicles and aircrafts. Therefore, the development of microalgae energy has an experimental significance to the development of low-carbon economy and energy efficiency.

The two-step method of traditional solvent extraction and transesterification often requires the consumption of a large amount of energy because of algae dewatering and grinding into powder. For biodiesel production from dry algae, algae drying accounts for the majority of the total energy consumption (84%) (Patil et al., 2012). Furthermore, the complex process of the traditional two-step method often requires a total of 0.5–1.5 h for extraction and transesterification. Johnson and Wen (2009) converted wet microalgae to biodiesel using conventional heating via the one-step method. They reported that the biodiesel content of bio-oil is only 7.76%, indicating low efficiency. Some researchers have used microwave irradiation to convert castor oil into biodiesel, and found that microwave irradiation is superior to conventional heating. The thermodynamic efficiency increases from 70% to 94%, and the reaction time decreases from 3 to 1 h (Yuan et al., 2009).

Some researchers have recently compared various methods for microalgal cell disruption, including autoclaving (125 °C, 1.25 MPa), bead-beating, microwaving, sonication, and 10% NaCl solution. The microwave oven method is reportedly the most simple and most effective for microalgae lipid extraction among the tested methods (Lee et al., 2010). Microwave-pretreated microalgae have higher bio-oil yield (Sostaric et al., 2012) and present several micro cracks in the cell wall. By contrast, the cell wall of native microalgae is slightly damaged (Dejoye et al., 2011). The largest biodiesel production can be obtained within 5 min using strontium oxide as a catalyst when microwave irradiation is used for biodiesel synthesis from dry microalgae via the one-step method (Koberg et al., 2011). However, the use of dry powder consumes enormous energy. Patil et al. (2011) used methanol as solvent and reactant to convert dry algae into biodiesel via the one-step method using microwave irradiation. They reported that approximately 4–5 min of reaction time is sufficient to complete extraction and transesterification. However, the cell disruption efficiency and biodiesel yield of microalgae cells have not been analyzed.

To address the large energy consumption of microalgae dewatering and to simplify the conventional two-step method for

* Corresponding author. Tel.: +86 571 87952889; fax: +86 571 87951616.
E-mail address: juncheng@zju.edu.cn (J. Cheng).

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biodiesel production, wet microalgae biomass was used to produce biodiesel directly by microwave irradiation. The effects of conventional heating, microwave irradiation, and the one-step and two-step methods on biodiesel production from wet microalgae biomass were investigated. The bio-oil and biodiesel yields obviously increased when microwave irradiation was used to convert wet microalgae into biodiesel.

2. Methods

2.1. Experimental materials

The algae (*Chlorella pyrenoidosa*) used in the experiments were purchased from the Institute of Hydrobiology, Chinese Academy of Sciences, Wuhan. The microalgae were cultivated with modified Brostol's medium containing 1 g/L $NaNO_3$, 0.075 g/L $K_2HPO_4{\cdot}3H_2O$, 0.15 g/L $MgSO_4{\cdot}7H_2O$, 0.025 g/L $CaCl_2{\cdot}2H_2O$, 0.35 g/L KH_2PO_4, 0.025 g/L NaCl, 0.005 g/L $FeCl_3{\cdot}6H_2O$, 1 mL/L A5, 40 mL/L soil extract, 1 mL/L Fe-ethylenediaminetetraacetic acid (EDTA), and 958 mL/L distilled water. The soil extract was the supernatant of 0.5 kg mud and 1 L of deionized water boiled for 2 h. The A5 solution contained 2.86 g/L H_3BO_3, 1.81 g/L $MnC1_2{\cdot}4H_2O$, 0.22 g/L $ZnSO_4{\cdot}4H_2O$, 79 mg/L $CuSO_4{\cdot}5\ H_2O$, and 39 mg/L $(NH)_6Mo_7O_{24}{\cdot}4H_2O$. Fe-EDTA contained 10 g/L Na_2EDTA, 0.81 g/L $FeCl_3{\cdot}6H_2O$, and 500 mL/L HCl (0.1 N). The microalgae were cultivated in a 5 L bioreactor with an air pump for 20 d. Illumination (2500 lux) was supplied at the surface of the bioreactors under 12/12 h of dark/light cycle. The microalgae were harvested via centrifugation. The water content of the resulting paste was approximately 80 wt.%. Chloroform, methanol, and sulfuric acid were purchased from Sinopharm Chemical Reagent (China).

2.2. Preparation of biodiesel from wet microalgae via the one-step method using microwave irradiation

A WX-4000 microwave digestion system (2.45 GHz, Shanghai Yiyao Microwave Chemistry Company) equipped with six 60 mL digestion reactors was used. The wet algal biomass stored at −20 °C was thawed at room temperature. Approximately 1 g biomass (water content, 80 wt.%) was placed in the digestion reactor and mixed with 4 mL of chloroform, 4 mL of methanol, and 0.2 mL of sulfuric acid. Then, the digestion reactor was sealed and heated via microwave irradiation for approximately 40 s to achieve the setting temperature. The algal biomass was maintained at the setting temperature depending on the setting reaction time. The output of the microwave oven was set at 500 W during the heating process and 400 W during the holding process. After the heating treatment, the mixture was allowed to cool for approximately 20 min and transferred to a centrifuge tube. Chloroform (4 mL) was used to wash the digestion reactor twice. The used chloroform solutions were combined in a centrifuge tube. Up to 15 mL of distilled water was added to the mixture, which was then centrifuged. The organic layer containing the biodiesel was transferred to a new centrifuge tube. Distilled water (10 mL) was added to the organic layer twice to ensure that the aqueous phase was completely removed. The two-phase mixture was again centrifuged. The organic layer was transferred into a pre-weighed glass vial, which was evaporated in a baking oven at 70 °C. The bio-oil mass was gravimetrically determined.

2.3. Preparation of biodiesel from wet microalgae via the two-step method using microwave irradiation

The two-step method was divided into extraction and transesterification. The experimental method for lipid extraction was similar to the one-step method. However, sulfuric acid was not used. For the transesterification process, the bio-oil obtained from the extraction process was again mixed with 4 mL of chloroform, 4 mL of methanol, and 0.2 mL of sulfuric acid as catalyst and heated via microwave irradiation. The subsequent steps were the same as the one-step method.

2.4. Preparation of biodiesel from wet microalgae via the two-step method using conventional heating

The extraction process was similar to the two-step method using microwave irradiation, except that the mixture was heated using a baking oven. The transesterification procedure was also similar to the process in Section 2.3, except that the mixture was heated using a baking oven at 60 °C for 30 min.

2.5. Biodiesel composition analysis

The algae-based bio-oil was analyzed using a gas chromatograph (Kexiao 1690, Hangzhou China) equipped with a capillary column (FFAP series, 30 m × 0.25 mm × 0.32 μm). The initial column temperature was 150 °C for 6 min and programmed to a final temperature of 230 °C at a heating rate of 4 °C/min. The temperature was held at 230 °C for 6 min. The bio-oil components were identified by comparing the retention times with those of the standards. The bio-oil was added with methyl nonadecanoate (C19:0) for quantitative analysis. All measurements were conducted in triplicate. The mean value and standard deviation were reported.

3. Results and discussion

3.1. Biodiesel yield improvement via the one-step method using microwave irradiation

Using microwave irradiation as a selective and instantaneous heating method has many obvious advantages for preparing biodiesel. Microwaves accelerate the disruption of microalgae cells and enable easier lipid release. Microwave irradiation is also a faster and more efficient heating process, which directly contributes to molecular diffusion and mass transfer. In addition, methanol is a strong microwave absorption solvent. Thus, microwave irradiation can promote the transesterification reaction. As shown in Table 1, the bio-oil and biodiesel yields of the two-step method using microwave irradiation were slightly higher than those of the two-step method using conventional heating because microwave irradiation, which is a selective and fast method, can simultaneously heat the interior and exterior of polar objects. Methanol, which contains an OH group, is a strong microwave absorption material. The dipole rotates at a high speed under the changing electrical field, which leads to molecular friction and local superheating which accelerates the reaction to complete faster (Patil et al., 2011).

The bio-oil and biodiesel yields of the one-step method using microwave irradiation were 31% and 26% higher than those of the two-step method using conventional heating. The thermodynamic efficiency of microwave irradiation was approximately 1.3 times higher than that of conventional heating. When using microwave irradiation to convert castor oil into biodiesel, the conversion efficiency is 94%. However, 70% conversion efficiency is obtained using conventional heating (Yuan et al., 2009), which is very similar to our results. For the one-step method using microwave irradiation, the transesterification reaction simultaneously occurs with lipid extraction. The lipid concentration gradient is the driving force for the extraction. The extracted lipids are transesterified at a higher rate under microwave irradiation, which further increases