

Hydrothermal carbonization of lipid extracted algae for hydrochar production and feasibility of using hydrochar as a solid fuel

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ABSTRACT

In this study, hydrothermal carbonization was conducted to convert lipid extracted algae (*Chlorella vulgaris*) from the algal biodiesel process to produce hydrochar, while evaluating its feasibility for use as a solid fuel. Hydrothermal carbonization was conducted at a relatively low temperature range, owing to the energy consumption during the process. Based on the results, the properties of lipid extracted algae as a solid fuel improved during hydrothermal carbonization, being comparable to the low-ranked coals. The hydrochars thus produced showed stable combustion characteristics at high temperature. Moreover, the sulfur and ash content in the lipid extracted algae decreased through devolatilization during hydrothermal carbonization, allowing the hydrochar (low sulfur and ash content) to be considered as a clean energy source. Thus, a sustainable integration process, combining the hydrochar production from lipid extracted algae with the algal biodiesel process, seems to be feasible for solid fuel production and disposal of waste.

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1. Introduction

Since fossil fuels are not only limited, but also cause air pollution, a number of biofuels have been developed as alternative energy sources to substitute them [1]. Among these biofuels, biodiesel production, particularly using microalgae as feedstock, is beneficial in terms of high growth rate [2], economical land usage (i.e., ability to be grown on sites not suitable for food crop cultivation) [3], high lipid content [4], and high carbon dioxide sequestration potential [5]. During biodiesel production, lipid extracted algae (LEA) is generated from the algal biodiesel production industries. The LEA residue, after the extraction of lipid, is commonly considered as waste. Therefore, an appropriate disposal method should be integrated in the algal biodiesel production process to achieve economic sustainability [6]. Previous studies have investigated the utilization of LEA as feedstock for the production of useful products (i.e., animal food, protein, and reduced sugar) [6,7] and renewable energy (i.e., biogas) through anaerobic digestion [8,9]. Since two or

more chemicals are used for lipid extraction from algae, LEA can become contaminated, necessitating the purification of LEA before its utilization due to the chemicals remaining in it [10]. Thus, the aforementioned utilization of LEA (i.e., as feed/food for consumption by animals and substrate for renewable energy production) may be inappropriate.

The Hydrothermal Carbonization (HTC) process is defined as a thermochemical process used to convert biodegradable biomass into biochar in the presence of moisture under moderate temperature (180–350 °C) and pressure (2–10 MPa) conditions [11]. It might be feasible to synthesize valuable carbon materials from the feedstock, while classifying the process according to the different operating conditions (i.e., temperature) [12]. A low-temperature HTC is commonly conducted at moderate temperature (up to 250 °C), consuming less energy than the high-temperature based process [13,14]. In this respect, a low-temperature HTC could be a plausible disposal solution for LEA in terms of efficient conversion of abandoned carbon source to useful biocarbon with low energy consumption and energy recovery potential.

Various types of biodegradable waste, such as microalgae, sewage sludge, municipal solid waste, livestock manure, and cellulosic biomass, can be converted to useful biocarbon through

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HTC treatment. After hydrothermal treatment, fuel properties (i.e., heating value, fuel ratio, and aromatic structure) of the produced biocarbon could be improved, compared to the untreated biomass [15–17]. The produced biocarbon could be incinerated as fuel for single/co-combustion and utilized as industrial material [18]. Although lipid is extracted from its original biomass (microalgae), LEA contains a high amount of carbon, with comparable calorific value. Since the HTC can be directly applied to moist or muddy biomass, without pre-treatment (i.e., drying) [19,20], it can be suitable for the treatment of LEA [21]. However, there are a limited number of studies on the treatment and disposal of LEA through HTC. The treatment efficiency and energy recovery potential of HTC for LEA should be studied to secure the sustainable application of HTC for LEA.

The main objective of this study was to produce an alternative solid fuel from LEA through the HTC process that can convert abandoned organic matter to hydrochar, while suggesting its integration with the energy recovery process, thus combining biodiesel production from microalgae and solid fuel production from LEA. In order to achieve this goal, a laboratory-scale HTC treatment process was operated under low temperature (240 °C) with LEA, while the characteristics of the produced hydrochars were investigated.

2. Materials and methods

2.1. Materials

In this study, *Chlorella vulgaris* (*C. vulgaris*) was selected as the microalgae species for lipid extraction. LEA obtained from *C. vulgaris* was used as the feedstock for HTC. *C. vulgaris* is a eukaryotic photosynthetic microorganism that grows in a short duration due to its simple structure. *C. vulgaris* is one of the most useful and well-known species of feedstock for biodiesel production due to its relatively high lipid content (in the range of 5–40%) [22]. In this study, *C. vulgaris* was obtained from a commercial market. The physicochemical properties of *C. vulgaris* used in this study showed a volatile solid content of 85% (dry basis, wt.), ash content of 6% (dry basis, wt.), and pH of 6.9. *C. vulgaris* generally consists of proteins, lipids, and carbohydrates, which account for 51–58, 5–40 (can contain up to 58 under unfavorable growth conditions), and 6–12% of dry biomass, respectively [2,23]. The amounts of proteins, lipids, and carbohydrates in *C. vulgaris* used in this study were comparable with the references.

The total lipids in *C. vulgaris* were extracted using Dimethyl Ether (DME). DME is the simplest ether, with the formula CH_3OCH_3 , being widely used for extracting lipids from a diverse species of microalgae. For lipid extraction, the *C. vulgaris* was oven-dried to remove moisture completely until constant weight was obtained (more than 12 h). The oven temperature of 70 °C was maintained during oven drying. Oven-dried *C. vulgaris* was ground into a fine powder. One gram of dried *C. vulgaris* was mixed with 20 mL of DME, being sustained for 30 min. Subsequently, the samples were centrifuged for 10 min at 3000 rpm, with the liquid phase being moved to a glass-vial. To evaporate solvent, algal slurry was placed in hood chamber with heating as 60 °C for 24 h. After evaporation, the remaining solid residue was considered as LEA.

2.2. Hydrothermal carbonization

HTC of LEA was carried out to study the effects of different process conditions on product (Fig. 1). Experiments were performed using a lab-scale reactor with a volume of 1 L, into which 300 mL of LEA feedstock, mixed with an equal amount of distilled water (total amount of reactant = 600 mL), was loaded. The reactor

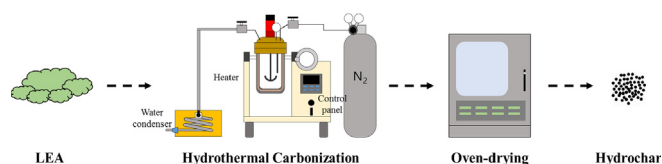


Fig. 1. Layout of experimental setup.

(batch type) consists of the stainless steel Teflon-lined vessel, heater, and steam condenser. Teflon-liner was employed to avoid damage of vessel wall by halogen and sulfur under hot-compressed water. Anaerobic condition was achieved by purging the reactor with nitrogen gas for sufficient time before the HTC experiments. The operating temperatures ranged between 180 and 240 °C (pressure was not controlled), with the reaction being maintained for 30 min in the presence of subcritical water. The contents of the reactor were mixed vigorously using an agitator, rotating at 200 rpm. After the HTC treatment, the residual steam was discharged from the reactor. The reaction products were collected in mixed both solid and liquid phases. The mixed products were oven-dried (105 °C, 12 h) and the hydrochars as dry matter were obtained. Hydrochar yield was calculated using the following equation (Eq. (1)).

$$\text{Hydrochar yield (\%)} = \frac{\text{Dry weight of Hydrochar}}{\text{Dry weight of LEA}} \times 100 \quad (1)$$

2.3. Analytical methods

The list of equipment for analysis and test methods are summarized in Table 1. Proximate analysis was carried out according to the Korea standard test method for coal and coke (KS E 3705). The elemental composition (e.g., carbon, hydrogen, oxygen, nitrogen, and sulfur contents) of the samples (i.e., LEA and the produced hydrochars) were determined using an elemental analyzer (Flash1112, Thermo Fisher Scientific, Germany). For elemental composition analysis, the samples were completely oven-dried at 70 °C, subsequently being ground into a fine powder. The higher heating values (HHVs) of LEA and hydrochars were determined by a calorimeter (Parr6400, Parr instrument, USA) according to the standard method for calorimetric analysis (EPA 5050). Results were obtained by taking the average of the repeated experiments, performed in triplicate. The structure of the produced biochar samples was analyzed by Fourier Transform Infrared (FTIR) spectroscopy (Vertex70, Bruker, Germany) in the resolution range of 4000–400 cm^{-1} . The FTIR spectra of the samples were converted into pellet form, using KBr (120 mg, 1% by wt.) as the reference material. Thermal Gravimetric Analysis (TGA) is commonly conducted to analyze combustion behavior [24]. In this study, TGA was carried out using a Thermogravimetric Analyzer (TGA Q5000, TA instruments, USA) at atmospheric pressure, with the air flowing at a rate of 25 mL/min. TG (weight loss) and DTG (rate of weight loss) of the biochar samples were observed continuously under non-isothermal conditions. The initial temperature was 40 °C, with a 20 °C/min step, up to 1000 °C. Finally, the structure of LEA and hydrochars were investigated by electron microscope imaging using Olympus BX51 (Olympus, Japan).

3. Results and discussion

3.1. Characteristics of hydrochars

The elemental composition of LEA and hydrochars are shown in

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