



Simultaneous production of bio-solid fuel and bio-crude from vegetal biomass using liquefied dimethyl ether



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HIGHLIGHTS

- DME method has robust dewatering ability for the high-moisture vegetal biomass.
- This method also efficiently removes the organic bio-crude from the vegetal biomass.
- The properties of dewatered biomass should be suitable for used as bio-solid fuel.
- DME method can efficiently extract lipids from lipid-rich vegetal biomass as same as hexane Soxhlet.

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ABSTRACT

Liquefied dimethyl ether (DME) was used for moisture and bio-crude extraction in 4 common vegetal biomasses. The process removed approximately 81.3–88.7% of the water from these biomasses and yielded 5.8–16.8% bio-crude of dry weight of sample. The properties of the original samples, dewatered bio-solids, extracted bio-crude and removed water were studied by elemental analysis, gel permeation chromatography (GPC), and gas chromatography-mass spectrometry (GC-MS), respectively. In addition, the oxygen activity of the bio-solids was investigated. The results indicate that both bio-solids and bio-crude could be further exploited for energy production and other purposes.

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1. Introduction

Fossil fuel depletion and global warming problem have led to the development of alternative energy source, particularly by utilising renewable resources such as municipal waste and agricultural vegetal biomass [1,2]. However, the vegetal biomass generally contains organic components (the so-called bio-crude) and excessive water, which cause environmental problems and requiring capital-intensive methods of biofuel production. Currently, the main methods for biofuel production from vegetal biomass are gasification and/or liquefaction [3], and/or making them bio-solid as refuse-derived fuels (RDF) [4]. The biomass with or without drying is usually treated by a physical or chemical method to recover their combustible chemical components such as lipids and hydrocarbons. An example of these methods is pyrolysis, which can convert biomass into useful liquid and gaseous fuel and solid char. However, the high energy consumption is still a

drawback of pyrolysis due to the large amount of heat required for this process [5]. RDF, on the other hand, has various merits such as easy transportation and combustible stability. However, direct combustion of RDF results in waste of value-added chemicals in the biomass and the production of environmental pollutants such as dioxins and polychlorinated biphenyls (PCBs) [6].

In previous studies, we have developed a new method, which uses liquefied dimethyl ether (DME) as extractant to remove the water from wet materials. In addition, simultaneously with dewatering, DME can also extract the bio-crude from the materials because DME is an organic solvent and can dissolve a wide range of organic components.

Thus far, this method has been studied on both laboratory and bench scale and reported on (i) drying of low-rank coal at normal and frozen temperature [7,8]; (ii) removal of PCBs from polluted soil [9]; (iii) extraction of lipids and hydrocarbons from high-moisture microalgae [10–12] and (iv) extraction of bioactive components from the green tea leave [13]. Our institute is also attempting to carry out a pilot-scale study on this technology. The advantages of the proposed method are that it is high energy-efficient and environmentally friendly, with its principle being based on the following DME characteristics: (i) High affinity

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to organic compositions and partial miscibility with water. The phase equilibrium relationship for the liquefied DME/water system is presented in detail in a previous paper [14]. At normal temperature, water is soluble in DME in the range of 7–8 wt.% [14]. Therefore, the weight of DME required for the extraction of water is $1/(0.07 - 0.08)$ times the weight of water. (ii) The standard boiling point of DME is $-24.8\text{ }^{\circ}\text{C}$, and it exists in a gaseous state under normal conditions. Its saturated vapour pressure is mentioned and explained in detail in Ref. [15]; for example, it is 0.51–0.59 MPa in the normal temperature range ($20\text{--}25\text{ }^{\circ}\text{C}$). (iii) The European Food Safety Authority concluded that there are no safety concerns with regards to the use of DME as an extractant in food processing [16].

The principle of energy-saving of this process is shown in Fig. 1. First, the process involves mixing the water/organic component-bearing materials with liquefied DME and subsequent extraction of water and bio-crude from the materials using DME. In the next step, DME in the mixture was evaporated at around room temperature and then the bio-crude are separated from water in the after-treatment process. For large-scale application, a heating source of unharnessed waste heat of about $40\text{ }^{\circ}\text{C}$ is desirable for DME evaporation. DME gas is then liquefied again at a slightly lower temperature for recirculation. In this stage, a cooling source of about $10\text{ }^{\circ}\text{C}$ such as geo-heat, which is the temperature within the first 50 m of the Earth's surface, is desirable [17,18].

Taking all these factors into consideration, we performed experiments to investigate the applicability of this method for dewatering and extraction of bio-crude for high-moisture vegetal biomass. In this study, 4 representative common vegetal biomasses including spent coffee ground, tea leaf waste, orange peel and gramineous weed were selected for testing. The former three are main industrial food wastes with very huge production worldwide, thus, the effective utilisation of them is important. For example, the world annually produces spent coffee grounds of around 6 million tons from the beverage factory [19]. According to a credible report, the spent coffee grounds contain around 10–15% oily substance depending on the coffee species, which can be easily converted to biodiesel [20]. As an unexplored biomass resource, the gramineous weed is an herbaceous plant which can be seen and available anywhere, therefore, it is an ideal sample to investigate the performance of DME method on such vegetal biomass. In this paper, the properties of dewatered bio-solids, extracted bio-crudes and removed water were analysed and evaluated. In addition, the oxygen reactivity of the dewatered bio-solids was examined.

2. Experimental

2.1. Materials

The spent coffee grounds (pulverized coffee bean) were supplied by a Japanese brewing company. The species of tea leaves

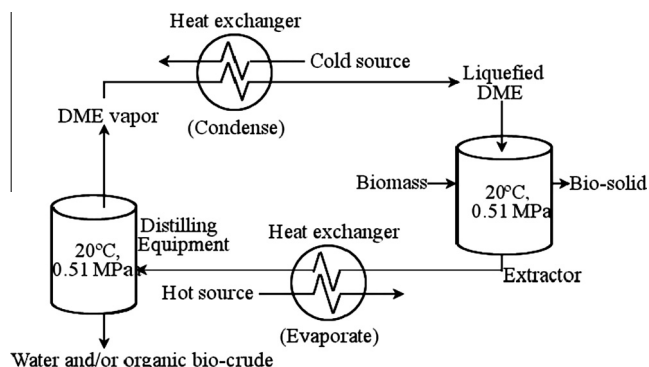


Fig. 1. Schematic illustration of the DME extraction method.

is *Camellia sinensis* obtained from a producing plant in Shizuoka, Japan. The initial water content and profile of the tested biomasses before and after DME extraction and the extracted bio-crudes are shown in Fig. 2. Because the size of the coffee grounds was larger than that of the extraction column, the coffee grounds were again pulverised to particle size to be able to load them into the extraction column. Gramineous weed and orange peel were cut into cylinders and rectangles. The tea leaves were loaded into the column in their received form. The following sizes of samples were average values obtained from 10 gauging stations, with maximum and minimum values. The diameter of pulverised coffee grounds was 2.5 (3.6–1.5) mm. The as-received tea-leaf sizes were between 1.0 (0.5–1.7) mm and was 13.6 (7.0–19.0) mm, and the thickness was 0.5 (0.1–1.1) mm. Orange peel was cut into sizes between 6.3 (4.8–7.5) mm and 19.2 (13.0–28.0) mm, and the thickness was 2.3 (1.3–3.1) mm. Gramineous weed was cylindrically cut into a diameter of 4.3 (between 1.5 and 5.2) mm, and the length was 28.1 (22.4–33.7) mm, and the thickness was 2.3 (1.3–3.1) mm.

2.2. Outline of extraction method

The experimental apparatus has been described in detail in our previous paper [10]. Fig. 3 is a schematic diagram of the Lab-scale DME extraction apparatus. It consists of 2 main parts: an extraction column (diameter, 11.6 mm; length, 190 mm; HPG-10-5, Taiatsu Techno Corp., Saitama, Japan) and a storage vessel for the mixture of DME, water, and bio-crude (HPG-96-3, Taiatsu Techno Corp.). The extraction column was loaded with the test sample. On average of 3 tests, the amounts of spent coffee ground, tea leaf waste, orange peel and gramineous weed were 6.85 (± 0.1) g, 4.84 (± 0.2) g, 5.11 (± 0.2) g and 5.21 (± 0.2) g, respectively. At the column outlet, extracted bio-crude passed through a filter (pore diameter $< 0.65\text{ }\mu\text{m}$). The DME flow rate was $10\text{ }(\pm 1)\text{ cm}^3\text{ min}^{-1}$, and the extraction temperature and pressure were $20\text{ }^{\circ}\text{C}$ and 0.51 MPa, respectively.

2.3. Property analysis

The proximate analysis, ultimate analysis, and high heating value (HHV) measurements were carried out for the original dry biomass samples and the bio-solids obtained from DME extraction. The analytical methods were according to the Japanese Industrial Standard (JIS) and were the same as those used in our previous study [10]. The biomass samples and dewatered bio-solids were pre-treated for this analysis by continual drying at $105\text{ }^{\circ}\text{C}$ up to constant weight in vacuum.

The concentrations of minor elements, Na and K, in the bio-crudes and removed water were determined by flame atomic absorption spectrometry (Z-2000; Hitachi High-Technologies Corp., Tokyo, Japan) according to the Japanese industrial standard methods JIS K 0102 48.2 (Na) and 49.2:2011 (K). The Ca and Mg concentrations were determined by inductively coupled plasma atomic emission spectroscopy (Optima3300XL; Perkin Elmer Inc., USA) according to JIS K 0102 50.3 (Ca) and 51.3:2011 (Mg). The P concentrations in the bio-crudes were determined by sulphuric-perchloric acid digestion molybdenum blue absorption spectroscopy (U-2001; Hitachi High-Technologies Corp., Tokyo, Japan) according to the analytical method JIS K 0102 46.3.2:2011. The P concentrations in the removed water were determined by potassium peroxodisulphate digestion molybdenum blue absorption spectroscopy (U-2001) according to the analytical method JIS K 0102 46.3.1:2011. The N concentrations in the removed water were determined by the copperised cadmium column method according to the analytical method JIS K 0102 45.4:2011.

The molecular weight distributions (MWDs) of the bio-crudes were determined by gel permeation chromatography (GPC) per-

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