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Research paper

Water removal from wood biomass by liquefied dimethyl ether for enhancing heating value

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ABSTRACT

Liquefied dimethyl ether (DME) was used as the dewatering solvent to remove water from *Tectona grandis* L.f. (*T. grandis* L.f.), *Shorea* sp., and *Cinnamomum camphora* (*C. camphora*) biomass wood. The experiments were performed at 27 °C (room temperature) and at a pressure of ~0.59 MPa using a batch system. The results showed that 13–25 g of liquefied DME removed 1 g of water from the wood chip biomass matrix with a water content of 10%–40%. The UV–vis spectra of the collected liquid extract indicated the presence of some wood biomass-derived compounds. The FT-IR spectra revealed that the functional group compositions of the wood chip biomass samples before and after DME dewatering were identical. In addition, the heating value of wood chip biomass samples increased by 754–1570 kJ/kg after dewatering.

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1. Introduction

Several energy sources can be used to produce electricity or other power forms, including fossil fuels, renewable sources, solar energy, and nuclear sources. Wood biomass is a renewable and sustainable energy source composed of organic materials (Chintala, 2018; Weldekidan et al., 2018; Proskurina et al., 2018). Although this energy source has long been used, to date the burning of wood collected by hand remains an important means to heat for cooking or other applications in many countries. When the wood biomass is used as an energy source, the generated energy is affected by several parameters including moisture content (McKendry, 2002; Nati et al., 2018; Lachowicz et al., 2018). Generally, the moisture content of wood biomass negatively influences its heating value and should be considered as an important factor.

Herein, liquefied dimethyl ether (DME) was used as a solvent to extract water from several wood species, including *T. grandis* L.f., *Shorea* sp., and *C. camphora*. DME is the simplest type of ether with the formula CH₃OCH₃ and is gaseous at room temperature. DME has a low normal boiling point, −24.8 °C and its saturated vapor pressure at 20 °C is approximately 0.51 MPa (Wu et al., 2011). The relative permittivity of this compound is approximately 1.08 and 5.34 at 30.5 °C in the gaseous and liquid state, respectively. In addition, DME is a hydrogen bond acceptor which forms weak hydrogen bonds and is partially miscible in water at

room temperature (Hollendorff and Knapp, 1988). Therefore, DME can be easily removed from water by simple flash distillation at mild temperatures. Because of these characteristics, liquefied DME has been used as a solvent to remove water from plants, foods (Li et al., 2014), high-water content solids, low rank coals (Kanda et al., 2008a,b; Kanda, 2008; Kanda and Makino, 2010), sewage sludge (Kanda et al., 2008a,b), and polymers (Kanda et al., 2009).

It is well-known that two forms of water exist in wood biomass (Rowell, 2005), free and bound water, which can be found in the cell cavities and cell walls of wood. Free water is the water present within the cell lumina, while bound water is the water that is adsorbed within the cell wall of the wood (cellulose and hemicellulose). Generally, free water in the cell lumina of wood can be easily removed, whereas bound water embedded in the chemical constituents of the wood containing hydroxyl groups forms strong intermolecular hydrogen bonds. Thus, to reduce the water content in wood, drying is required. Accordingly, energy is required for the evaporation of free and bound water (Mujumdar, 2014; Chen et al., 2015; Stenstrom, 2019). For DME dewatering, the drying process developed herein differs from conventional methods based on drying where water is evaporated. DME dewatering was performed at room temperature and the wood biomass was dewatered using liquefied DME. The method developed herein will be important for wood biomass treatment as it is inexpensive and efficient.

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Table 1

Energy consumption of the wood chip biomass dewatering process by using oven (a) and liquefied DME (b).

| (a) Wood chip biomass dewatering by using oven.* | | | |
|--|------------------|------------|-------------------------|
| Wood species | Temperature (°C) | Time (min) | Energy consumption (kJ) |
| <i>C. camphora</i> | 35 | 73 | 8760 |
| <i>Shorea</i> sp. | | 57 | 6840 |
| <i>T. grandis</i> L.f. | | 120 | 14 400 |
| * Initial water content is 10% | | | |
| * Time is the time required to achieve the constant weight of wood chip biomass. | | | |
| * The oven power is 2000 w | | | |
| (b) Wood chip biomass dewatering by using liquefied DME.** | | | |
| Wood species | Temperature (°C) | Time (min) | Energy consumption (kJ) |
| <i>C. camphora</i> | 39 | 30 | 1800 |
| <i>Shorea</i> sp. | | | |
| <i>T. grandis</i> L.f. | | | |
| ** Initial water content is 10 to 40%. | | | |
| ** The heater power is 1000 w | | | |
| ** The liquefied DME can be recycled. | | | |

2. Materials and methods

2.1. Materials

Three hardwood species were obtained from a local wood processing company (Surabaya, Indonesia) were used as starting materials (*T. grandis* L.f., *Shorea* sp., and *C. camphora*). The wood chip biomasses were shredded using a laboratory mill and passed through stainless steel sieves with apertures of 5.6 and 3.35 mm. The samples were impregnated with water to achieve a 10%–40% moisture content in a plastic box for 5 days to obtain uniformly wet wood chip biomasses. The moisture content of the wood chip biomasses was subsequently determined from the weight of the oven-dried (105 °C) samples and expressed as a total weight percent (wood containing water). The energy consumption of the wood chip biomass dewatering process was described in Table 1. The DME used herein was purchased from PT Bumi Tangerang Gas Industry, Serang, Indonesia, while the oxygen and nitrogen were obtained from PT. Samator Gresik (Bambe), Indonesia.

2.2. Methods

Fig. 1 illustrates the DME dewatering apparatus for the laboratory-scale experiment that was used to remove water from the wood chip biomasses. This apparatus consisted of a dewatering column (10 cm³), needle valve to tune the DME flow rate, and collector storage tank (96 cm³). These main parts were purchased from Taiatsu Techno Corp., Saitama, Japan. In all experiments, 1.5 g of wet wood chip biomasses (moisture content: 10%–40%) was loaded into the dewatering column at the lower half, and the upper half of dewatering column was subsequently filled with glass beads. A filter with a pore size of 0.65 µm was placed in the dewatering column outlet. The DME dewatering process was performed at approximately 27 °C (room temperature) and ~0.59 MPa (Wu et al., 2011) using a semi-batch system and a DME flow rate of 10 mL min^{−1}. Initially, the liquefied DME was fed to the wood chip biomasses from the bottom of the dewatering column. Prior to starting the experiments, the air in the dewatering column was purged with DME gas via carefully opening and closing the inlet and outlet valves. Accordingly, the amount of water removed in this step was ignored. After filling the apparatus with various amounts of liquefied DME (10–30 g), the liquefied DME was contacted with the wood chip biomasses for 30 min by closing the inlet and outlet valves. Hence, the DME dewatering process time was estimated to be 30 min. At the end of experiments, the liquefied DME could be removed completely

from the wood chip biomasses and the extract via vaporization at room temperature (Hoshino et al., 2017).

The extracted solutions were analyzed using UV–vis spectrophotometry (UV–Vis Genesys 10S, Thermo Fisher Scientific, Waltham, MA) between 190 and 1100 nm. The samples were then placed in a quartz cuvette with a 1 cm path length and inserted into the UV–vis spectrophotometry device. Because water can strongly absorb UV light at wavelengths of <200 nm (Hu et al., 2019), prior to the analysis, a blank solution was required for initial calibration and was prepared using deionized water. UV–vis analysis is a simple and effective method for characterizing chemical compound and yields important information regarding the chemical structure of an analyte. During each DME dewatering trial, the collected solid products were analyzed using a Spectrum Two FT-IR spectrophotometer (Perkin–Elmer, Ltd., England), characterized by thermogravimetric-differential thermal analysis (TGA-50, Shimadzu, Japan), and subjected to bomb calorimetry (IKA Oxygen Bomb Calorimeter C 6000, Westtune, China). For FT-IR characterization, the spectra were obtained in attenuated total reflectance (ATR) mode (golden single reflection ATR system, P/N 10 500 series, Specac) at a 4 cm^{−1} resolution. The FT-IR setup was assembled using a standard optical system with KBr (potassium bromide) windows and a universal attenuated total reflectance (UATR) sampling accessory for a spectral collection. Therefore, the collected solid products were characterized and fed directly between two KBr window plates. For heating value analysis, the collected solid products (0.5 g) were placed inside the bomb calorimeter in a special container and ignited using an ignition wire (electrical ignition). This equipment consisted of the pressurized oxygen container placed in a container loaded with water. The water container was equipped with a precise thermocouple and the heating value was determined by measuring the water temperature increase.

3. Results and discussion

Fig. 2 shows the amount of water removed from the wood chip biomasses after DME dewatering with various amounts of liquefied DME. When 10 g of liquefied DME was added, water removal from *T. grandis* L.f., *Shorea* sp., and *C. camphora* reached 68.80, 67.58, and 79.10%, respectively. The extent of water removal from each wood chip biomass increased with increasing liquefied DME consumption, approaching 82.00, 80.04, and 81.08% when 30 g of liquefied DME was used for dewatering of *T. grandis* L.f., *Shorea* sp., and *C. camphora* wood chips, respectively. Under these conditions, liquefied DME with high dissolving ability and low viscosity

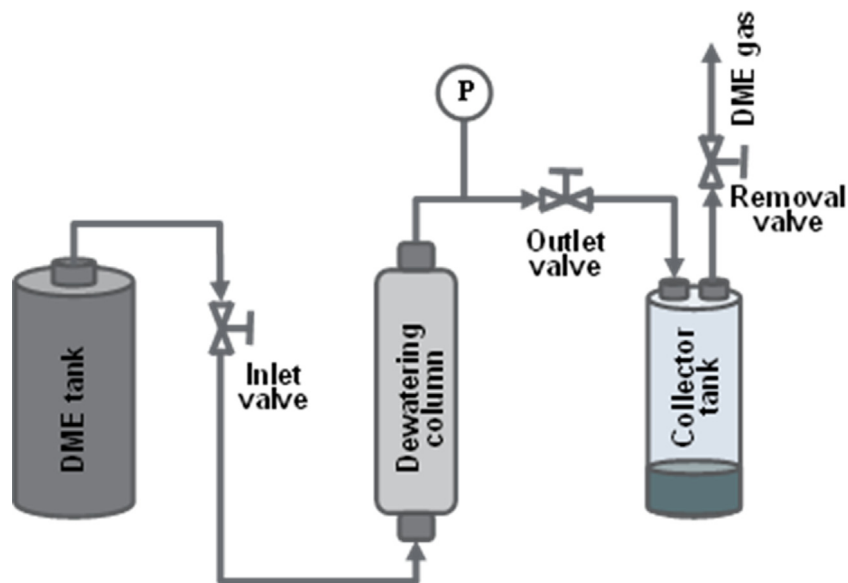


Fig. 1. The laboratory-scale apparatus scheme of the wood chip biomass dewatering by liquefied DME.

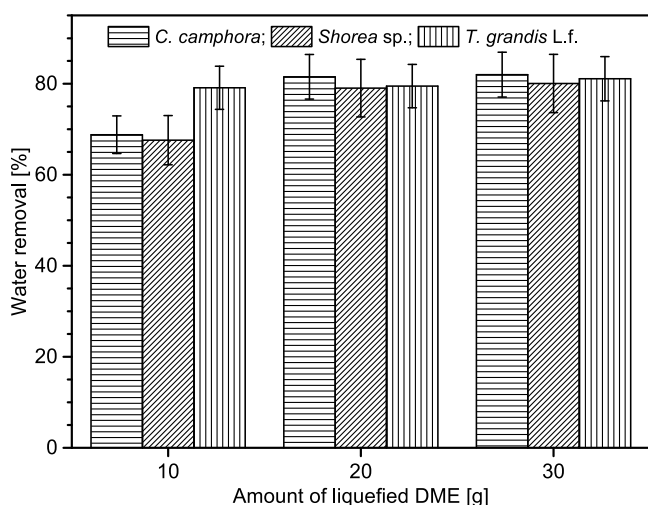


Fig. 2. Water removal from the wood chip biomasses by DME dewatering process.

can enter into the cells of the wood chip biomass. Subsequently, it flows out together with water and other components from the wood chip biomass matrix (Rowell, 2005; Kanda et al., 2014, 2015). In addition, DME and water are miscible with each other over the range of 7–8 wt% at room temperature (Hollendorff and Knapp, 1988). This is likely the reason why the liquefied DME can remove water from the *T. grandis* L.f., *Shorea* sp., and *C. camphora* wood chips.

Similar to extraction, the DME dewatering process, in principle, could be used for the removal of water and other soluble components from an insoluble material, either liquid or solid. In addition, it is well-known that wood biomass consists of various substances, including lignin, hemicellulose, and cellulose, each of which contains distinct groups of compounds when processed individually. Thus, their extraction may also occur when the liquefied DME was contacted with the wood chip biomasses. To examine the extractive components removed together with water

during DME dewatering, the collected liquid products were analyzed directly by UV–vis spectrophotometry after water dilution. Fig. 3 shows the UV–vis spectra of the water removal samples containing the extracted wood chip biomasses components from *T. grandis* L.f., *Shorea* sp., and *C. camphora* after DME dewatering. As shown in Fig. 3, the predominant peaks were observed at 205–210, 250, and 280 nm. Thus, lignin derived compounds were observed in the collected liquid products from each wood chip biomass sample, as indicated by the peaks at 205–210 nm. In addition, the peaks 250 and 280 nm arose from aldehyde groups, such as hydroxymethylfurfural and furfural, which were generated from cellulose groups during DME dewatering (Machmudah et al., 2015). However, it should be noted that the detailed information of the extracted components is not presented herein.

Fig. 4 shows the thermogravimetric curves for the different wood chip biomasses. The collected solid products were placed in a thermogravimetry apparatus and heated at $10\text{ }^{\circ}\text{C min}^{-1}$ from room temperature ($26\text{--}28\text{ }^{\circ}\text{C}$) to $600\text{ }^{\circ}\text{C}$. During analysis, the chamber was purged with air at a flow rate of 50 mL min^{-1} . Generally, air is composed of a mixture of oxygen, nitrogen, carbon dioxide, and other gases, where oxygen and nitrogen are the main components. Therefore, synthetic air was formed by mixing nitrogen and oxygen gases in a ratio of 20% oxygen and 80% nitrogen. This analysis provides information for the quantitative measurement of weight loss associated with the thermally induced transition and allows thermal stability and volatile components to be determined (Matsunaga et al., 2014; Jayaraman et al., 2017). Accordingly, the thermal degradation processes of *T. grandis* L.f., *Shorea* sp., and *C. camphora* before and after DME dewatering were examined using this analysis method. As shown in Fig. 4, the thermogravimetric curves of the different wood chip biomasses have three distinct stages under an air atmosphere. First, degradation occurred at $<260\text{ }^{\circ}\text{C}$ where a slight decrease of the wood chip biomass sample weight was observed due to water removal (drying) and decay of light volatile compounds. The second stage occurred between 260 and $460\text{ }^{\circ}\text{C}$ where a significant weight decrease for each wood chip sample was clearly observed. Generally, the decomposition of wood biomass components including cellulose, hemicellulose, and lignin occurred at this stage. The final stage involved the combustion of the heavy components

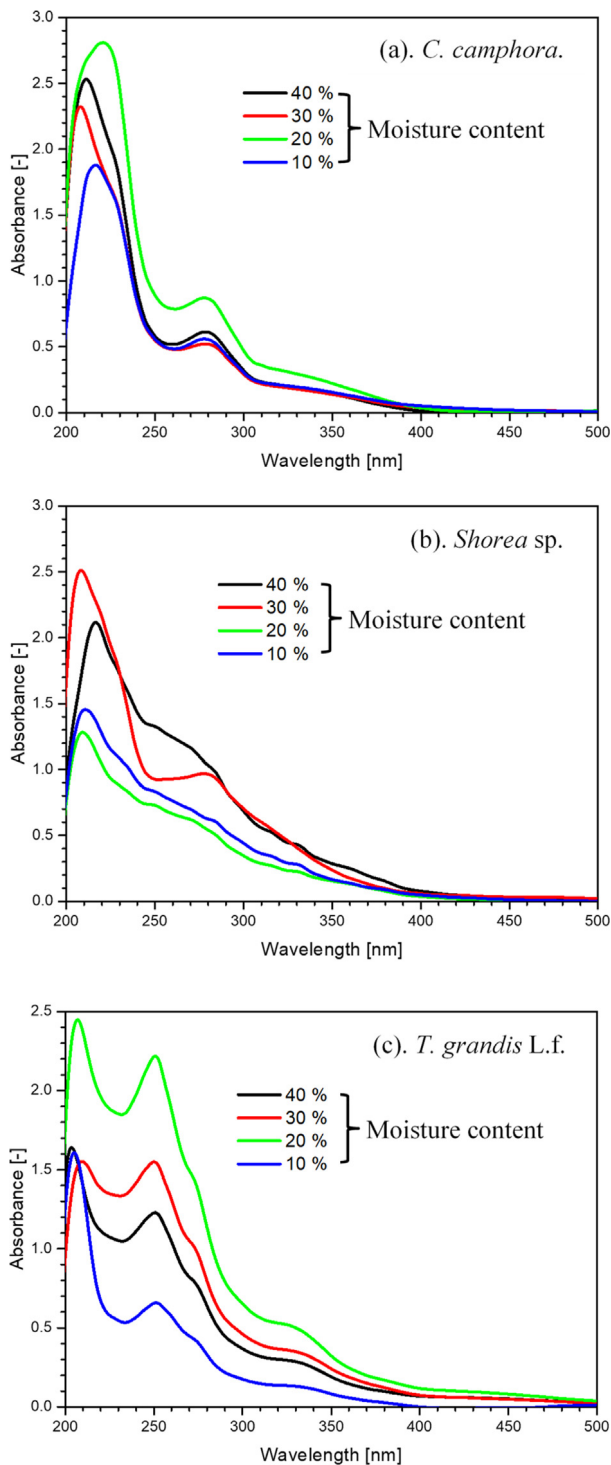


Fig. 3. UV-vis spectra of extractive components.

of the wood chip biomass and occurred at >460 °C. From Fig. 4, it is clear that the thermal decomposition behaviors of *T. grandis* L.f., *Shorea* sp., and *C. camphora* before and after DME dewatering were quite different. This is likely due to the water removal that included other biomass-derived substances from the wood chip biomasses during DME dewatering treatment, with the remaining contents exhibiting various thermal behaviors (Matsunaga et al., 2014).

During the drying process of wood biomass, water will typically move from zones of containing high moisture content to

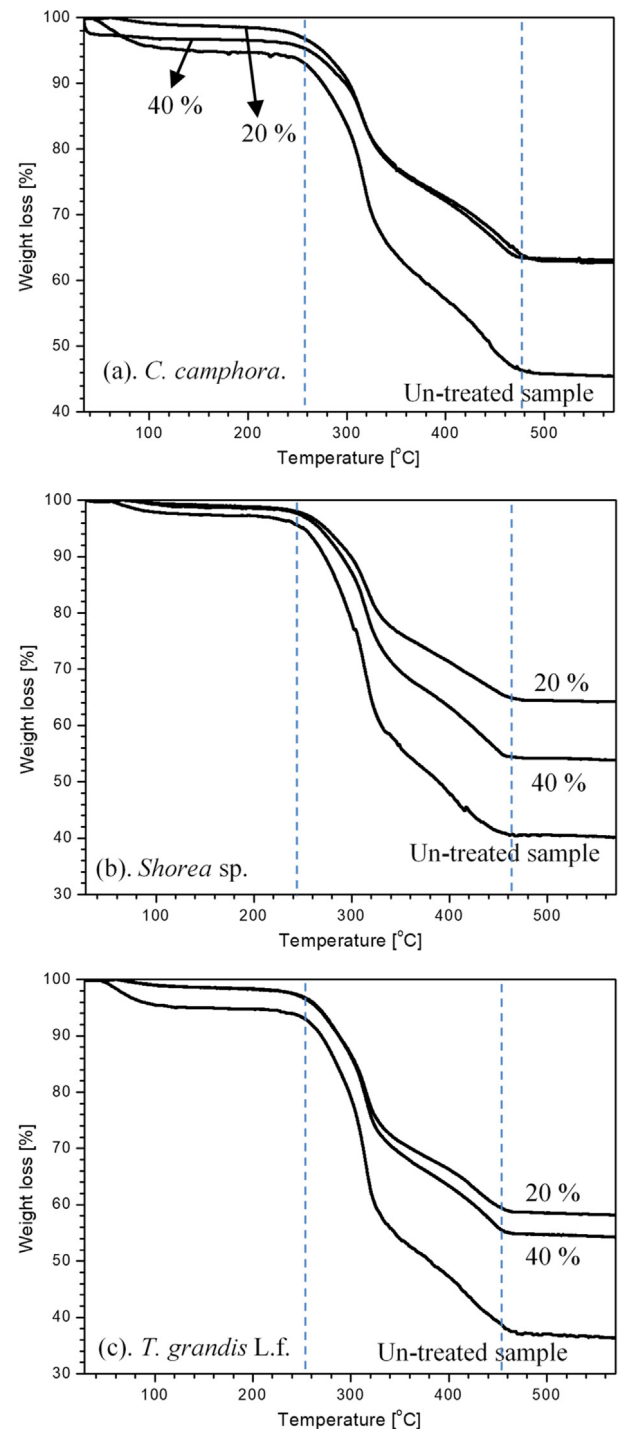


Fig. 4. TG curves of wood chip biomasses before and after treatment by DME dewatering process.

those with lower moisture contents. Thus, the wood biomass surface is drier than the interior of the materials if the moisture is to be released (Ross, 2010). For drying of wood biomass, the first water to be released is free water. The bound water associated with wood biomass substances is not removed until all free water has been released. Therefore, a change in the wood biomass structures may occur during water removal. Herein, the chemical structures of *T. grandis* L.f., *Shorea* sp., and *C. camphora* before and after DME dewatering were characterized by FT-IR in a range from 4000 to 400 cm^{-1} . This is a typical analytical

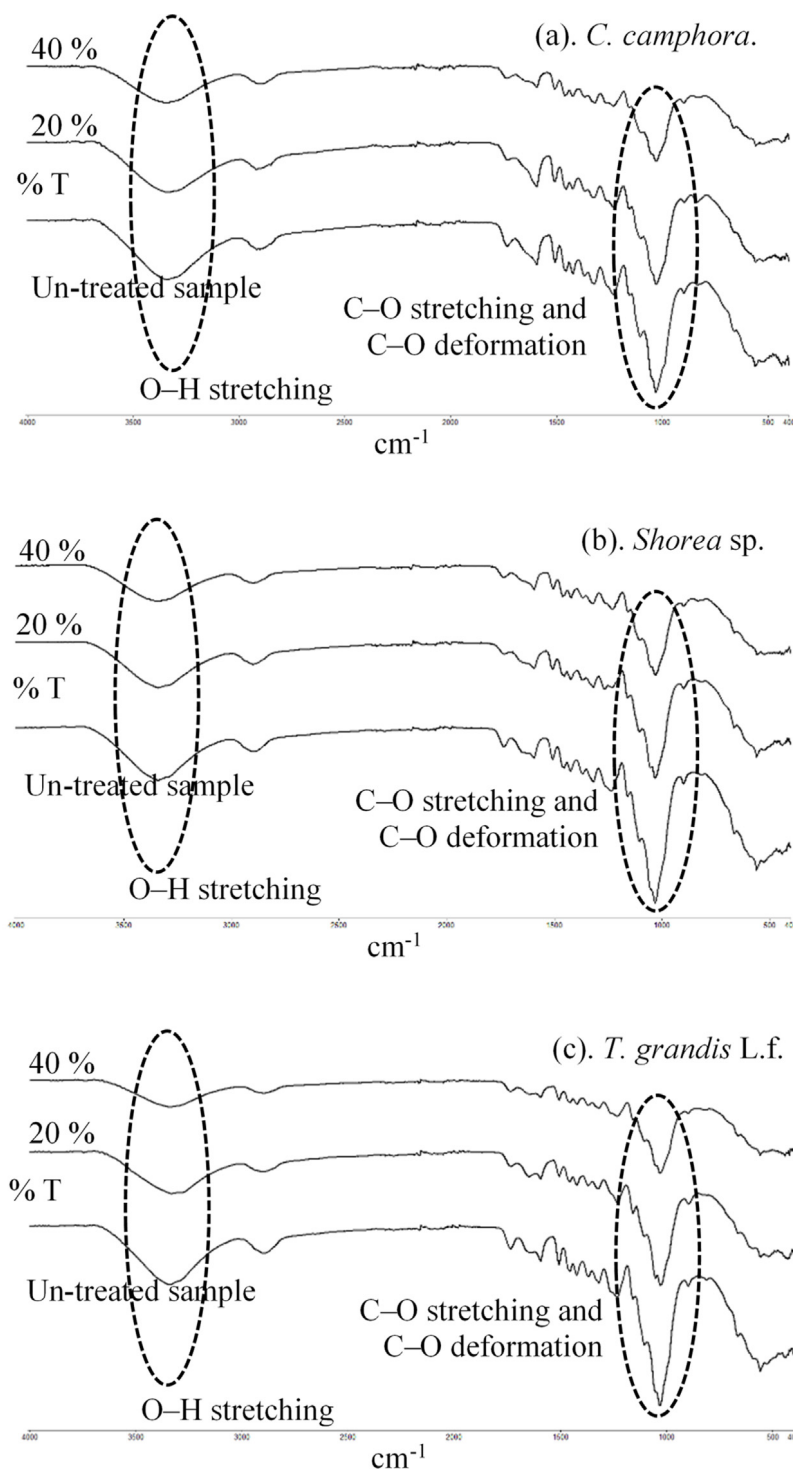


Fig. 5. FT-IR spectrum of wood chip biomasses before and after treatment by DME dewatering process.

method that allows for the identification of unknown substances and the chemical bond types of the constituent compounds in samples. Fig. 5 shows the FT-IR spectra of *T. grandis* L.f., *Shorea* sp., and *C. camphora* samples before and after DME dewatering. It is clear that these wood chips comprised lignin, hemicellulose, and cellulose as main wood biomass components with mainly alcohol, ketone, aromatics, esters, and alkene functional groups (Matsunaga et al., 2014; Kodama et al., 2015). Table 2 summarizes the functional groups and peak positions of all infrared bands for the wood biomasses. The FT-IR spectra of wood chip biomass samples before and after DME dewatering were similar and varied

only in peak intensity. This indicated that their functional group compositions did not significantly differ after DME dewatering. Thus, it is clear that water was successfully removed from the *T. grandis* L.f., *Shorea* sp., and *C. camphora* wood biomass matrices by liquefied DME at room temperature.

The liquefied DME may dissolve various substances including polar and non-polar compounds. It is known that liquefied DME is a good solvent for many compounds containing hydrogen-bonded and can act as a hydrogen bond acceptor, hydrogen bonding with solutes. At room temperature, liquefied DME has a low viscosity and can enter into wood chip biomass cells without disrupting

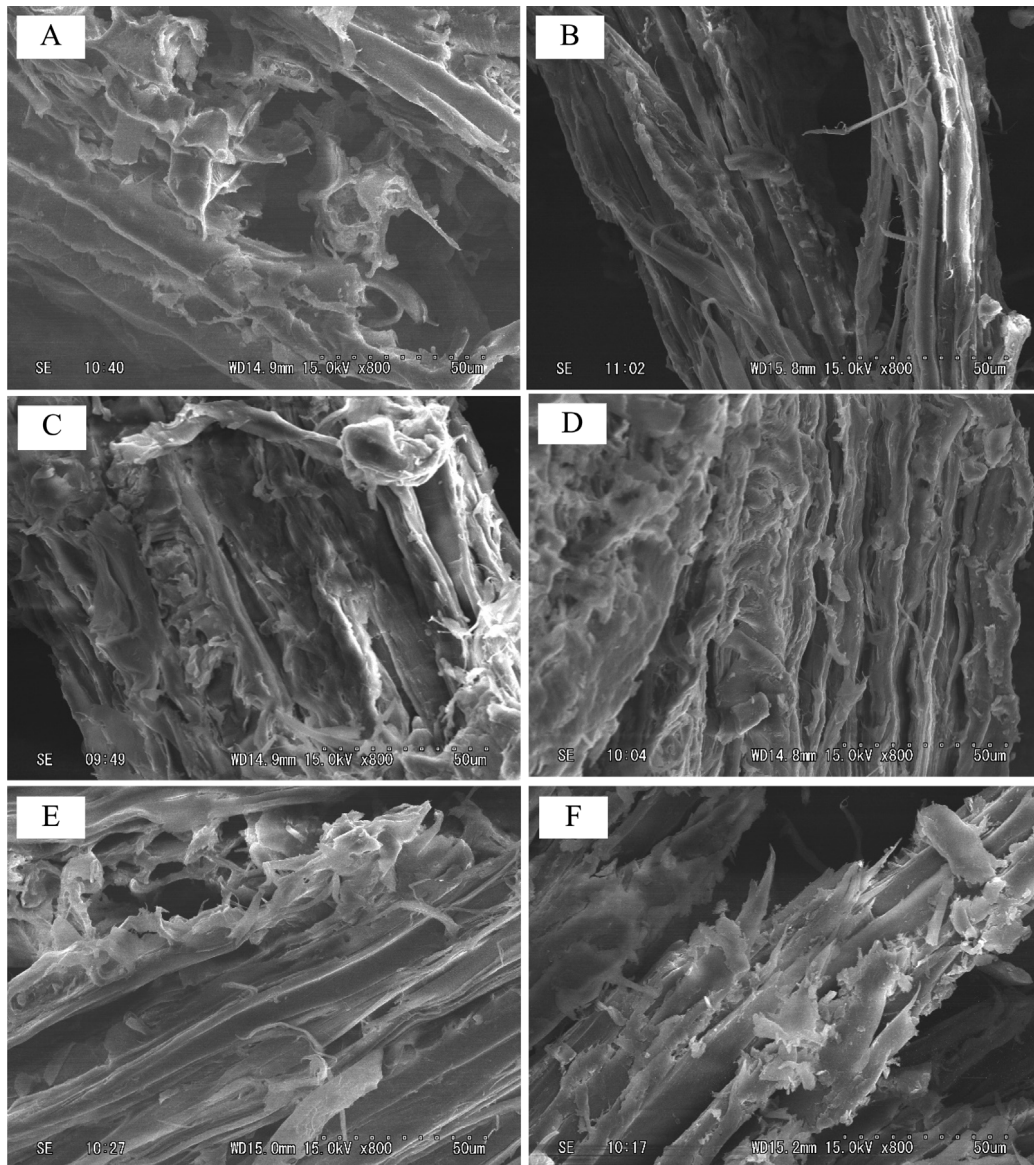


Fig. 6. SEM images of wood chip biomasses before (A, C, and E) and after (B, D, and F) treatment by DME dewatering process for *C. camphora*, *Shorea* sp., and *T. grandis* L.f., respectively.

cell walls. In addition, liquefied DME can also exit the wood chip biomass matrix together with water and other substances without any cell wall disruption (Kanda et al., 2014, 2015). Fig. 6 shows SEM images of the wood chip biomasses with 40% moisture content before and after treatment by DME dewatering. Before DME dewatering, the surface morphology of the wood chip biomass exhibited some clear border and did not show the presence of surface cracks or pores. After treatment by liquefied DME, their physical structures were not damaged in any of the wood chip biomass samples. This indicated that the liquefied DME treatment extracted the water and other substances from the wood chips without destroying their cell walls. Chemat and Vian (2014) reported the extraction mechanism including dewatering using liquefied DME as an extraction media. The authors also showed that initially, the water and/or target substances in the biomass matrix were extracted using liquefied DME resulting in a mixture of DME, water, and organic substances. In addition, the authors stated that the concentration of organic substances and water in the liquefied DME increased, achieving saturation while the biomass matrix was simultaneously dried. In the final

step, DME in the mixed solution was vaporized and water and organic substances were then separated. Chen et al. (2020) also reported a similar mechanism for water removal from sludge containing water and organic and inorganic matters that was generated from the second process of the wastewater treatment plant. They explained that the liquefied DME dissolved the substances (polysaccharides, lipids, etc.) in the sludge and at the same time, some water was also miscible with liquefied DME. The liquefied DME and the mixed solution will flow out together from the sludge due to the different vessel pressures during the process. Next, the liquefied DME can be separated from the mixed solution in the separation vessel via the vaporization process.

To understand the efficacy of dewatering using DME, the heating value of wood biomass before and after dewatering were measured using a bomb calorimeter. The wood biomass heating values are listed in Table 3. Based on the bomb calorimetry analysis, the heating values of *T. grandis* L.f., *C. camphora*, and *Shorea* sp. increased to 754, 1160, and 1570 kJ/kg, respectively, after dewatering. This indicated that DME can remove water content and increase the higher heating value (HHV) of the wood biomass.

Table 2
Main functional groups of wood biomass.

| Wave number [cm ⁻¹] | Functional groups | Compounds |
|---------------------------------|------------------------------------|----------------------------|
| 3600–3000 | O–H stretching | Acid, methanol |
| 2860–2970 | C–H _n stretching | Alkyl, aliphatic, aromatic |
| 1700–1730, 1510–1560 | C=O stretching | Ketone and carbonyl |
| 1632 | C=C | Benzene stretching ring |
| 1613, 1450 | C=C stretching | Aromatic skeletal mode |
| 1470–1430 | O–CH ₃ | Methoxyl–O–CH ₃ |
| 1440–1400 | O–H bending | Acid |
| 1402 | C–H bending | |
| 1232 | C–O–C stretching | Aryl-alkyl ether linkage |
| 1215 | C–O stretching | Phenol |
| 1170, 1082 | C–O–C stretching vibration | Pyranose ring skeletal |
| 1108 | O–H association | C–OH |
| 1060 | C–O stretching and C–O deformation | C–OH (ethanol) |
| 700–900 | C–H | Aromatic hydrogen |
| 700–650 | C–C stretching | |

Table 3
Heating value of wood chip biomasses before and after DME dewatering process.

| Wood species | Heating value (kJ/kg) | |
|------------------------|-----------------------|-----------------|
| | Before treatment | After treatment |
| <i>C. camphora</i> | 17 136.56 | 18 296.32 |
| <i>Shorea</i> sp. | 16 052.18 | 17 622.24 |
| <i>T. grandis</i> L.f. | 16 981.65 | 17 735.28 |

4. Conclusions

Water removal from *T. grandis* L.f., *Shorea* sp., and *C. camphora* as wood biomass matrices using the liquefied DME was performed at room temperature in batch processes. Approximately 13–25 g of liquefied DME can remove 1 g of water from wood chip biomass matrices containing 10–40% water. The UV–vis spectra of the collected liquid extract showed that wood biomass-derived compounds were present. The FT-IR spectra indicated that the functional group compositions of the wood chip biomass samples before and after DME dewatering were similar. The heating value of the wood biomasses increased after dewatering. Based on these results, DME dewatering is suitable for water removal from wood chip biomass to enhance energy source quality.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRedit authorship contribution statement

Siti Machmudah: Conceptualization, Validation, Supervision, Funding acquisition. **Dimas Tiar Wicaksono:** Investigation, Formal analysis, Visualization. **Mary Happy:** Investigation, Formal analysis, Visualization. **Sugeng Winardi:** Supervision, Data curation. **Wahyudiono:** Methodology, Writing - original draft. **Hideki Kanda:** Resources. **Motonobu Goto:** Writing - review & editing.

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