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Simultaneous Extraction and Emulsification of Food Waste Liquefaction Bio-Oil

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Abstract: Biomass-derived bio-oil is a sustainable and renewable energy resource, and liquefaction is a potential conversion way to produce bio-oil. Emulsification is a physical upgrading technology, which blends immiscible liquids into a homogeneous emulsion through the addition of an emulsifier. Liquefaction bio-oil from food waste is characterized by its high pour point when compared to diesel fuel. In order to partially replace diesel fuel by liquefaction bio-oil, this study aimed to develop a method to simultaneously extract and emulsify the bio-oil using a commercial surfactant (Atlox 4914, CRODA, Snaith, UK). The solubility and stability of the emulsions at various operating conditions such as the bio-oil-to-emulsifier ratio (B/E ratio), storage temperature and duration, and co-surfactant (methanol) addition were analyzed. The results demonstrate that higher amounts of bio-oil (7 g) and emulsifier (7 g) at a B/E ratio = 1 in an emulsion have a higher solubility (66.48 wt %). When the B/E ratio was decreased from 1 to 0.556, the bio-oil solubility was enhanced by 45.79%, even though the storage duration was up to 7 days. Compared to the emulsion stored at room temperature (25 °C), its storage at 100 °C presented a higher solubility, especially at higher B/E ratios. Moreover, when methanol was added as a co-surfactant during emulsification at higher B/E ratios (0.714 to 1), it rendered better solubility (58.83–70.96 wt %). Overall, the emulsified oil showed greater stability after the extraction-emulsification process.

Keywords: emulsification; liquefaction; bio-oils; co-surfactant; surfactant; diesel

1. Introduction

In the 21st century, food wastes in excess of one billion metric tons are generated worldwide every year from various sources and such great amounts lead to a severe environmental problem. Failure to recycle or treat food wastes properly will lead to the emission of methane (CH₄) and carbon dioxide (CO₂) during bio-degradation. These greenhouse gases may cause human health and environmental problems [1].

In recent years, there has been an improvement in thermochemical conversion equipment, which can transform wastes into biofuels or bioenergy products [2,3]. Food waste is considered a valuable source of energy due to its high biodegradability and high organic matter content [4,5]. Nevertheless, there is a great challenge in the conversion of food wastes into biofuels by traditional thermochemical conversions such as pyrolysis and gasification. The high moisture content of food waste requires more time and energy for drying before the conversion proceeds.

Hydrothermal technology (HTL) provides opportunities for more energy efficient valorization of wet biomass in that water is used directly as a reaction medium, thus, bypassing the drying process [6,7]. After HTL is performed, a separation procedure is needed to obtain the oil phase, also known as the liquefaction bio-oil. The separation method includes using a gravimetric separation funnel or extraction with an organic solvent such as tetrahydrofuran (THF), ethyl acetate, and dichloromethane (DCM) [8,9]. The liquefaction bio-oil is a mixture of several hundred organic compounds such as acids, alcohols, aldehydes, amides, esters, ketones, phenols, and lignin oligomers [10,11]. The liquefaction bio-oil has undesirable features for industrial applications such as a high viscosity and high ash content. This implies that the bio-oil composition is complex, making it relatively difficult to control the performance during the combustion process.

Emulsification is an effective way for upgrading bio-oil and blending it with other fuels [12,13]. Emulsification technology is popular for upgrading pyrolysis oil. Since diesel and bio-oil have polar molecules, the emulsifier or surfactants can disperse them both to form a stable and homogeneous emulsion [12]. The emulsification of bio-oils or water in diesel using various emulsifiers is summarized in Table 1. Michio [14] studied the emulsion of diesel oil and pyrolysis oil from hardwood and investigated the effects of five operation parameters (bio-oil concentration, surfactant concentration, residence time, motor speed and emulsification temperature). The results were optimized by a statistical model and the dominant variables were identified for minimal stratification formation. These dominant parameters were bio-oil concentration, and surfactant and power input. Chiaramonti [15] studied diesel/bio-oil emulsions with four kinds of pyrolysis oils from Canadian oak, beech wood, California pine, and pine wood. The results suggested that it is necessary to use fresh bio-oil and micronize these products to obtain a stable emulsion. Guo [16] used a KDL-5 molecular distillation apparatus to separate the middle fraction and the heavy fraction of the crude bio-oil. The blending of the diesel and bio-oil was conducted by ultrasonic and ultrasonic-mechanical emulsification. The droplet size distribution was measured using a Malvern nanometer particle size analyzer for the three emulsions (heavy fraction with 40–300 nm, middle fraction with 15–60 nm and crude bio-oil with 8–25 nm) and had the same tendency with the stability time 14 min, 216 min and 31 days, respectively. As for the emulsion on the liquefaction bio-oil, Lijian. [17] employed micro-emulsion technologies to dissolve three kinds of sewage sludge liquefaction bio-oil extracted by different solvents into diesel. The experiments showed that the bio-oils obtained at a higher liquefaction temperature (360 °C) benefited the micro-emulsion.

Table 1. Literature of bio-oil and liquefaction oil in diesel emulsions.

Pyrolysis Bio-Oil Content	Mixing Method	Emulsifier and Co-Surfactant	Stability	Ref.
10–30 wt %	Micro-emulsifier (800–1750 rpm and 5–20 min)	Hypermer B246SF/2234 (Croda International)	3 to 42 days	[14]
25–75 wt %	Variable speed electrical motor (1100 W)	Polymeric surfactants/short chain additives (n-octanol)	3 days	[15]
10 wt %	Ultrasonic (20 kHz; 2 min) and Ultrasonic-mechanical (20 kHz; 2 min; and 5000 rpm; 5 min)	Span80 (sorbitan monooleate)/Tween 80 (Polyoxyethylenesorbitan monolaurate)/Span 85 (sorbitan trioleate)	27 to 31 days	[16]
2–40 wt %	Homogenizer (8800–16,700 rpm)	Span 80 (sorbitan monooleate)/Tween 80 (Polyoxyethylenesorbitan monolaurate)	5 days	[18]
Liquefaction Bio-Oil Content	Mixing Method	Emulsifier or Co-Surfactant	Stability	Ref.
5 wt %	Manual shaking (1 min) and centrifugation (6000 rpm; 20 min)	Span 80 (sorbitan monooleate)	-	[17]
5 to 30 wt %	Vortex mixer (3000 rpm; 2 min)	Atlox4914 (Croda International)/Methanol	7 days	This study

Despite some useful information provided from these studies, it should be underlined that there is still a lack of emulsification information for the liquefaction of bio-oil from food waste. Moreover, the mechanisms of extraction and emulsification are related to the surface tension, viscosity, and polarity between the molecules [15–17]. Thus, this study aimed to investigate the feasibility of the simultaneous emulsification-extraction of the food waste liquefaction bio-oil and diesel with the addition of an emulsifier. This process can combine liquefaction bio-oil extraction and emulsification in one step, which can reduce the experiment cost and time. The details of the proportion of the liquefaction bio-oil in the emulsion after mixing with diesel are also addressed.

2. Materials and Methods

2.1. Materials

The raw liquefaction bio-oil was produced from the hydrothermal liquefaction process of food waste, which was collected from the local food waste recycling field. It included rhizomes and peels of several kinds of vegetables and fruits, which were obtained from livelihood waste. Before the start of the liquefaction process, the size of the food waste (800 g) was reduced using a blender to intensify the reaction surface area. The food waste had a high moisture content (74%) for the liquefaction reaction, thus, dismissing additional water. Then, the food waste slurry was pretreated with a 5 wt % potassium carbonate (K_2CO_3 , alkali catalyst) by heating the mixture to 100 °C for 1 h. Subsequently, the temperature was raised to 320 °C and held for 30 min as the conditions of liquefaction. The process yielded products at three different phases: gas, liquid, and solid. The water vapor in the reactor was removed by cooling to 105 °C under atmospheric pressure. The leftover liquid/solid mixture was heated to 100 °C for 4 h in the oven to remove the excess water content in the mixture. Finally, the remaining residue was considered as the raw liquefaction bio-oil having an average conversion rate of 40 wt %.

The diesel fuel used for experiments was commercially available and sourced from petrol station of China Petroleum Corporation (CPC). In the emulsification-extraction process, the liquefaction oil was dispersed into the diesel, leading to the bulk of the emulsion volume. The emulsifier used in the experiments was Atlox 4914 obtained from Croda International Plc. Methanol (>99 vol %) was used as the co-surfactant. The properties of the bio-oil, diesel, Atlox 4914, and co-surfactant such as density, viscosity, and higher heating value (HHV) are tabulated in Table 2.

Table 2. Properties of the bio-oil, diesel, surfactant, and co-surfactant.

Compound	Density ($kg\ m^{-3}$)	Viscosity (mPa s)	HHV (MJ kg^{-1})
Bio-oil	1100.0	4916	31.41
Diesel	832.0	1.6–5.8	44.80
Atlox 4914	970.0	-	37.64
Methanol	791.0	0.533	20.40

2.2. The Procedure for Preparing the Sample

To determine the minimum volume required for the emulsifier, a fixed quantity of 20 g of diesel was mixed with various amounts of the emulsifier (based on B/E ratio) in a 50 mL centrifuge tube. A vortex mixer (Model: VM-2000, Digisystem Laboratory Instruments Inc., Taipei, Taiwan) was used to mix the diesel and emulsifier for 1 min at a speed of 3000 rpm. The liquefaction bio-oil was in the form of the solid phase at room temperature, so it was preheated to 100 °C in the oven to transform it into a liquid before the emulsification-extraction process. Thereafter, the liquid raw liquefaction bio-oil was added into the diesel-emulsifier mixture and mixed by the vortex mixer for 2 min at a speed of 3000 rpm. Then, the emulsion samples were stored at room temperature (25 °C). Samples, which needed higher-temperature condition, were stored in an oven at 100 °C. The co-surfactant could reduce

the surface tension of bio-oil and enhance the performance of the emulsions [13]. Therefore, further emulsion experiments examined the effect of the adjunction of the co-surfactant by adding one more step in the process of sample preparation. After the emulsions of diesel, emulsifier, and bio-oil were prepared, methanol was added and mixed for 1 min at a speed of 3000 rpm. The detailed process is shown in Figure 1.

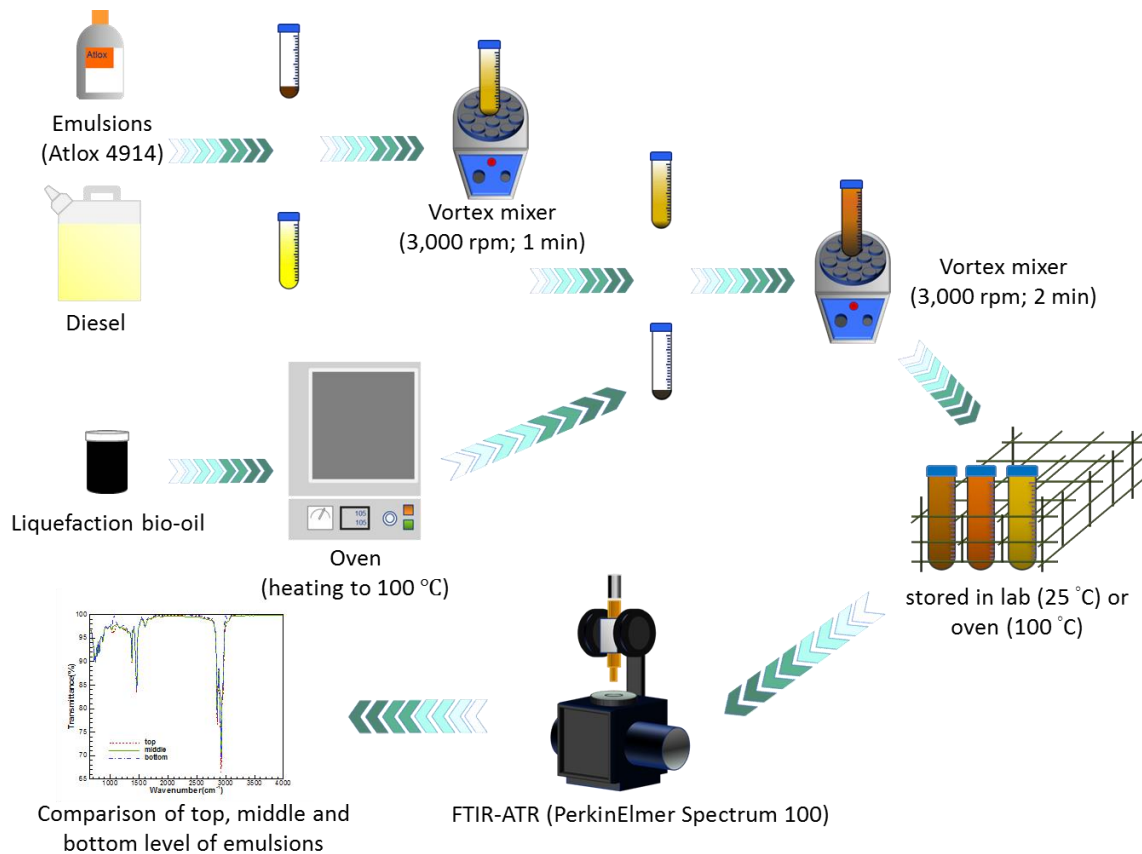


Figure 1. Scheme of emulsion preparation and stability analysis.

2.3. Analytical Methods

There are two directions to investigate the performance of the extraction-emulsification experiment: quantitative and qualitative methods. The quantitative research is to identify the amount of bio-oil dissolved in diesel. The deposited bio-oil is in the solid phase, so it is easy to separate it from the liquid emulsion and weigh the amount. In order to measure the extent of diesel-solvable bio-oil, a parameter called solubility was defined as

$$\text{Solubility (\%)} = \frac{m_{\text{added bio-oil}} - m_{\text{insolvable bio-oil}}}{m_{\text{added bio-oil}}} \times 100\% \quad (1)$$

where $m_{\text{added bio-oil}}$ stands for the added mass of bio-oil and $m_{\text{insolvable bio-oil}}$ denotes the mass of deposited bio-oil in the bottom of the tube.

Qualitative research is to evaluate the emulsion stability. Several methods can be used such as measuring the pH value, moisture content, the viscosity of the emulsion, or droplet size distribution in the emulsion [14,15]. However, there is no standard procedure for examining the long-term stability [15]. In this study, Fourier transform infrared spectroscopy (FTIR) coupled with attenuated total reflection (ATR) was used to examine the stability and homogeneity of the emulsions through the identification of the functional groups present in the emulsions [12,13,19]. In some cases, even though the emulsion seemed to be uniform by visual inspection, the FTIR spectra were different, revealing the

phase separation in the emulsion. For a homogeneous emulsion, the FTIR spectra at different places would overlap. Therefore, the emulsion was divided into three regions, namely, the top, middle, and bottom liquid levels in the sample tube and the three regions were analyzed using FTIR [20].

3. Results and Discussion

3.1. FTIR Spectra of Atlox, Bio-Oil, Diesel, and Methanol

FTIR analysis of the input components (Atlox, food waste liquefaction bio-oil, diesel, and methanol) was performed and the spectra are shown in Figure 2. Diesel, methanol, and Atlox 4914 are commercially available substances with distinct chemical compositions. To identify the stratification of the bio-oil in the emulsions, the chemical composition of the liquefaction bio-oil was studied. From the obtained spectra and functional groups summarized in Table 3, the liquefaction bio-oil contained C-H-(CH₂) (732 cm⁻¹), C-O (1034 cm⁻¹), C-O (1258 cm⁻¹), C-H-(CH₃) (1414 cm⁻¹), CH₃ (2918 cm⁻¹), and polymeric O-H (3320 cm⁻¹) functional groups.

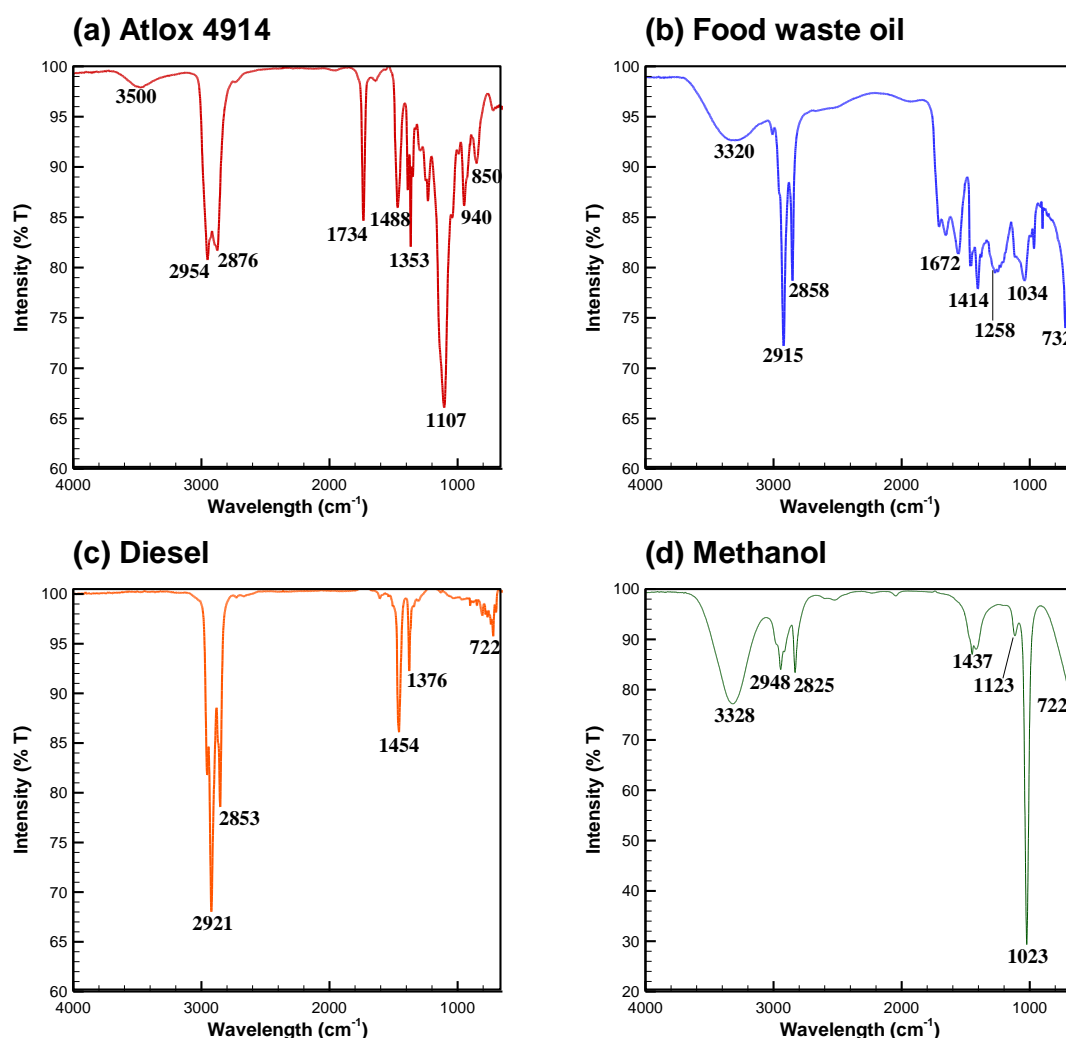


Figure 2. FTIR spectra of (a) Atlox, (b) bio-oil, (c) diesel, and (d) methanol.

Table 3. Main functional groups of emulsifiers, co-surfactants and liquefaction bio-oil.

Wave Number Range (cm ⁻¹)	Functional Group	Mode of Vibration	Reference
3550–3450	Dimetric O-H	Stretching	[21]
3400–3230	Polymetric O-H	Stretching	[21]
2949–2915	Aliphatic C-H	Stretching	[21,22]
2924–2858	Aliphatic C-H	Stretching	[23]
2835–2815	CH ₃ -O-CH ₃ (ethers)	Stretching	[21]
1740–1732	C=O (ester)	Stretching	[23]
1651–1640	C=C (aromatic)	Skeletal	[24]
1652–1579	C-C (ring)	Stretching	[21]
1486–1446	C-H- (CH ₃)/(CH ₂)	Symmetric Asymmetric	[24]
1461–1377	C-H- (CH ₃)	Symmetric	[24]
1290–1211	C-O	Stretching	[21]
1230–1140	C-O (phenol)	Stretching	[21]
1239–1100	C-O-C (ester)	Stretching	[22,23]
1090–1049	C-O	Stretching	[21]
1043–1006	C-O	Stretching	[24]
970–960	C=C-H	Bending	[21]
730–710	C-H-(CH ₂)	Bending	[21]

3.2. Bio-Oil Solubility and Emulsion Stability at B/E Ratio = 1

By mixing various volumetric ratios of bio-oil/emulsifier and observing the emulsion's stability, Figure 3 examines the solubility of the bio-oil in the emulsions over time. In all cases, the B/E ratio was 1 but there were different amounts of bio-oil and emulsifier in the various samples in the emulsions. In all instances, there was an increase in the amount of bio-oil in the sediment over time. The solubility in all cases at 1 h duration was as high as at 70 wt %, except for the case of 1 g of bio-oil with 55.70 wt %. The separation of bio-oil after 1 day was significant in the case of 1 g bio-oil and only 16.61 wt % of it remained dissolved into the emulsion. With an increase in storage time, the sediment of the cases involving 1, 2, and 3 g of bio-oil became pronounced. However, the solubility of the cases involving 4, 5, and 6 g of bio-oil with B/E = 1 remained above 55 wt % on the 7th day, implying that the solubility of the emulsion improved greatly when the amounts of surfactant and bio-oil were increased. On the 7th day, the maximum solubility in the case of 6 g bio-oil with B/E = 1 was 66.48 wt %.

The FTIR spectra at three regions (i.e., top, middle, and bottom liquid levels in the tube) in the emulsion under 2 g bio-oil with B/E = 1 for 1 h to 7 days are displayed in Figure 4. The FTIR spectra appear to almost overlap and coincide with each other, confirming homogeneity and successful emulsification [12].

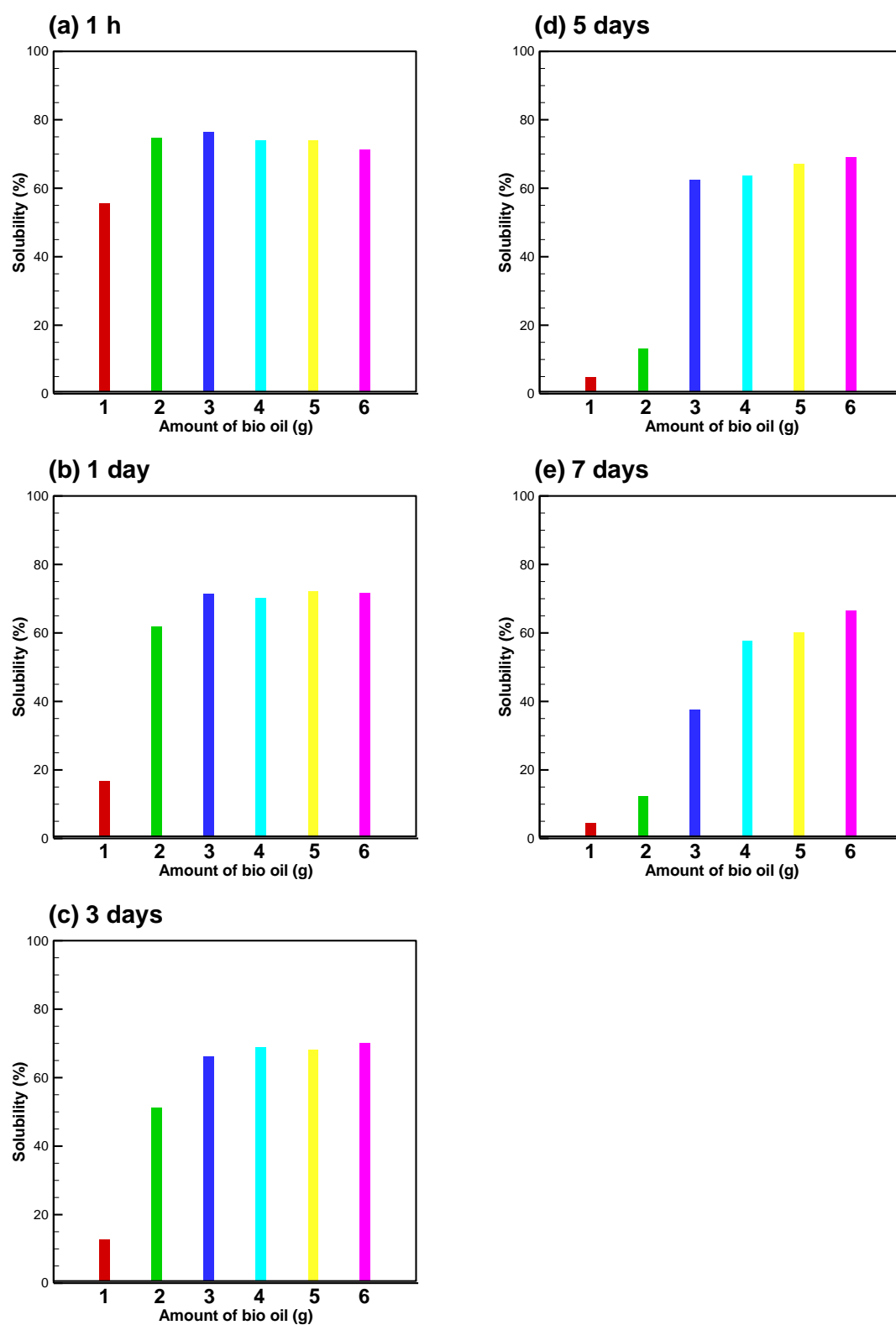


Figure 3. The solubility of the emulsions with different bio-oil amounts ($B/E = 1$) (a) 1 h, (b) 1 day, (c) 3 days, (d) 5 days, and (e) 7 days.

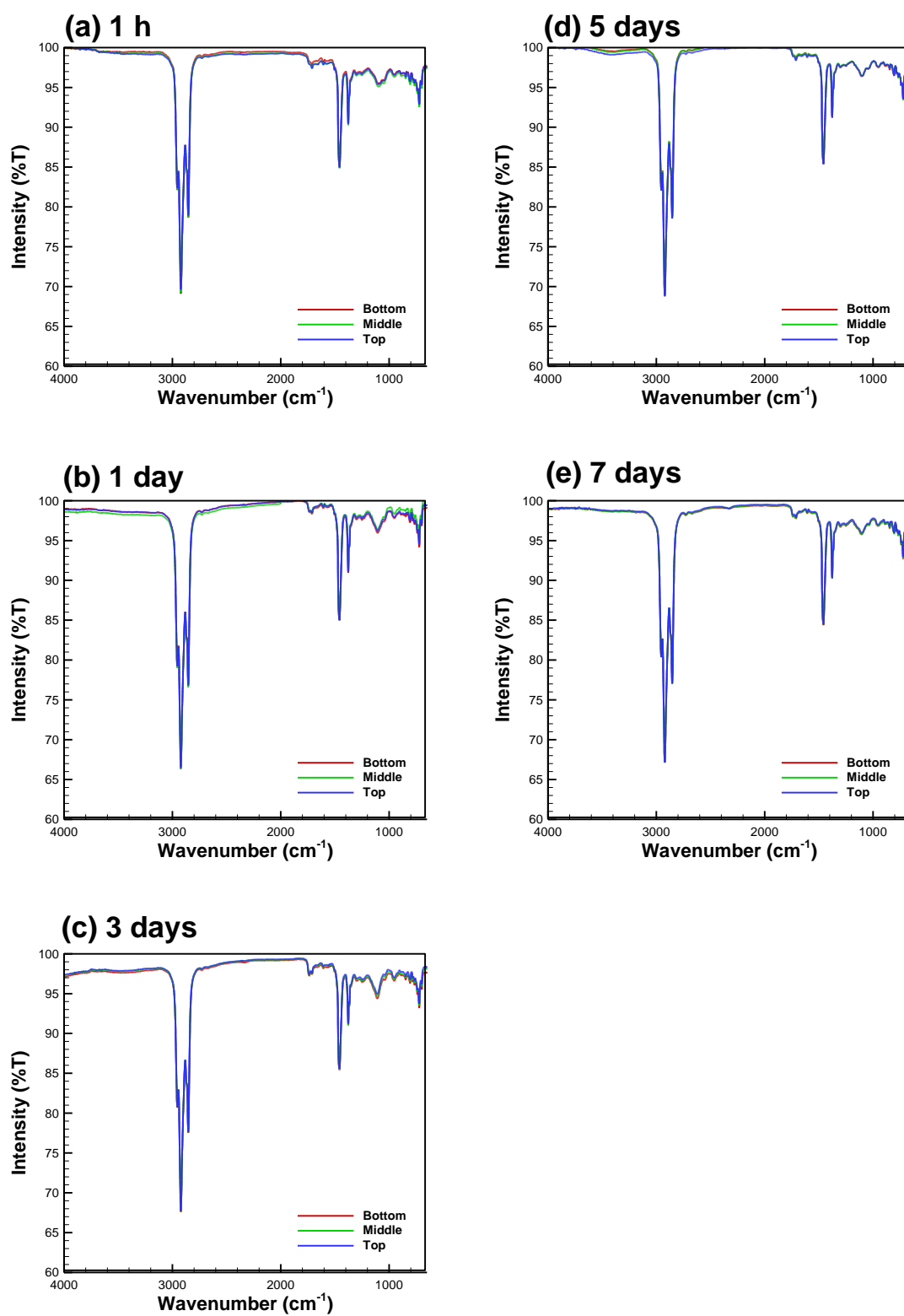


Figure 4. Fourier transform infrared spectroscopy (FTIR) spectra of emulsion (2 g bio-oil, B/E = 1) at three regions for (a) 1 h, (b) 1 day, (c) 3 days, (d) 5 days, and (e) 7 days.

3.3. Bio-Oil Solubility at B/E Ratios between 0.556 and 1

The separation of the emulsion in the cases of a lower amount of bio-oil with $B/E = 1$ was more pronounced compared to those with a higher amount of bio-oil. To study the solubility of the bio-oil, the solubility at the B/E ratios of 0.556–1 was investigated. The surfactant's concentration was one of the most important factors influencing the solubility of emulsions. The bio-oil and diesel contents were fixed at 2 g and 20 g, respectively. An increase in the amount of the emulsifier or a decrease in the B/E ratio caused more bio-oil to dissolve into the emulsion (Figure 5). The solubility at $B/E = 1$ on the 7th day was only 12.02 wt %. When the B/E ratio decreased to 0.556, the solubility increased to 57.81 wt %, implying that a higher emulsifier concentration rendered a higher solubility of the bio-oil into the emulsion.

The FTIR spectra at three regions (i.e., top, middle, and bottom liquid levels in the tube) in the emulsion with 2 g bio-oil and $B/E = 1$ for 7 days are displayed in Figure 4e, showing the stability of the emulsion. The FTIR spectra almost overlap and coincide with each other, confirming homogeneity and higher stability of the emulsion.

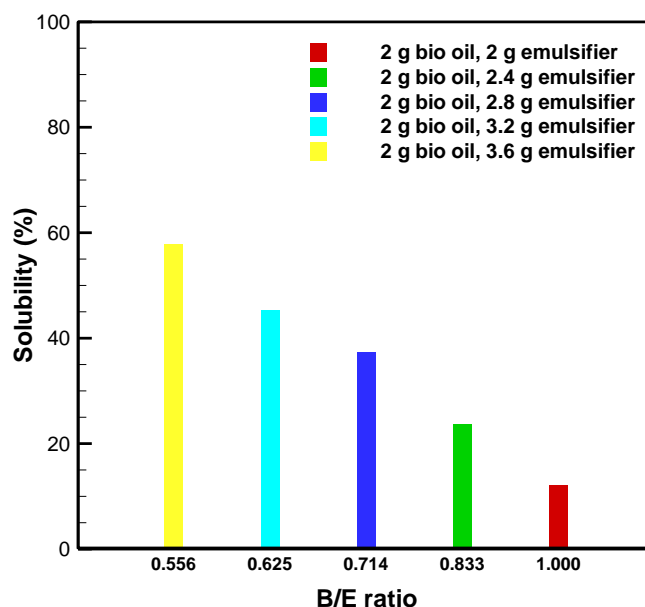


Figure 5. The solubility of the emulsions with different B/E ratios (2 g bio-oil) after 7 days.

3.4. Temperature Effect

The fluidity of the bio-oil was low at room temperature. To increase the fluidity of the emulsion during storage, the emulsion was heated at 100 °C in the oven for 7 days. In general, both the viscosity and interfacial tension decreased with increasing temperature, making the emulsification easier [15]. This resulted in an increased degree of solubility at 100 °C compared to that at room temperature. Compared to the storage of the emulsion at room temperature, the solubility in the case of 2 g bio-oil with $B/E = 1$ at 100 °C promoted the solubility to 59.86 wt % from 12.02 wt % at room temperature (Figure 6). This clearly indicates that the effect of the bio-oil temperature at $B/E = 1$ played an important role in improving its solubility. However, the impact of the storage temperature in the case of $B/E = 0.556$ was not of the same magnitude; the solubility being merely intensified from 57.81 wt % at room temperature to 63.45 wt % at 100 °C, accounting for an increase of 6.64 wt %. Overall, the temperature effect on the solubility of bio-oil increased when the B/E ratio increased.

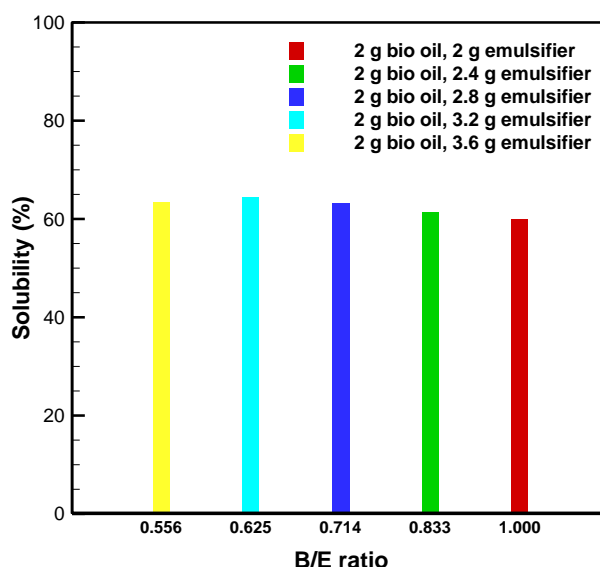


Figure 6. The solubility of bio-oil at different B/E ratios (2 g of bio-oil, fixed amount) at 100 °C for 7 days.

The FTIR spectra at three regions (i.e., top, middle, and bottom liquid levels in the tube) in the emulsion at the storage temperature of 100 °C with 2 g bio-oil and B/E = 1 for 7 days are displayed in Figure 7. The FTIR spectra also almost overlap and coincide with each other, showing the homogeneity and high stability of the emulsion again.

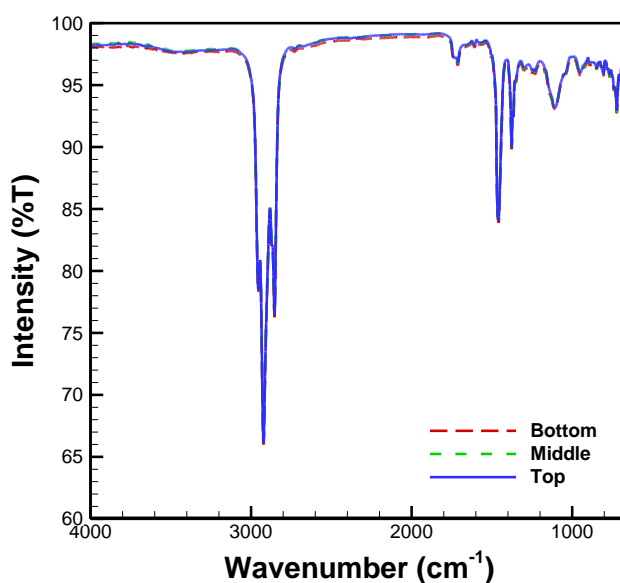


Figure 7. FTIR spectra of the emulsion with 2 g bio-oil and B/E = 1 under 100 °C at 7 days.

3.5. Co-Surfactant (Methanol) Effect

In general, the amount of the emulsifier must be increased to improve the bio-oil usage in the emulsion. The co-surfactants are surface-active compounds that can lower the surface tension of bio-oil or the dispersed phase, making them suitable for emulsification [25]. In this study, methanol was used as the co-surfactant, resulting from its influence on increasing the emulsion's stability [13]. In each experimental run, 1 g of methanol was loaded into the emulsions. The bio-oil solubility in the case of 2 g bio-oil with B/E = 1 at a duration of 7 days increased to 46.66 wt % (Figure 8), as opposed to 12.02 wt % without the addition of methanol. This evidence suggests that methanol can significantly

facilitate the emulsification performance. The impact of the co-surfactant in the case of $B/E = 0.556$ was not pronounced. The solubility with the co-surfactant (70.96 wt %) was higher than that without methanol (57.81 wt %) by 13.15 wt %. The co-surfactants effect on the solubility of the bio-oil increased when the B/E ratio increased.

The FTIR spectra at the three regions (i.e., top, middle, and bottom liquid levels in the tube) in the emulsion after adding 1 g methanol with 2 g bio-oil and $B/E = 1$ for 7 days are displayed in Figure 9. The FTIR spectra also show the high homogeneity and stability of the emulsion. By comparing the influences of the two factors (storage temperature and co-surfactant) on the solubility of the bio-oil from $B/E = 0.556$ to 1 (Figures 6 and 8), when the B/E ratio was between 0.714 and 1, the impact of the storage temperature on the solubility was beyond the co-surfactant. On the other hand, when the B/E ratio ranged from 0.556 to 0.625, a converse behavior was observed.

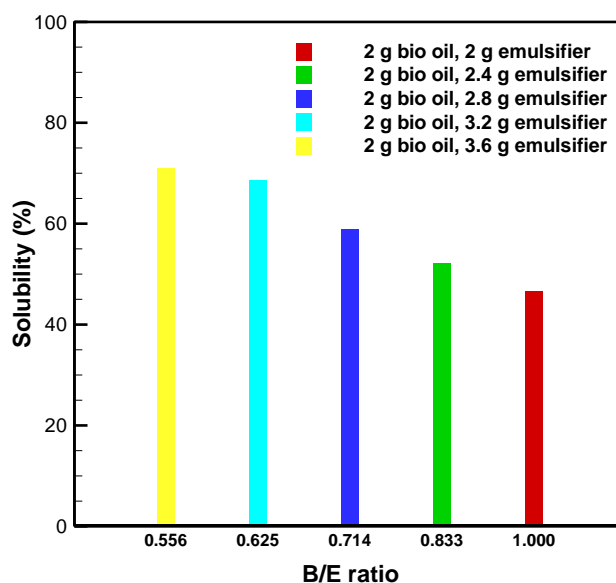


Figure 8. The solubility of bio-oil at different B/E ratios with 1 g methanol.

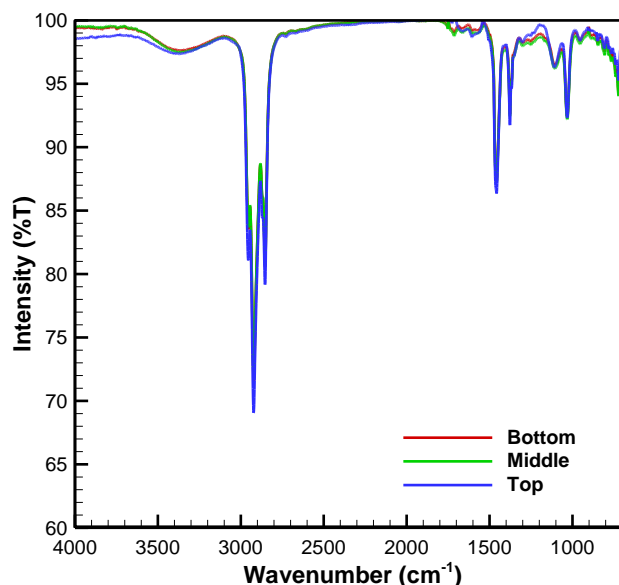


Figure 9. FTIR spectra of the emulsion with 2 g bio-oil, $B/E = 1$, and 1 g methanol for 7 days.

4. Conclusions

The simultaneous emulsification-extraction process of liquefaction bio-oil and diesel with the aid of a commercial emulsifier (Atlox 4914) and co-surfactant (methanol) have been explored where 20 g of diesel was used. When the bio-oil amount increased from 1 g to 6 g with the fixed B/E ratio of 1, its solubility increased from 55.70 wt % to 71.35 wt %. However, the solubility with 1 g bio-oil ($B/E = 1$) decreased from 55.70 wt % to 4.41 wt % after the 7 days storage and decreased from 71.35 wt % to 66.48 wt % with 6 g bio-oil ($B/E = 1$). This suggests that performing emulsification at a larger scale may facilitate the solubility. When a higher amount of emulsifier was used with the B/E ratio decreasing from 1 to 0.556, the bio-oil solubility also increased from 12.02 wt % to 57.81 wt % after a 7-day duration. To reduce the viscosity of the emulsions, the emulsions with B/E ratios ranging from 0.556 to 1 were stored in an oven at 100 °C for 7 days. The storage temperature of 100 °C had more impact on the solubility at higher B/E ratios, especially for the case of $B/E = 1$, which enhanced the solubility by a factor of 47.84% as compared to room temperature (25 °C). Moreover, the addition of a co-surfactant (methanol) at $B/E = 1$ at room temperature intensified the bio-oil solubility from 12.02 wt % (without addition) to 46.66 wt %. When the liquefaction bio-oil was extracted and emulsified in the emulsions, their higher stability after 7-day storage was achieved from the FTIR analysis. The results of the study indicate that hydrothermal liquefaction can be applied to waste-to-fuel technology and the liquefaction bio-oil from food waste has a high potential to replace the diesel fuel used in industrial furnaces and boilers.

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Conflicts of Interest: The authors declare no conflicts of interest

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