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Nanoscale Devices

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Microwave-Assisted Solvothermal Liquefaction of Kenaf Stalks for Bio-oil Production

Anita Ramli, Lai Weng Kin, Nur Akila Syakida Idayu Khairul Anuar, Normawati M. Yunus, and Nurul Jannah Abd Rahman

Abstract In this study, microwave-assisted solvothermal liquefaction was investigated to produce bio-oil from Kenaf stalks. Optimization of process parameters was performed using concentrated sulfuric acid as catalyst at biomass to solvent ratio of 1:10, catalyst loading of 10% and microwave power of 300 W with constant stirring at 200 rpm. The maximum bio-oil yield of 87% was achieved at temperature of 180 °C for duration of 20 min, in which ethanol was used as solvent. Different types of catalysts were also tested to produce bio-oil at the optimum process conditions. It was found that synthesized ionic liquid [BMIM][Cl] catalyst could give the highest quality of bio-oil in terms of heating value (26.55 MJ/kg) with considerable bio-oil yield.

Keywords Bio-oil · *Kenaf stalks* · Solvothermal liquefaction · Catalyst · Ionic liquid · Microwave

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1 Introduction

The rapid depletion of fossil fuels, as well as the aftermath of fossil fuel usage have led researchers around the world to search for a promising renewable energy. Bio-oil derived from plants and animals is one of the potential alternatives, which has gained widespread recognition around the globe. Bio-oil is a mixture of various hydrocarbons compounds, usually containing acids, alcohols, aldehydes, esters, ketones, phenols and lignin-derived oligomers. Bio-oil is commonly used as substitute for fuel in engines, boilers and turbines. According to Mohan et al. [1], bio-oils emits negligible amount of SO_x and relatively lower amount of NO_x, as compared to the fossil fuels. In addition, bio-oil can also be used for production of adhesives, surfactants, pharmaceuticals and bio-degradable polymers.

In the recent years, various technologies have been developed to produce liquid bio-oil products from lignocellulosic biomass. These biomass conversion technologies are divided into two main categories, biochemical conversion and thermochemical conversion [2]. As compared to biochemical conversion, thermochemical conversion is processed at relatively higher temperature. Furthermore, the process is usually performed under pressurized condition and in inert atmosphere. The thermochemical conversion can be further classified into different methods namely, pyrolysis, liquefaction, gasification and combustion. Among the technologies, the solvothermal liquefaction seems to be a more promising method due to its low operating temperature (523–647 K) and pressure (40–220 bar) as compared to the pyrolysis method [3].

The liquefaction of biomass to produce bio-oil often contained many shortcomings, as compared to the conventional fossil fuels, such as high oxygen content, high acid value and low heating value [3]. Therefore, the production of bio-oil requires the process parameters to be optimized to obtain maximum yield and high quality of final product. It is interesting to note that, the yield and the quality of bio-oil synthesized via microwave irradiation have not been investigated thoroughly. The microwave irradiation offers the advantage of extremely rapid heating throughout the volume of the reaction mixture because it penetrates and produces a volumetrically distributed heat source.

In this study, the optimization of process parameters for liquefaction of Kenaf stalks was investigated using solvothermal liquefaction process under the irradiation of microwave to maximize the energy efficiency, as well as reducing the process duration. The optimum reaction conditions where a maximum bio-oil yield was achieved were then utilised to further study the effect of catalyst types on liquefaction yield and bio-oil quality. The catalysts used include homogeneous sulfuric acid, heterogeneous ZSM-5 and ionic liquid [BMIM][Cl] catalysts. The synthesis and characterization of the ionic liquid have been covered in this study.

2 Methodology

2.1 Synthesis of Ionic Liquid [BMIM][Cl]

20 g of 1-methylimidazole was transferred into a 3-neck round-bottom flask. 22.5 g of 1-chlorobutane was added into the round-bottom flask. The mixture was then heated to 80 °C under reflux for 48 h in a silicon oil bath. After the reaction, the sample was cooled to room temperature and washed five times with ethyl acetate. The remaining ethyl acetate in the solution was further removed using rotary evaporator at 50 °C under vacuum for 5 h.

2.2 Characterization of Ionic Liquid [BMIM][Cl]

The molecular structure and conformation of the synthesized ionic liquid of 1-Butyl-3-methylimidazolium Chloride ([BMIM][Cl]) was analysed using 1H-NMR spectrometer (Bruker Aviance III NMR) at 500 MHz. 25 mg of the ionic liquid was mixed with 0.65 mL of dimethyl sulfoxide (DMSO) as solvent and transferred into NMR tube for the analysis.

The moisture content was measured by titration (Metler Toledo V30 Karl Fischer Titrator). 0.3 g of the ionic liquid was added into a vial and tightly sealed. The vial was then heated up at 110 °C for 10 min. A blank (empty vial) was also prepared. The result obtained for the [BMIM][Cl] sample was in relevance of the blank sample.

2.3 Optimization of Solvothermal Liquefaction Process

Kenaf stalks were purchased from Lembaga Kenaf dan Tembakau Negara (LKTN), Malaysia. The Kenaf stalks were grinded and sieved to <0.4 mm particle size. The solvothermal liquefaction process was carried out using Milestone Advanced Microwave Synthesis (MA143). The liquefaction process was started off by loading Kenaf stalks and ethanol at ratio of 1:10. Then, 0.2 g of concentrated sulfuric acid (H_2SO_4) catalyst was added into vessel. A magnetic bar stirrer was then placed into the vessel. The vessel was tightly capped and placed into microwave.

The liquefaction process was subjected to different reaction temperature (140–200 °C), duration (10–25 min) and type of solvent [ethanol, water and ethanol–water (1:1)]. After the reaction, the products were allowed to cool down to room temperature. Then, the liquefied products were dissolved in 100 mL of solvent, under constant stirring for 4 h at 400 rpm. The products were then filtered through Whatman 1442–110 filter paper using a vacuum filtration pump, to separate the liquid and solid residues. The solid residues were further washed with solvent to ensure maximum liquid products has been extracted. The liquid products were then rotary

evaporated at 50 °C under reduced pressure of 120 mbar to remove the solvent. The final product, which was a black viscous liquid was obtained.

2.4 Effect of Catalysts

The solvothermal liquefaction process was then carried out at optimum reaction condition using different type of catalysts of commercial heterogeneous Zeolite Socony Mobil-5 (ZSM-5; Zeolyst), and synthesized ionic liquid [BMIM][Cl].

2.5 Qualitative and Quantitative Analysis

The synthesized bio-oil was qualitatively analyzed using GC-MS (Shimadzu GCMS-QP2020) with a capillary polar wax column (30.00 m × 0.25 mm × 0.25 μm). The pressure was 83.50 kPa. The injection volume had the total flow of 34.50 mL/min. The oven temperature was fixed at 40 °C and was hold for 1 min, before ramping it up to 250 °C (6 °C/min) and hold for 24 min. The split mode with the ratio of 20:1 was employed, while the injection temperature was set at 250 °C. Helium gas was used as the carrier gas with the flow rate of 1.50 mL/min.

The carbon, hydrogen, nitrogen and sulfur contents were quantitatively measured using CHNS analyser (CHNS Elementar Variomicro Cube). The elemental composition obtained from the CHNS were used to compute the higher heating values (HHV) using Dulong formula:

$$\mathrm{HHV\ (MJ/kg)} = 0.3383\mathrm{ZC} + 1.422(\mathrm{ZH} - \mathrm{ZO}/8) \tag{1}$$

where ZC, ZH and ZO represent the weight percentage of carbon, hydrogen and oxygen content present in the bio-oil, respectively.

3 Result

3.1 Characterization of Catalyst

Figure 1 shows the NMR spectrum of synthesized ionic liquid [BMIM][Cl]. As can be seen, the result obtained was as follows: 1H-NMR (500 MHz, DMSO, ppm): δ 9.500 (s, 0H), 7.877–7.800 (d, 1H), 4.216–4.187 (t, 2H), 3.880 (s, 0H), 1.793–1.734 (p, 4H), 1.283–1.208 (m, 5H), 0.901 (t, 2H).

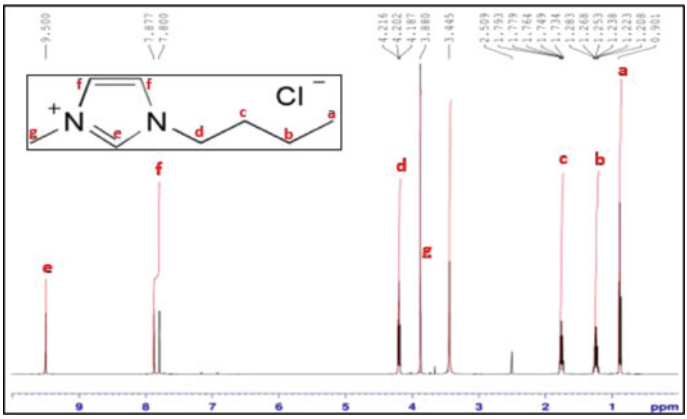

Fig. 1 ^{1}H-NMR spectrum of synthesized [BMIM][Cl]

From the result, there were few foreign peaks appear in the spectrum. At δ 3.445, the signal refers to the moisture present in the sample. This suggested that the synthesized [BMIM][Cl] may contain some moisture as impurity. In addition, another singlet (δ 2.509) was noticed which could be due to the impurity of DMSO solvent.

The moisture content of the ionic liquid [BMIM][Cl] was determined to be 2.505%. The low moisture content obtained is highly desirable as water acts as an impurity which may affect the results.

3.2 Optimization Study

Figure 2 shows the effect of temperature on bio-oil yield using ethanol as solvent for 20 min and catalysed by H_2SO_4. It was observed that the bio-oil yield increased as the temperature was increased from 140 to 180 °C. According to Reddy et al. [4], the higher the temperature, the easier the bonds were broken into fragments and subsequently, undergo hydrolysis and repolymerization reactions to form bio-oil. Also, at high temperature, the solid products depolymerized while the gas products aggregated to bio-oils [5] . However, when the reaction temperature was further increased to 200 °C, the bio-oil yield decreased significantly. This signified that the liquid bio-oil was converted into gaseous products at temperature above 180 °C, resulting in loss of bio-oil yield. It was also reported that higher temperature result in higher rate of reaction and favours free radical reaction which leads to formation of gaseous products [6].

Figure 3 shows the effect of liquefaction time on bio-oil yield using ethanol as solvent at optimum temperature (180 °C) and catalysed by H_2SO_4. From the result, it was observed that the bio-oil yield increases at prolonged reaction time, from 10 to 20 min. This phenomenon was occurred due to the competition between the

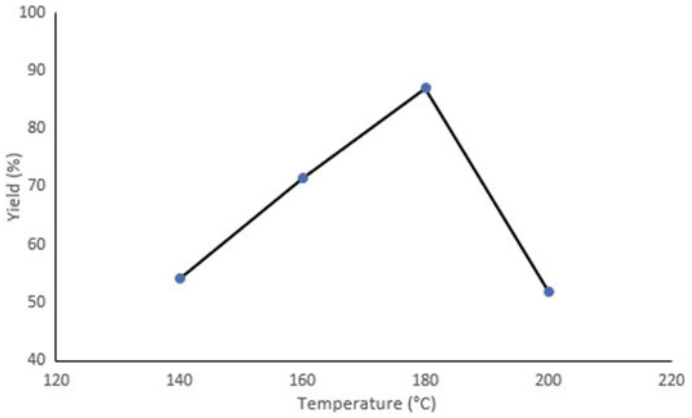

Fig. 2 The effect of temperature on bio-oil yield

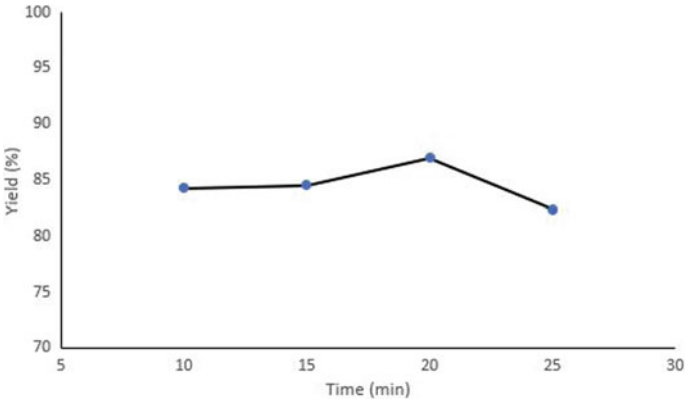

Fig. 3 The effect of liquefaction time on bio-oil yield

degradation of feedstock and repolymerization of bio-oil reactions during liquefaction process. At shorter liquefaction time, the degradation outweighs the repolymerization reaction. At higher liquefaction time, the repolymerization of bio-oil fragments dominates the liquefaction process, therefore producing liquified bio-oil [2]. However, when the liquefaction time was further increased to 25 min, significant decrease in bio-oil yield was observed.

Figure 4 shows the effect of different solvent system on bio-oil yield at optimum temperature (180 °C) for 20 min and catalysed by H_2SO_4. It was found that ethanol solvent resulted in the highest liquefaction yield, followed by ethanol-water and water. The high liquefaction yield from ethanol could be due to the present of hydrogen donor to hydrogenate the biomass fragments during liquefaction process [7]. This inhibits the condensation, cyclization or repolymerization of free radicals,

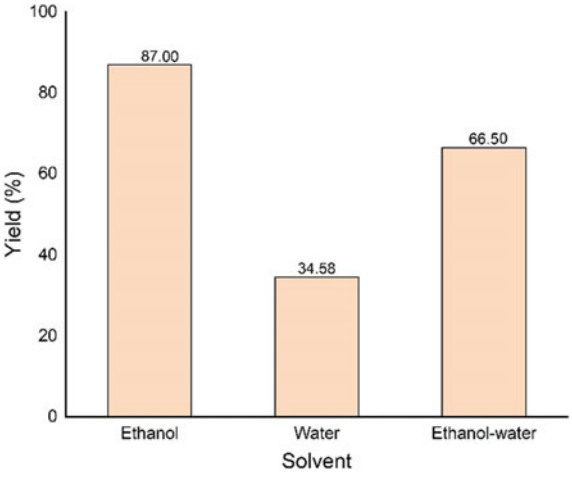

Fig. 4 The effect of solvent system on bio-oil yield

therefore reducing the formation of char. Ethanol also can react with acidic components in the bio-oil to produce esters [8]. Similarly, Liu and Zhang [9] also compared the solvent efficiency on liquefaction yield between ethanol, acetone and water and it was found that the ethanol solvent produced the highest liquefaction yield.

3.3 Effect of Catalysts

In this part, different types of catalysts were tested to produce bio-oil at optimum reaction condition of 180 °C for 20 min and ethanol as solvent system, as shown in Fig. 5. Maximum liquefaction yield of 87% was achieved using H_2SO_4 catalyst followed by the synthesised ionic liquid [BMIM][Cl] catalyst (43.17%). However, significant decrease in liquefaction yield was observed when heterogeneous catalyst of ZSM-5 was employed in the liquefaction process. According to Behrendt et al. [10], homogeneous catalysts in general are more active as compared to the heterogeneous catalysts, resulting in higher conversion of the biomass to bio-oil. In addition, H_2SO_4 is known for its high acidity and oxidization properties, allowing the catalyst to break down the molecules of the biomass, promoting the depolymerization reaction which results in higher yield of bio-oil [2]. On the other hand, Li et al. [11] reported that addition of ionic liquid will influence the solvent polarity and enhance the interaction between the solvent and biomass which resulted in increasing liquefaction yield. Also, ionic liquid could disrupt hydrogen bond structure and lead to the increase in number of active intermediates. These intermediates will be dissolved and stabilized in the solvent, which result in high bio-oil yield.

Table 1 shows the elemental composition of bio-oil produced at optimum reaction condition using different types of catalysts along with their heating value. It has been observed that the type of catalysts heavily influenced the elemental compositions and

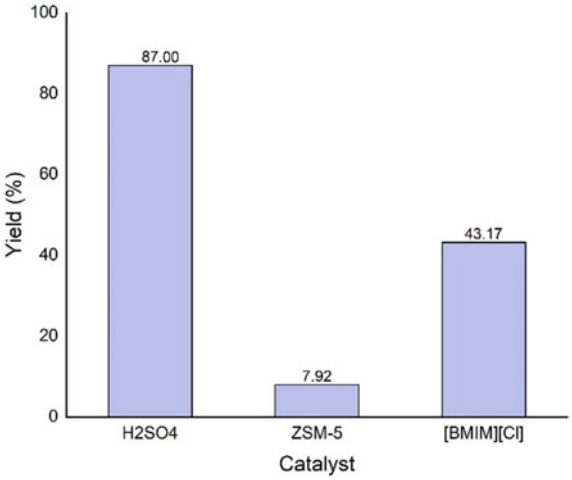

Fig. 5 The effect of catalyst types on bio-oil yield

Table 1 Elemental composition and heating value of bio-oil using different types of catalysts

Catalyst	Elemental compositions (wt%)					HHV (mJ/kg)
	C	H	N	O	S	
H_2SO_4	42.3	6.91	0.09	50.71	0.00	15.11
ZSM-5	52.05	8.28	0.53	38.59	0.45	22.50
[BMIM][Cl]	53.41	9.39	9.56	27.35	0.30	26.55

the heating value. The homogeneous H_2SO_4 catalyst resulted in the lowest heating value as compared to the other catalysts. The low heating value could be due to the reduced amount of carbon content contained in the bio-oil. On the other hand, when the liquefaction process was catalysed by ionic liquid [BMIM][Cl], the carbon content and the heating value of the bio-oil increased significantly. The heterogeneous ZSM-5 catalyst resulted in better quality of bio-oil in terms of heating value as compared to the H_2SO_4 catalyst. However, as previously mentioned, the yield of the ZSM-5 catalysed bio-oil was lower compared to the use of H_2SO_4 and [BMIM][Cl] as the catalyst.

From GC-MS analysis, it has been found that different types of catalysts resulted in the different compositions of bio-oil. The H_2SO_4-catalysed bio-oil produced mainly esters (41.43%), followed by ketones (16.18%), C5 sugars and its derivatives (14.52%), aldehydes (4.37%), nitrogen-containing compound (2.29%) and alkanes (1.76%). As for the ZSM-5-catalysed bio-oil, it produced a large fraction of ethers (33.42%), mostly due to the large yield of 1,2-dimethoxy-4-(1-methoxy-1-propenyl) benzene. It also produced a considerable number of phenolic compounds (13.82%), carboxylic acids (7.28%), aldehydes (5.83%) and nitrogen-containing compounds

(3.93%). As for the [BMIM][Cl]-catalysed bio-oil, it mainly consists of nitrogen-containing compounds (38.33%), mostly due to the formation of imidazole derivatives. This was further complemented with the high nitrogen content obtained from CHNS result, as shown in Table 1. The bio-oil also contained ethers (24.98%), ketones (6.6%), phenolic compounds (4.67%), aldehydes (4.18%), esters (3.29%) and carboxylic acids (2.3%).

4 Conclusion

Kenaf stalks have been successfully liquefied via solvothermal liquefaction method to produce bio-oil. The optimum process condition was determined under microwave irradiation using H_2SO_4 as the catalyst. At the optimum process condition, the synthesized ionic liquid [BMIM][Cl] was found to give considerable bio-oil yield with higher amount of carbon content and heating value as compared to H_2SO_4 and ZSM-5 catalysts.

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