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To cite this article: A N Awang *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **932** 012002

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# Torrefaction of *Leucaena Leucocephala* in a fixed-bed reactor

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**Abstract.** Raw biomass contained low energy and high moisture content which contributed to its limitation as energy source. However, the biomass properties can be improved via torrefaction method. Torrefaction is a biomass pretreatment method that is performed between 200- 300 °C using slow heating rate in an inert condition. In this study, *Leucaena Leucocephala* (*Leucaena*) was torrefied in a fixed-bed reactor at 20 °C/min from 200- 300 °C at holding times of 20 min. The changes in the elemental and chemical properties of *Leucaena* were studied using a few methods of analysis such as proximate, elemental analysis and fourier transform infrared (FTIR) spectrophotometer. When the torrefaction temperature was increased from 200 to 300 °C, the oxygen to carbon (O/C) and hydrogen to carbon (H/C) ratio of torrefied *Leucaena* reduced significantly. From FTIR, the broad peak at wavenumber of approximately 3400 cm<sup>-1</sup> due to the presence of OH group reduced in its intensity upon increasing torrefaction temperature from 200 to 300 °C which indicated that the degree of hydrophobicity of torrefied *Leucaena* had improved.

## 1. Introduction

Malaysia is a tropical country that is blessed with abundance of biomass. Malaysia has generated approximately 168 million tons of biomass annually which included palm wastes, rice husk, sugarcane wastes, coconut fibers as well as municipal [1]. The biomass has been converted into several valuable products in various applications such as in furniture, building and renewable energy industries. Biomass energy had contributed almost 16% of energy consumption in Malaysia [2]. The energy from biomass is considered as one of the largest renewable, potentially sustainable and environmental friendly that eventually overcome the problems associated with the consumption of fossil fuel [3].

Although the raw biomass is usually used for energy conversion in power plant, however it is least preferable. Raw biomass contains low energy density and high moisture content, that requires higher loading to produce same amount of energy compare with fossil fuel [3, 4]. The higher moisture content reduced the combustion temperature, thus decreased the thermal efficiency of biomass [5]. Improving the chemical properties of the biomass itself is an important route to solve these problems prior for energy purpose. One of the methods to improve the biomass properties is through torrefaction process.

Torrefaction is a thermochemical pretreatment method whereby biomass is subjected to thermal heating in an inert condition between temperature range of 200 to 300 °C at low heating rate and long residence time [6, 7]. Torrefaction reduces the water content in biomass and increases the calorific value of biomass [8].

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Biomass has been reported to undergo chemical transformations during torrefaction due to the chemical and thermal reactivity that occurred [9]. Therefore, the evaluation of torrefied biomass is essential.

Sulaiman et al. (2016) performed torrefaction of empty fruit bunch (EFB) in a vertical tubular reactor at several temperatures of 220, 260 and 300 °C and the reaction times were 30, 60 and 90 mins [10]. They reported that upon increasing torrefaction temperature, the calorific value and carbon content of torrefied EFB had increased. Toscano et al. (2015) conducted torrefaction of tomato peels in a heated bench-top reactor towards several torrefaction temperatures from 214- 316 °C within 30-60 mins [11]. They reported that torrefaction process on tomato peels had produced solid fuel with higher carbon and lower oxygen than raw material. Na et al. (2015) studied the torrefaction process and its effect on the chemical and physical properties of yellow poplar by varying torrefaction temperatures and times [12]. They stated that torrefaction reduced the moisture uptake capacity as well as increased the calorific value when the temperature was increased. Ibrahim et al. (2013) has also been studied the effect of process parameters on the physicochemical properties of torrefied biomass [13]. They suggested that the effect of torrefaction temperatures was greater than residence times. They also reported that torrefaction decrease the R-OH group in biomass while C=O group increased. Matali et al. (2016) had studied torrefaction process using non-woody and woody biomass in a quartz tube reactor [14]. They reported that as temperature increased, the energy reduced due to the decreasing of solid mass yield, thus improving higher heating value compared to raw biomass. Chen et al. (2015) performed torrefaction of cotton stalk in tubular furnace by varying torrefaction temperature [15]. They stated that higher temperature contributed to high hydrophobicity of torrefied cotton stalk in which the oxygen containing functional groups were removed. The carbon content also increased greatly with the heating value. Zheng et al. (2013) conducted torrefaction of corncobs in an auger reactor at several temperatures and residence times [16]. They studied the effect of torrefaction on the behaviour of bio-oil from pyrolysis. They reported that the bio-oil using torrefied sample in pyrolysis contained lower acetic acid compared to the bio-oil using raw biomass. Hence, it showed that the torrefaction method successfully reduce the amount of acetic acid in the biomass.

Many reported studies on biomass torrefaction focused on agricultural wastes such as EFB [17, 18], mesocarp fibre [19] and rice straw [20]. However, the information regarding torrefaction of energy crops such as *Leucaena* with regard to the changes in torrefied sample in terms of its chemical properties and comprehensive evaluation on the existence of functional groups by FTIR spectroscopy is very scarce. *Leucaena* is of particular interest since it is a fast growing tree that can be planted in tropics condition such as in Malaysia, Thailand and Indonesia. Therefore, this paper is aimed towards reporting the effect of torrefaction temperatures on the chemical and physical properties of *Leucaena* as well determination of functional groups by FTIR technique on raw and torrefied *Leucaena* samples.

## 2. Materials and methods

### 2.1. Sample preparation

*Leucaena* stem was obtained from *Leucaena* tree located in Padang Besar, Perlis, Malaysia. The stem was chopped manually prior to size reduction. The sample was washed using tap water to remove any impurities and dried at 80 °C in an oven for 24 hours. The dried sample was ground using grinder and sieved between sizes of 1 mm- 1.7 mm. Prior to the experiment, the sample was kept in air tight container.

### 2.2. Torrefaction process

Torrefaction of dried sample was performed in a fixed-bed reactor. The dried sample of 10 g was fed in the reactor tube and was properly sealed with gasket at the top and bottom tube. Before starting the experiment, the nitrogen gas was purged into the reactor for 10 mins to ensure inert environment. The flow rate of gas was fixed at 150 ml/min. Torrefaction was conducted at different temperatures of 200,

220, 240, 260, 280 and 300 °C for 20 mins. The heating rate of 20 °C/min was kept constant for each runs.

### 2.3. Proximate analysis

The proximate analysis of raw *Leucaena* sample were conducted according to American Society for Testing and Materials (ASTM) method. The moisture content (MC), volatile matter (VM) and ash content (ASH) were determined according the ASTM E871-82, E872-82 and D1102-84 respectively, while the fixed carbon content (FC) was estimated by difference as according to Equation 1.

The proximate analysis of torrefied sample were performed in accordance to ASTM D1762-84 with slight modification, while fixed carbon was calculated by difference as Equation 1. Each analysis was conducted in triplicate.

$$FC = 100 - MC - VM - ASH \quad (1)$$

### 2.4. Calculation for carbon, hydrogen and oxygen content

The content of carbon (C), hydrogen (H) and oxygen (O) were determined by using a correlation between ultimate analysis and proximate analysis proposed by [21]. The correlations were listed below, where VM, FC and ASH corresponded to volatile matter, fixed carbon and ash content, respectively.

$$C = -35.9972 + 0.7698VM + 1.3269FC + 0.3250ASH \quad (2)$$

$$H = 55.3678 - 0.4830VM - 0.5319FC - 0.5600ASH \quad (3)$$

$$O = 223.6805 - 1.7226VM - 2.2296FC - 2.2463ASH \quad (4)$$

### 2.5. FTIR analysis

The infrared spectra of raw and torrefied sample were carried out using Perkin Elmer Frontier FTIR spectrophotometer at wave numbers of 4000 to 600 cm<sup>-1</sup>. The samples were prepared using potassium bromide (KBr) at 3:1 ratio between KBr to sample.

## 3. Results and discussion

### 3.1. Proximate analysis

The results of the proximate analysis of raw and torrefied *Leucaena* are shown in Table 1. It can be observed that the torrefaction pretreatment has successfully reduced the moisture and volatile matter contents. Moisture is released during torrefaction due to the destruction of hydroxide radical, thus making the biomass loses its capacity to form hydrogen bond with water. Moreover, hemicellulose underwent decomposition between 200-300 °C, hence leading to the devolatilization reaction [22]. Meanwhile, the ash content reduces from torrefaction temperature of 200-240 °C and increases from torrefaction temperature of 260 °C to 300 °C. Ash content is attributed to the inorganic residue (minerals) remaining after organic material undergo complete oxidation [23]. The inconsistent trend of the ash content of torrefied *Leucaena* is in good agreement with [24] which mentioned that this a typical trend for woody biomass. They also concluded that the reason for inconsistent trend because of the soft texture of biomass at lower temperature (200-240 °C) while at higher temperature (260-280 °C) the texture became harder due to the free water removal. When comparing between torrefied *Leucaena* itself, the increasing trend are observed for each increment in torrefaction temperature due to the increasing of mass loss occurs during torrefaction [25].

Furthermore, the volatile matter content decreases from 200 to 300 °C for at the expense of increasing fixed carbon content. This is due to the removal of volatile during torrefaction that increase when temperature is increased. The fixed carbon content shows an increasing trend when the torrefaction temperature is increased from 200 °C to 300 °C. The fixed carbon content increase when increasing torrefaction temperature due to the cell wall structure is destroyed [26].

Torrefaction of *Leucaena* at 300 °C has produced solid material with a volatile matter content comparable to lignite with a higher fixed carbon content. Anthracite has the highest fixed carbon content of 84.40 wt% which indicates that it is the most prominent solid fuel.

**Table 1.** Proximate analysis of raw and torrefied *Leucaena*.

Torrefaction temperature (°C)	Moisture content (wt %)	Ash content (wt %)	Volatile matter content (wt %)	Fixed carbon content (wt %)
Raw	5.35	1.15	90.21	3.29
200	4.58	1.03	86.53	7.86
220	4.04	1.04	84.16	10.76
240	3.90	1.06	83.17	11.87
260	3.89	1.34	75.44	19.33
280	3.85	1.48	60.25	34.43
300	3.59	2.52	46.12	47.77
Lignite <sup>a</sup>	-	10.26	48.76	40.98
Anthracite <sup>b</sup>	-	11.20	4.40	84.40
Bituminous coal <sup>b</sup>	-	7.80	40.30	51.90

<sup>a</sup>[5]; <sup>b</sup>[27]

### 3.2. Calculation for C, H and O content

Based on the elemental content in the raw and torrefied *Leucaena*, correlation equations were applied to calculate the C, H and O contents. The values of elemental contents are shown as in Table 2 with the corresponding O/C and H/C ratios. In general, torrefaction results in the *Leucaena* enhancement of C content at the expense of reducing O and H contents. Removal of oxygenated compounds were achieved via decarboxylation and dehydration that happened at higher temperature [28]. The experimental result is in good agreement with [16] and they suggested that deoxygenation is the primary reaction occurred during torrefaction. The O content is likely to be released in the form of H<sub>2</sub>O, CO, CO<sub>2</sub> and oxygen-containing organic compounds.

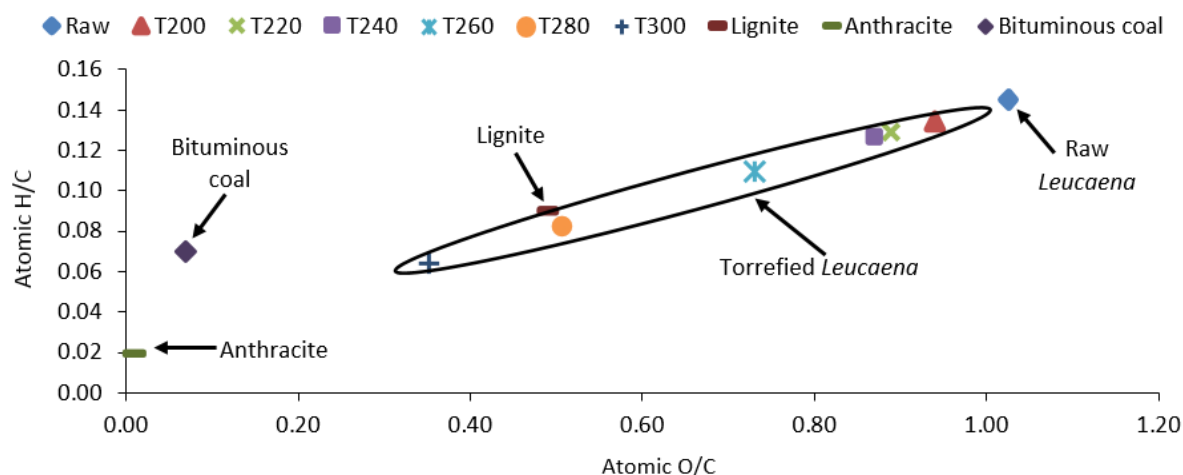
In addition, the variation in elemental composition of raw and torrefied *Leucaena* was also analyzed using Van Krevelen diagram as illustrated in Figure 1. Data points for lignite, anthracite and bituminous coal are described as well. It can be seen that the properties of torrefied sample obtained at torrefaction temperature of 280 °C approached that of lignite. This indicates that torrefied *Leucaena* at torrefaction temperature of 280 °C is a prominent renewable source of energy that is comparable to non-renewable source of energy like lignite. While the atomic composition of torrefied sample at 300 °C fell below lignite but higher than anthracite. The changes is in good agreement with [13].

**Table 2.** Elemental analysis based on correlation equations.

Torrefaction temperature (°C)	C	H	O	O/C ratio	H/C ratio
Raw	45.28	6.56	46.44	1.03	0.14
200 °C	47.45	6.38	44.57	0.94	0.13
220 °C	48.77	6.26	43.37	0.89	0.13
240 °C	49.30	6.22	42.87	0.87	0.13
260 °C	53.32	5.83	38.94	0.73	0.11
280 °C	61.65	5.08	31.24	0.51	0.08
300 °C	68.47	4.36	24.06	0.35	0.06
Lignite <sup>a</sup>	61.64	5.72	31.13	0.51	0.09
Anthracite <sup>b</sup>	84.90	1.90	0.80	0.01	0.02

Bituminous coal <sup>b</sup>	77.50	5.30	5.70	0.07	0.07
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<sup>a</sup>[5]; <sup>b</sup>[27]



**Figure 1.** Van Krevelen diagram for raw, torrefied *Leucaena*, lignite, anthracite and bituminous coal.

### 3.3. FTIR analysis

The FTIR spectra of raw and torrefied *Leucaena* at 20 min holding time, 20 °C/min heating rate, 150 ml/min N<sub>2</sub> flow rate and six different torrefaction temperatures (200, 220, 240, 260, 280 and 300 °C) are presented in Figure 3 while the possible functional group identified by FTIR are listed in Table 3. FTIR analysis is employed to determine the functional groups of *Leucaena* under different torrefaction temperatures. The effect of torrefaction temperature towards the functional groups evolution can be easily noticed according to these spectra.

The most prominent peak in the wavenumber range of 3600 to 3200 cm<sup>-1</sup> is present in both raw and torrefied *Leucaena*. This broad peak is attributed to the intra- and inter molecular hydrogen and characteristic peak of crystalline cellulose [5]. The peak was referred to the carboxylic, phenolic and alcoholic groups [29]. However, at higher torrefaction temperature, the intensity of the peak has decreased. The decreasing peak intensity is in line with temperature and can be associated with the removal of OH thus, it contains less amount of OH group. The loss of this functional group is due to the dehydroxylation and condensation reaction occur during torrefaction [15]. Consequently, it improved the hydrophobicity of torrefied *Leucaena* [12]. Torrefaction of *Leucaena* at 300 °C has produced solid material with the improved hydrophobicity property.

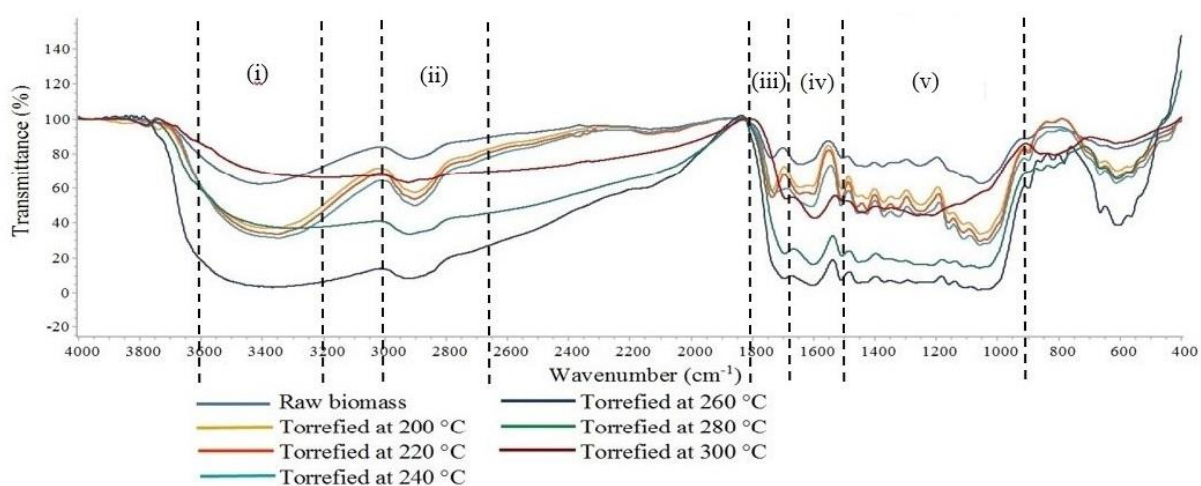
The peak between 3000- 2650 cm<sup>-1</sup> is assigned to C-H stretching vibration in CH<sub>3</sub>- and CH<sub>2</sub>- [30]. This stretching is referred to the aliphatic content in the samples. The raw and torrefied *Leucaena* at 200 °C to 280 °C showed similar intensity at this peak, however at torrefaction temperature of 300 °C the intensity has reduced. Thus, it shows that the aliphatic content in torrefied *Leucaena* at 300 °C has reduced due to the carbonaceous material that has been converted at higher temperature since hemicellulose and cellulose were degraded at this temperature region [31].

The sharp peak at wavenumber of 1800-1675 cm<sup>-1</sup> corresponds to the presence of C=O carbonyl group. The reduction in the intensity of transmittance peak from torrefied *Leucaena* at temperatures of 260, 280 and 300 °C might be due to the deacetylation during torrefaction which caused the removal of ester group in hemicellulose [9]. The decreasing intensity might be due to the liberation of more oxygen-containing functional groups from hemicellulose and lignin derivatives [32].

The transmittance peak at 1675-1550 cm<sup>-1</sup> is assigned to C=C stretching vibration of alkene group. The torrefied *Leucaena* at 260, 280 and 300 °C show increased intensity of this band. The formation

of non-polar and unsaturated compounds caused by the degradation of hemicellulose that explain the observed trends [13]. It was observed that the major effect of torrefaction process was towards the degradation of hemicellulose.

The band at approximately  $1550\text{-}900\text{ cm}^{-1}$  referred to the aromatic C-H group skeletal vibration. The raw and torrefied *Leucaena* at 200, 220, 240, 260 and 280 °C show the band which indicate that they have the same functional groups. However, for the sample torrefied at 300 °C, the band was almost negligible. This is because lignin was degraded at 300 °C [12]. Based on [33], lignin started to decompose at temperature region of 280- 500 °C due to the cleavage of ether bonds and scissioning of carbon-carbon bonds, thus produce phenols.



**Figure 2.** FTIR spectrum of raw and torrefied *Leucaena*.

**Table 3.** Possible functional group identified by FTIR.

Wavenumber ( $\text{cm}^{-1}$ )	Possible assignment
Peak at 3600-3200	OH vibration of carboxylic and alcoholic groups
Peak at 3000-2650	C-H stretching vibration
Peak at 1800-1675	C=O carbonyl double bond stretching
Peak at 1675-1500	C=C stretching vibration of alkene group
Band at 1500-900	C-H in plane bending vibration

#### 4. Conclusion

Torrefaction of *Leucaena* was conducted in a fixed-bed reactor at different torrefaction temperatures of 200-300 °C. Torrefaction had decreased the O content of *Leucaena*. The elemental and chemical properties of torrefied sample were also investigated for different torrefaction temperatures. It was observed that the raw and torrefied *Leucaena* had similar evolution patterns, but differ in degree of intensity of functional groups. Torrefaction temperature has a strong effect on the existence of functional groups in torrefied samples. From all torrefied *Leucaena*, the sample at torrefaction temperature of 300 °C experienced considerable functional groups loss. Overall, the proximate analysis as well as C, H and O calculation are in good agreement with FTIR spectra patterns, with regard to the degradation of hemicellulosic compounds.

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### Acknowledgement

The authors would like to express their gratitude to the Department of Chemical Engineering Technology, Faculty of Engineering Technology (FETech) and Institute of Sustainable Agrotechnology (INSAT) of Universiti Malaysia Perlis.

