Durability Studies of Solid Pellets from Torrefied Leucaena Leucocephala

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Abstract. Torrefaction of Leucaena Leucocephala was performed in a fixed bed reactor at temperature of 300 °C and 20 minutes holding time under inert environment. The torrefied Leucaena Leucocephala was pelletized with starch as the binder. Two parameters were investigated such as different ratio of starch addition to torrefied Leucaena Leucocephala pellets and the thickness of torrefied Leucaena Leucocephala pellets towards its durability. The ratios of starch addition to torrefied Leucaena Leucocephala pellets studied were 0, 5, 10, 15, 20 and 25 wt%. Meanwhile, the studied thickness of torrefied Leucaena Leucocephala pellets were 0.3, 0.4, 0.5, 0.6 and 0.7 cm. It was observed that when the ratio of starch addition to torrefied Leucaena Leucocephala pellets was varied from 0-25 wt%, the durability increased steadily and the highest durability of 56.06% was achieved at 20 wt%. When the thickness of torrefied Leucaena Leucocephala pellets was increased from 0.3-0.7 cm, the highest durability of 93.18% was achieved at 0.5 cm thickness. Scanning Electron Microscope (SEM) analysis was performed on the torrefied Leucaena Leucocephala pellets of 0 and 20 wt% ratio of starch addition. SEM images show the existence of some minor pores on the surface of torrefied Leucaena Leucocephala pellet at 250X and 1000X magnifications.

1. Introduction

Fossil fuel combustion leads to the emission of harmful gases containing sulfur (S), nitrogen (N), and carbon (C). The acid rain is formed by the combination of oxides of sulfur, nitrogen and carbon with water. The hazardous impact of the fossil fuel utilization on the environment has been the driving factor in search of renewable energy that could substitute fossil fuel [1]. Biomass is a prominent solution to this problem as it is a renewable fuel which is abundant [2].

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Pyrolysis and torrefaction are well known biomassic thermochemical processing technology with several advantages that enable manipulation on the process parameters like reaction temperature, holding time, heating rate and gas flowrates towards maximizing desired products such as biochar/torrefied biomass, bio-oil and non-condensable gases. The investigation of process parameters in biomass pyrolysis have been reported that indicates the potential acquirement of bio-oil from biomass [3-8].

One of the example of biomass resource is *Leucaena Leucocephala*. *Leucaena Leucocephala* is a fast growing tree and is considered as a potential energy crop [2]. Biomass is known for having high moisture content and low calorific value. Therefore, torrefaction is introduced to solve this problem before going through pelletization. Torrefaction is a thermal pretreatment between low temperature range of 200°C to 300°C with short residence or holding time and conducted under inert environment such as in oxygen-free environment under atmospheric pressure [9,10]. Torrefaction of biomass such as *Leucaena Leucocephala*, oil palm empty fruit bunches, mesocarp fiber and kernel shell have been reported that indicated the mass yield of torrefied biomass decreased over increasing torrefaction temperature [11 -13]. Handling of torrefied biomass under loose condition seems tedious. This can be solved by pelletizing the torrefied biomass. Pelletization converts torrefied biomass into a regular shape product like pellets, briquettes and cubes. This is beneficial because it assists the management and transportation of the torrefied biomass [13,14]

In the pelletization process, usually binder is added to either raw or torrefied biomass to improve its durability. Durability of pelletized raw and torrefied biomass has been reported elsewhere [15, 16]. Starch is a prominent binder in the pelletization of torrefied biomass. There is scarce information that focused on the effect of torrefied biomass, especially *Leucaena Leucocephala* towards the durability of the pellets and its surface morphological properties upon mixing with starch as binder. Therefore, this paper aims to report about the durability and surface morphological properties of torrefied *Leucaena Leucocephala* upon mixing with starch as binder at a selected ratio of starch addition to torrefied *Leucaena Leucocephala*.

### 2. Materials and Methods

#### 2.1. Sample preparation

*Leucaena Leucocephala* sample was collected from a village named Kg MADI in Jejawi, Perlis, Malaysia. The bulky *Leucaena Leucocephala* biomass samples were cut and chopped. After that, the chopped samples were crushed by crusher into chip-sized samples. *Leucaena Leucocephala* was placed in the oven at temperature of 80°C for 24 hours. Then, the chip-sized samples were ground into small particle sizes using a grinder. Then, the samples were sieved using Retsch sieve shaker in order to obtain the particle size in the range of 500-710 μm which was selected for torrefaction process.

#### 2.2. Torrefaction Process

Torrefaction process was conducted using a fixed-bed reactor for several times in order to obtain a certain amount of torrefied sample which was sufficient for subsequent studies. The total mass of torrefied *Leucaena Leucocephala* produced was approximately 100g.

Approximately 10g of *Leucaena Leucocephala* was fed into the fixed-bed reactor. The bottom part of the fixed-bed reactor was attached to a conical flask to collect the bio-oil during torrefaction. The conical flask was connected with a pipe in order to discharge the waste gas. The conical flask was placed in an iced-box to condense volatile gas from the process. The torrefaction process was operated at torrefaction temperature of 300°C and holding time of 20 minutes. Nitrogen gas purged the reactor for 15 minutes at a rate of 150 ml/min in order to provide inert condition within the reactor [11]. After
torrefaction experiments completed, the fixed-bed reactor was turned off and it was allowed to cool at room temperature. The torrefied sample was taken out from the reactor [17].

2.3. Pelletization of Torrefied Leucaena Leucocephala

The pelletization process was performed using a mould in which 1.6 g of sample with starch addition was properly inserted into the mould until full. Then, the mould was placed into the press machine and the pressure was applied to the mould in order to form the pellet. The pressure applied was 7 bar. After applying the pressure, the mould was left for 2 minutes in order to allow the biomass to form a stable and strong pellet inside the mould. Lastly, the pellet was taken out from the mould and ready for durability testing. In pelletization, the height of the pellets was manipulated with 5 different heights whereas the diameter of the pellets was fixed. After conducting the durability test, the optimum height of the pellet was obtained with minimum weight loss. Then, starch was used as binder and mixed with torrefied Leucaena Leucocephala. Next, the torrefied Leucaena Leucocephala with starch was ready for pelletization process. The ratio of starch addition to torrefied Leucaena Leucocephala was varied at 5, 10, 15, 20 and 25 wt% respectively. From the first experiment, it was identified that the highest durability was obtained at the ratio of starch addition to torrefied Leucaena Leucocephala of 20 wt%. Therefore, this ratio was kept constant for the current study. In order to investigate the thickness of torrefied Leucaena Leucocephala pellets, the initial mass of torrefied Leucaena Leucocephala were 2.0, 1.8, 1.6, 1.4 and 1.2 g. The studied thickness of the pellets were 0.3, 0.4, 0.5, 0.6 and 0.7 cm.

2.4. Durability Analysis of Torrefied Leucaena Leucocephala Pellets

The Retsch sieve shaker was used to test the durability properties of pellets. The sample used in this process was the torrefied Leucaena Leucocephala. This method known as tumbling process that the sample was tumbled for 15 minutes. The samples were placed in the top of stack sieve shaker. The initial weight of pellets was measured before sieving whereas the final weight of pellets was measured after sieving. The weight of the pellets was recorded before and after the tumbling process. This was because the changes of mass must be measured and calculated in order to find the durability percentage of the biomass pellet. The durability percentage is calculated using equation 1:

\[
\text{Durability} = \frac{\text{Final mass of torrefied pellets}}{\text{Initial mass of torrefied pellets}} \times 100\% \tag{1}
\]

The durability test was repeated twice for every ratio of starch addition to the torrefied sample. Hence, the results obtained from two runs for different conditions were used to calculate the average durability.

2.5. Scanning Electron Microscope (SEM)

The surface morphology investigation was performed by using scanning electron microscope (SEM) (Hitachi TM 3000). The pellets were mounted onto SEM stubs layer which was the layer with sticky tape. The stub was placed in sputter coater which was Auto line cutter, JEOL-1600 for five minutes for coating with platinum to give high quality of being reflective during scanning process.

3. Results and Discussion

3.1. Durability Analysis of Different Ratio of Starch Addition to Torrefied Leucaena Leucocephala Pellets

Figure 1 shows the average durability of the different ratio of starch addition to torrefied Leucaena Leucocephala pellets. When the ratio of starch addition was 0 wt%, the durability of the pellets was 0 %. This indicates that the structure of the torrefied Leucaena Leucocephala pellet was definitely weak in the absence of binder because it is brittle and lack of solid bridges between torrefied particles [11]. Torrefaction process at torrefaction temperature of 300 °C also has resulted in the loss of natural binder from the hemicellulosic fraction of Leucaena Leucocephala. Therefore, it requires the presence
of binder in order to increase its durability. When the ratio of starch addition to torrefied *Leucaena Leucocephala* pellet was increased to 10 wt% and 15 wt%, the average durability of the pellet increased to 10% and 22.95 % respectively. The highest average durability of 56.06 % was achieved at starch addition to torrefied *Leucaena Leucocephala* pellet of 20 wt%. However, the average durability decreased slightly to 40.50 % once the ratio of starch addition to torrefied *Leucaena Leucocephala* pellet increased to 25 wt%.

3.2. Durability Analysis of Different Thickness of Torrefied Leucaena Leucocephala Pellets

Figure 2 shows the average durability against thickness of torrefied *Leucaena Leucocephala* pellets at different thickness of 0.3-0.7 cm. When the thickness of the pellet was increased from 0.3 to 0.4 cm, the average durability of the pellet decreased from approximately 91.0 % to 86.95 % respectively. However, when the thickness of the pellet was increased to 0.5 cm, its durability increased to 93.18%. This is the highest value of average durability of the pellet with a thickness of 0.5 cm. It is worth noted that when the thickness of the pellet was increased further to 0.6 and 0.7 cm, the average durability decreased further to 92.90 % and 90.33 % respectively.

![Figure 1](image1.png)

**Figure 1.** The graph of average durability against ratio of starch addition to torrefied *Leucaena Leucocephala* pellets.

![Figure 2](image2.png)

**Figure 2.** The graph of average durability against thickness of torrefied *Leucaena Leucocephala* pellet

3.3. Scanning Electron Microscope (SEM) Analysis

The SEM images of torrefied *Leucaena Leucocephala* pellet with 0 and 20 wt% ratio of starch addition are as shown in Figure 3 respectively. The magnifications for 0 and 20 wt% pellets are 250x/1000x and 500x/1000x. The pellet with 0 wt% ratio of starch addition is referred as torrefied *Leucaena Leucocephala* pellets.
SEM images in Figure 3 show the existence of some minor pores on the surface of torrefied *Leucaena Leucocephala* pellet at 250X and 1000X magnification. Pore formation occur during torrefaction process mainly due to the contribution of volatiles removal process [11].

The surface morphological analysis on the torrefied *Leucaena Leucocephala* pellets with ratio of starch addition of 20 wt% indicates the absence of pores at 500 X magnification. The structure stacks with other layers due to the pressure that was applied during the pelletization of the torrefied biomass. At 1000X magnification, it is identified the existence of minute particles with definite size and shape in an orderly arrangement. This is suggested as the appearance of starch particles that become compacted during pelletization process. It is also suggested that the orderly arrangement of starch particles has filled up the pores that exist on the surface of torrefied *Leucaena Leucocephala*. This could be due to the good binding between starch particles and torrefied *Leucaena Leucocephala* at that particular ratio. Consequently, this had improved the durability of the torrefied *Leucaena Leucocephala* pellet.

| Sample                  | Torrefied *Leucaena Leucocephala* pellet | Torrefied *Leucaena Leucocephala* pellet with ratio starch addition of 20 wt% |
|-------------------------|------------------------------------------|---------------------------------------------------------------------------------|
| Magnification           | 250X                                     | 1000X                                                                           |

Figure 3: Scanning Electron Micrographs of torrefied *Leucaena Leucocephala* pellet and *Leucaena Leucocephala* pellet with ratio starch addition of 20 wt%

4. Conclusion

Torrefaction of *Leucaena Leucocephala* has been conducted a fixed bed reactor at torrefaction temperature of 300 °C to produce torrefied *Leucaena Leucocephala* that underwent pelletization in the presence of starch as the binder. Durability analysis on the different ratio of starch addition to torrefied *Leucaena Leucocephala* pellets indicates that at 20 wt% ratio of starch addition to torrefied *Leucaena Leucocephala*.
Leucaena Leucocephala, the durability of 56.06% was the highest. Meanwhile, when the thickness of torrefied Leucaena Leucocephala pellets was 0.5 cm, the highest durability of 93.18% was achieved. Scanning Electron Microscope (SEM) analysis showed the existence of minute particles with definite size and shape in a certain orderly arrangement at 1000X magnification on the surface of the pellet with ratio of 20 wt% starch addition to torrefied Leucaena Leucocephala.

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