Biopellet from demineralized oil palm trunk

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Abstract. Oil palm trunk (OPT) is a potential raw material for biopellet manufacturing. This study aimed to reduce the ash content of biopellet through pre-treatment with sulfuric acid. The moisture content, durability, ash content, density, and calorific values of the biopellet were determined according to DIN EN 14961-2 and 51731 standards. Scanning electron microscopy (SEM) indicated the presence of inter-particle interlocking in the highly durable biopellet. Thermal analysis indicated that the mass and water loss, hemicellulose, cellulose, and lignin decomposition occurred at 76.12 ºC, 113.97-200 ºC, 310-360 ºC, and >400 ºC, respectively. Biopellet produced retained a moisture content of 3.40-8.90%, the durability of 97.75-99.38%, ash content after pre-treatment with H₂SO₄ of 1.02-1.47%, control ash content of 2.20-3.31%, the density of 1.03-1.30 g/cm³, and the calorific value of 3954-4608 kcal/kg. The biopellet quality fulfilled the requirements of DIN EN 14961-2, 51731, and SNI 8021-2014 standard, except for the ash content of the control.

1. Introduction
Ministry of Agriculture [1] reported that the total area of Indonesia’s oil palm plantation in 2015 was 13.5 million Ha; an 11.8% increase from that in 2014. Along with the increasing area, since 2010, rejuvenation of oil palm plantation is done for at least 100.000 Ha annually. Rejuvenation programs produce much oil palm biomass waste. The wastes are potential biomass for fossil-based fuel alternatives. The use of biomass-based fuel does not increase greenhouse gasses and CO₂ emission [2].

Biomass is convertible to high-density pellet beneficial handling cost, cleanliness, transportation, and storage. Biomass densification is a practical technology to convert biomass into pellet and briquette. Biopellet can be made from agricultural wastes, solid waste of biogas production, and wood smaller than briquette. DIN 51731 Standard [3] defines the pellet measurement as less than 50 mm in length and 4 – 10 mm in diameter. Important parameters for the quality of biopellet include moisture content, density, ash content, durability, and calorific value. A high moisture and ash content tend to decrease the calorific value of biopellet [4], and high fixed carbon content increases the calorific value [5]. Biopellet durability is crucial in its storage and long-distance transportation. Pre-heating, particle size, raw material sources, moisture content, pelletizing temperature, densification pressure, and densification duration determine the durability of biopellets [6, 7]. The pellet density sharply increases during initial densification, followed by a gradual increment with increasing densification load [8]. Lignin retains many advantageous properties for its application in biopellet production. The lignin increases the durability of biopellet [9], probably due to its thermoplastic properties that soften when heated and function as a natural adhesive for biopellet production [10].
Even though the lignin content of the oil palm bark is higher than its xylem, the addition of bark in the production of oil palm biopellet failed to fulfill DIN 14961-2 and DIN requirements 51731 on the durability, density, and ash content [11]. Biopellet durability and density are influenced by densification load [12, 13].

The higher ash content in the bark brought about the higher content of ash in the biopellet, as well [11]. Ash hinders interparticle bonding and reduces the energy content of biopellet. Therefore, reducing ash content in the biomass could increase the quality of biopellet. Demineralization of biomass before densification has been done with water [14, 15], deionized water, nitric acid, and orthophosphoric acid [16], and hydrochloric acid, and sulphuric acid [16, 17]. Acidic washing is more effective than water washing to reduce ash content [15, 16, 18, 19]. Washing biomass with hydrochloric acid, nitric acid, and sulphuric acid removes Ca and Mg [16]. Acid concentration and soaking time influence demineralization, in which increases concentration and soaking duration reduce the ash content [18].

In the present research, the oil palm biomass was demineralized with sulphuric acid at various concentrations and soaking durations. The pre-treatment is carried out to reduce the ash content and to increase the calorific content of the biopellet.

2. Methods
2.1. Raw material preparation
Oil palm trunk of approximately 25 years old was divided into bottom, middle, and top divisions. All divisions were barked and chipped. The chips were air-dried and milled with a Willey mill. The oil palm trunk meal was then sieved into 20–40 mesh, 40–60 mesh, and 60–80 mesh. Oil palm trunk meal for biopellet was demineralized with sulphuric acid and then dried to a moisture content of 10% [19]. Figure 1 indicates the outline of the experiments.

2.2. Demineralization
The ash content of all samples before demineralization was determined using raw material of 40–60 mesh. Demineralization was carried out by soaking samples in sulphuric acid of 0 (control), 1, and 5% concentration. Sulfuric acid volume was 10 mL/g samples under a soaking temperature of 27 ± 2 °C [16]. Upon soaking stage, samples were washed to neutrality and dried to a moisture content of 10%.

2.3. Biopellet formation
In the preparation of biopellet, oil palm trunk meal was mixed with oil palm bark meal. The bark content was varied at 0, 10, and 30%. Densification was carried out by pellet mill BEJE-UPTK 08 (Indonesia) of 200 kg/h capacity with 252 holes. Each hole diameter is 5 mm, and a power of 8200 watts drove the mill. Before densification, the raw material was well mixed with 500 mL of water per kg raw material. Densification was carried out at 130 °C. Upon densification, the resulting biopellet was conditioned for one day. Table 1 indicates the biopellet composition produced in the present work.

![Figure 1. Experimental outline.](image-url)
Table 1. The composition of oil palm trunk meal and oil palm bark meal for the preparation of biopellet.

| Biopellet formulation | Bottom division | Xylem (trunk) content (%) | Middle division | Upper-division | Bark content |
|-----------------------|----------------|--------------------------|----------------|--------------|--------------|
| 1                     | 50             | 30                       | 20             | 0            |
| 2                     | 50             | 30                       | 10             | 10           |
| 3                     | 40             | 20                       | 10             | 30           |

2.4. Quality parameters of biopellet

Moisture content, density, ash content, durability, and calorific values are biopellet quality parameters determined in the current work. Other supporting data such as SEM and thermal behavior analysis were also determined. Moisture content was determined by the oven drying method of a sample at 103 ± 2 ºC until a constant weight was obtained. The displacement method previously applied by Rabier et al. [20] was used to determine the biopellet density, and ash content determination was based on the TAPPI T 211 om-93 standard procedure [21]. Biopellet durability was measured following the ASABE standard [22], and its calorific value was measured with Bomb Calorimeter LECO UK AC-500.

Scanning electron microscopy method by SEM, JSM-6510LV, JEOL (USA) with 50x and 500x magnification was applied to determine a piece of visual information on the interparticle bonding of the biopellet. The thermal properties of the biopellet were determined by TG-50 (ZG-127, Shimadzu). Analysis was conducted in the range of 30-600 ºC at a heating rate of 10 ºC /min with Alumina (α-Al2O3) powder as control.

2.5. Data analysis

A factorial complete random design with three factors, i.e., particle size (20-40 mesh, 40-60 mesh, and 60-80 mesh), acid concentration (0, 1, 5%), and bark content (0, 10, and 30%) was applied. The data were obtained in triplicate, and Duncan’s multiple range test (MRT) was further used to analyze the effect of treatments.

3. Results and Discussion

3.1. Ash content of the oil palm biomass

Ash is inorganic or minerals component of biomass. The high ash content of biomass bring about slagging, fouling, agglomeration, and deposition [23]. The present results showed that ash content is significantly influenced by trunk division, acid concentration, and interaction. Duncan MRT indicated that before demineralization, the ash content of the trunk (2.03 – 2.89%) was lower than the ash content of its bark (6.43%) (figure 2). The ash content of biomass bark is higher than that of its wood [24]. A similar finding has also been reported by Wistara et al. [11], where the ash content of oil palm bark was higher than the ash content of its meristem.

![Figure 2](image-url)  
**Figure 2.** Ash content of demineralized meristem and bark component of oil palm (the same letter indicates a nonsignificant difference, and the different letter indicates a significantly different).
Ash content significantly reduced with increasing acid concentration. The ash content of oil palm biomass demineralized with 1% $\text{H}_2\text{SO}_4$ was in the range of 0.62 – 4.27% higher than those demineralized with 5% $\text{H}_2\text{SO}_4$, which was in the range of 0.42 – 2.15%. Washing with sulfuric acid was proven to reduce as much as 34.20% ash content accompanied with a 7.10% increased calorific value [17]. Abundant $\text{H}^+$ ions in sulfuric acid promote the extraction of both water-soluble and water-insoluble minerals of biomass, leading to the reduction of ash content [25].

3.2. Biopellet quality parameters

Analysis of variance was carried out to evaluate the influence of acid concentration, bark content, and particle size on the quality parameters of biopellet. Table 2 indicates the result of the analysis of variance on the procured data.

3.2.1. Moisture content. Moisture content is an essential parameter in biopellet quality. Biopellet moisture content obtained in the present research was in the range of 3.40 – 8.90% (figure 3). The values satisfied DIN EN 14961-2 standard of lower or equal to 10%. The moisture content was significantly influenced by particle size, acid concentration, bark content, and interactions (table 2). The moisture content of biopellet prepared with a 60-mesh particle demineralized with 5% sulfuric acid and contain 10% bark was the lowest (3.40%), and the highest (8.90%) was with 20 mesh particles without demineralization and bark addition. Increasing particle size tends to increase the moisture content of biopellet [26]. Wistara et al. [27] has proven that biopellet prepared with 20 mesh particles retained a higher moisture content than that prepared with 40 mesh particles. Biopellet with a bark content of 30% tended to have a higher moisture content. A similar finding for the biopellet of Pinus pinaster and Pinus halepensis has been previously reported [28].
Table 2. The analysis of variance of the biopellet quality parameters.

| Treatments                  | Moisture content | Durability | Ash content | Density |
|-----------------------------|------------------|------------|-------------|---------|
| Particle size (A)           | 0.000            | 0.005      | 0.000       | 0.000   |
| Acid concentration (B)      | 0.000            | 0.008      | 0.000       | 0.000   |
| Bark content (C)            | 0.000            | 0.000      | 0.000       | 0.067   |
| A*B                         | 0.000            | 0.006      | 0.002       | 0.872   |
| A*C                         | 0.008            | 0.420      | 0.926       | 0.132   |
| B*C                         | 0.000            | 0.017      | 0.000       | 0.001   |
| A*B*C                       | 0.000            | 0.035      | 0.939       | 0.865   |

Note: P-value ≤ 0.05 is significant and P-value > 0.05 is insignificant at confidence level of 95%.

3.2.2. Durability. The durability of biopellet was found in the range of 97.75-99.38% (figure 4). It satisfied the requirement of DIN EN 14961-2, which must be ≥96.5%. Biopellet prepared using a 20 mesh particle size demineralized with 5% acid concentration without any addition of bark achieved the lowest durability of 97.75%. Meanwhile, the highest biopellet durability was obtained with 60 mesh particle size demineralized at 1% and bark content of 30%.

Figure 4. The effect of bark content and acid concentration on the durability of oil palm biomass biopellet. Particle size: (a) 20 – 40 mesh, (b) 40 – 60 mesh, and (c) 60 – 80 mesh.
Densification load is an essential factor influencing biopellet durability—a higher densification load results in a higher biopellet durability [13]. The present results indicated that the density of biopellet prepared with the particle size of 20 – 40 mesh and 40 – 60 mesh were not significantly different; however, they were different from that of biopellet made by 60 mesh particle. Biopellet of finer particles tends to retain higher durability. It has been reported by Lee et al. [6] for biopellet of *Larix kaempferi* and *Liriodendron tulipifera* wood that biopellet prepared by the particle size of lower than 1.41 mm was more durable than that prepared by 1.41 – 3.17 mm particle size. Bark content determined the durability of biopellet. The addition of 30% bark resulted in the highest biopellet durability. The bark contains a higher amount of lignin that is thought to increase interparticle bonding in biopellet. At high densification temperature, high content of lignin increased biopellet durability [29]. Figure 5 indicates that biopellet with higher durability (bark content of 30%) retains a denser interparticle bonding than that of biopellet with lower durability (without bark).

![Figure 5. SEM micrograph of biopellet with 30% bark content [(a) at 100x and (b) at 500x magnification], and biopellet without the addition of bark [(c) at 100x and (d) at 500x magnification].](image)

3.2.3. Ash content of biopellet. The use of biomass containing many minerals brings about calorific and combustion-related problems [15]. The ash content of the presently produced biopellet was in the range of 1.01 – 3.31% (figure 6). Demineralization of oil palm biomass succeeded in producing biopellet with satisfying ash content according to DIN EN 14961-2 standard requirement of 1.5% or lower. The ash content of biopellet tended to increase with the severity of biomass milling. It is indicated by a higher ash content found in biopellet made of 60 mesh particles compared to those made by 20 and 40 mesh particles. However, the ash content of pine biopellet with a larger than 0.95 mm particle size was lower than that made of lower than 0.95 mm particle size [25].

Sulfuric acid concentration significantly reduced the ash content of biopellet (figure 7). The ash content of biopellet prepared from demineralized oil palm biomass is significantly lower than biopellet
prepared from untreated oil palm biomass. Strong acid effectively degrades minerals in biomass [16, 30].

![Figure 6](image.png)

**Figure 6.** The ash content of biopellet prepared from oil palm biomass.

![Figure 7](image.png)

**Figure 7.** The effect of bark content and acid concentration of the ash content of biopellet.

3.2.4. **Density.** The resulting bio pellets density was in the range of 1.03-1.30 g/cm³ (figure 8), which fulfilled the DIN 51731 standard of 1.00-1.40 g/cm³. Densification load and temperature influence the biopellet density—the density increases with increasing densification pressure and temperature [13, 31]. Particle size influenced biopellet density, in which finer particles were resulting in a higher density. It has been reported that pellets prepared from 60 – 100 mesh particles retain a higher density than those produced from 40 – 60 mesh [32].

![Figure 8](image.png)

**Figure 8.** The effect of sulfuric acid concentration, bark content, and particle size on the density of bio pellet.
Sulfuric acid concentration and the addition of 10% and 30% bark did not influence biopellet density. A 10% addition of bark is required to increase the biopellet density (figure 9). A similar finding for *Pine halepensis* and *Pine pinaster* biopellet has been reported by Arce et al. [28]. The increase of biopellet density with the addition of bark could be brought about by the cementing properties of the lignin.

3.2.5. Calorific value. In the present research, the calorific value measurement was carried out for biopellet prepared with 60–80 mesh particle size; 0 and 5% sulfuric acid concentration; and 0, 10, and 30% bark content. The resulting measurement indicated that the calorific value of biopellet ranged from 3954–4608 kcal/kg, satisfying the requirement of DIN EN 14961-2 standard of 3821–4538 kcal/kg. It can be seen from figure 10 that biopellet with a 30% bark content demineralized with 5% sulfuric acid retains the highest calorific value, and the lowest was that without bark and non-demineralized.

3.3. Thermal properties analysis
Thermal analysis was carried out for biopellet prepared with 60–80 mesh particles, demineralized with 5% sulfuric acid, and bark content of 30%. The thermograph of the sample is depicted in figure 11.
Figure 11. Thermograph of oil palm biopellet.

It can be seen that an exothermic peak occurred at 76.12 °C, with a slight weight loss indicating water evaporation of the sample. Sample degradation started at 113.97-200 °C, which is the range temperature of hemicellulose degradation. A significant weight loss indicating the decomposition of cellulose occurred at 310-360 °C. Pasangulapati et al. [33] stated that evaporation of water commonly occurs at below 125 °C, followed by the decomposition of hemicellulose (below 275 °C, lignin (200-340 °C), and cellulose (250-360 °C). Based on the presented thermograph in figure 11, densification should be done at below 200 °C. Densification above 200 °C could decompose lignin components [26] that potentially bring about a significant weight loss.

4. Conclusions
Demineralization of oil palm biomass significantly reduced ash content and increased the calorific value of the resulting biopellet. The quality of the resulting biopellet satisfying the requirement of DIN EN 14961-2, 51731 standards, except for the ash content of non-demineralized raw materials. The quality of biopellet was partly brought about by a well-formed particle interlocking, as shown by the SEM micrograph. The highest quality of biopellet was produced with 60 – 80 mesh particle size demineralized with 5% H2SO4 and 30% bark.

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