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Optimizing Nitric Acid Leaching Conditions for Ash and Potassium Reduction in Empty Fruit Bunches-Based Biomass Energy

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ABSTRACT

Oil palm empty fruit bunches (EFB) represent an abundant biomass waste in Indonesia with high potential as a solid biofuel. However, the high ash content and the presence of alkali metals such as potassium in EFB contribute to low combustion efficiency and increase the risks of fouling and slagging. This study aims to reduce the ash and potassium content in EFB-based biomass fuel through demineralization using nitric acid (HNO_3). Acid demineralization was conducted using various acid concentrations (1%, 3%, and 5%) and soaking durations (10, 30, and 60 minutes). The parameters analyzed include ash content, calorific value, potassium content, as well as proximate and ultimate analysis. The results indicate that demineralization using 1% nitric acid for 10 minutes is the optimum condition, successfully reducing the ash content from 7.93% to 3,43% and reduced potassium by 77% (0,087%- 0,02%). In addition, the fix carbon increased (16,11%-18,09%), sulfur decreased (0,115%-0,086%), and the calorific value increased to 4355,83 cal/g. This treatment also increased the carbon and hydrogen content while reducing sulfur content, thereby improving thermal quality and reducing emissions. Acid demineralization using low-concentration nitric acid is effective in reducing ash and potassium content, thereby enhancing the performance of EFB-based biopellets as a biomass energy source.

Keyword: Ash, Biopellet, Demineralization, Nitric Acid, Oil Palm Empty Fruit Bunches (EFB), Potassium

1. Introduction

Biomass is the fourth largest source of energy in the world. Biomass provides around 11-12% of global primary energy consumption [1]. One of the most abundant types of biomass in Indonesia is oil palm (Elaeis guineensis). The area of oil palm plantations has been increasing year by year. In 2023, the area of oil palm plantations in Indonesia increased to 16.83 million hectares compared to 14.58 million hectares in 2020 [2]. making Indonesia the world's largest producer of crude palm oil (CPO) [3]. The increase in oil palm plantation area is accompanied by an increase in biomass waste from oil palm mills, consisting of empty fruit bunches (EFB), fiber, and palm kernel shells. EFB waste accounts for 22% of the weight of fresh fruit bunches (FFB) and is the largest percentage of solid waste [4]. The chemical components of EFB consist of cellulose (23.70–65.00%), hemicellulose (20.58–33.52%), lignin (14.10–30.45%), and extractives (3.21–3.70%), making it a significant potential energy source [5]. The high cellulose and hemicellulose content contributes to calorie value, while lignin acts as a natural binder in the compaction process. Therefore, this chemical composition supports the use of EFB as a potential raw material for biopellet production.

However, EFB has a weakness, namely a very high moisture content of ≤60% [6], which can reduce combustion efficiency. In addition, EFB contains ash and minerals such as high alkali metals with a dominant mineral content of potassium at 48.94% of the ash weight [7]. The high potassium content in EFB is beneficial when used as fertilizer [8]. However, as a fuel, the ash content and inorganic elements such as alkali metals like potassium (K), sulfur (S), and chlorine (Cl) produced from biomass combustion contribute to corrosion, slagging, and fouling of boiler furnaces and stoves [9]. The formation of slagging and fouling can reduce overall energy conversion efficiency. Therefore, the mineral content in fuel must be reduced or even eliminated [10].

Demineralization biomass is an effective process for removing ash and alkali metal content from biomass. Leaching with strong acid solutions is more effective than leaching with water for removing ash content and alkali metal [11]-[14]. In the study by [11], rice straw treated with immersion in several acid solutions showed a significant reduction in ash content, with the highest reduction occurring in immersion using nitric acid (HNO_3) solution, decreasing from 14.7% to 6.69%. This aligns with the research by [15], where the use of low-concentration nitric acid was able to dissolve the mineral content forming ash in corn and soybean biomass to very low levels and increase the calorific value of the biomass. Leaching with an acid solution removes both water-soluble and water insoluble ash content, resulting in low ash content. The demineralization process is influenced by acid concentration and soaking time. According to previous research, most of the ash content reduction during the demineralization process occurs within the first 30 minutes [12]. After this duration, the effectiveness of demineralization decreases significantly [16]. Therefore, appropriate acid concentration and soaking time are required to achieve optimal results, thereby producing high-quality fuel sources.

Therefore, this research aims to investigate the optimum conditions for the demineralization process in producing low ash and alkali metal EFB biomass. The factors of washing the acid solution with different concentrations and soaking times were evaluated using measurement parameters in the form of calorific value data, ash content, and alkali metal composition before and after demineralization treatment.

2. Materials and Methods

2.1 Preparation of Raw Materials

Empty palm fruit bunches (EFB) were obtained from the palm oil mill of PT. Perkebunan Nusantara VIII, located in Cigudeg, Bogor, Indonesia (6°33'04.46"S 106°32'17.78"E). The empty palm fruit bunches were chopped using a chipper machine (Pallmann, Germany). Next, they are ground into smaller particles using a ring flaker (Pallmann, Germany). The particles are then sieved using a sieve shaker (Dahan, China). The particle size of EFB used in this study passed 60 mesh [17].

2.2 Pre-treatment Demineralization

Demineralization was carried out by immersing empty oil palm bunches in varying concentrations of nitric acid (1%, 3%, and 5%) and varying soaking times in nitric acid solution (10, 30, and 60 minutes). The volume of nitric acid solution was 10 mL/g of sample at a soaking temperature of 27 ± 2 °C [11]. After the soaking stage, the samples were washed with water until neutral and dried to a moisture content of < 10% [18].

2.3 Characterization of EFB and EFB Biopellets

The moisture content, ash content, calorific value, and ultimate and proximate analysis of EFB have been evaluated. Moisture content was calculated by drying the sample at a temperature of 103±2 ºC for 24 hours [13]. Ash content and calorific value were determined according to ASTM E-1755-01 and ASTM D 2015–96 using an IKAC 6000 bomb calorimeter. The ultimate analysis of EFB samples was measured according to ASTM D 3176 using an Eager 300 elemental analyzer. Potassium content was measured using an Atomic Absorption Spectrophotometer (AAS) Thermo iCE 3000 series. The proximate analysis of EFB samples was calculated according to SNI 06-3730. Testing of biopellet characteristics, including moisture content, ash content, volatile matter content, fixed carbon content, and calorific value, was conducted in accordance with SNI 8675-2018.

2.4 Data analysis

The statistical analysis used was a completely randomized design (CRD) with two factors: nitric acid concentration and soaking time. The nitric acid concentration levels were 0, 1, 3, and 5%, and the soaking times were 0, 10, 30, and 60 minutes. Each treatment was replicated three times. The effect of the factors on the response was examined using analysis of variance at a 95% confidence level, and Duncan's multiple range test was performed to analyze the effect of the treatments.

3. Results and Discussion

3.1 Ash Content

The efficiency of the EFB demineralization process was observed through ash content analysis. Figure 1 shows the results of ash content reduction at various acid concentrations and different soaking times. The ash content value of untreated EFB was 10.62%. The highest ash content reduction was observed in the immersion using a 3% nitric acid solution with a soaking time of 10 minutes, resulting in an ash content of 5.45%. Statistical analysis revealed that acid concentration and soaking time did not significantly affect ash content but significantly affected the control value ($P \leq 0.05$). This is consistent with previous research indicating that demineralization effectively dissolves certain alkaline minerals such as potassium, sodium, and magnesium, resulting in a decrease in ash content of the sample at low acid solution concentrations (1%) [19]. Furthermore, these results are supported by the characteristic of ash having higher solubility at lower pH (higher acidity) [20]. Furthermore, demineralization using nitric acid on corn and soybean biomass can significantly reduce ash content [12]-[15].

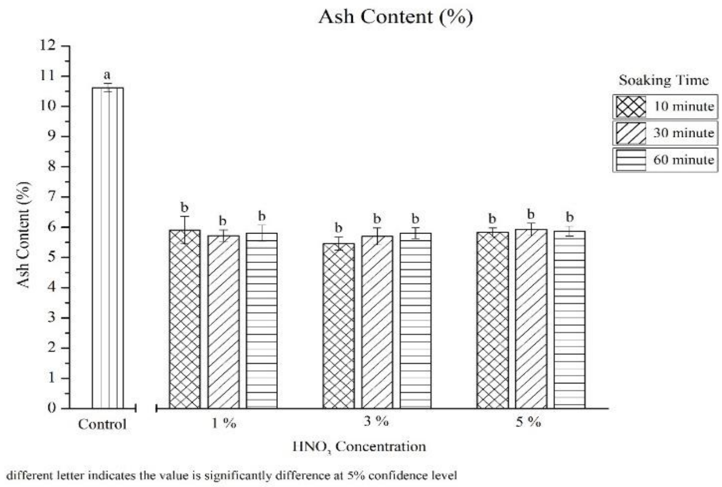
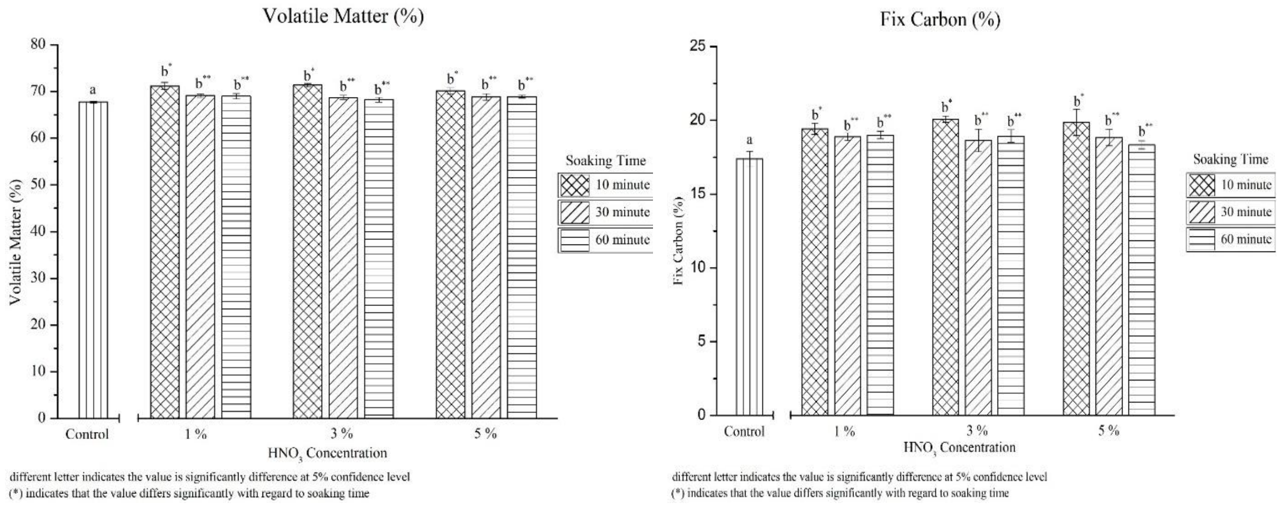


Figure 1. Effect of HNO_3 concentration and soaking time on the reduction of ash content in EFB

A significant reduction in ash content has a positive impact on the thermal quality of biomass. Lower ash content correlates with increased calorific value and reduces the potential for corrosion, deposition, agglomeration, and contamination in thermal conversion systems [21].

3.2 Volatile Matter and Fixed Carbon

Figure 2 shows the effect of demineralization treatment on the volatile matter and fix carbon content in empty palm fruit bunches. Based on the graph, the volatile matter content ranged from 68 to 72%, and fix carbon content ranged from 17.5 to 20%. Untreated EFB (control) showed the lowest volatile matter and fix carbon content. Leaching treatment at various acid concentrations increased the volatile matter and fix carbon content. Leaching treatment with 3% nitric acid for 10 minutes resulted in the highest increase, reaching 72% volatile matter and 20% fix carbon content. This contributes to an increase in calorific value, combustion quality, and energy efficiency of EFB as an alternative fuel [22]. The increase in volatile matter and fix carbon content is due to the mineral content in biomass being washed out during the leaching process and the reduction in ash content after demineralization [11]. Statistical analysis results indicate that acid concentration does not significantly affect the increase in volatile matter and fix carbon content. However, soaking time significantly affects the increase in volatile matter and fix carbon content ($P \leq 0.05$). A soaking time of 10 minutes is an effective process for increasing volatile matter and fix carbon content.



(a) **(b)**

Figure 2. Effect of HNO_3 concentration and soaking time on (a) volatile matter and (b) fixed carbon in EFB

This increase in volatile matter content can have a positive impact on the ignition process and combustion rate. However, flame stability must also be considered, as the dominance of volatiles can cause fluctuations in the heterogeneous combustion phase and produce unburned gas residues, which can potentially increase emissions of pollutants such as CO and unburned hydrocarbons if oxidation conditions are not optimal [10].

3.3 Calorific Value

The results of the experiment showed that the calorific value ranged from 4295 to 4569 cal/g (Figure 3). The highest calorific value was found in EFB that was treated with 3% nitric acid for 10 minutes. The nitric acid leaching treatment increased the calorific value of EFB compared to untreated EFB. This is consistent with the increase in fix carbon content, which is the primary contributor to calorific value due to the high energy content in the carbon structure [23]. Demineralization with nitric acid can degrade alkali metal minerals, and the direct loss of mineral material contributes to the increase in calorific value [24]. Low ash content increases the calorific value. Ash content has a negative correlation with the calorific value [25]. Statistical analysis results indicate that acid concentration and soaking time do not significantly affect the calorific value but significantly affect the control value.

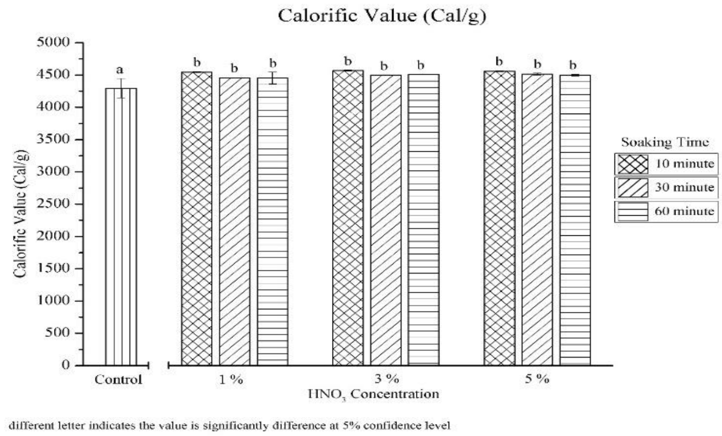


Figure 3. Effect of HNO_3 concentration and soaking time on the calorific value of EFB

3.4 Ultimate Analysis

The ultimate analysis of EFB fiber with and without treatment is shown in Table 1. The organic components of biomass are composed of various hydrocarbons, which are mostly made up of C and H atoms, and the molecular mass of C is much heavier than that of H [11]. In the control sample, the carbon content was recorded at 42.80%, while the demineralization treatment with nitric acid increased the carbon content to 46.50% at a nitric acid concentration of 3% for 10 minutes. Based on the data in Table 1, the leaching treatment with nitric acid solution tends to increase the carbon, hydrogen, and oxygen content. However, the increase in carbon is more dominant than that of oxygen and hydrogen. Therefore, even though the concentrations of O and H increase, their proportion relative to carbon becomes smaller. This is due to the presence of dissolved inorganic and alkali metal content, as well as the drying process after leaching, which can increase the proportion of carbon and hydrogen content [26]. The increase in carbon and hydrogen content is also correlated with the increase in the calorific value of EFB after treatment with nitric acid solution [27]. Soaking treatment with nitric acid solution can reduce sulfur content in EFB. In this study, the control sulfur content of 0.115% was successfully reduced to 0.086% after 1% nitric acid treatment for 60 minutes. This improves the quality of EFB fiber for use as low-emission fuel. During combustion, sulfur in biomass oxidizes into SOx, which can pollute the environment, and also contributes to the formation of corrosive ash, making sulfur undesirable in combustion [28].

Table 1. Ultimate analysis of demineralized EFB at varying HNO_3 concentrations and soaking Times

Sample	Carbon (C)	Hydrogen (H)	Nitrogen (N)	Oxygen (O)	Sulfur (S)
Control	42.80 (3.03)	5.65 (0.47)	1.52 (0.29)	39.36 (3.13)	0.120 (0.0006)
HNO_3 1%, 10 minute	46.00 (1.61)	5.76 (0.25)	1.55 (0.32)	40.79 (1.11)	0.092 (0.0011)
HNO_3 1%, 30 minute	42.80 (0.92)	5.49 (0.69)	1.58 (0.11)	44.38 (1.27)	0.091 (0.0007)
HNO_3 1%, 60 minute	45.73 (1.04)	5.81 (0.23)	1.58 (0.28)	41.05 (1.11)	0.086 (0.0004)
HNO_3 3%, 10 minute	46.50 (2.37)	5.92 (0.27)	1.66 (0.25)	40.40 (2.59)	0.087 (0.0007)
HNO_3 3%, 30 minute	45.15 (0.80)	5.78 (0.28)	1.87 (0.06)	41.44 (1.13)	0.095 (0.0016)
HNO_3 3%, 60 minute	45.62 (0.82)	5.84 (0.06)	1.58 (0.15)	41.13 (0.58)	0.087 (0.0006)
HNO_3 5%, 10 minute	46.04 (1.33)	5.87 (0.27)	1.76 (0.14)	40.45 (1.61)	0.089 (0.0024)
HNO_3 5%, 30 minute	45.63 (1.16)	5.81 (0.12)	1.41 (0.35)	41.22 (0.81)	0.088 (0.0007)
HNO_3 5%, 60 minute	45.74 (1.01)	5.85 (0.25)	1.70 (0.12)	40.81 (1.15)	0.087 (0.0007)

*Values in parentheses are standard deviations

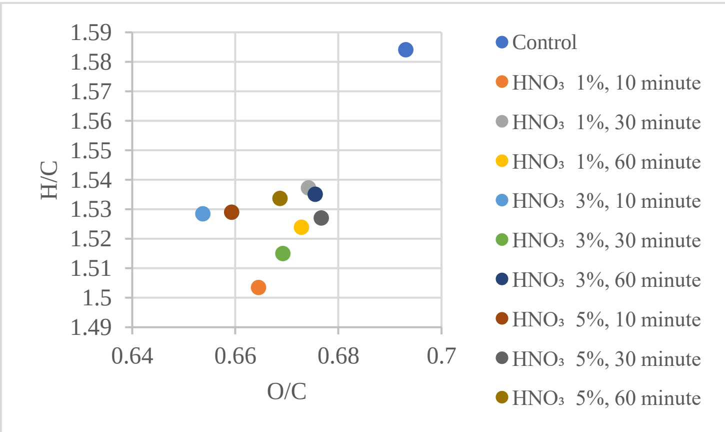


Figure 4. Van Krevelen diagram of demineralized EFB at varying HNO_3 concentrations and soaking times

Furthermore, the interpretation of the O/C and H/C molar ratios is used to assess the thermal stability and energy properties of biomass. The O/C ratio is negatively correlated with the calorific value, where a lower O/C ratio indicates that the biomass has a higher energy content (Figure 4) [29]. Based on the Van Krevelen graph, the control EFB has the highest O/C ratio (0.695) and H/C ratio (1.58), indicating high oxygen content and low energy stability. After demineralization treatment, the points on the graph shifted to the lower left, indicating a greater increase in the carbon fraction relative to H and O. Treatment with 1% nitric acid for 10 minutes showed an O/C ratio of 0.665 and an H/C ratio of 1.50, while treatment with 3% nitric acid for 10 minutes showed an O/C ratio of 0.653 and an H/C ratio of 1.52. Leaching in nitric acid causes the dissolution of alkali metal ions and the partial destruction of mineral bonds within the lignocellulose matrix. The loss of these minerals enriches the relative content of C atoms compared to H and O through decarboxylation and dehydration reactions during the demineralization process [30]. This causes the O/C and H/C ratios to decrease. The movement of these points indicates an improvement in the thermal quality of the biomass [31]. This ratio aligns with the findings of [32], who stated that biomass with O/C < 0.7 and H/C < 1.6 is suitable for energy applications such as high-quality pellets or briquettes.

3.5 Potassium content

The alkali content is very high in EFB raw materials (± 50%), making this biomass prone to fouling and slagging [33]. The high concentration of potassium in biomass fuel tend to result in easy formation of compounds with low melting points. Potassium content is important for indicating the potential for ash melting or ash deposition. Alkali metals and alkaline earth metals are present in organic structures. They are typically stored as water-soluble fractions, making them easily removable by water washing [21]. Figure 5 shows that the potassium content of untreated EFB is 0.087%. Leaching treatment at various acid concentrations significantly reduces potassium content. Leaching in 1% nitric acid for 30 minutes reduced the potassium content to the lowest level of 0.02%, representing a 77% decrease compared to the control. Nitric acid (HNO_3) converts potassium compounds that were originally insoluble into water-soluble nitrate salts. H^+ ions from HNO_3 replace alkali metal ions such as K^+ in the biomass structure through ion exchange and mineral dissolution mechanisms, resulting in water-soluble KNO_3 that is easily released and washed out of the biomass matrix[34]. At a concentration of 5% HNO_3, there was an increase in potassium content in the solid residue. This condition indicates that at high concentrations, the process is no longer dominated by mineral dissolution, but also by partial degradation of the lignocellulose structure and dissolution of organic components, causing significant biomass mass loss. The potassium content expressed in weight percent may increase due to the concentration effect. Thus, the use of HNO_3 at moderate concentrations is more effective in reducing potassium content than higher concentrations. This is consistent with the research by [11], which found that washing with strong acid solutions is effective for removing both water-soluble and water-insoluble ash and alkali metal content. The reduction in potassium content in EFB biopellets improves fuel quality by reducing the potential for slagging, fouling, and corrosion during the combustion process. In addition, low potassium content contributes to an increase in ash melting point and operational stability of the combustion system [13].

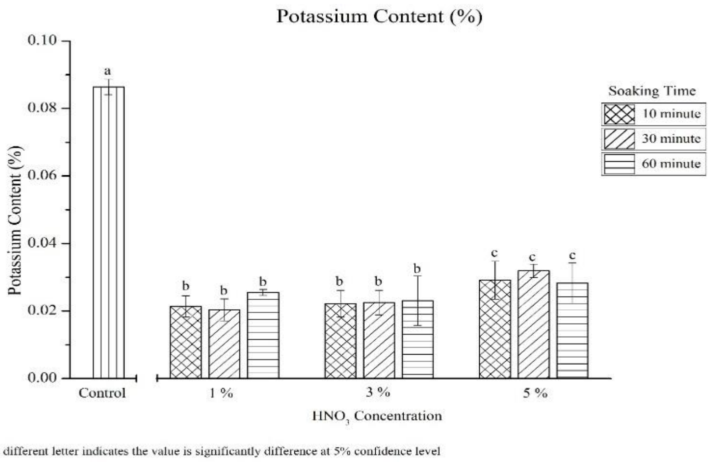


Figure 5. Effect of HNO_3 concentration and soaking time on the potassium content of EFB

3.6 Characteristics of Biopellets

The density of biopellets is an important parameter because it is directly related to combustion efficiency and ease of energy distribution. High-density solid fuel will ease storage but reduce the combustion rate due to smaller pores [35]. Densification pressure and temperature affect biopellet density, with density increasing as densification pressure and temperature increase [36]. According to SNI 8675-2018 standards, the minimum density required is 0.6 g/cm³ for household biopellets and 0.8 g/cm³ for industrial biopellets. Test results show that biopellets from EFB have a density of 0.93 g/cm³ in the control sample and increase slightly to 0.94 g/cm³ after treatment by immersion in a 1% nitric acid solution for 10 minutes. This value is above the minimum standard limit of SNI for both household and industrial use, thus meeting the quality requirements. High density can increase volumetric heat value and facilitate transportation and storage [37].

Moisture content plays an important role in determining the calorific value of biopellets. High moisture content can reduce combustion efficiency because some of the initial energy is used to evaporate water before the heat from combustion can be optimally utilized [38], [39]. According to SNI 8675-2018, the maximum permissible moisture content is 10% for household-scale biopellets and 12% for industrial-scale biopellets.

Table 2 shows that the moisture content of the control biopellets was 4%, while biopellets treated with 1% nitric acid for 10 minutes had a moisture content of 4.45%. Moisture content exceeding the SNI quality standard can cause biopellets to be difficult to burn, reduce combustion efficiency, and increase the risk of fungal growth due to damp conditions.

Table 2. Characteristics of EFB biopellets

Parameter	**Unit, min/max**	**SNI 8675-2018**		**Biopellet treatment**	
		Household	**Industry**	**Control**	**HNO_3 1%, 10 minute**
Density	g/cm3, min	0.6	0.8	$0.93_{(0.014)}$	$0.94_{(0.016)}$
Moisture content	%, max	10	12	$4.09_{(0.072)}$	$4.45_{(0.054)}$
Volatile matter	%, max	75	80	$71.87_{(0.306)}$	$74.02_{(0.159)}$
Ash content	%, max	5	5	$7.93_{(0.238)}$	$3.43_{(0.077)}$
Fix carbon	%, min	14	14	$16.11_{(0.103)}$	$18.09_{(0.201)}$
Calorific value	Cal/g, min	4000	4000	$4353.44_{(0.089)}$	$4355.83_{(0,215)}$

*Values in parentheses are standard deviations

In solid fuels, volatile matter content plays a crucial role in accelerating the combustion process of biopellets and stabilizing the flame [40]. A high volatile matter content generally indicates that the fuel is easier to ignite and requires lower ignition energy [41]. During combustion, the volatile components of biomass evaporate and are released as gases, such as hydrocarbons, alcohols, aldehydes, and organic acids [42]. These components contribute to heat generation, influence combustion rates, and affect the formation of emissions (e.g., particulates and nitrogen oxides) and flame stability, ultimately impacting combustion efficiency.

Based on test results, the control EFB biopellets had a volatile matter content of ±71% and a fixed carbon content of ±16%. After demineralization using a 1% nitric acid solution for 10 minutes, the volatile matter content increased to ±74% and the fixed carbon content to ±18%. These values meet the requirements of SNI 8675–2018, which are a volatile matter content <80% and fixed carbon content >14%. Pre-treatment with nitric acid was found to enhance energy distribution by reducing non-energetic components (ash) and enriching the volatile and fixed carbon fractions. The increase in fix carbon is influenced by the decrease in ash content and the increase in the relative proportion of carbon after demineralization. Higher bound carbon content indicates greater calorific value potential and more stable combustion, which are important characteristics for high-quality solid fuel [43].

High ash content in biopellets can reduce combustion efficiency because the heat generated is partially absorbed by the ash layer, and it can also cause deposits to form in the furnace, increasing the risk of corrosion [44]. Test results show that untreated EFB biopellets have an ash content of 8%, while biopellets whose raw materials were soaked in a 1% nitric acid solution for 10 minutes showed a decrease in ash content to 3.4%. This value is below the maximum ash content limit set in SNI 8675-2018, which is 5%. This reduction in ash content indicates the effectiveness of nitric acid treatment in dissolving inorganic minerals, such as potassium, sodium, calcium, and magnesium, which are the main components of ash in lignocellulosic biomass [11], [19]. Lower ash content contributes to improved combustion quality, reduced air pollutant emissions, and prevention of slagging and fouling in thermal systems [45].

Calorific value is a key factor in the success of biopellet production as an alternative fuel. The higher the calorific value of a biopellet, the better its combustion efficiency. The results of this study indicate that EFB biopellets have a calorific value of 18.227 MJ/kg under control conditions and increase slightly to 18.237 MJ/kg after demineralization treatment with 1% nitric acid for 10 minutes. Both values are above the SNI 8675-2018 standard, which requires a minimum calorific value of 16.5 MJ/kg, so it can be concluded that EFB-based biopellets have sufficient energy quality for domestic and industrial applications. Theoretically, this increase in calorific value is due to the high fixed carbon content in the solid fuel [46]. The increase in fixed carbon content in the solid fuel is caused by the reduction in moisture and inorganic mineral (ash) content during the demineralization process [24].

Analysis results show that the demineralization treatment using a 1% HNO_3 solution for 10 minutes not only significantly reduces ash content but also increases the calorific value of biopellets. The reduction in ash content decreases the non-energetic fraction in the raw material, thereby increasing the proportion of fix carbon and enabling more optimal utilization of combustion energy. This higher calorific value supports combustion efficiency and improves the overall quality of the biopellets. In general, the treated biopellets meet most of the quality parameters of SNI 8675:2018, both for household and industrial scales. These findings confirm that acid leaching with nitric acid is an effective pre-treatment strategy to improve the quality of biopellets from EFB waste and has the potential to expand their use as a lignocellulosic-based bioenergy source.

4. Conclusions

This research showed that EFB demineralization using HNO_3 solution effectively reduced ash and potassium content, while improving the thermal quality and combustion characteristics of biopellets. Optimal treatment was achieved at an HNO_3 concentration of 1% with a soaking time of 10 minutes, which reduced ash content to 3.43–5.45%, decreased potassium content by 77% from 0.087% to 0.02%, and increased calorific value from 4295 cal/g to 4546.26 cal/g. This treatment also increased the fix carbon and hydrogen content, and decreased sulfur content, thereby potentially reducing emissions and corrosion risks. The characteristics of the treated biopellets meet most of the quality requirements of SNI 8675-2018 for household and industrial use. Thus, acid leaching using low-concentration HNO_3 can be recommended as an economical and environmentally friendly pre-treatment strategy to improve the quality of EFB-based biopellets and reduce the potential for slagging and fouling issues in biomass combustion applications.

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Conflicts of Interest: The authors declare no conflict of interest.

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