

RESEARCH ARTICLE

Investigating the role of oxygen and residence time in the torrefaction of empty fruit bunch

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ABSTRACT - This research investigates the oxidative torrefaction of Empty Fruit Bunch (EFB) as a renewable energy source. EFB samples from the LCSB palm oil mill were torrefied at 250°C with residence times (RT) of 10, 20, and 30 minutes and oxygen concentrations (OC) of 0, 5, 15, and 21%. Each experiment was repeated three times. Visual observations indicated that the EFB colour darkens with increasing RT, particularly at 5% OC, where torrefaction was found to be optimal. Higher OCs of 15% and 21% led to combustion but were ineffective due to insufficient incineration. Mass yield decreased with increasing RT due to moisture and volatile loss. The torrefaction severity index (TSI) showed a linear increase in the enhancement factor with increasing TSI, while energy yield decreased as the enhancement factor exceeded mass yield. The optimal condition for torrefaction was identified as 5% OC and 30 minutes RT, balancing energy efficiency and mass retention.

INTRODUCTION 1.0

Renewable energy is crucial for overcoming climate change and decreasing dependence on fossil fuels. Biomass is a versatile and abundant natural resource that can replace fossil fuels and reduce greenhouse gas emissions [1]. Oil palm, a significant commodity in Malaysia, supports the country's economy and agriculture. During palm oil production, various types of biomass waste are generated, including empty fruit bunches (EFB), mesocarp fibres, trunks, palm kernel shells, and oil palm fronds [2]. EFB is a particularly promising feedstock for biofuels in the global energy market [3]. Previously, EFB was burned and turned into ash for fertilizer, causing uncontrolled incineration and air pollution which has made the Environmental Quality Act of 1974 ban this method [4]. The Malaysian government aims to increase renewable energy production and emphasizes sustainability in palm oil production. The estimated biomass production from palm oil mills for EFB from fresh fruit bunches (FFB) can be up to 25%, depending on process efficiency [5]. Malaysia produces approximately 168 million tons of oil palm biomass per year, with oil palm waste accounting for 94% of the biomass feedstock. By 2020, solid oil palm biomass was expected to increase to 85-110 million tons [6], indicating a growing abundance of EFB. However, the properties of raw EFB, such as high moisture and oxygen content, low heating value, low energy density, low grindability, and irregular size and shape, make it unsuitable for direct use as fuel [7][8]. Thus, raw biomass needs to undergo a pretreatment process to improve its properties as a fuel.

Torrefaction is a pretreatment process that enhances biomass properties for use as fuel. In torrefaction, organic material is degraded at temperatures ranging from 200–300°C in a nitrogen or inert environment [9]. This thermochemical process improves biomass by eliminating volatile matters, reducing moisture content [12, 13], increasing the higher heating value (HHV) [12] and enhancing the energy density [13]. There are different types of torrefaction, including dry torrefaction, wet torrefaction, and steam torrefaction [14]. Dry torrefaction can be further divided into inert torrefaction and oxidative torrefaction. In inert torrefaction, nitrogen is used as the carrier gas, while in oxidative torrefaction, oxygen [15] or flue gas [16] is used. Several parameters influence the torrefaction process, such as temperature, residence time, heating rate, moisture content, and oxygen concentrations in oxidative torrefaction [17]. Torrefaction has been extensively explored in previous research. For example, a study by Jifara & Mekuria [18] investigated the inert torrefaction of corncob and khat stem at temperatures of 200, 250, and 300°C for residence times of 15, 30, and 45 minutes. They found that the optimal conditions for the highest energy yield from corncobs were 300°C for 30 minutes, resulting in a 98.5% energy yield. For khat stems, the best conditions were 300°C for 45 minutes, yielding a 94.9% energy yield.

Sui et al. [19] focused on the oxidative torrefaction of cotton stalks at 220-330°C with oxygen concentrations ranging from 0-21%. The results indicated that the optimized conditions for producing torrefied products as fuel are at temperatures of 260-270°C with 2-3% oxygen concentrations. Soh et al. [20] investigated the impact of wet torrefaction using a conventional acid digestion vessel on EFB and oil palm trunks (OPT) at temperatures of 180, 210, and 240°C with residence times of 2.0, 2.5, and 3.0 hours. The study found that the mass yields of both samples decreased with increasing temperature and residence time due to the degradation of hemicellulose and cellulose [21]. Despite extensive studies on the oxidative torrefaction of various biomass types such as densified woody biomass [22], bamboo processing residues [23], spent coffee grounds [15] and corn stalks [24], relatively little research has focused on oil palm wastes. For example, Uemura et al. [25] studied the oxidative torrefaction of EFB, but their research focused solely on the effect of

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temperature. They did not report on the impact of oxygen concentrations, did not analyze the process based on the torrefaction severity index (TSI), and consistently fixed the duration of the process at 30 minutes. Similarly, Zhang et al. [26] focused on the effect of torrefaction on TSI but used a different type of biomass. Consequently, there is no information on how oxygen concentrations and residence time affect the torrefaction process of EFB, nor is there information on the TSI analysis of EFB.

Thus, the objective of this research is to study the oxidative torrefaction of EFB as a renewable energy source by examining the effects of oxygen concentration (0, 5, 15, and 21%) and residence time (10, 20, and 30 minutes) on the mass and energy yield of EFB. Additionally, a TSI analysis on the torrefied EFB samples was conducted.

2.0 MATERIALS AND METHOD

2.1 Materials

The main materials for this study were palm oil empty fruit bunch which was obtained from LCSB Lepar Mill. The oxygen and nitrogen gases were purchased from Azam Synergy Sdn. Bhd. and Air Products Malaysia Sdn. Bhd., respectively. Pre-treatment was done by drying the samples inside an oven at a temperature of 105°C for 24 hours until the mass of the samples was constant. The samples were then grounded and sieved into the size of 500 µm.

2.2 Torrefaction

Before the samples were torrefied, approximately 5g of each sample was weighed and put inside the inner tubular reactor. A schematic diagram of the setup for the torrefaction experiment is shown in **Error! Reference source not f ound.** Oxygen and nitrogen concentrations were set, and the heater for the torrefaction reactor was switched on. As the temperature approached 250° C, the timer was started. Once the residence time was reached, the gas valve was shut, and the heater was switched off. The torrefaction reactor was allowed to cool down to 40° C. In this experiment, the torrefaction process was conducted with residence times varying from 10, 20, and 30 minutes, and oxygen concentrations of 0%, 5%, 15%, and 21%. A 0% oxygen concentration indicates non-oxidative torrefaction, which was performed for comparison with oxidative torrefaction.



Figure 1. Flow process diagram of methanolic process of bioactive molecules

2.3 Mass Yield

Mass yield is done to determine how much useful product is obtained after the torrefaction is done. The masses of raw and torrefied samples were measured using an analytical weighing balance. The samples were weighed as soon as the samples were taken out of the inner tubular reactor to minimise exposure to moisture in the air. After being weighed, the samples were immediately preserved. Mass yield is determined using Eqn. (1) [27]:

Mass Yield,
$$M_Y$$
 (wt%)= $\left(\frac{m_{torr}}{m_{raw}}\right) \times 100\%$ (1)

where m_{torr} is the mass of the torrefied sample and m_{raw} is the mass of the raw sample, both in g.

2.3 Torrefaction Performance and Severity

Torrefaction severity index (TSI) is defined as the ratio of the biomass' weight loss at the torrefaction condition to the weight loss at the maximum condition [28]. TSI is a useful method for determining the quality of torrefied products and assessing the extent of thermal degradation during the torrefaction process [31, 32]. TSI is calculated using Eqn. (2) [31] as follows:

Torrefaction severity index (TSI) =
$$\frac{100 - MY(t_{OC})}{100 - MY(t_{C})_{OC}}$$
(2)

where $MY(t)_{OC}$ is the mass yield at a residence time and oxygen concentration, $MY(t_c)_{oc,c}$ is the mass yield for severe conditions. Enhancement factors are also analysed to assess the performance of the torrefaction process by reflecting the change in HHV during the torrefaction process [32].

3.0 RESULTS AND DISCUSSION

3.1 Visual Observation

The colour changes of EFB is an intuitive indicator that changes when the feedstock is thermally degraded [14]. Figure 2 shows the samples under different conditions of torrefaction. Based on Figure 2, the colour of the biomass becomes darker as the residence time of 10, 20 and 30 minutes increases due to devolatilization, an increase in carbon content, the decomposition of hemicellulose, and the reduction in moisture content during torrefaction [33]. Similar trends were reported by Wulandari et al. [34], who observed that the colour of rubberwood turned from raw brown to dark brown with increasing residence time. Orisaleye et al. [35] also found that the highest residence time has turned corncobs to a black colour. The presence of O₂ in an oxidizing atmosphere accelerates the decomposition of the samples, resulting in a higher decomposition rate and superior physical characteristics of the torrefied sample compared to those observed under inert conditions [36]. However, at a residence time of 30 minutes, the colour rank is as follows: 0% < 21% < 15% < 5%, with 5% oxygen concentration showing the darkest colour. This is because at oxygen concentrations of 15% and 21%, the biomass undergoes combustion rather than torrefaction [37]. However, because the process operates at low temperatures, it is ineffective due to inadequate incineration [38]. The torrefaction process, particularly at 5% oxygen concentration, effectively enhances the biomass properties. At this optimal concentration of oxygen, the process promotes significant thermal decomposition [39] and carbonization [40], leading to a darker colour and improved fuel characteristics without reaching combustion temperatures. Similar results were observed by Xi et al. [41] where Chinese fir residues exhibited a darker colour across oxygen concentrations ranging from 0% to 6%.



Figure 2. Samples under different conditions

3.2 Mass Yield

Figure 3 shows the mass yield at various oxygen concentrations and residence times respectively. From Figure 3, the mass yield decreases as the residence time (RT) increases. For 0% oxygen concentration (OC), the mass yield shows a small drop from 95.91% at 10 minutes RT to 93.87% at 30 minutes RT. For 5% OC, the mass yield exhibits a significant drop from 95.01% at 10 minutes RT to 68.63% at 30 minutes RT. Meanwhile, for 15% OC, the mass yield decreases from 95.30% at 10 minutes RT to 82.84% at 30 minutes RT. For 21% OC, the mass yield drops steadily from 94.53% at 10 minutes RT to 88.86% at 30 minutes RT. For 21% OC, the mass yield drops steadily from 94.53% at 10 minutes RT to 88.86% at 30 minutes RT. These results highlight the effectiveness of torrefaction at different oxygen concentrations and residence times. Specifically, the substantial decrease in mass yield at 5% OC and 30 minutes RT indicates efficient removal of volatiles and moisture, leading to a more energy-dense fuel [42]. Cao et. al [23] also compared the oxygen level of 0-15% on the oxidative torrefaction of bamboo processing residue and found that 5% of OC was the best condition for the formation of alcohol. This demonstrates the potential of torrefaction, particularly at optimal conditions, to significantly enhance the properties of biomass as a renewable energy source.

Figure 4 shows the mass yield at various residence times (RT). Based on Figure 4, for the RT of 10 minutes, there is no significant difference in mass yield across the various O2 concentrations. The mass yield remains consistent, ranging from 94.53% to 95.91%. Meanwhile, for RT 20 minutes, the mass yield drops from OC 0% to OC 15% and increases for OC 21%. Lastly, for RT 30 minutes, the OC drops significantly from OC 0% to OC 5% and increases gradually. The decrease in mass yields is caused by the removal of moisture and the release of volatile compounds [43]. When oxygen is present, using an oxidizing atmosphere instead of an inert one accelerates the decomposition of the samples, as evidenced by the increased decomposition rate values [36]. In this study, the optimum OC in oxidative torrefaction is less than 15%, resulting in small drops in mass yields for OC 15% and 21%. These findings highlight the effectiveness of oxidative torrefaction in improving biomass properties. The controlled removal of moisture and volatiles at specific conditions enhances the energy density of the biomass, making it a more efficient renewable energy source.



Figure 3. Mass yield at various OC



Figure 4. Mass yield at various residence time

3.3 Torrefaction Performance and Severity

Studies have shown that the Torrefaction Severity Index (TSI) can be linearly correlated with fundamental properties and energy performance, particularly in lignocellulosic materials [44]. According to Figure 4, the maximum weight loss recorded in this experiment is 31.51%, which occurred under torrefaction conditions of 5% oxygen concentration and a residence time of 30 minutes. Therefore, this is taken as a critical condition in evaluating TSI as shown in Figures 5 and 6.



Figure 5. Distribution and regression of enhancement factor vs TSI



Figure 6. Distribution and Regression of Energy Yield vs TSI

Figure 5 shows a relationship of y=0.0776x + 1.0196 with regression of $R^2 = 0.9899$. This shows that the enhancement factor increases linearly with increasing TSI. However, for 5% oxygen concentration with 30 minutes RT, the enhancement factor is the highest due to the high TSI value. This is because the condition shows the highest mass yield drop of 33.58% whereas the other condition only shows a mass yield drop in the range of 0.30% to 10.40%. This shows that at this condition, the physical quantity is sensitive to TSI. Figure 4.5 shows the distribution and regression of energy yield vs TSI.

The regression lines of energy yield with negative slopes imply that the decrease in mass yield exceeds the increase in the enhancement factor. Based on Figure 6, the relationship between energy yield and TSI is y = -9.5491x + 102.41

with a regression of $R^2 = 1$. The enhancement factor and energy yield correlate well, as the regression is more than 0.9899. Although the majority of the conditions show more than 95% of energy yield, when the TSI is less than 1.1, the energy densification is reduced even though the mass yield is high, indicating that the energy densification is inadequate [45]. Therefore, three conditions have TSI > 1.1: oxygen concentration 5% (30 minutes), 15% (20 minutes), and 15% (30 minutes). However, since an oxygen concentration of 15% may lead to biomass combustion, a 5% oxygen concentration and 30 minutes RT are optimal.

4.0 CONCLUSION

The present study on the oxidative torrefaction of palm oil EFB may be useful for analysing the optimum conditions for torrefying this biomass. Based on visual observation, the biomass appears darker as the residence time increases. The changes in colour in the biomass are due to changes in chemical composition, as the decomposition of hemicellulose generates various by-products. In conclusion, considering the mass yield and TSI of the biomass, it is found that a 5% oxygen concentration and a 30-minute residence time represent the optimum conditions for oxidative torrefaction. However, more studies are needed to find the best oxygen concentration below 15%, focusing more on the effects of torrefaction. Additionally, the influence of temperature needs to be investigated in future studies, as it can affect the type of torrefaction and thus impact the TSI value.

CONFLICT OF INTERESTS

We declare no conflict of interest.

AUTHORS CONTRIBUTION

M.K.A Mohd Fauzi (Investigation, Writing - review and editing, Data curation)

G.R. Hernaez (Investigation, Writing - original draft, Methodology, Visualization)

R. Abdul Rasid (Funding Acquisition, Writing - review and editing, Project Administration, Supervision)

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