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Torrefaction of Hydrotreated Palm Kernel Shell and Mesocarp Fiber Mixture

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ABSTRACT

This paper examines the torrefaction of a hydrotreated mixture of palm shell and mesocarp fiber and its physicochemical and thermal properties after torrefaction. The weight ratio was fixed at 60:40 (palm shell to mesocarp fiber) for the untreated and hydrotreated mixture of palm shell and mesocarp fiber. The untreated and hydrotreated mixture was subsequently torrefied from 250 to 300°C. The results revealed that the physical appearance of the untreated and hydrotreated mixture darkened after torrefaction. Higher torrefaction temperature increased the mass loss, fixed carbon, ash and heating value of torrefied products. The ash content of torrefied hydrotreated mixture decreased by 42% (maximum) if compared to the torrefied untreated mixture. Likewise, the heating value of the torrefied hydrotreated mixture (23 MJ/kg) was higher than the untreated mixture. However, the moisture content and volatile matter gradually decreased with increasing torrefaction temperature. The mass loss and volatile matter of the torrefied hydrotreated mixture were considerably improved compared to the torrefied untreated mixture. Overall, the results showed that hydrotreatment resulted in a promising torrefaction end-products. Therefore, combined hydrotreatment and torrefaction is a practical and promising technique for producing low-ash oil palm biomass.

Keywords: *Oil palm shell, mesocarp fiber, hydrotreatment, torrefaction, ash, heating value*

1.0 INTRODUCTION

The large consumption of rapidly depleting fossil fuels has caused serious damage to ecosystems and reserves worldwide. Great efforts to reduce these issues such as the widespread development and deployment of renewable energy technologies (RETs) have shown outstanding results [1]. Generally, RETs are derived from abundant sources such as hydro, biomass, wind, and solar. In Malaysia, oil palm cultivation generates tremendous biomass resources amounting to 25.64 million tonnes based on data from 2016 [2]. According to Loh [3], only 10% of oil is extracted from oil palm while 90% is biomass waste.

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Typically, the biomass commonly extracted include palm kernel shell (PKS), palm mesocarp fiber (PMF), and empty fruit bunch (EFB) [2]. Interestingly, oil palm biomass has the potential for wide applications, such as manufacturing of furniture [2], fertilizer [4] and energy [5]. PKS and PMF are reportedly used as raw material for power generation due to favourable combustion performance [5]. However, the performance of raw PKS and PMF for energy production is prone to operational problems [2, 6]. This is due to several factors such as high ash and moisture content, as well as low heating value and low energy density [6]. EFB, on the other hand, is considerably underutilized, despite its potential for power generation after pre-treatment [6–9]. For the case of PKS and PMF, there are few treatments that offer a promising end-product for energy production such as hydrotreatment (water washing) and torrefaction. The study on torrefaction is considerably established and has received significant interest among researchers compared to hydrotreatment.

Likewise, the presence of alkali and alkaline earth metals (AAEMs) in biomass affects its solid fuel properties and conversion efficiency in energy production systems [10]. Potassium (K), sodium (Na) and chloride (Cl) are the major AAEM compounds that contribute to high ash content, slagging, fouling and corrosion at high temperatures [10, 11]. In large part, these issues are inimical to energy recovery from solid biomass fuel. However, hydrotreatment has a potential to reduce the AAEMs from biomass [11, 12] as demonstrated by some researchers in the literature [10–13]. Hydrotreatment was found to effectively decrease the ash content of biomass by 20 to 75% through spraying, soaking, and stirring [10, 12, 14–17]. Hence, the removal of AAEMs slightly increased the high heating value of the biomass after hydrotreatment [14–16].

Torrefaction is a considerably effective treatment for enhancing the properties of raw biomass [2, 6]. Torrefaction typically occurs under an inert or oxidative atmosphere and mild temperatures from 200 to 300°C for 15 to 60 minutes [2, 6, 7, 18, 19]. The products of torrefaction typically consist of torrefied biomass, bio-oil and gases [2]. Generally, torrefaction degrades the lignocellulosic biomass components resulting in a promising end-product with coal-like or wood properties [2]. These include a considerably high energy density and higher heating value (HHV, MJ/kg) along with improved grindability and hydrophobicity [2, 6, 18, 19]. The outlined properties are largely influenced by the torrefaction parameters; temperature, residence time and type of biomass [6]. The torrefaction of oil palm biomass such as EFB, PKS and PMF was firstly reported by Uemura *et al.* [6]. The torrefied oil palm biomass reportedly revealed high mass loss, mass yield, and HHV of 52-77%, 56-96%, and 18-22 MJ/kg, respectively.

The review of literature indicates torrefaction is a practical treatment technique for improving raw biomass properties and generating quality end-products for energy production. However, comprehensive studies on the hydrotreatment, torrefaction, and fuel characteristics of (washed or hydrotreated) PKS and MSF is still limited in the literature. Therefore, this study seeks to investigate the torrefaction of hydrotreated mixture of PKS and PMF at selected operating conditions. In addition, the fuel characteristics of the untreated and hydrotreated torrefied mixtures such as mass loss, HHV, moisture, volatile matter and ash are presented.

2.0 MATERIALS AND METHODS

2.1 Materials

The palm kernel shell (PKS) and palm mesocarp fiber (PMF) residues were collected from a near palm oil mill in southern part of Johor. The residues were subsequently grounded and sieved into smaller particles below than 500 µm [19]. Next, the residues were mixed according to the weight ratio of 60:40 for PKS:PMF. The mixing was performed using a homogenizer shaker (Model: RETSCH mixer mill MM 400, Germany) at 160 rpm for two hours.

2.2 Hydrotreatment Procedure

Figure 1 shows the experimental set up for hydrotreatment and filtration process adopted in this study. The mixture of PKS and PMF was stirred homogeneously in distilled water at constant stirring speed, time, and temperature at 360 rpm, 2 hours and 90°C, respectively [12, 14]. At the designated conditions, the sediment was left to cool under an ambient condition and subsequently filtered by vacuum filtration method as shown in Figure 1(b). Finally, the hydrotreated mixture of PKS and PMF (residue) was oven dried at a constant temperature of 105°C for 8 hours. The dried hydrotreated mixture of PKS and PMF were subsequently placed in a desiccator prior to the torrefaction. The weight of the dried residue before torrefaction was 8 g.

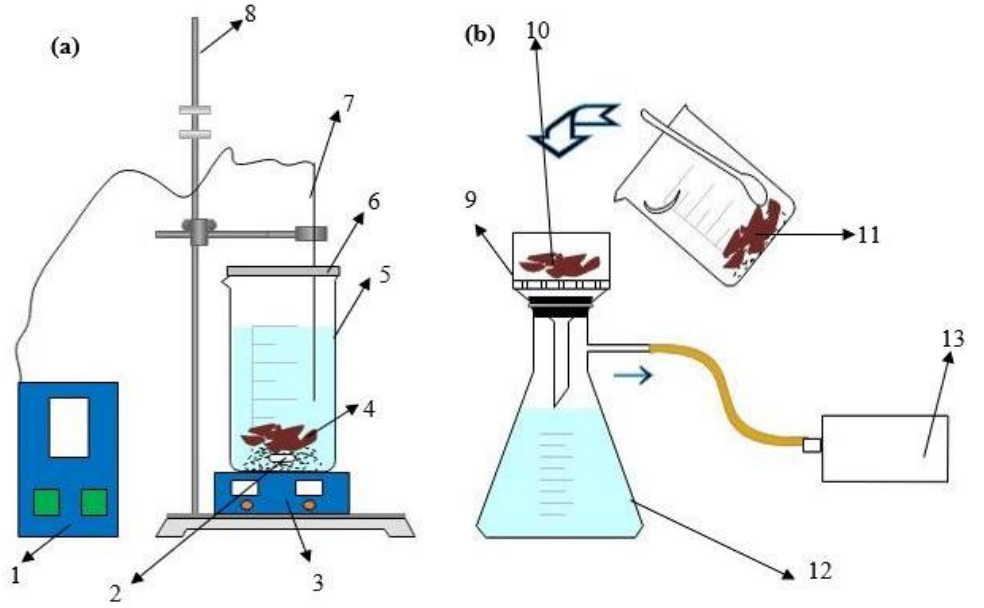

Figure 1: (a) Hydrotreatment experimental set-up and (b) filtration process: (1) PID temperature controller; (2) magnetic stirrer bar; (3) magnetic hot plate; (4) sediment; (5) beaker; (6) metal sheath lid; (7) K-type thermocouple; (8) retort stand; (9) *Büchner* funnel; (10) residue; (11) sediment; (12) conical flask; (13) vacuum pump [12]

2.3 Torrefaction Set-up and Procedures

The experimental set up for torrefaction is presented in Figure 2. Next, the dried residue was carefully loaded into a stainless-steel crucible and 1 L/min of nitrogen was subsequently used to purge the reactor. The purging was conducted of about one hour to create an inert environment. Once the purging has been completed, the heater of the reactor was set to 250°C and immediately turned on to perform torrefaction for 30 minutes at the designated temperature. After the completion of torrefaction treatment, the heater temperature was set to 60°C to allow the torrefied product to cool down and subsequently was removed from the reactor. Finally, the torrefied product was placed inside a desiccator before further analysis. The torrefaction process was repeated at 275°C and 300°C. The torrefaction procedures were adopted from Fuad *et al.* [18].

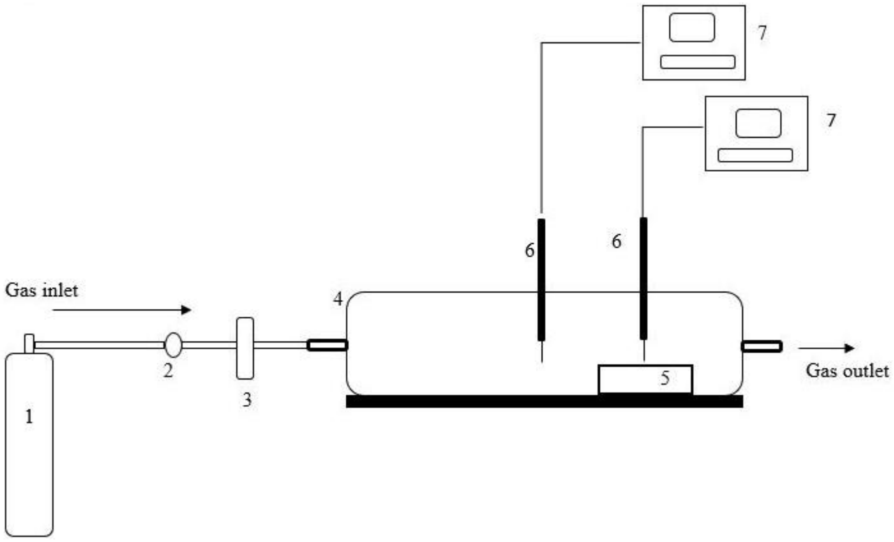

Figure 2: Schematic diagram for the torrefaction experimental setup: (1) Nitrogen gas cylinder; (2) pressure regulator; (3) flow rate meter; (4) tubular reactor; (5) stainless steel crucible; (6) K type thermocouple; (7) PID temperature controller [20]

2.4 Products Analysis

The physical and combustion properties of the torrefied samples were subsequently analysed. The physical properties consisted of physical appearance and mass loss whereas the combustion properties consisted of moisture, volatile matter, ash, fixed carbon and heating value. The mass loss of the hydrotreated mixture of PKS and PMF was calculated from the Bridgeman *et al*. [21] relation as shown in Equation (1).

$$\text{Mass loss (g)} = (\text{mass of mixture before torrefaction} - \text{mass of mixture after torrefaction})] / \text{mass of mixture before torrefaction} \quad (1)$$

The proximate analysis was performed and presented in dry basis according to the American Society for Testing and Materials (ASTM) standards D3173 (moisture content) [22], D3174 (ash content) [23], D3175 (volatile matter) [24]. The fixed carbon was determined by difference as stated in Equation (2). The high heating value (HHV) was determined by using *Isoperibol* bomb calorimeter (LECO, AC 350). All the results were duplicated and reported on average.

$$\text{Fixed carbon} = 100 - (\text{Moisture content \%} + \text{ash content \%} + \text{volatile matter \%}) \quad (2)$$

3.0 RESULTS AND DISCUSSION

3.1 Physical Appearance

Table 1 shows the physical appearance of the unwashed mixture of PKS and PMF before and after torrefaction. As observed, the color of the sample changed from brownish to darker shades at higher torrefaction temperatures [6]. Similar changes were observed for hydrotreated or washed samples as shown in Table 2. The change in color of the PKS and PMF mixture during torrefaction is due to the increase in fixed carbon and carbon content [25, 26].

Table 1: Appearance of unwashed PKS-PMF mixtures before and after torrefaction

Torrefaction of Unwashed PKS and PMF mixture			
Torrefied Temperature	250°C	275°C	300°C
Before Torrefaction			
After Torrefaction			

Table 2: Appearance of washed PKS-PMF mixtures before and after torrefaction

Torrefaction of Washed PKS and PMF mixture			
Torrefied Temperature	250°C	275°C	300°C
Before Torrefaction			
After Torrefaction			

3.2 Mass Loss

Figure 3 shows the mass loss of the untreated and hydrotreated PKS and PMF mixtures gradually increased with increasing torrefaction temperature from 250 to 300°C.

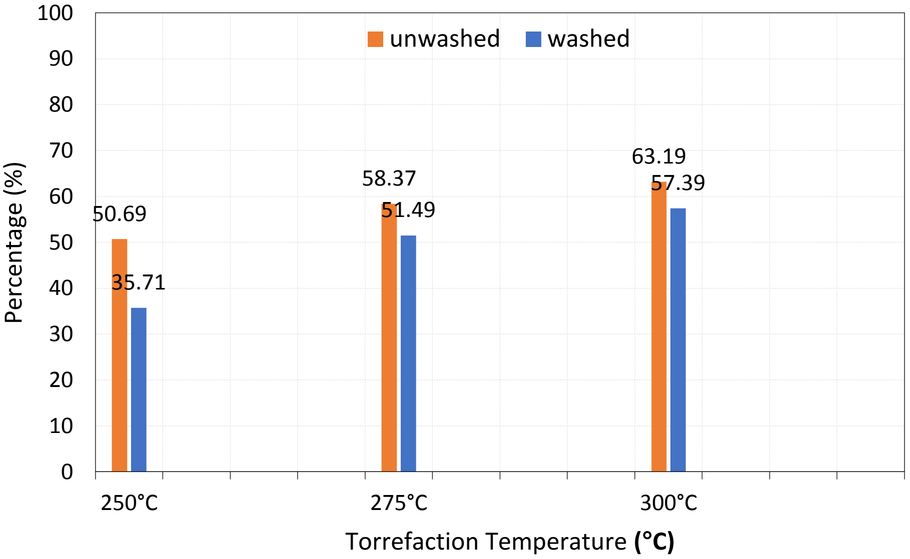

Figure 3: Mass loss of untreated and hydrotreated PKS and PMF mixture after torrefaction

The mass loss occurred due to the massive degradation of hemicellulose, cellulose and some lignin [18]. Therefore, this, in turn, resulting in the conversion and release of volatile matter [27]. The effect of washing is evident in the marginal mass loss of the hydrotreated (washed) mixture of PKS and PMF if compared to the untreated (unwashed) mixture after torrefaction. The removal of major AAEMs from biomass may have hampered significant mass loss in the hydrotreated PKS and PMF mixture after torrefaction. This is in good agreement with Cen *et al*. [28].

The study by Cen *et al*. examined the effect of combined water washing and torrefaction of corn stalk through thermogravimetric analysis (TGA) [28]. The study observed that the removal AAEMs delayed pyrolysis reaction along with the initial decomposition temperature, maximum loss rate, shoulder peaks and residual mass [28]. This phenomenon is supported by researchers who posit that AAEMs such as potassium act as a catalyst thereby enhancing the significant degradation during pyrolysis [29] and combustion [30].

3.3 Heating Value

Figure 4 presents the higher heating value (HHV) of the raw (untreated) and the torrefied hydrotreated PKS and PMF mixture after torrefaction at 300°C.

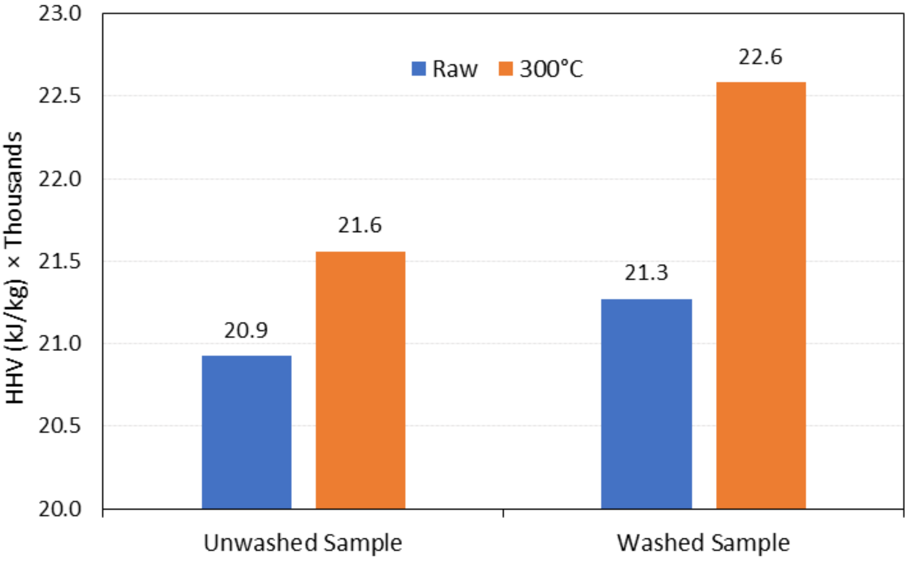

Figure 4: HHV of raw and torrefied hydrotreated PKS and PMF mixture

The results indicate that the HHV (22.6 MJ/kg) of the torrefied hydrotreated mixture of PKS and PMF is significantly higher than the torrefied raw (untreated) PKS and PMF mixture. The effect of AAEMs removal during hydrotreatment may be responsible for the marginal increase in HHV of the torrefied hydrotreated mixture of PKS and PMF. This is in good agreement with other studies reported in the literature [15, 31]. Interestingly, the HHV of a hydrotreated mixture of PKS and PMF is massively increased after torrefaction at 300°C.

3.4 Proximate Analysis

Figures 5 and 6 show the proximate analysis of the raw and the hydrotreated mixture of PKS and PMF, respectively. Torrefaction of the untreated and hydrotreated mixture of PKS and PMF decreased the volatile matter (74% to 39% and 74 to 44%, respectively). Interestingly, the volatile matter for hydrotreated is considerably higher than the hydrotreated variant. This finding confirms that hydrotreatment effectively removed some major AAEMs that act as a catalyst [28, 30, 32]. Besides, the torrefaction process also caused a massive release of volatile matter in the un-hydrotreated mixtures of PKS and PMF due to lignocellulosic degradation [18, 26]. However, the other properties of untreated and hydrotreated mixtures such as ash (4 to 10% and 3 to 6%, respectively) and fixed carbon (18 to 46% and 15 to 44%, respectively) gradually increased at higher temperatures. Besides, torrefaction has also played an important role in improving the properties of a hydrotreated mixture of PKS and PMF compared to the untreated mixture.

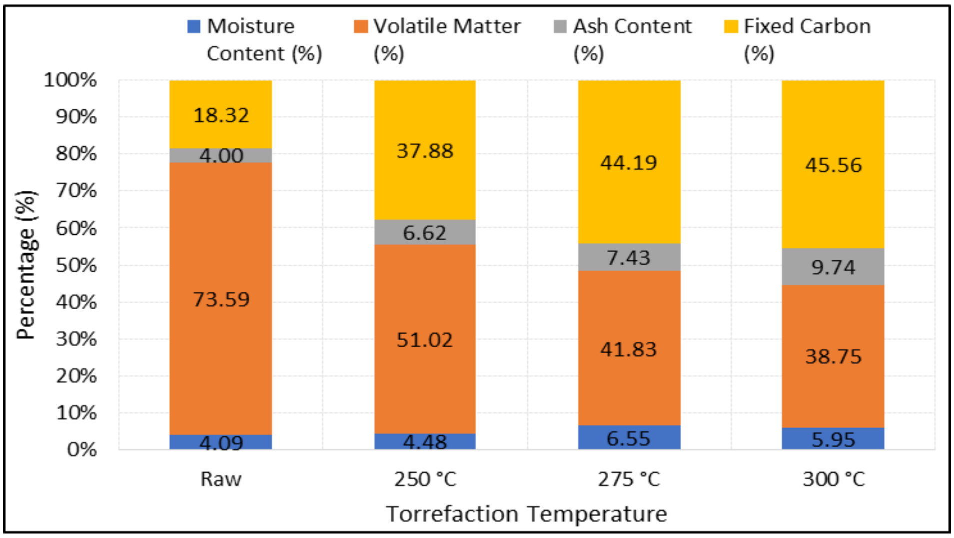

Figure 5: Proximate analysis of the untreated PKS and PMF mixture

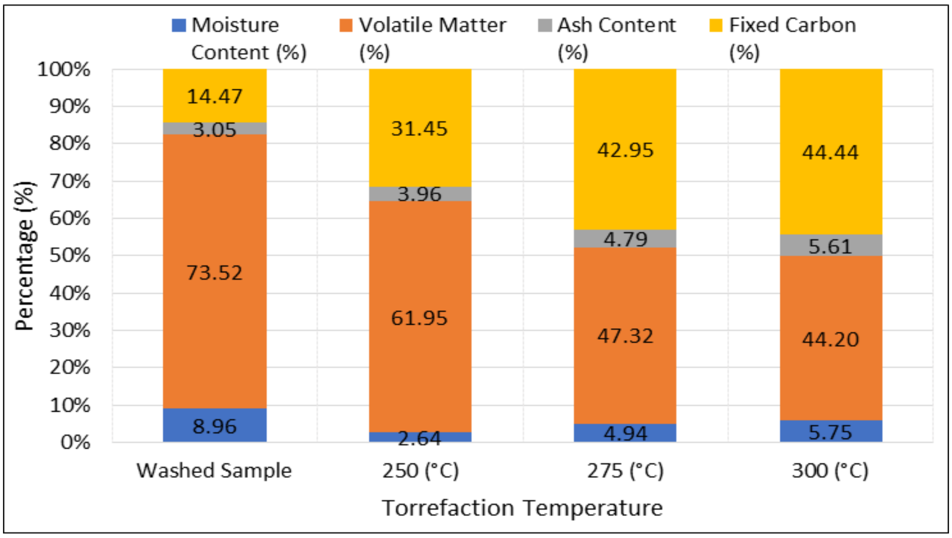

Figure 6: Proximate analysis of the hydrotreated PKS and PMF mixture

Lastly, the ash content of the hydrotreated mixture of PKS and PMF for all torrefaction conditions are improved if compared to the untreated mixture which has not been reported in any other study. Interestingly, the hydrotreatment of the biomass results in a positive impact by producing low-ash torrefied solid biomass fuel. Therefore, hydrotreatment is considerably effective in removing the AAEMs that form ash as also has been reported by other researchers [10–12, 14–17].

4.0 CONCLUSION

In the present study, investigation on the mixtures of PKS and PMF that have been hydrotreated and torrefied was performed for different torrefaction temperature. The HHV, ash, volatile matter, and mass loss of torrefied hydrotreated mixture of PKS and

PMF were improved if compared to the torrefied untreated mixture in this study. Meanwhile, the physical appearance of torrefied products changed from brown to black due to the increase in the fixed carbon of the torrefied products. In general, hydrotreatment positively improved most of the properties of the torrefied PKS and PMF mixture. Finally, the combined hydrotreatment and torrefaction is evidently effective for improving the biomass properties for future solid fuel production.

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