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Effect of temperature and residence time on the characteristics of torrefied food

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Abstract. Malaysia generated food waste (FW) is approximately 15 000 tons every day and expected to increase every year. Currently, Malaysia is applying the landfilling method to dispose all of the MSW which cause potential threat towards the environment and society. FW may be converted into more valuable products through reutilizing as a renewable energy source through torrefaction. The objective of this study is to investigate the effect of torrefaction temperature and residence time towards the characteristics of torrefied FW. The torrefaction process was carried out in a fixed bed reactor with continuous flow of nitrogen, at various torrefaction temperature (260 to 320 °C) and residence time (15 to 60 minutes). The torrefied FW was evaluated in terms of its mass yield, thermal behaviour and the produced functional groups through the Fourier transform infrared spectroscopy (FTIR). It can be observed that the higher the torrefaction temperature and residence time reduces the mass yield. Meanwhile, the FTIR spectra for raw and torrefied FW at different residence time was executed the functional group of C=C, C=O, C-O and C-H were present in sample. From this study, it is established that there were significant effects of both parameters towards the characteristic of FW.

1. Introduction

Urbanization and population growth have results in increasing amount of solid waste in Malaysia [1]. The daily rate of the solid waste generates was increased gradually from 2000 until 2012, and expected to continue increase up to 41,565 tons in 2025 [2,3]. The constitution of the solid waste in Malaysia are complex which consists of food waste (FW), plastic, bottle, glass, papers, metal, diapers and fabric [4]. The highest amount of waste present in solid form is FW which is about 60%. 4.404 million tons of FW was generated in 2005 and it is estimated to increase to 6.54 million tons in 2020 [5].

Till now, Malaysia still applied landfilling method to dispose all these waste where more than 80% of the wastes are dumped into landfills [2,4]. This increasing trend of solid waste generation has potential threat towards environment, society and the economy. The most serious effect on environment are soil contamination, leachate, air pollution and also green-house gases emission [1]. To overcome all these problems, the FW must be converted into value added products in which FW is reutilized to secondary usage or bio-transformation of waste [6]. However, the characteristics of raw FW is not suitable to directly use as energy source since raw FW has lower higher heating value (HHV), grindability and fuel qualities [7], which will reduce the efficiency of the process. Therefore, torrefaction process that can be applied to improve the properties of raw FW.

Torrefaction is the conversion of biomass into biochar that occur at low temperatures between 200 to 300 °C in inert environment [8]. There are a few studies that have focused on FW as raw material, for example Yuan et al. [7] in Southern China. The FW was torrefied at temperature range of 250 to 450



°C for 30 min. The HHV showed an increasing trend from torrefaction temperature of 250 until 300 °C, after which, the HHV dropped as the temperature continue to increase. This may be the result of the pyrolysis reaction that was just starting. The highest HHV was 19.34 MJ/kg, at 300 °C compared to the HHV of the raw FW at 17.5 MJ/kg. The fixed carbon and ash content in torrefied samples increased with increasing torrefaction temperature compared with raw sample, which was from 14.63 to 28.51% and from 24.94 to 46.24%, respectively. Meanwhile, the volatile content substantially decreased with an increasing torrefaction temperature which was from 60.63 to 25.25%. In terms of the proximate analysis, the carbon (C) content was increased significantly as the torrefaction temperature increased, while the oxygen (O) content was decreased rapidly across torrefaction temperature as the functional group that contained oxygen was easily cracked. The hydrogen (H) content was decreased as a result of hydrocarbon released. Torrefaction also caused the slight changed in nitrogen (N) content and lower amount of sulphur (S). The energy yield also has been discussed in this paper, which the energy yield was decreased gradually until torrefaction temperature increased up to 300 °C. The value was between 70.48 to 87.89%. This happen because of at the temperature range studied, most of the energy was stored in the torrefied sample. The energy yield was below 60% during torrefaction temperature above 300 °C, which means that too much energy was lost and consumed during torrefaction process. The surface structure of the samples was also studied, where by the result of the torrefied sample had smoother surface compared to raw sample.

Poudel et al. [9] studied the torrefaction of FW in South Korea, in terms residence time effect towards the characteristic of torrefied samples. Torrefaction temperatures used was between 250 to 400 °C. The volatile content, mass yield and energy yield were decreased as the torrefaction residence time increased. Meanwhile, the ash content increased with an increasing of torrefaction residence time. In the paper, the authors mentioned that torrefaction residence time had less an effect on torrefaction compare to temperature. This is due to significant decrease in moisture content and volatile matter of the sample. The mass loss increased with longer torrefaction residence time was applied because of decomposition of the less reactive component, while the several reactive components were release during beginning of the torrefaction process.

In another study, Rago et al. [8] also studied about FW which was collected from cafeteria of the University Of Mauritius. The FW was torrefied at temperature between 225 to 300 °C for 2 different residence time which were 1 and 3 hours. The HHV at 300 °C for 1 hour and 274 °C for 3 hours showed a high value of 23.83 MJ/kg and 24.00 MJ/kg respectively. Both conditions showed drastic increased compared to the HHV of raw FW which was 15.08 MJ/kg. The mass loss that has been observed show the more loss of mass occurred at higher residence time at the same temperature. However, the different in mass loss at torrefaction temperature exceeding 250 °C was not drastic which proved the previous study stated that the effect of temperature predominated than residence time. In this work also mentioned that the additional increment in residence time does not affect the properties of torrefied FW. The most mass loss was occurred at torrefaction temperature 300 °C and 3 hours with less than 50% mass loss. This work stated that torrefaction condition 300 °C and 3 hours was energetically inefficient as it will give impact on mass yield, energy yield and the economics of the process. Besides, the energy yield was dropped with increasing in torrefaction temperature and residence time. The lowest energy yield of 56.27% and 48.32% were obtained under condition 300 °C at both durations. Even if the lowest energy yield was attained at 3 hour torrefaction process, the dropped of energy yield was more dramatic on various temperature compared residence time.

The proximate analysis also studied which the moisture content and volatile matter were reduced as the torrefaction temperature and residence time increased. Meanwhile, torrefaction process enhanced the fixed carbon and ash content at increasing torrefaction temperature and residence time. The least volatile matter content and highest fixed carbon content were attained at 300 °C during both torrefaction residence time. The different of volatile matter between 1 hour and 3 hours was significant, 30.26% and 5.02% respectively. The unusual low content of volatile was deemed unsuitable for pretreating the FW. Prominently, the fixed carbon value between 1 hour and 3 hours was insignificant different with 55.86% and 54.02% respectively. The ultimate analysis was also discussed where the element of C was increased and parallel drop in O and H element. The C contain increased from range 41.70% to 55.80% and the highest C contain was obtained at 300 °C and 1 hour. Meanwhile, the lowest O and H content was

achieved at torrefaction temperature 300 °C at both torrefaction residence time. Though, the O contained different between 1 hour and 3 hour was significant, 10.93% and 1.67% respectively. Yet, the different of H content was insignificant between both torrefaction residence time

Although there are already several works on the torrefaction of FW, there were limited studies that focused on Malaysia's FW. On the top of that, there were no studies have been conducted to focus on the improvement of FW on based on available functional groups. Therefore, this study seeks to investigate the effect of torrefaction temperature and residence time on the characteristic of FW that collected from cafeterias in University Malaysia Pahang, Malaysia.

2. Methodology

2.1. Material

FW was collected from 4 different cafeterias in University Malaysia Pahang, Malaysia. All these sample had been mixed, then used as sample in this study. Samples were collected at lunch hour between 12 to 1 pm. Samples from each cafeteria were identified. The FW was taken as it is without any specific type of food. The FW was dried in the oven for 24 hours at 105 °C to reduce its moisture content. After that, the dried FW was grinded by using blender. Next, the samples were sieved to make sure that the particles size range from 0.5 mm to 2.0 mm. The sample was stored in air- tight container prior to further use. Table 1 showed the raw

2.2. Experimental Procedure

20 grams of FW was inserted into a tubular reactor and the reactor is placed inside the furnace. The reactor was flushed with N₂ at 2 L/min for 10 minutes to maintain inert atmosphere in the reactor. After that, the temperature was set to 260 °C for the first run. The torrefaction temperature was varied from 260 °C until 320 °C with 20 °C increment. Each run lasted for 15, 30, 45 and 60 min after the sample temperature reached the desired temperature. Subsequently, the reactor was cooled down with N₂ before sample was taken out and weighed. Each run was triplicate to ensure the accuracy of the results. Figure 1 shows the schematic flow diagram of torrefaction for process.

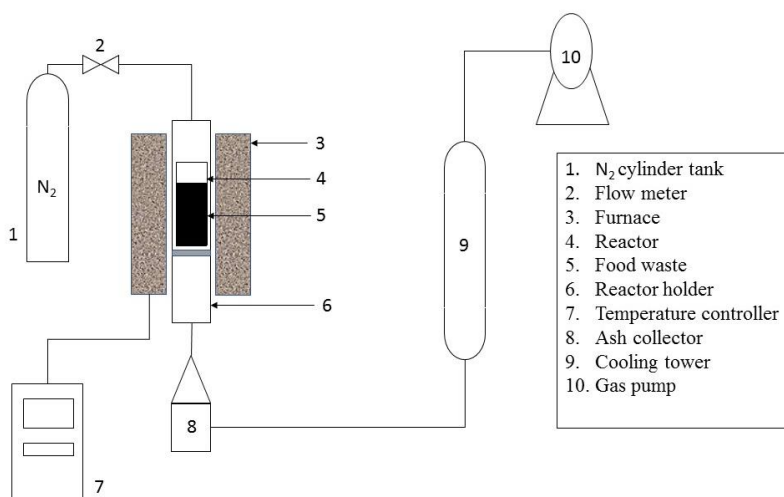


Figure 1. The schematic diagram of torrefaction process.

2.3. Analysis

2.3.1. *Mass yield.* Mass yield in this study was measured by equation (1) [7]. This is used to evaluate the loss of mass during torrefaction.

$$\text{Mass yield } (Y_M) = \frac{\text{mass after torrefaction}}{\text{mass before torrefaction}} \times 100 \% \quad (1)$$

2.3.2. Fourier transform infrared spectroscopy (FTIR). FTIR analysis is used to determine the functional group based on the carbon chemical functional groups. The analysis was conducted by Nicolet iS5 Thermo Scientific. Samples were placed in the sample slider that filled with KBr. Data acquisition was performed in the range $4000 - 400 \text{ cm}^{-1}$.

2.3.3. Thermogravimetric analysis. Thermogravimetric analysis was conducted with TA TGA Q500-0617. The samples were heated at a heating rate of $10 \text{ }^\circ\text{C}/\text{min}$ and the temperature ranging from $30 \text{ }^\circ\text{C}$ to $900 \text{ }^\circ\text{C}$. TGA analysis were carried out with nitrogen flow of $10 \text{ ml}/\text{min}$. This analysis is used to analyse the mass loss of the raw and torrefied FW during the temperature range mentioned.

2.3.4. Scanning electron microscopy (SEM). SEM was used to analyze the surface morphology of raw and torrefied FW. This analysis was conducted by Hitachi TM3030 plus Tabletop Microscope in Cariff Lab, University Malaysia Pahang. The samples were investigated the morphology surface structure at an accelerating voltage of 15 kV and with magnification of (250x, 500x and 1000x).

3. Result and Discussion

3.1. Physical Observation

The most obvious observation that can be made was the changed of the color of dark-blackish FW as shown in Table 1. The color of the sample changed from light-brownish to dark-blackish across residence time at each temperature. At lower residence time (15 min), all the color of sample across the temperature were dark-brownish in color. At 60 min, all the product turns into blackish color at all temperatures. Meanwhile, the product was into darker-brown sample at 260 and $280 \text{ }^\circ\text{C}$ at torrefaction residence time of 30 min, while the color at 300 and $320 \text{ }^\circ\text{C}$ were blackish in color. This observation was also experienced by Mualina et al. [10] where the color of OPF used become darker from the initial brownish color as torrefaction temperature increased. The darker of the sample color indicated the mass loss in biomass. In the paper also stated that the biomass color changed into darker when residence time increased. The color changed was due to destruction of connective, non-structural organic compounds and also variation of C content in the remaining torrefied sample [11].

3.2. Mass Yield

Figure 2 shows the effect of the residence time on mass yield at various temperatures and residence times. It can be observed that mass yield decreased with increasing residence time at each torrefaction temperature. The acceptable range of mass yield for any type of biomass is 24 to 95% of its original weight [8]. As shown in Figure 2, the longer the residence time did not affect the mass yield at lower temperature. There was a gradual decline of mass yield at the lowest torrefaction temperature of $260 \text{ }^\circ\text{C}$. Meanwhile, the mass yield was decreased drastically at $280 \text{ }^\circ\text{C}$ between 30 and 45 min with 9.93% difference. This can be caused by the decomposition of some reactive components [12]. The mass yield continues to decrease between 15 and 30 min at the same torrefaction temperature. At $300 \text{ }^\circ\text{C}$, the mass yield dropped drastically after 15 min, with 13.33% difference. However, the mass yield showed insignificant of decline at torrefaction of residence time 45 min with only less than 1% difference. This mean that the loss of reactive components process at 30 and 45 min residence time is the same [13]. Nevertheless, at 60 min, the mass yield drastically decreased again which achieved the lowest mass yield in this study, 66.82%. At torrefaction temperature of $320 \text{ }^\circ\text{C}$, the mass yield experienced drastic decrease between 15 and 30 min $300 \text{ }^\circ\text{C}$, with 17.06% difference. In this study, the impact of residence time towards mass yield was more significant compared to torrefaction temperature, since at longer residence time, the mass yield was lower.

Table 1. The color change of raw and torrefied FW at various temperatures and residence times.

Condition	15 min	30 min	45 min	60 min
Raw				
260 °C				
280 °C				
300 °C				
320 °C				

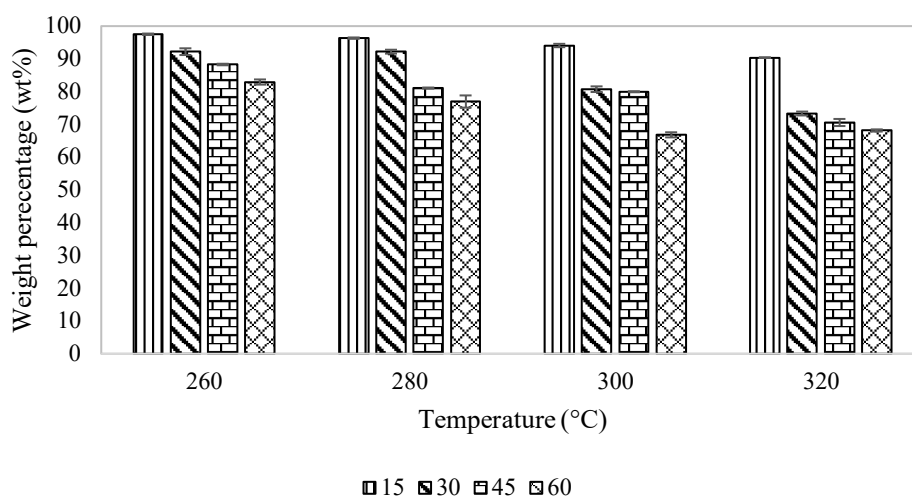


Figure 2. The mass yield across the residence time at specific temperature.

3.3. Functional groups

Figure 3 (a) shows that the FTIR spectra of raw and torrefied sample across the torrefaction temperature for 30 min. The intensity of the peak in the graph was attributed to the release of volatile [9]. All the peak became more intense when the torrefaction temperature increased. The CO₂ peak was slightly appeared which at range 2250 to 2500 cm⁻¹ for raw sample until during temperature 260 °C. This was due to more pronounced degradation of biomass structure at mild temperature [9]. However, the CO₂ peak was not existed during severe torrefaction temperature. This is proved that at higher torrefaction temperature, there is no CO₂ components released. Between wavenumber of 1750 to 2000 cm⁻¹, the peak was indicated for C=O and C=C bond stretching [9]. The intensity of the peaks was increased as the torrefaction temperature increased. This may cause by tar formation which the release of phenol, aliphatic compound and carbonyl compounds occurred [16]. Meanwhile, the peaks between wavenumber of 1000 to 1500 cm⁻¹ which contained C-O and C-H compound were also increased across the torrefaction temperature. This is because of the release of the phenols and aliphatic compounds and reshuffle of structural in biomass [9]. This can be said that at higher torrefaction temperature, more carbonyl and aromatic compound was released. During torrefaction at higher temperature, emission of carbonyl and aromatic compound was higher which the reason of the decreasing mass and energy yield.

FTIR spectra obtained at torrefaction temperature of 320 °C at various residence times were analysed as shown in Figure 3 (b). At wavelength between 2250 to 2500 cm⁻¹, the peak intensity is increased before 30 min. This occurred due to the released of H₂O during intermolecular dehydration reaction process [14]. The H₂O molecule did not exit after 15 min which all the H₂O molecule was totally dispersed during torrefaction after 30 minutes. At wavelength between 1750 to 2000 cm⁻¹, the peak intensity was increased as the torrefaction residence time increased. This peak is attributed to C=C and C=O bond stretching which lead to the presence of carbonyl and aromatic compounds [13]. According to Kai et al. [14], the stretching of the peak represent the adsorption of aldehyde, ketone and acids group into torrefied product. The peak intensity at wavelength between 1000 to 1500 cm⁻¹ was also increased as the torrefaction residence time increased. This peak is associated to the stretching of C-C, C-H and C-O bonds. This stretching occurred due to the removal of aldehyde, ketone and acids groups, yet substituted with the presence of methyl and methylene groups of alkane and alkenes in torrefied FW [15].

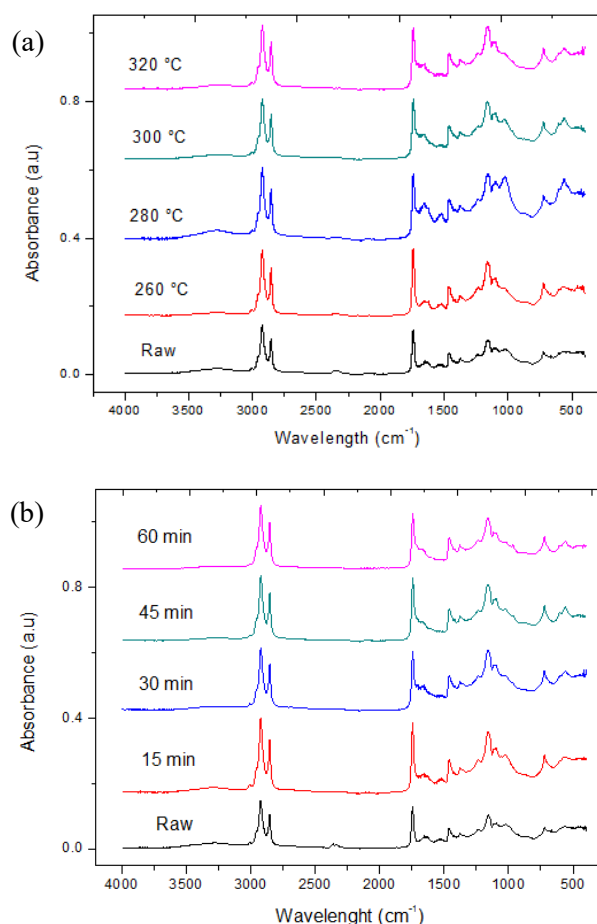


Figure 3. (a) The FTIR spectra of raw with raw and torrefied sample across the torrefaction temperature at 30 min. (b) The FTIR spectra of raw with raw and torrefied sample across the torrefaction residence time at 320 °C.

3.4. Thermal properties

A study to investigate the thermal decomposition of torrefied samples during torrefaction process was carried out. Thermogravimetric experimental curves (TG) for raw and torrefied FW are shown in Figure 4. It showed that the weight loss was decreased as the torrefaction residence time increased at torrefaction temperature 320 °C. During torrefaction temperature 300 °C, the weight loss at 30 and 60 min showed insignificant change. At temperature before 200 °C, the weight loss between raw and torrefied FW was insignificant, except for torrefied FW at 320 °C 30 min. This can be explained that at this temperature, dehydration process occurred which the amount of water content in torrefied FW at 320 °C 30 min was lower than others [16].

Between temperature 200 °C and 300 °C, the weight loss for all torrefaction condition started to have more obvious change. The most weight loss between this temperature was raw FW. Torrefied FW at 320 °C 30 min still had the least weight loss among the other torrefied FW. The weight loss between this temperature was included in the first slope, which means that, the dehydration process was still occurring where the raw FW has the highest amount of water [13]. At the second slope, which is between 300 °C to 500 °C, the weight loss continued to decrease as the torrefaction residence time increased, the decomposition of the volatile components occurred [13]. It can be observed that this occurrence is more rapid compared to the first slope. This is also observed by Qiu et al. [17] which may involve with the release and combustion of the volatiles. In this study, between temperature of 300 °C and 400 °C where the highest less weight loss was determined at 300 °C & 30 min. However, after temperature 400 °C, the least weight loss was experienced by torrefied FW at 320 °C, 60 min. This can be explained that

torrefied FW at 320 °C & 60 min has the least amount of volatile components in the sample [18]. On the other hand, 320 °C & 60 min has the least mass yield which can be related to the least volatile components presented for all samples. At third stage which is between temperature 500 °C to 900 °C, the weight continued to decrease for all samples. During this stage, the torrefied FW at 320 °C & 60 min still has the least weight, which is caused by limited volatile emission compared to raw and other torrefied FW [18]. This can be concluded that, higher emission of volatile component was occurred during the torrefaction process at 320 °C & 60 min which contributed to the least weight loss during the thermal decomposition study.

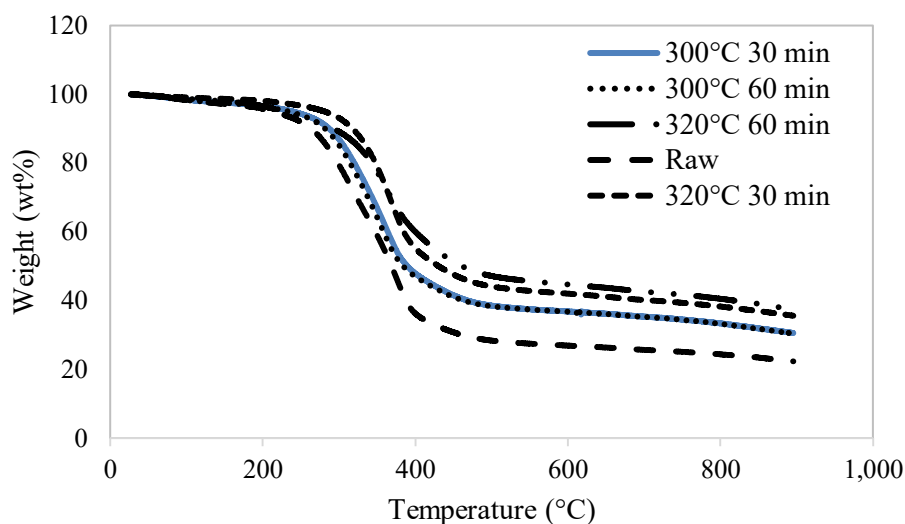


Figure 4. The mass loss across the residence time at specific temperature.

3.5. Surface Morphology

Comparison of SEM images between raw and torrefied FW are shown in Figure 5 at 250x, 500x and 1000x magnification. The surface of the torrefied FW was smoother compared to raw FW. The microparticles that are present on the surface of raw FW were diminished at 320 °C for 30 min. This may be due to the decomposition of particles and also re-arrangement of the polymer structure [19], causing the sample to become more brittle and harder as the torrefaction temperature was increased. Similar observation was made by [7] where the surface of the torrefied sample was found to be smoother. On the other hand, [20] discovered that at mild torrefaction temperature, the sample surface was not significantly changed. However, microparticles were starting to disappear on the torrefied sample structure at 300 °C, which was thought to be due to the combustion on the internal structure of the sample.

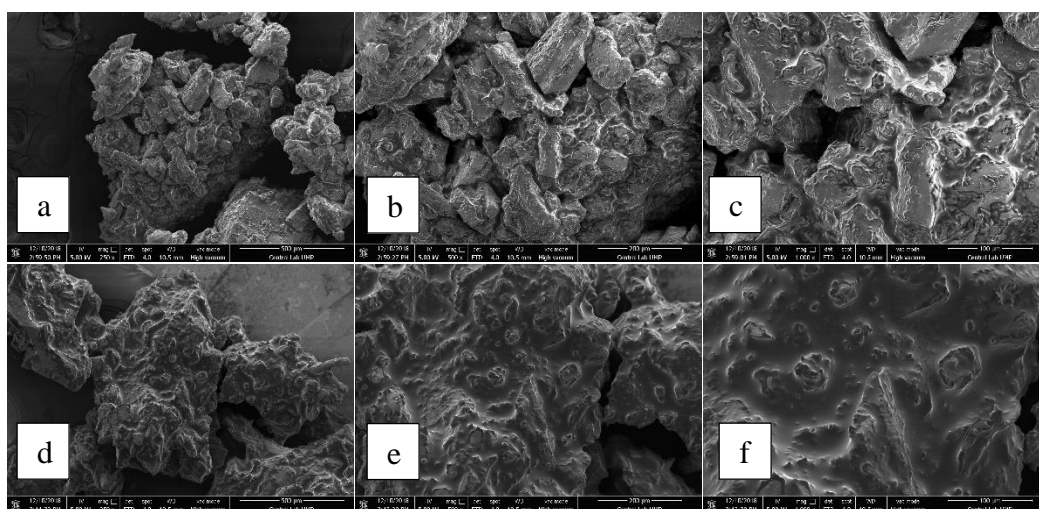


Figure 5. SEM images of (a) raw FW at 250x, (b) raw FW at 500x, (c) raw FW at 1000x, (d) torrefied FW at 320 °C for 30 min at 250x, (e) torrefied FW at 320 °C for 30 min at 500x (f) torrefied FW at 320 °C for 30 min at 1000x.

4. Conclusion

From this study, it has been established that torrefaction of the FW was affected by temperature and residence time which the lowest mass yield was observed at the most severe temperature of 300 °C at the longest residence time of 60 min with value 66.83%. The characteristics of fuel FW can be seen through FTIR analysis which the release of carbonyl and aliphatic compounds are responsible for the fuel formation. Other than that, the thermal treatment helps in improving the FW's characteristics by the weight loss which help rearrange the structure of fuel through volatile released.

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