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# Effect of Oxidative Torrefaction on Characteristics of Treated Corncob Pellets

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**Keywords**：agricultural residues, biomass energy, pyrolysis, thermal engineering.

## ABSTRACT

Oxidative torrefaction of corncob pellets was carried out in a fixed bed thermal reactor with 40±0.5 g of samples. All experiment works were performed under oxidative conditions with 0 to 18% oxygen in nitrogen atmosphere, temperatures at 220, 260, and 300 °C for reaction time of 5 to 20 min. Experimental results showed that mass yields were significantly reduced with increasing temperature at given oxygen concentration and reaction period. Similarly, increase in torrefaction time was found to significantly reduce mass yields. However, effect of oxygen content was not observed to affect mass loss markedly. In this work, change in chemical composition, energy density, water uptake and grindability were also investigated. Oxidative torrefaction was found to improve quality of torrefied biomass in terms of increased carbon content, higher heating value and water resistance, and reduced energy requirement for size reduction.

## INTRODUCTION

Thailand is among the largest economy in Southeast Asia with a population of nearly 70 million people. The domestic energy demand has increased sharply during the past few decades, whose majority of energy supply is from foreign imports. Coal, oil and natural gas are the three primary sources of energy. Thailand imports crude petroleum oil and natural gas at about 80% and 40% of the annual consumption (Chaichana and Kaewtathip, 2016). To improve its national energy security, economic affordability and environmental sustainability, the Royal Thai government has developed the national integrated energy blueprint, making the energy efficiency, oil, gas and power development, and alternative energy development plans aligned with each other. The blueprint has targeted 30% of total final energy consumption from renewables by 2036. Biomass and bioenergy will play a critical role in achieving these goals. For biomass, the ambitious targets are set at 5,570 MW of electricity and 22,100 ktoe of heat. In 2014, biomass energy accounted for about 2450 MW of electricity generation and about 5100 ktoe of heat generation in the country (EPPO, 2015).

With limited petroleum resources, the country is heavily based on agriculture. Thailand ranks among the World top producers of agricultural products such as paddy rice, sugar cane, cassava, corn, palm oil and rubber. Large amount of agricultural wastes that could be used as energy is generated each year. In 2014, available biomass residues in Thailand that remained untapped were approximated at more than 31 million tons. With a development plan by the Thai Ministry of Agriculture and Cooperatives, biomass residue potential is projected at around 80 million tons annually (IRENA, 2017). Biomass from agricultural residues can be used as fuel for direct heat production. But direct use of raw biomass is not straightforward, because in its natural forms, it has high water content, low bulk density, low energetic content, wide variety of shape and size, tendency to absorb moisture, and ease of degradation during storage. Physical conversion by densification proved to be simple and effective in mitigating some of these issues for solid biomass (Wongsiriamnuay and Tippayawong, 2015; Piboon et al., 2017). However, upgrading by torrefaction can be a simple, yet useful method to further improve the quality of raw biomass.

Torrefaction is a low temperature thermal degradation process, usually conducted in an inert atmosphere at temperature between 200ºC to 300ºC (Saadon et al., 2014). Torrefaction has been

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considered as an effective method to improve the quality of raw biomass feedstock prior to further applications in combustion, gasification, or pyrolysis (Basu, 2013). For the past 10 years, there have been a great number of published reports about biomass torrefaction. Main topics include (i) physical and chemical properties of solid products and their characteristics (Asadullah et al., 2014; Chen et al., 2015; Bach and Skreiberg, 2016; Bach et al., 2016), (ii) torrefaction process modeling and kinetics (Sarvaramini et al., 2013; Bach et al., 2017a), (iii) techno-economic analysis on torrefaction (Batidzirai et al., 2013; Svanberg et al., 2013), (iv) subsequent utilization on thermochemical conversion routes and applications (Chen et al., 2014a; Gil et al., 2015; Starfelt et al., 2015; Bach et al., 2017b), (v) combined pretreatment on torrefaction with other processes (Zhang eta l., 2016; Chen et al., 2017a; 2017b).

Known benefits of torrefaction are reduction of oxygen functional groups (Chen et al., 2012), improvement in carbon content, hence, higher heating value (HHV) (Ciolkosz and Wallace, 2011), and increase in grinding ability (Arias et al., 2008) and increase in water resistance (Chen et al., 2014b; Mei et al., 2015). Although torrefaction is considered to be one of the most modern methods for upgrading biomass materials, this technology still requires the inert gases and thermal energy (Yan et al., 2010). Most biomass torrefaction research works focused on composition of solid yields and the products distribution (Bridgeman et al., 2008; Medic et al., 2012). Volatiles generated can be recycled to use for torrefaction with no need for input thermal energy and more inert gases, leading to savings of resources and expenses (Bergman et al., 2005). For example, High heating value of oxidative torrefaction of oil palm fiber pellets at 275ºC increased when compared to non-oxidative torrefaction (Chen et al., 2016), Significant diffusion resistance in the mineral layer can favor substantial reduction in the oxygen flux to the biomass from the environment (Leontieva et al., 2018). Oxidative torrefaction on nutshells also exhibited strongly linear distribution in the van Krevelen diagram, and the carbon enrichment was a feasible index to describe weight loss (Zhang et al., 2019). At 280 °C, the high oxygen concentration affected some properties of biomass (Rousset et al., 2012), The effects of oxygen concentration on the weight loss, energy yield and torrefied biomass properties were more significant at 300 °C than at 240 °C (Wang et al., 2018). Some of works on oxidative torrefaction utilized oxygen concentration in inert gas in the range of 0% to 10% for oil palm fiber (Chen et al., 2016), 0% to 6% for sawdust (Wang et al., 2013) and only 3% for sugarcane bagasse (Conag et al., 2017). The solid phase conversion increased with increasing temperature and oxygen or carbon concentration, but was not significantly affected by biomass size (Uemura et al., 2015). However, little has been done about oxygen in torrefaction, because its content in inert gas was quite low (Rousset et al., 2012; Lu et al., 2012; Uemura et al., 2013; Conag et al., 2017). Furthermore, effectiveness of oxidative torrefaction to improve quality of biomass material is still in question.

In order to address these problems, the main objective of this research was to investigate effect of oxidative torrefaction on characteristics of treated corncob pellets. In the present work, thermal treatment of the pelletized biomass was carried out in a fixed bed thermal reactor under oxidative conditions by mixing oxygen and nitrogen. Effects of torrefaction temperature, reaction time and oxygen content in inert gas were examined to investigate changes in physio-chemical properties of torrefied biomass samples such as elemental composition, HHV, energy density, water uptake ability, and grindability.

## MATERIALS AND METHODS

### Sample preparation and characterization

The biomass samples used as the feedstock for all experiments were corncobs obtained from local farms in northern Thailand. These samples were first ground to the range of 8 – 10 mm in size in a rotary cutting mill and dried in the sun for removing moistures. They were densified into pellet form with diameter of 7 mm and about 20 mm long. After that, the samples were closely stored in air-tight zipper bag, before sending for analysis. The main composition that included the results of proximate and ultimate analysis are shown in Table 1. The pelletized biomass appeared to have equilibrium moisture content of about 10% . HHV was calculated from main elemental components using a published mathematical model (Demirbas and Demirbas, 2004).

### Torrefaction experiment

The biomass pellets were tested in the fixed bed thermal reactor, comprising a carrier gas unit, a furnace, and thermocouple, shown in Fig 1. The thermal reactor was used to study the change on biomass samples under gas oxygen atmosphere condition (Onsree et al., 2018; 2019). It was made of stainless steel at 50 mm diameter and 400 mm height

Table 1. Properties of the corncob feedstock.

| | | |
|---|---|---|
| Proximate analysis (% wt.) as-received basis | Moisture | 10.7 |
| | Volatile matter | 67.3 |
| | Fixed carbon | 16.5 |
| | Ash | 5.5 |
| | VM/FC | 4.1 |
| Elemental analysis (% wt.) dry basis | Carbon | 40.7 |
| | Hydrogen | 5.6 |
| | Nitrogen | 0.3 |
| | Oxygen | 53.4 |
| HHV (MJ/kg) | | 16.1 |

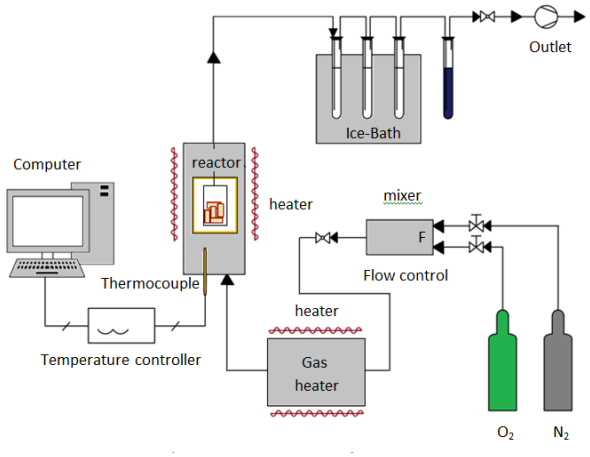



Fig. 1. Schematics of torrefaction experimental setup under oxidative condition.

whose external wall was wrapped around by fiber board insulator and a 3 kW electrical heating tape. The heater was regulated by a digital temperature controller and the data logger. Inside the reactor, cylindrical basket with 35 mm in diameter and 80 mm high was hang. Gas/tar was collected in 8 test tubes, which were connected to the reactor system. For each experimental run, the sample of 40 ± 0.5 g was used. Experimental conditions were varied for 0, 6, 12, and 18% of oxygen in nitrogen with a fixed gas flow rate at 3.0 L/min. The reaction time in the reactor were 5, 10, 15, and 20 min. Temperatures conditions were 220, 260, and 300 °C.

The products from torrefaction were solid, liquid or gas. Each experimental condition was repeated for at least three times and the yields of the resulting products were obtained. The weight of the pyrolysis products was calculated from material balance. Solid yield was defined as weight of remaining solid product after torrefaction, compared to original weight input. Liquid yield was that collected in a test tube. The product weight balance was based from eqs. (1-3), where $Y_S, Y_L, Y_G$ are solid, liquid and gas yields (%), respectively, $m_s$ and $m_L$ are solid and liquid weights after torrefaction (g), $m_{feed}$ is original raw pellet weight (g). Gas yield was determined from $Y_S, Y_L$.

$$Y_S(\%) = \frac{m_s}{m_{feed}} \text{ x } 100, \quad (1)$$

$$Y_L(\%) = \frac{m_L}{m_{feed}} \text{ x } 100, \quad (2)$$

$$Y_G(\%) = 100 - (Y_L + Y_S) \quad (3)$$

**Product analysis**

The solid products obtained were sent for ultimate analysis, where carbon, hydrogen and nitrogen were determined using an elemental analyzer (model Flash 1112 EA Series). The O/C and H/ C atomic ratios were calculated. HHV was calculated from a mathematical model (Demirbas and Demirbas, 2004), shown in eq. (4). Energy yield ($Y_E$) was evaluated using HHV from the torrefied sample and the raw pellet divided by the solid yield of the sample, shown in eq. (5).

$$HHV = 0.3856\,(C+H) - 1.6938, \quad (4)$$

$$Y_E = Y_S \frac{HHV_f}{HHV_i} \quad (5)$$

For determination of moisture uptake ability, torrefied pellets were initially placed on an aluminum plate and incubated at 105°C in an oven (model Binder DE115) about 36 h for completely removing moisture in the samples. The samples were then weighed and quickly put on a tray in a 900 ml glass jar, containing saturated potassium sulfate solution (Tumuluru et al., 2011). The glass jar was conditioned at a fixed 35°C, providing constant humidity. The samples were weighed at 1, 2, 3, 6, 12, 24, 36, 48, 60 and 72 h interval. Moisture uptake was determined from the weight gained by the originally dried sample due to moisture absorption from saturated solution. The moisture uptake expression is shown in eq. (6), where U is moisture uptake (%), and $m_i$ and $m_f$ are sample weights before and after conditioning with saturated solution (g).

$$U = \frac{m_f - m_i}{m_i} \quad (6)$$

Grinding energy and sieve analysis were used to find energy consumption for grinding per mass of torrefied pellets. A commercial coffee grinder (model Fiorenzato M.C. F5) was used to grind torrefied pellets, coupled with a power analyzer (000.1 to 999.9 W ± 1%) with MN93A Clamp 5 A (500 mA to 6 A ± 0.7%). Power (P) used and time taken to grind the sample (t) were collected and integrated to find the work needed. The grinding energy (E) was grinding energy per unit mass processed, calculated from eq. (7).

$$E = \frac{Pt}{m} \quad (7)$$

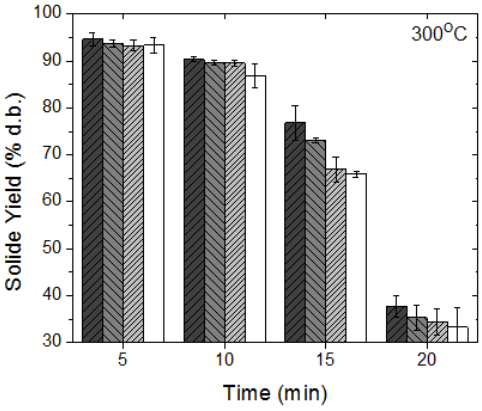
For particle distribution, an automatic sieve shaker (model D407) was employed with nine sieve sizes at 2000, 1180, 850, 600, 425, 300, 212, 150, 75 µm, following ASTM E11 standard. For each size fraction, the collected mass was determined, against total sample mass, eq. (8), where $f_m$ is mass fraction (%), $m_d$ is the mass in each sieve size, and $m_{ts}$ is total weight before sieving.

$$f_m = \frac{m_d}{m_{ts}} \quad (8)$$

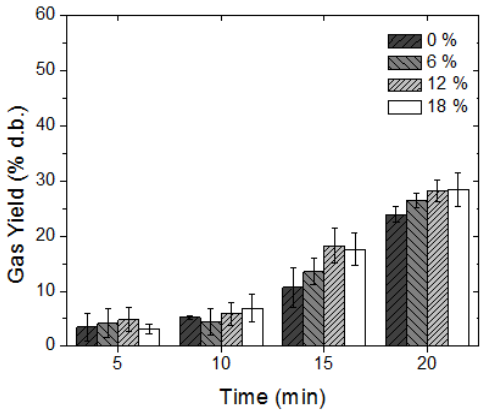
## RESULTS AND DISCUSSION

### Yields of torrefied products

Experimental results are shown in Fig. 2(a), (b) and (c) for solid yields after torrefaction at different conditions of temperature, reaction time and oxygen concentration. They are average values, with error bars showing standard deviation. It can be seen that solid yields showed a decrease when temperature, time and oxygen content were increased. At relatively mild conditions of low temperature, short time and low oxygen content, solid yields rarely exhibited any significant changes with these variables.




(a)

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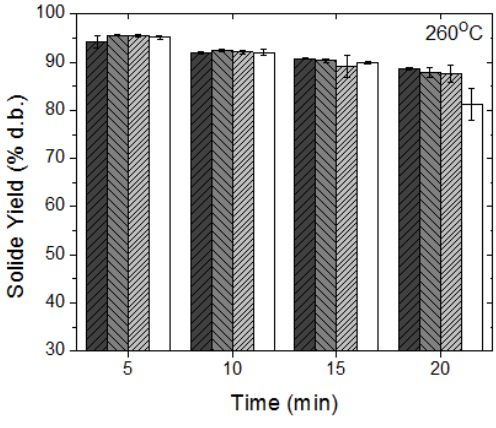


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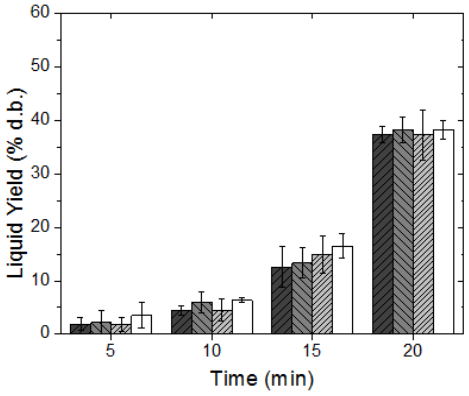
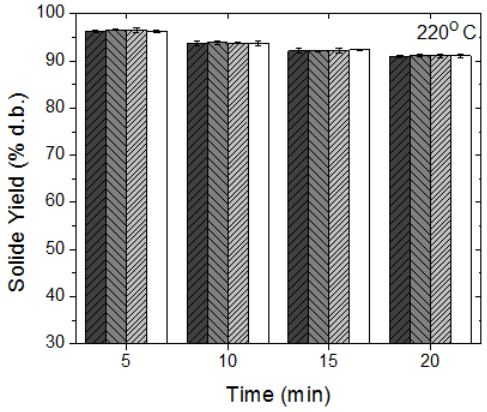



(d)




(e)

Fig. 2. Variation of products yields from torrefaction of corncob pellets with time and oxygen content in inert gas; solid yields at (a) 300°C (b) 260°C (c) 220°C, and (d) gas yields at 300°C, and (e) liquid yields at 300°C.

At higher degrees of severity for torrefaction, marked change in solid yields was observed, especially for high temperature. At mild temperatures of 260°C and below, effect of oxygen content on solid yields seemed to be negligible, while increasing reaction time appeared to affect mass loss by only 10-12%. This implied that only dehydration occurred in this ranges. At 300°C, significant mass loss was found for longer reaction time, reduction of up to 35% and 65% at 15 and 20 min, respectively. Around 27-35% of original weight was lost when the pelletized biomass was torrefied at 6-18% oxygen content and 300°C for 15 min condition. This was rather big, compared to a loss of 15% weight at 3% oxygen content and 300°C for 15 min, reported for sugarcane bagasse (Conag et al., 2017). For higher oxygen content, change in mass loss at this temperature was also noticeable, but in a lesser degree than reaction time. Gas and liquid yields are also shown in Fig. 2(d) and (e) for 300°C. Gas and liquid yields were found to be inversely in accord with solid yields. Loss of solid yields was translated directly into gas and liquid, due to devolatilization of

pelletized biomass. Initially, liquid yields were found to be slightly less than gas yields. But at 20 min of reaction time, liquid yields appeared to be higher than gas, for all conditions.

**Elemental composition and energy yields**

Ultimate analysis results are shown in Fig. 3(a), (b) and (c) for carbon, hydrogen and nitrogen as function of temperature, time and oxygen content in the treated gas.

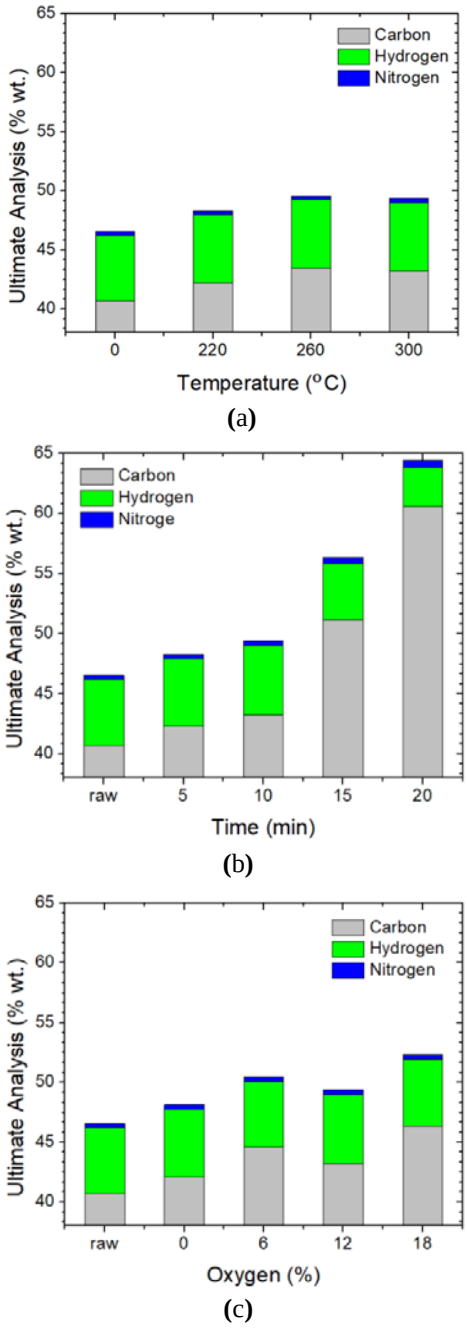



Fig. 3. Ultimate analyses of torrefied products for varying (a) temperature, (b) reaction time, and (c) oxygen content in inert gas.

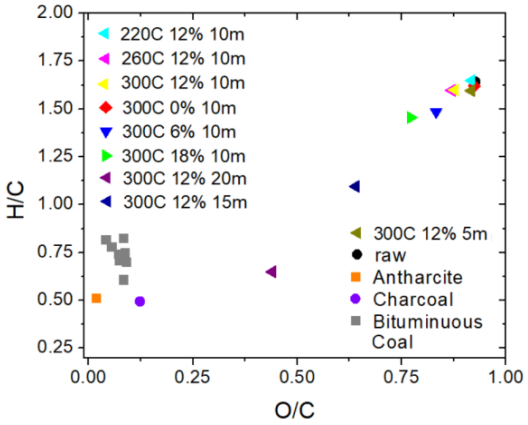



Fig. 4. van Krevelen diagram for torrefied pellets.

It was observed that increases in temperature and oxygen concentration resulted in slight improvement in carbon content of the torrefied pellets from about 41 to 45% . Meanwhile, changing reaction time from 5 min to 20 min led to marked increase in carbon content from about 41 to 60%. Relationships between the O/C and H/C atomic ratios at selected conditions are also shown as van Krevelen plots for raw and torrefied corncob pellets in Fig. 4. Difference between raw corncobs, torrefied corncob pellets and various coals was clearly demonstrated. As anticipated, the raw material was found at the furthest top - right corner, while coals were near to the bottom-left corner. Changes in the O/C and H/C ratios illustrated a tendency towards charcoal, depending on the degree of torrefaction severity.

Fig. 5 shows HHV of the torrefied pellets at selected conditions. At relatively mild conditions of low temperature and short reaction time, change in HHV of the torrefied biomass was small, from about 16 to 17 MJ/kg. Remarkable improvements in HHV were observed for the treated biomass pellets at high torrefaction severity conditions i.e. high temperature, long residence time and high oxygen content. HHV of 18 to 23 MJ/kg was evident.

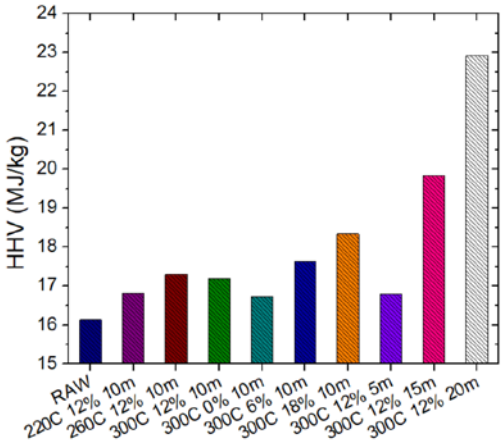



Fig. 5. HHV of torrefied solid products

An increase of more than 43% could be realized at the most severe case. Nonetheless, increase in HHV usually went against solid yields. Consideration of both HHV and solid yields simultaneously could be done with energy yield, shown in Fig. 6. Energy yields of most torrefied pellets were found to be about 92-97% from original raw materials, similar to those reported for sawdust (Wang et al., 2013). Those biomass pellets that were torrefied near the most severe conditions (300ºC, 15-20 min, and high oxygen content) appeared to have markedly low energy yields of 82 and 49%, due largely to low mass yields obtained at these conditions.

**Moisture uptake ability**

Time dependent moisture uptake ability of the solid fuels is shown in Fig. 7. They are shown as a function of torrefaction temperature, time and oxygen content in inert gas. Values for the raw pellets are also shown as reference water resistance performance. The dried raw biomass pellet was observed to re-absorb moisture of about 15.5% of its original weight and stabilize within 48 h. Almost all torrefied pellets exhibited improvement in resistance to uptake moisture, between 10-12%. Similar values were reported for the torrefied wood pellets (Sokhansanj et al., 2010). For those pellets that underwent thermal treatment at high oxygen content, high temperature and long reaction time tended to have low moisture uptake ability or high water resistance. At 12% oxygen, 300°C and 15 min condition, 100% increase in water resistance from the raw pellets was realized.

**Grindability and particle size distribution**

Grinding energy required for size reduction of the torrefied corncob pellets and their resultant particle size distribution are shown in Fig. 8. The grinding energy was illustrated as a function of solid

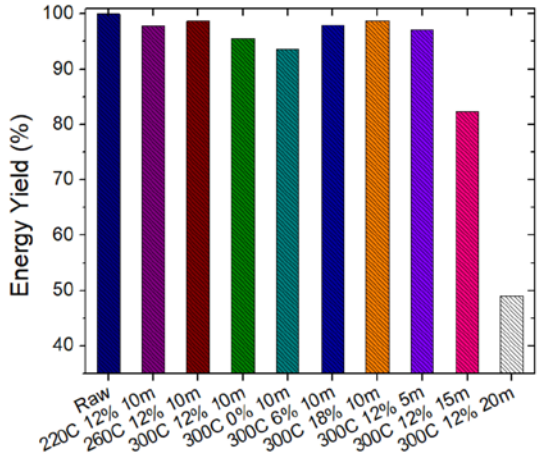
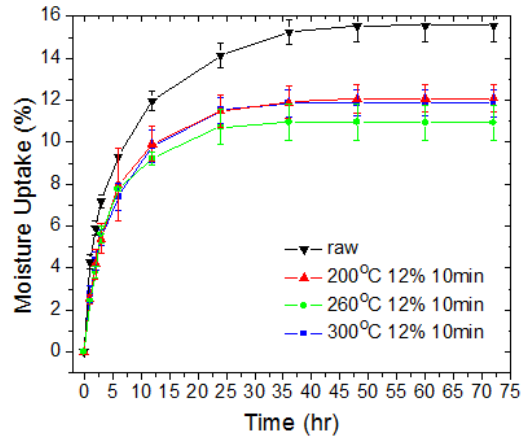
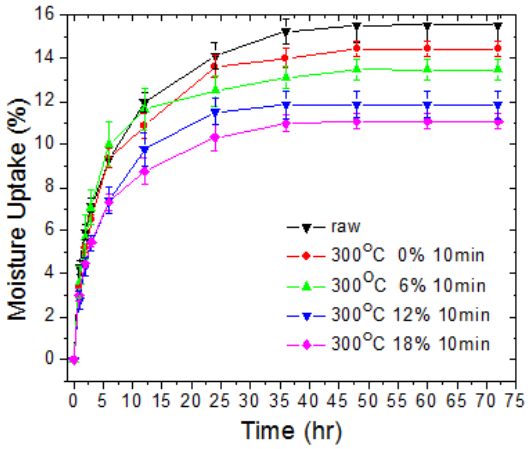


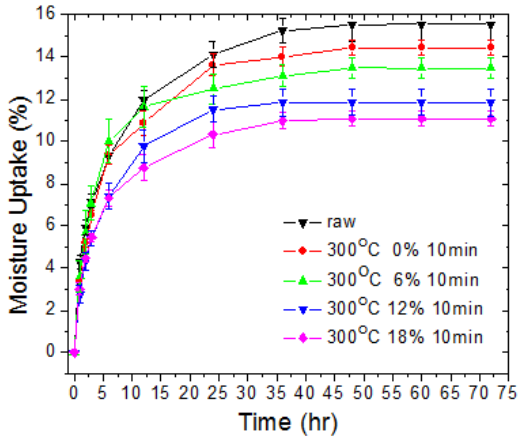

Fig. 6. Energy yields of solid products from torrefaction of corncob pellets.




(a)




(b)




(c)

Fig. 7. Moisture uptake ability of torrefied corncob pellets; variation with (a) temperature, (b) reaction time, and (c) oxygen content in inert gas.

product mass lost after torrefaction. It was confirmed that torrefaction process enabled lower energy requirement for size reduction of the solid products. Energy required to process the raw biomass was about 675 J/g on average. Most conditions at low

temperatures and long reaction time or high temperature at short reaction time were found to have mass loss of lower than 15%, corresponding to about 350 J/g or more. This was almost 50% reduction in energy consumed. Wang et al. (2013) reported a 50% reduction in the grinding energy required when the pellets were treated at 225ºC.

Near the most severe torrefaction conditions (300ºC, 15 and 20 min), reduction in the grinding energy by 78% at 33% mass loss, and more than 92% at 65% mass loss were found, in comparison with the untreated corncob pellets. This was in agreement with those reported by Repellin et al. (2010), who reported that torrefied spruce and beech woods with 28% of mass loss demonstrated lessening of grinding energy by 93%.

Particle size distribution from sieve analysis, illustrated in Fig. 8, is also useful in determining the grindability of torrefied biomass. Compared to the untreated biomass pellets, the size distribution histrograms were found to move to finer particle sizes. Thermal treatment by torrefaction proved to encourage smaller particles after size reduction. For instance, there was about 22% of raw pellets with particle sizes between 800 and 425 µm, whereas there was more than 30% of torrefied pellets in the same size range. Similar observation was observed by other published reports (Arias et al., 2008).

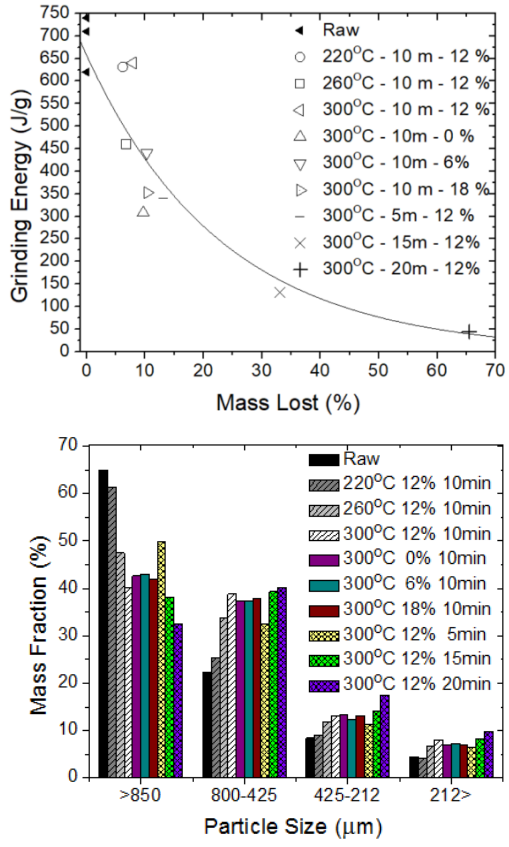



Fig. 8. Grinding energy and sieve analysis results of torrefied corncob pellets.

## CONCLUSIONS

In this work, torrefaction of pelletized agricultural residues was experimentally conducted under oxidative atmosphere. Effect of varying torrefaction temperature, reaction time and oxygen concentration in inert gas on some physico-chemical properties of the solid products was investigated. It was found that the product yields had tendency to decrease with increasing in the severity of thermal treatment conditions, which was directly dependent on temperature, residence time and oxygen concentration. Effects of reaction temperature and time were more significant in reducing solid mass yields of treated biomass pellets than the effect of oxygen content in inert gas. The presence of oxygen was found to affect HHV and water resistance of the torrefied pellets slightly, but effect on grinding energy and particle size distribution were not significant. At typical torrefaction conditions of 260ºC, 5 to 15 min and oxygen over 12% and 300ºC, 10 min or less with oxygen, the solid mass losses were about 5-15%, the HHV and water resistance improved slightly, the energy yields remained about 90-95%, and the grinding energy reduced almost by half.

From overall analysis of the findings, it could be concluded that oxidative torrefaction enabled improvement in physico-chemical properties of the pelletized biomass by removing the moisture and the light volatiles, increasing the carbon and the energetic content, and altering the mechanical and the water resistance performances. Harnessing solid agricultural residues for energy may become much more widespread with torrefaction as a simple, appropriate and promising pretreatment technology.

## ACKNOWLEDGMENT

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