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A Parametric Study of Torrefaction Technology of Agricultural Residues in Indonesia

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ABSTRACT

Enhancing the value of biomass residues holds promise for mitigating open burning. However, biomass utilization as an energy feedstock encounters its own set of challenges owing to inherent properties. To address these concerns, torrefaction, an essential thermal pretreatment process for carbonaceous materials, emerges as a viable solution. In a laboratory experiment conducted in a static tube reactor, torrefaction was investigated at temperatures of 250°C for 45 minutes and 300°C for 5 minutes. The findings revealed that rice straw, corncob, and cassava stalk exhibit properties exceptionally suited for utilization as energy feedstock. Notably, at 300°C corncob attains a carbon content of 58.10%, a fixed carbon content of 34.35%, and a calorific value of 22.46 MJ/kg.

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INTRODUCTION

Indonesia, a significant worldwide contributor to agricultural goods, confronts an enduring challenge associated with post-harvest open burning. An annual estimation suggests that approximately 45 million metric tons of residues from these crops are susceptible to open burning practices [1]. This practice is responsible for emitting air pollutants, the assessment of which indicates a contribution of approximately 12-14% to the overall global warming potential attributed to open burning of crop residues on a global scale [2].

This academic endeavor explores the potential of biomass residue enhancement to mitigate open burning occurrences. While processing biomass, scientists encountered technical challenges that present significant hurdles, which can be effectively addressed through torrefaction technologies. Torrefaction, a thermal pretreatment process of carbonaceous materials conducted at temperatures ranging from 200-300°C under inert conditions, produces a product characterized by solid mass content within the range of 50-97%, energy content yields ranging from 62-97%, and moisture content below 10%. Importantly, torrefaction enhances energy density, carbon

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content, and calorific value by 15-25%, while simultaneously reducing grinding energy requirements by approximately 70% [3]–[6].

Torrefaction, as highlighted by the previous researcher, primarily leads to the degradation of hemicelluloses, with an expectation of similar outcomes from both hardwood (e.g., alder, beech, maple, and oak) and softwood (e.g., cedar, juniper, pine, and spruce) characterized by comparable hemicellulose content [7]–[9]. However, this anticipated parallelism in results does not materialize, as evidenced by significantly disparate mass yields, as reported by Prins et al. [10]. The variation is attributed to a hemicellulose subcomponent known as xylan, with hardwoods containing 80–90% xylan, in contrast to the 15–30% found in softwoods [11], [12]. Beyond hemicellulose content, hardwoods and softwoods differ in various aspects. During torrefaction, hardwoods release acetic acid and water, while softwoods emit formic acid. Furthermore, hardwoods exhibit a higher energy density compared to softwoods, as indicated by Prins [10], [13].

Nam et al. investigated the torrefaction process involving two distinct crop residues, namely rice straw and cotton stalk. The notable discrepancy in xylan content, the principal constituent of hemicellulose, between these residues—rice straw containing four times more xylan than cotton stalk—resulted in a pronounced disparity in weight loss, with rice straw exhibiting a more substantial reduction[14]. Additionally, Eseltine et al. explored torrefaction dynamics in two woody biomasses, Juniper and Mesquite, characterized by their wood types, the former being a softwood and the latter a hardwood. In this context, Mesquite displayed a heightened loss in lignin peak compared to Juniper, while Juniper exhibited a greater cellulose peak loss. The observed thermal degradation variance is attributed to the inherent distinctions in wood types, specifically the classification of Juniper as a softwood and Mesquite as a hardwood [15].

Torrefaction is also influenced by various parameters, with temperature exerting the most significant impact, as elucidated [10], [13], [16]. The degradation process of biomass is fundamentally contingent on temperature, and while time and temperature can be interchangeable for certain product properties, the overall influence of torrefaction temperature surpasses that of residence time and time-temperature interaction terms [17].

Elevated temperatures during torrefaction result in lower mass and energy yields but contribute to higher energy density, as documented by Bridgeman et al. and Medic et al. [18], [19]. This outcome is attributed to an increase in fixed carbon content, with the fraction of fixed carbon escalating with rising temperatures.

In contrast, the impact of residence time on torrefaction is less pronounced compared to temperature, where longer residence times yield lower solid output and higher energy density. However, this effect tends to diminish after approximately one hour [20]. Additionally, Arias asserts that the mass loss during torrefaction between 30 minutes and 3 hours is relatively similar [21].In this study, established crop residues liable to open burning from Indonesia were selected for torrefaction. A comprehensive parametric study of fast torrefaction at 300℃ for 5 minutes and slow torrefaction at 250℃ for 45 minutes was subsequently conducted, utilizing a static tube reactor. The primary objective was to investigate the potential benefits that selected crop residues, typically subject to open burning, may offer as an alternative energy feedstock, thereby mitigating the open burning practices.

MATERIAL AND METHODS

Sample Preparation and Selection

The process of sample preparation involves various steps such as sampling, moisture analysis, drying, cutting, grinding, and property analysis. These steps aim to identify the optimal samples for use as energy feedstock, considering their specific properties. The selection procedure is depicted in **Figure 1**.

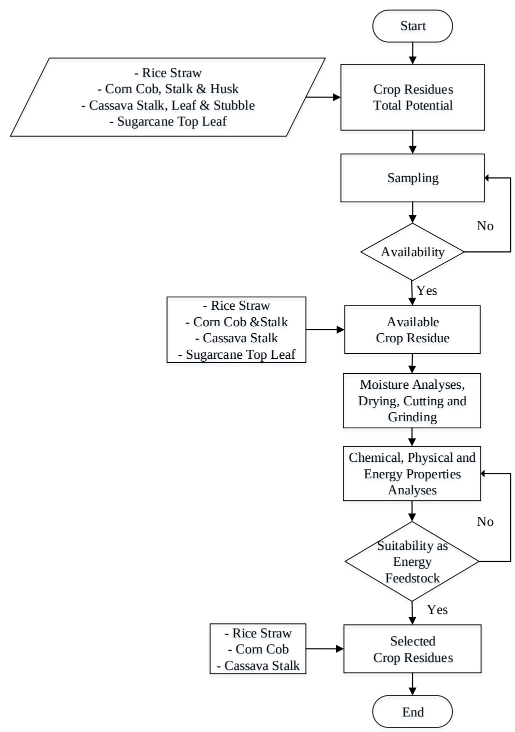

Figure 1. Flowchart sample preparation.

Rice straw was gathered from the Metro district in Lampung Province, while corncob, corn stalk, and corn leaf husk were obtained from Tegineneng in the Pesawaran district of Lampung Province. It's important to note that only corn cob and corn stalk could be transported to Thailand, as corn peels and leaf-husk harbored fungus and small insects. These were prohibited by the Quarantine

Department due to the potential risk of pest propagation. Cassava stalk was procured from the Jati Agung district in Southern Lampung Province. Finally, sugarcane tops and leaves were assembled from the Bunga Mayang district in Northern Lampung Province. The collection of all these samples is visualized in **Figure 2**.

Figure 2. Samples of crop residues (a) Rice straw, (b) Corncob, (c) Cornstalk, (d) Cassava stalk, (e) Sugarcane top and leaf

Sample Elemental Analysis

The elemental analysis for crop residues and torrefied crop residues covered the ultimate, proximate, calorific value, bulk density, emissions gas, and combustion behavior analyses. The analysis procedures are based on ASTM standards and utilized through the instrument, as shown in **Table 1**.

Table 1. Instrumentation for Torrefied Agricultural Crop Residues Compositional Analysis

Biomass Constituent	Apparatus
Ultimate Analysis	Thermo-Finnigan, Flash EA 1112 Series
Proximate Analysis	PerkinElmer, TGA Pyris1
Calorific values	LECO AC-350
Bulk Density	Standardize box
Emissions	490 Micro GC
Combustion behavior	TGA-50, SHIMADZU

Apparatus Setup and Procedure

Two different conditions (slow and fast torrefaction) had been prepared. The slow torrefaction was operated at 250℃ for 45 minutes, while 300℃for 5 minutes was for fast torrefaction. The schematic diagram of the static tubular reactor is in **Figure 3**.

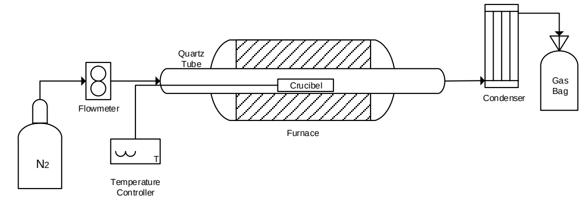

Figure 3. Schematic Diagram of the Static Tubular Reactor.

The experimental procedure entailed a sequential series of steps. Initially, samples were loaded into a tube situated atop a furnace and allowed to reside for the prescribed duration as determined by the required residence time. Both extremities of the tube were effectively sealed with a silicon bed. Subsequently, an inert atmosphere was established by purging nitrogen into the tube at a flow rate of 100 ml/min following a 10-minute purging phase. Throughout torrefaction, temperature measurement employed a thermocouple. Control over temperature, residence time, and a heating rate of 10°C/min was exercised through a dedicated controller. Emitted volatiles and gases followed through an outlet port during the procedure. Upon reaching the specified temperature, the residence time commenced, during which hot gas was collected in a gas bag for immediate analysis using gas chromatography. Subsequent cooling facilitated the analysis of torrefied samples, encompassing ultimate, proximate, and calorific values, densities, and lignocellulosic composition assessments.

RESULTS AND DISCUSSION

Chemical Properties

Table 2 presents the carbon content of various materials, revealing that cassava stalk boasts the highest carbon content at 48.66%. Following closely are corncob, sugarcane top leaves, rice straw, and corn stalk, registering carbon contents of 48.22%, 46.04%, 44.24%, and 40.95%, respectively. This carbon content trend correlates almost proportionally with their respective calorific values. Cassava stalk commands the leading position in calorific value at 22.75 MJ/kg, succeeded by corncob at 22.48 MJ/kg, sugarcane top leaves at 20.80 MJ/kg, corn stalk at 17.35 MJ/kg, and finally rice straw at 15.11 MJ/kg. It is noteworthy that rice straw exhibits the lowest calorific value due to its notably high ash content of 22.06%, while corn stalks ash content stands at a mere 0.98%. Despite its impact on volatile matter and fixed carbon, the elevated ash content holds potential benefits as a catalyst in thermal conversion processes [22], [23].

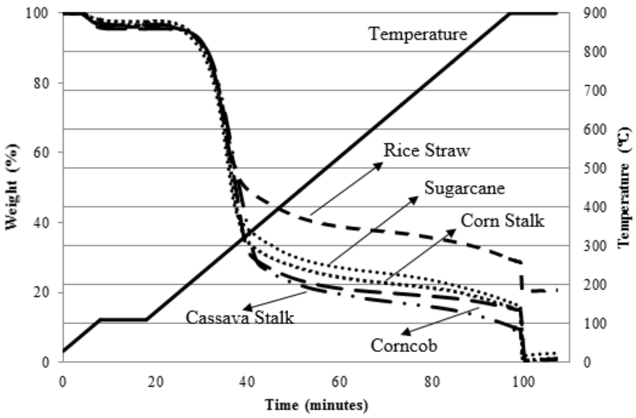

Figure 4. TGA graph of raw agricultural crop residues.

In **Figure 4**, it is evident that the initial crop residue to undergo reactions in the process is corn stalk, while corn cob participates as the final residue. Notably, during the process, cassava exhibits a higher conversion rate primarily due to its substantial volatile matter content of 89.10%. On the contrary, rice straw demonstrates the lowest conversion rate, attributed to its elevated ash content of 22.06%. Regrettably, in terms of volatile matter (VM), fixed carbon (FC), and ash content, no significant differentiation is observed between corn cob and corn stalk, both registering values of 83.85%, 15.36%, and 0.78% for corncob, and 83.91%, 15.11%, and 0.98% for corn stalk, respectively. As a result, a single representative from the corn residues was chosen.

Among the available crop residues – rice straw, corncob, corn stalk, cassava stalk, and sugarcane top and leaf – only three were chosen as the most suitable options for energy feedstock. The selection process hinged on their chemical properties, energy characteristics, and mass potential, outlined as follows. Despite its lower calorific value, rice straw was favored due to its significantly higher mass potential. In contrast, while sugarcane exhibited a higher calorific value than rice straw and corn stalk, its mass potential was the lowest, leading to its elimination. Considering corn cob and corn stalk, despite their similar volatile matter and fixed carbon content, corn stalk's heating value and carbon content ranked second lowest, leading to its exclusion. In conclusion, rice straw (RS), corncob (CC), and cassava stalk (CS) stood out as suitable samples for energy feedstock in the context of torrefaction.

Table 2. Chemical properties of raw agricultural crop residues before torrefaction.

Samples	**Ultimate Analyses (% d.a.f)**				**Proximate Analyses (% dry basis)**			**HHV_{db} (MJ/kg)**
	C	**H**	**N**	**O**	**VM**	**FC**	**ASH**	
Rice Straw (RS)	44.24	7.95	1.24	46.57	69.13	8.81	22.06	15.11
Corncob (CC)	48.22	8.70	0.53	48.22	83.85	15.36	0.78	22.48
Cassava Stalk (CS)	48.66	8.85	1.38	48.66	89.10	9.34	1.56	22.75
Corn Stalk	40.95	7.26	0.63	40.95	83.91	15.11	0.98	17.35
Sugarcane Top Leaf	46.04	8.48	1.42	44.06	82.08	15.01	2.91	20.80

Table 3. Chemical properties of raw agricultural crop residues after torrefaction at 250 °C.

Samples	**Ultimate Analyses (% d.a.f)**				**Proximate Analyses (% dry basis)**			**HHV_{db} (MJ/kg)**
	C	**H**	**N**	**O**	**VM**	**FC**	**ASH**	
RS	49.58	6.24	1.27	42.91	58.11	14.93	26.96	14.19
CC	51.35	5.94	0.40	42.31	73.98	23.70	2.32	20.02
CS	50.06	6.43	1.18	42.34	83.59	13.24	3.18	19.93

Table 4. Chemical properties of raw agricultural crop residues after torrefaction at 300 °C.

Samples	**Ultimate Analyses (% d.a.f)**				**Proximate Analyses (% dry basis)**			**HHV_{db} (MJ/kg)**
	C	**H**	**N**	**O**	**VM**	**FC**	**ASH**	
RS	57.32	5.92	1.61	35.14	47.30	20.83	31.87	15.22
CC	58.10	5.36	0.58	35.95	63.99	34.35	1.66	22.46
CS	54.85	5.94	1.44	37.77	70.82	24.34	4.84	21.04

During the torrefaction process, a discernible decrease in the volatile matter was observed, which exhibited a direct correlation with elevated temperatures and prolonged retention periods. Conversely, the levels of fixed carbon and ash content demonstrated an ascending trajectory. Furthermore, there was a conspicuous augmentation in the proportion of carbon content concomitant with the escalation of temperature. A reduction in the hydrogen percentage was noted, attributed to the liberation of steam, methane, and ethane.

Notably, the proportion of oxygen content also underwent a decline, attributable to the production of carbon dioxide and carbon monoxide, as elucidated in **Table 3** and **Table 4** [24].

In terms of crop residue classification, the corncob exhibited the highest carbon content, fixed carbon, and calorific values, exerting a discernible influence on the other parameters, across various temperatures and reactor types, followed by cassava stalk and rice straw. The result was linear to Prins et al., non-woody biomass demonstrated an increase in higher heating value (HHV) during torrefaction, ranging from 0.4% to 14.9%, primarily attributed to heightened carbon content [25]. Mukhtar et al. stated that the higher the temperature and residence time in torrefaction, the higher the yield of HHV, due to the dehydration, devolatilization, and depolymerization of biomass [26]. For instance, at 300°C, the carbon content of corncob reached 58.10%, its fixed carbon content was 34.35%, and its calorific value was 22.46 MJ/kg. In contrast, rice straw displayed values of only 57.32%, 20.83%, and 15.22 MJ/kg for carbon content, fixed carbon content, and calorific value, respectively.

Furthermore, an increasing trend in carbon content was observed as the temperature rose, while hydrogen and oxygen content exhibited a concomitant decrease. Volatile matter diminished initially, followed by an increase in fixed carbon and ash content with escalating temperatures. For example, in the case of cassava stalk at 250°C, the carbon content was 50.06 wt.%, hydrogen content was 6.43 wt.%, and oxygen content was 42.34 wt.%. At 300°C, these values changed to a carbon content of 54.85 wt.%, a hydrogen content of 5.94 wt.%, and an oxygen content of 37.77%, denoting an increase in carbon content by 9.57%, a decrease in hydrogen content by 7.62%, and a decrease in oxygen content by 10.79%. These variations were attributed to the formation of methane, steam, carbon monoxide, and carbon dioxide [27].

Physical Properties

Figure 5 shows the different colors of raw biomass and torrefied biomass. Raw corncob (CC) has a light yellow color and cassava stalk (CS) has a dark yellow color, while rice straw (RS) has a light chocolate color. After torrefaction, the three-biomass changed to dark chocolate at 250 °C and black color at 300 °C. The biomass color change after torrefaction indicates that increase of C carbon as well as a decreasing weight loss of biomass [28].

These results are in accordance with findings by Hidayat, subjecting the wood to heat treatment was able to induce alterations in its color as a consequence of hemicellulose degradation [29]. A corresponding investigation undertaken by Yulianto affirmed that the color transformation observed in oil palm empty fruit bunch pellets occurs, manifesting as a shift to a darker or black hue subsequent to the process of torrefaction[30]. Hadiyane also asserted that the change in color occurs due to temperature variations, leading to lignin relocation and chemical degradation of wood components [31].

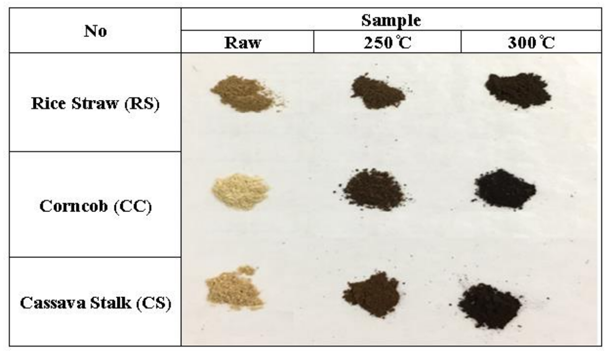

Figure 5. Visualization of the sample before and after torrefaction.

TGA Analysis

Figure 6 shows the weight loss behavior of crop residues during the combustion process in the TGA as well as the DTG curve. The curve has two well-defined peaks. The first peak was mostly related to the combustion of volatile matter, which was mostly dominated by cellulose and hemicellulose. Further, the second peak was related to the combustion of fixed carbon and lignin which the same result was also found by previous studies [32], [33]

The combustion process of volatile matter in rice straw exhibited the lowest power generation in comparison to corncob and cassava stalk, both prior to and after torrefaction. As demonstrated by the example of raw crop residues, rice straw produced power at a rate of 0.016 Watt, followed by corncob (0.034 Watt) and cassava stalk (0.036 Watt).

Conversely, the power generation pattern underwent a minor alteration during the combustion of fixed carbon. Rice straw generated the least power, followed by cassava stalk and corncob. This disparity is attributed to the distinct compositions of volatile matter and fixed carbon within each type of crop residue.

The power generation during the combustion of volatile matter in torrefied crop residues declined due to the liberation of volatiles during the preceding torrefaction process. Conversely, power generation during the combustion of fixed carbon experienced a notable increase owing to the formation of fixed carbon during torrefaction. For instance, in the case of cassava stalk, power generation during the combustion of volatile matter decreased by approximately 17%, reaching 0.03 Watt. In contrast, power generation during the combustion of fixed carbon increased by 70%, reaching 0.017 Watt.

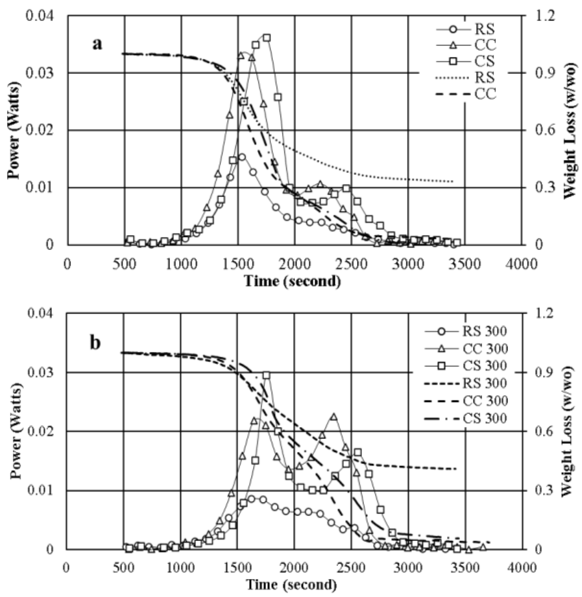

Figure 6. DTG curve of combustion Process:
(a) raw agricultural crop residues &
(b) Torrefied raw agricultural crop residues.

Additionally, it was observed that rice straw exhibited the earliest onset of weight loss during the combustion process, followed by corncob and cassava stalk. This pattern persisted in both the weight loss attributed to volatile and fixed carbon, regardless of whether the analysis was conducted on raw crop residues or torrefied counterparts.

Emission Analysis

Unlike corncob and cassava stalks, the ratio of CO/CO_2 of rice straw is higher than others. The ratio at 300°C is higher than 250°C because of the increasing formation of emissions with the increasing of temperature as shown in **Figure 7**. This ratio is also affected by the composition of carbon, hydrogen, and oxygen content of each crop residue. In the case of cassava, it has the lowest carbon and oxygen content compared to the other two crop residues, thus it has the lowest ratio of CO/CO_2. In relation to crop residues, cassava emitted less emissions followed by corncob and rice straw. For example, at 300°C cassava emits 26.85 ml of CO_2 followed by corn cob (28.15 ml) and rice straw (28.94 ml). The cassava stalk also emits the lowest emissions of carbon monoxide (10 ml) followed by corncob (15 ml) and rice straw (18 ml). Therefore, in terms of emissions, cassava stalk is the most prominent sample for torrefactions compared to the other two samples of crop residues. Nevertheless, cassava is a perennial plantation, thus the sustainability of its supply was not secured.

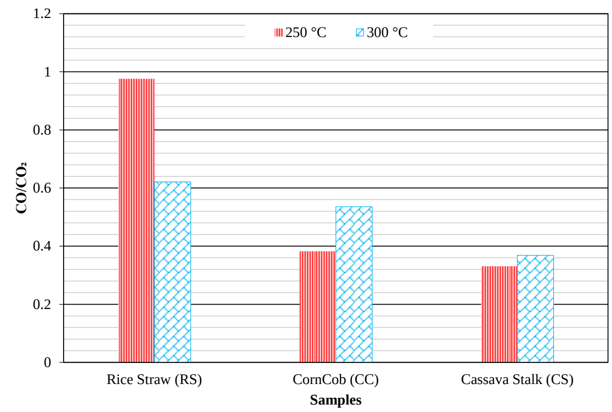

Figure 7. Carbon ratio of samples after torrefaction.

CONCLUSIONS

There are several points from this study to be concluded as follows:

- Proximate-ultimate analysis and higher heating value (HHV) assessment reveal notable distinctions among the examined biomass samples. Across varying temperatures, corncob consistently exhibits the highest levels of carbon content, fixed carbon, and calorific values. Notably, at 300°C corncob attains a carbon content of 58.10%, a fixed carbon content of 34.35%, and a calorific value of 22.46 MJ/kg. In contrast, rice straw presents comparatively lower values, registering at 52.43% carbon content, 20.31% fixed carbon, and 16.56 MJ/kg heating value.
- These results show that torrefaction can contribute to mitigating open burning by increasing the energy properties of crop residues and so be potentially valuable as energy feedstock. However, the supply of crop residues is influenced by seasonal production, and this is an issue for the industry which would require a constant supply in such feedstock as an energy source.

AUTHOR CONTRIBUTIONS

AA & PR are the research conceptor and design. BAF, DBIN, EFD, and HB contribute to the data collection data and experiment. A, ERF, F, EWT, MS, I, and HS contribute to the data analysis and interpretation. AA writes the initial manuscript. AA, HS, and PR perform the critical revision of the article. AA and PR ensure final approval of the article.

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