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Parametric study of rice husk torrefaction for the development of sustainable solid fuel

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Abstract

Torrefaction is the thermal treatment of lignocellulosic biomass at temperatures between 200 ℃ to 300 ℃ under inert or low oxygen environment. This process produces a dark brown, brittle substance that is hydrophobic, has high energy density, and less prone to biological degradation. The removal of oxygen-containing compounds is the primary purpose of torrefaction. Oxygen to carbon ratio is highly correlated to the biomass heating value. An increase in oxygen to carbon ratio from 0.1 to 0.7, results to a 60% reduction in higher heating value of the biomass. Removal of oxygen-containing compounds lowers the oxygen to carbon ratio thus increasing the higher heating value of torrefied biomass. These improved properties are significant for thermochemical conversion processes such as combustion and gasification particularly for power generation purposes. This study examines the impact of residence time, temperature, and particle size on torrefied rice husk, using a bench-scale batch reactor. Simultaneous variation of temperature between 240 ℃ and 295 ℃; residence time between 30 minutes and 60 minutes; and particle size between 1.19mm and 2.38mm were done. The results showed significant improvement in higher heating value of rice husk which increased by up to 25% from 13.4MJ/kg to 16.8MJ/kg. The fixed carbon increased up to 107% from 12.8 to 26.6 weight percent in dry basis. Analysis of results show that process temperature significantly affects the fuel properties of torrefied rice husk compared to residence time, particle size, and the interaction of these factors.

Keywords: torrefaction, rice husk, thermal pretreatment, biomass energy

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NOMENCLATURE

RRH = Raw Rice Husk
TRH = Torrefied Rice Husk
EMC = Equilibrium Moisture Content
OH = Hydroxyl group
HHV = Higher Heating Value
ANOVA = Analysis of Variance
TGA = Thermogravimetric Analysis
DTG = Derivative Thermogravimetric Analysis

1. Introduction

The Philippines is an emerging economy. With an annual growth rate of 6%, domestic consumption of products, services, and energy is expected to rise. Assuming a business-as-usual scenario, coal will be the leading source of energy contributing 69% to the total power generation mix by 2040 [1]. Heavy importation of this fossil fuel will continue in parallel to improving economic conditions. This will pose both environmental and energy security challenges for the country in the absence of policies requiring

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implementation of advanced coal technologies and quality improvement standards. Adapting a bio-based economy can significantly benefit the Philippines given its abundant biomass resources. The Philippine government initiates this shift to sustainability through the establishment of the National Renewable Energy Program that aims to increase renewable energy-based capacity for power generation. In its 20-year roadmap (2010-2030), delivery of additional 277MW of biomass power capacities is a priority.

Rice husk is among the top three biomass resources in the Philippines. It is typically used as an insulation material, animal bedding, or as a fertilizer additive. These non-energy uses consume about 1% of the total annual rice husk produced in the country leaving 99% for energy uses [2]. Despite the maturity of combustion and gasification technologies, problems still persist in the conversion of rice husk to power. Low energy density, affinity for water, and its high inherent moisture are some of the challenges faced by end-users. Transportation costs on the other hand, create a huge impact on the overall cost of recovering energy from agricultural waste [3].

Torrefaction is a technology that can address the above-mentioned barriers in the utilization of rice husk or any lignocellulosic feedstock for power generation. Slow-heating a biomass to 200-300 ℃, under atmospheric pressure, in an inert or low oxygen environment, degrades the fibrous lignocellulosic network of the plant walls and releases volatile substances. Overall, the final product has a lower mass; higher energy density; and less affinity for moisture in comparison to its original form [3].

This research studies the impact of temperature, residence time, and particle size on the heating value, solid yield, energy yield, energy density, and fixed carbon content of torrefied rice husk, using a batch reactor. Data derived from this experimental research will contribute to the existing body of knowledge on the characterization of agricultural residues and its potential as alternative fuel in the power sector.

2. Rice Husk Torrefaction Experiment

2.1. Torrefaction set-up

A schematic diagram of the torrefaction set-up is shown in Figure 1. This system can be subdivided into three sub-units namely:

- The torrefaction unit;
- The condensate collection unit; and
- The gas scrubbing unit

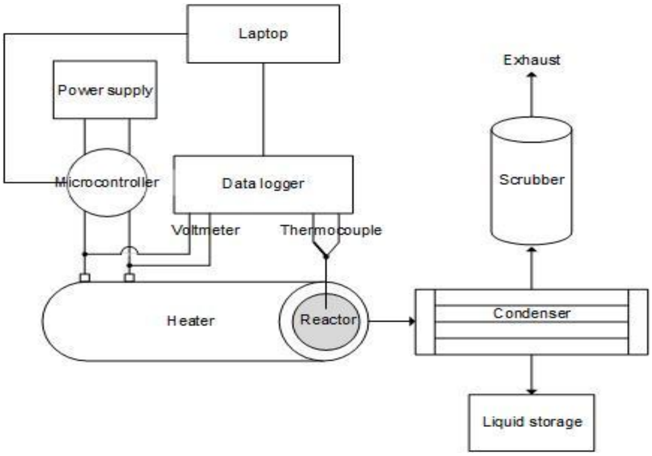


Fig. 1. Schematic Diagram of the Batch Torrefaction System

The torrefaction unit is a 42-liter stainless steel cylindrical vessel with a detachable cover on one end. RRH was loaded into the reactor then sealed. The batch reactor was heated using electric heating bands located on the outer surface of the vessel. Torrefaction temperature was controlled using a proportional integral derivative controller, set to ramp from ambient temperature to the desired temperature, at the rate of 10 ℃ per minute. When the target temperature was reached the heater turns on or off, based on a reference temperature, to maintain the target temperature inside the batch reactor at the required residence time. During torrefaction, gases that evolved are directed to the condenser through a 3/8 inch (outer diameter) copper tubing coil. This coil was submerged in water at room temperature. The liquid product that exits the coil was collected at the bottom of the condenser. Non-condensable gases were directed to a series of scrubbers for cleaning, and then released to the atmosphere. A flow meter was used to measure the flow rate of non-condensable gases that evolved in each experimental run.

After each trial, the torrefaction unit was allowed to cool to room temperature before collecting the TRH samples. For data gathering, a type-K thermocouple was fitted through a thermowell to properly measure the temperature inside the reactor. The thermocouple then sends the reading to the Fluke Hydra Series II logger that is connected to a computer. Voltage readings were also monitored during each run. Sufficient power was available in the laboratory where the torrefaction reactor was stationed.

2.2. Material preparation

Air dried samples of rice husk were collected from General Mamerto Natividad, Nueva Ecija, Philippines and stored in the laboratory at room temperature. The physico-chemical properties of the rice husk used in this study, such as moisture content, volatile matter, and HHV, are summarized in Table 1:

Table 1. Proximate analysis results and the HHV for RRH

Composition	As-received	Dry Basis
Moisture, wt%	7.35	-
Volatile Matter, wt%	56.2	60.7
Ash Content, wt%	24.5	26.4
Fixed Carbon, wt%	11.8	12.7
HHV, MJ/kg	12.4	13.4

The RRH samples were sieved into two different sized fractions: 2.38mm and 1.19mm using Tyler sieve numbers 8, 10, 12, 16. The RRH retained at sieve number 8 and sieve number 16 were collected and divided into twenty-four parts: 12 parts retained in sieve number 8 and 12 parts retained in sieve number 16. The weight of each partition is 200 grams, each stored in resealable plastic bags and stored in the laboratory at ambient conditions.

2.3. Operating conditions

Torrefaction temperature, residence time, and particle size are three factors that influence the devolatilisation rate of biomass. The simultaneous impact of these independent variables on the volatile matter, fixed carbon, hydrophobicity, solid yield, energy yield, and energy density was studied using a 2^3 factorial design with three replicates. Table 2 shows the factorial design for RRH torrefaction while Table 3 shows the design matrix for this study:

Table 2. 2^3 Factorial experimental design for rice husk torrefaction

Factor	Level 1	Level 2
Temperature, ℃	240	295
Residence time, min	30	60
Particle size, mm	Retained at mesh no. 16	Retained at mesh no. 8

Table 3. 2^3 Factorial experimental design matrix for rice husk torrefaction

Run Order	Temperature, ℃	Residence time, min	Particle size, mm
1	240	60	retained at mesh 8
2	240	30	retained at mesh 16
3	240	30	retained at mesh 16
4	295	60	retained at mesh 8
5	240	30	retained at mesh 8
6	240	60	retained at mesh 16
7	240	30	retained at mesh 8
8	295	30	retained at mesh 8
9	295	60	retained at mesh 16
10	240	60	retained at mesh 8
11	240	60	retained at mesh 16
12	240	60	retained at mesh 16
13	295	30	retained at mesh 8
14	295	60	retained at mesh 16
15	295	30	retained at mesh 16
16	240	60	retained at mesh 8
17	295	30	retained at mesh 16
18	295	60	retained at mesh 8
19	240	30	retained at mesh 8
20	295	60	retained at mesh 8
21	295	60	retained at mesh 16
22	240	30	retained at mesh 16
23	295	30	retained at mesh 16
24	295	30	retained at mesh 8

2.4. Sample analysis

The proximate analysis of RRH and TRH was performed according to the standard procedure of the American Society for Testing and Materials (ASTM E1131-08) using TA Instruments Q500 thermogravimetric analyzer. The calorific value of RRH and TRH samples was determined using the Parr 6200 Oxygen Bomb Calorimeter at the Department of Chemical Engineering.

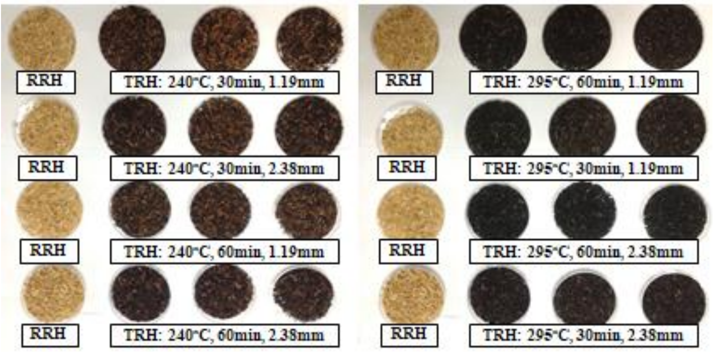
For the hydrophobicity test, the exposure method was used where TRH samples were stored for two weeks in a high relative humidity environment. This condition was achieved by preparing saturated K_2SO_4 solution (relative humidity of 97.3% ± 0.45) in an enclosed container. The K_2SO_4 used for the hydrophobicity test set-up is in powder form with an assay of 99.9% purity (trace metals basis).

3. Results and Discussion

To determine which among the factors (temperature, residence time, and particle size) and the corresponding interactions had significant effect on the fuel properties of TRH, statistical analyses of the data for TRH were conducted using the Minitab 17 software. Results showed that temperature had the most significant effect on the changes in volatile matter, fixed carbon, heating value, hydrophobicity and the process performance indicators (solid yield, energy yield, energy density).

3.1. Solid product composition and structure

After conducting the torrefaction trials, a distinct change in the physical appearance of the rice husk samples was observed. Fig. 2 shows photos of the RRH and TRH. Rice husk changed its color from golden yellow to brown to black. RRH samples that were torrefied at 240 ℃ had a dark brown color while RRH samples torrefied at 295 ℃ turned black similar to carbonized biomass.



(a) (b)

Fig. 2. a. RRH and TRH at 240 ℃; b. RRH and TRH at 295 ℃

This change in color reflects the chemical compositional changes occurring in the major components of rice husk namely hemicellulose, cellulose, and lignin. The main reactions that changed the biomass color are the following [4]:

- Devolatilization and carbonization of hemicellulose;
- Depolymerization and softening of lignin;
- Depolymerization and devolatilization of cellulose

As temperature increases, the color change of rice husk becomes more prominent due to the occurrence of these reactions. At higher torrefaction temperature more volatiles are released as hemicellulose and cellulose degrades, allowing the higher percent removal of oxygenated and hydrogenated compounds. This phenomenon leaves the torrefied solid product high in fixed carbon content. Figure 3 shows the TGA and DTG of RRH.

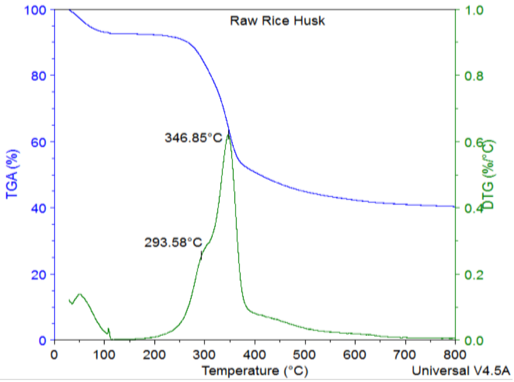


Fig. 3. TGA and DTG Curves of RRH

The blue line pertains to the TGA curve while the green line pertains to the DTG curve. Two peaks can be observed in the DTG plot of RRH at the temperature range of 200 ℃ to 400 ℃. The first temperature peak is at 293.58 ℃, while the second temperature peak is at 346.85 ℃. Shafizade et al. reported that hemicellulose typically decomposes between 150 ℃ to 350 ℃; cellulose decomposes between 275 ℃ and 350 ℃; and lignin decomposes gradually between 250 ℃ and 500 ℃ [5]. For RRH, the first peak at 293.58 ℃ is attributed to the decomposition of hemicellulose and the initial stages of cellulose and lignin decomposition. The peak at 346.85 ℃ is attributed to the decomposition of cellulose. The chosen torrefaction temperatures, 240 ℃ and 295 ℃ fall within the temperature region where hemicellulose and cellulose degrades. At 240 ℃, a portion of the hemicellulose content of RRH has degraded while at

295 ℃, portions of lignin, cellulose and all hemicellulose content has degraded.

The TGA and DTG curves of the RRH were superimposed with the TGA and DTG curves of the TRH samples. These are shown in Figures 4 and 5 respectively. In the TGA curves, it is observed that the weight of RRH drops from 100% to 40% of the original mass at 800 ℃. For TRH processed at 240 ℃, the weights drop from 100% to 45%-50% while for TRH processed at 295 ℃, the weight drops to 55%-65% of the original mass.

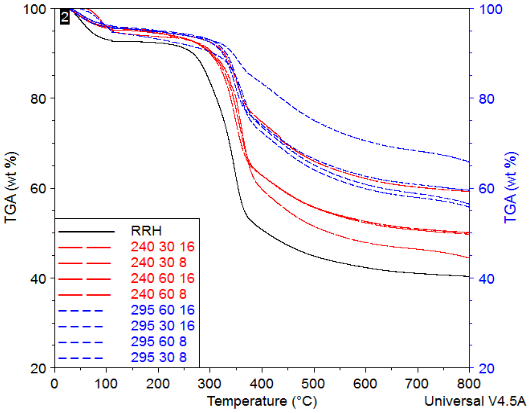


Fig. 4. TGA Curves of RRH and TRH at various conditions

Comparing the TGA plot of RRH with the TGA plots of TRH, an upward shift of the curves can be seen. As torrefaction temperature increases, mass loss in the TRH decreases. Torrefaction of RRH at 295 ℃ results to the release of more volatile compounds as hemicellulose degrades completely, leaving a higher percentage of solids in the TRH.

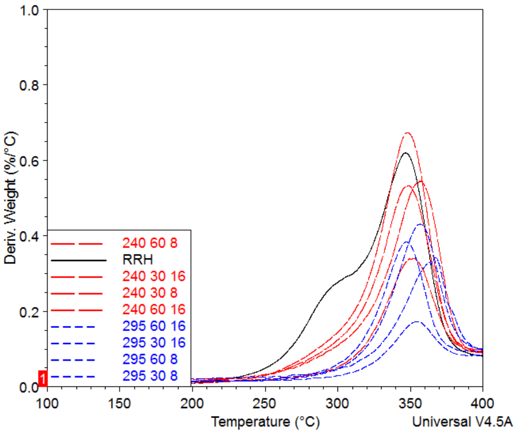


Fig. 5. DTG Curves of RRH and TRH at various conditions

Upon reviewing the 295 ℃ DTG curves, only the cellulose peak can be observed indicating complete degradation of the hemicellulose in the TRH. Furthermore, the maximum values of the cellulose peaks shown are decreasing as torrefaction temperature is increased. It is concluded that in addition to the complete degradation of hemicellulose at 295 ℃, torrefaction at this temperature destroys a portion of the cellulose in rice husk.

3.2. Proximate analysis and heating value

The proximate analysis and higher heating value of RRH and TRH are summarized in Table 4. After torrefaction, the lowest observed volatile matter content was 31.5% at 295 ℃, 60-min residence time, and a particle size of 2.38mm. At 240 ℃, the lowest volatile matter was 49%. This is a 19.2% decrease with respect to the original volatile content (in dry basis). ANOVA results showed that temperature, has the most significant effect on volatile matter content compared to residence time and particle size. At 295 ℃, the volatile matter decreased up to 48.1%. Torrefaction at 295 ℃ releases more volatile substances as the hemicellulose and cellulose content of rice husk degrade. For fixed carbon the highest observed fixed carbon content was 26.6% (dry basis) at 295 ℃, 60-min residence time, and a particle size of 2.38mm.

Table 4. Proximate analysis and higher heating valus of RRH and TRH

Torrefaction Conditions	Ash, wt % (dry basis)	VM, wt % (dry basis)	FC, wt % (dry basis)	HHV, MJ/kg (dry basis)
Raw Rice Husk	26.5	60.7	12.8	13.4
240, 30, 1.19	36.4	49.0	14.7	15.6
240, 30, 2.38	30.0	52.9	17.1	15.2
240, 60, 1.19	31.4	50.3	18.3	15.4
240, 60, 2.38	32.4	49.0	18.6	15.5
295, 30, 1.19	38.7	37.8	23.5	16.4
295, 30, 2.38	35.9	42.4	21.7	16.3
295, 60, 1.19	36.5	41.0	22.6	15.8
295, 60, 2.38	41.9	31.5	26.6	16.8

The initial HHV of the RRH was 13.4MJ/kg (dry basis). For all conditions considered in the study, an increase in the HHV was observed. These corresponding increases are summarized in Table 5. After torrefaction, the highest observed HHV was 16.8MJ/kg (dry basis) at 295 ℃, 60-min residence time, and a particle size of 2.38mm.

Table 5. Percent change of HHV of TRH

Torrefaction Conditions	HHV, MJ/kg (dry basis)	Increase
Raw Rice Husk	13.4	-
240, 30, 1.19	15.6	16.4%
240, 30, 2.38	15.2	13.4%
240, 60, 1.19	15.4	14.9%
240, 60, 2.38	15.5	15.7%
295, 30, 1.19	16.4	22.3%
295, 30, 2.38	16.3	21.6%
295, 60, 1.19	15.8	17.9%
295, 60, 2.38	16.8	25.4%

As temperature increases, the HHV of the TRH increases due to the increase in the degree of degradation of the RRH components. Oxygenated and hydrogenated compounds are removed through devolatilization of the cellulose and hemicellulose components of rice husk, thus leaving the solid fuel with high concentration of fixed carbon. Fixed carbon has the most contribution to the heating value of a solid fuel. It is concluded that the HHV of the TRH is a result of the higher concentration of fixed carbon in the torrefied samples compared to RRH. The proximate analysis for carbonized rice husk given by Paethanom et al., [6] shows comparable results to that of TRH. Fixed carbon for carbonized rice husk is at 26.4% while that of TRH is at 26.6% maximum. These results show that torrefaction can achieve comparable fixed carbon requirement at a lower process temperature. Table 6 shows the proximate analysis results of RRH, TRH, and carbonized rice husk.

Table 6: Proximate analysis of RRH, TRH, and carbonized rice husk

Proximate Analysis	Ash, wt % (dry basis)	VM, wt % (dry basis)	FC, wt % (dry basis)
Raw Rice Husk	26.5	60.7	12.8
Carbonized Rice Husk at 600°C	51.69	21.94	26.37
Torrefied Rice Husk			
240, 30, 1.19	36.4	49.0	14.7
240, 30, 2.38	30.0	52.9	17.1
240, 60, 1.19	31.4	50.3	18.3
240, 60, 2.38	32.4	49.0	18.6
295, 30, 1.19	38.7	37.8	23.5
295, 30, 2.38	35.9	42.4	21.7
295, 60, 1.19	36.5	41.0	22.6
295, 60, 2.38	41.9	31.5	26.6

3.3. Hydrophobicity

Hydrophobicity or the uptake of water by a biomass is attributed to the presence of OH groups in its structure. Torrefaction makes rice husk hydrophobic by destroying these OH groups to lessen its hydrogen bonding capacity. The hydrophobic characteristic of TRH samples was determined through exposure test in saturated K_2SO_4 solution with a relative humidity of 97%. As shown in Table 7, the moisture content of all samples increased however there is lower moisture absorption of TRH processed at 295 °C compared to TRH processed at 240 °C and RRH. Note that the moisture reported here is not the moisture content of the solid product but rather the EMC of the TRH.

Table 7. EMC of RRH and TRH at 97% Relative Humidity (in weight percent)

	Exposure Hours							
Torrefaction Conditions	0	24	48	144	168	192	312	384
	Equilibrium Moisture Content							
240,30,16	0%	7%	9%	11%	11%	11%	12%	12%
240,30,8	0%	7%	9%	12%	12%	12%	12%	13%
240,60,16	0%	7%	9%	11%	11%	11%	12%	12%
240,60,8	0%	7%	9%	11%	11%	11%	12%	12%
295,30,16	0%	6%	8%	9%	10%	10%	10%	11%
295,30,8	0%	7%	8%	10%	10%	10%	11%	11%
295,60,16	0%	6%	8%	10%	10%	10%	10%	11%
295,60,8	0%	6%	8%	9%	9%	10%	10%	10%
RRH	0%	12%	14%	16%	16%	16%	16%	17%

3.4. Process performance indicators: Solid yield, energy yield, energy density

The solid yield of TRH was calculated using equation 1. Solid yield range is from 62% to 81.5%. Increasing the torrefaction temperature decreases the solid yield. This observation is explained by the decomposition of hemicellulose at 240 ℃ and 295 ℃. The TGA curve of RRH shows that at the peak temperature 293.58 ℃ hemicellulose may have achieved complete degradation. Thus, more volatile substances and moisture are removed from TRH samples processed at 295 ℃ compared to TRH samples processed at 240 ℃. Similar results are obtained by Sadaka et al., [7], Pimchuai et. al. [8], and Prins et al. [9].

$$Solid\ yield_{dry\ basis} = \frac{mass\ of\ torrefied\ biomass\ on\ dry\ basis}{mass\ of\ raw\ biomass\ on\ dry\ basis} \tag{1}$$

Energy yield is the fraction of original energy retained in the biomass after subjecting it to torrefaction. In the process, energy-lean components evolve from the biomass in the form of condensable and non-condensable gases, leaving high-energy components in the solid product. Equation 2 shows the relationship for calculating energy yield:

$$Energy\ yield = Solid\ yield * \frac{heating\ value\ of\ torrefied\ biomass}{heating\ value\ of\ raw\ biomass} \tag{2}$$

The calculated energy yield of the TRH ranges from 77.5% to 94.5%. The ANOVA showed that temperature and the interaction between temperature and residence time have the most significant effect on the energy yield of the TRH samples.

Energy density is a measure of the amount of energy released when a unit mass of torrefied biomass is combusted and its product is cooled [10]. It is a parameter that is often associated with terms such as heating value and specific energy. During torrefaction, energy-lean components of the biomass such as CO_2, H_2O, and other non-condensable gases become part of the lost mass of the solid product. A significant portion of the solids (mostly fixed carbon) and its corresponding energy content is retained after torrefaction. An energy yield greater than unity is favorable. Energy density of TRH was calculated using equation 3:

$$Energy\ density = \frac{Energy\ yield}{Solid\ yield} \tag{3}$$

The energy density of TRH ranged from 1.16 to 1.30. The highest observed energy density is at 295 ℃, 60-min residence time, and a particle size of 2.38mm. As the torrefaction temperature increases, the energy density of the solid product increases. At higher temperature the emission of CO, CO2, and H2O results to the decrease of the hydrogen and oxygen content of the RRH. Removal of these compounds increases the energy density since more fixed carbon are retained in the solid product.

4. Conclusion

An electrically-heated, 42-liter batch reactor with a condensate and gas collection system has been fabricated and used in this study. It is equipped with a temperature controller designed to achieve a ramp rate of 10 ℃ per minute and maintain the internal reactor temperature at 240 ℃ or 295 ℃. The Fluke Hydra Series II Data Logger recorded temperature readings throughout every experimental run. These data were retrieved by connecting a computer to the data logger.

The study showed that torrefaction resulted in significant improvement in higher heating value of rice husk which increased by up to 25% from 13.4MJ/kg to 16.8MJ/kg. The fixed carbon increased up to 107% from 12.8 to 26.6 weight percent in dry basis. Volatile matter decreased by 48% (weight % in dry basis) while the EMC of TRH decreased by 41% (weight %). The parametric study revealed that temperature had the most significant effect on heating value, solid yield, energy yield, and hydrophobicity of the TRH compared to residence time, particle size and the interactions of these three factors. Higher temperature was found more favorable for increasing fixed carbon, heating value, and hydrophobicity of the TRH.

Acknowledgements

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Appendix A: Raw Data for HHV Solid Yield, Energy Yield, Proximate Analysis

A.1. Raw Data for Solid Product Property Results

Run no.	Temp, ℃	Time min	Size, mm	HHV, MJ/kg	Solid Yield, %	Energy Yield, %	Energy Density	Moisture, %	VM, %	FC, %	Ash, %
2	240	30	1.19	14.65	81	95	1.18	5.87	53	10	31
3	240	30	1.19	15.00	68	82	1.21	4.40	40	18	38
5	240	30	2.38	13.84	84	94	1.11	5.32	48	20	26
6	240	60	1.19	14.18	80	91	1.14	5.54	47	20	27
9	295	60	1.19	15.55	65	81	1.25	5.36	36	27	32
10	240	60	2.38	14.26	83	95	1.15	4.90	45	22	27
11	240	60	1.19	15.09	78	94	1.21	4.83	49	14	32
13	295	30	2.38	15.29	69	85	1.23	5.03	39	26	30
14	295	60	1.19	14.54	63	74	1.17	4.17	42	16	38
15	295	30	1.19	15.92	67	86	1.28	4.42	34	20	41
16	240	60	2.38	15.21	77	95	1.22	4.55	48	13	34

17	295	30	1.19	15.58	70	88	1.25	4.06	38	25	33
18	295	60	2.38	16.22	63	82	1.31	4.17	33	20	43
19	240	30	2.38	15.00	79	95	1.21	4.83	52	12	31
20	295	60	2.38	15.99	61	79	1.29	4.27	28	31	37
24	295	30	2.38	15.80	68	87	1.27	4.49	41	16	39

A.2. Raw Data for Solid Product Property Results

Composition in % weight	**Raw Rice Husk**	**240°C, 30min, 1.19mm**	**240°C, 30min, 2.38mm**	**240°C, 60min, 1.19mm**	**240°C, 60min, 2.38mm**	**295°C, 30min, 1.19mm**	**295°C, 30min, 2.38mm**	**295°C, 60min, 1.19mm**	**295°C, 60min, 2.38mm**
Moisture	7.35	5.14 ± 0.010	5.07 ± 0.003	5.18 ± 0.005	4.73 ± 0.002	4.24 ± 0.003	4.76 ± 0.004	4.76 ± 0.008	4.22 ± 0.001
Ash	24.6	34.5 ± 0.050	28.5 ± 0.034	29.7 ± 0.034	30.9 ± 0.049	37.1 ± 0.062	34.2 ± 0.062	34.8 ± 0.043	40.1 ± 0.039
VM	56.2	46.4 ± 0.093	50.2 ± 0.029	47.7 ± 0.011	46.6 ± 0.020	36.2 ± 0.027	40.4 ± 0.012	39.0 ± 0.047	30.1 ± 0.036
FC	11.9	13.9 ± 0.053	16.2 ± 0.060	17.4 ± 0.041	17.8 ± 0.066	22.5 ± 0.038	20.6 ± 0.071	21.5 ± 0.081	25.5 ± 0.075