Characterisation and Thermochemical Conversion of Rice Husk for Biochar Production

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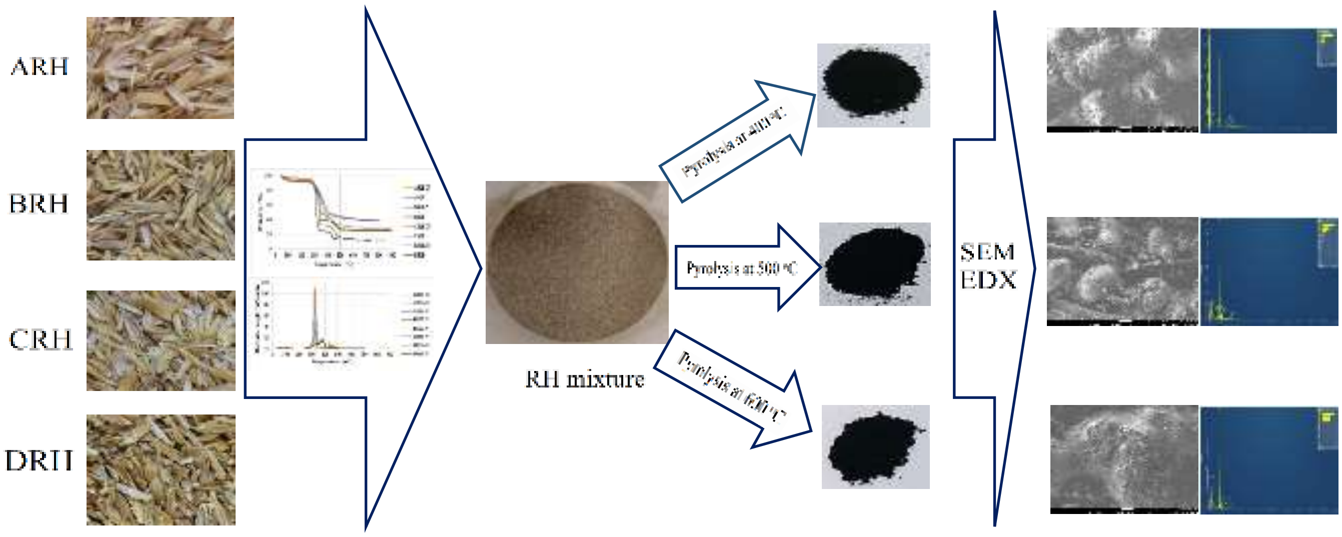
Received: 29.05.2018 Accepted:10.07.2018

Abstract- We present a thermochemical conversion of four varieties of rice husk samples i.e. Laila (ARH), Pusu (BRH), Bandul Beminyak (CRH) and Sendakan (DRH), collected from Brunei Darussalam. An extensive proximate and elemental analysis of the obtained samples is conducted. In addition, we thoroughly investigate temperature parameter as a function of weight loss for all samples via thermogravimetric analysis during respective pyrolysis and combustion. The study illustrated that three thermal decomposition stages occur during both pyrolysis and combustion. Hydrocarbon converts to volatiles in the second and third thermal decomposition stages. For the second and third stages of both pyrolysis and combustion, inhibitive and accelerative interactions, with temperatures ranging between 250 °C to 400 °C, and 430 °C to 900 °C in the former, respectively. And in the latter, the temperature varies between 250 °C to 350 °C, and 350 °C to 900 °C, respectively. The aim of this experimental work is to produce and characterise biochar from sample mixtures by pyrolysis in a fixed bed reactor. The experiments are conducted at different temperatures, ranging within 400, 500 and 600 °C. The most yield of biochar is found to be 49.5 wt. % at 400 °C. The bio-chars morphological structure and elemental contents are investigated by SEM and EDX to examine the physical and chemical changes.

Graphical abstract:



Keywords Rice husk, Thermochemical conversion, TGA, DTG, SEM.

1. Introduction

The development of clean, renewable, and sustainable energy has obtained more attention due to its low-cost greenhouse gas (GHG) emissions and the depletion of fossil fuel reserves [1-6]. Rapid rising population, industrialization and economic development has driven an increase in energy demand [7-9]. Fossil fuel has been used as a primary source of energy but its global impact on environment has expanded a significant search for renewable energy sources [10-13]. Rice husk (RH) can possibly have a huge effect on domestic fuel supplies in the rice producing countries like Brunei Darussalam and thus will help to meet Brunei's Vision 2035 for achieving the renewable energy goals [14]. Moreover, the production of biofuels and value-added products from RH can reduce the GHG emission which is important for Brunei Darussalam [15-17].

Rice is an important crop grown widely around the world [1]. The total world rice production is about 500 million tons per year [18]. Brunei total rice production in 2016 was 1,500 tonnes, and the Department of Agriculture and Agri-food aims to increase this to 11,500 tonnes by 2020 [19]. During rice processing, RH (taking up about 20 % of the total rice weight) and rice straws (consisting of remnant leaves and dry stalks) are the main biomass wastes or by-products, thus RH is an important source of energy [20-21].

Recent studies on thermochemical process of biomass, in particular the pyrolysis of rice husks has concentrated on the impact of pyrolysis conditions such as carrier gas, catalysis, heating rate, and particle size [1,22-25]. The quest for finding a solution to the continuous depletion of petrochemical reserves has compelled researchers to seek for alternative renewable energy sources. Biomass has proved to be the best alternative solution to the highlighted challenge [26-29]. Juxtaposed with other renewable energy sources worldwide, biomass has a considerably high annual production.

Biochar, a compound chiefly made up of carbon, is a residue that is obtained after heating biomass via pyrolysis in an oxygen-free environment. Biochar has a plethora of both chemical and physical properties owing to the fact that it is obtainable from a variety of biomass residues with respect to pyrolytic conditions. It contains organic material with a different composition from scarcely pyrolyzed lignin to a carbonized material, from low temperatures to high temperature, respectively. The end product of the pyrolysis process consists of biochar, bio-oil and syngas [8,30-31]. Biochar has shown a high potential to counterbalance carbon emanations by long-haul carbon sequestration with extra horticultural advantage [32].

Figure 1. Rice Husk samples.

Thermochemical conversion of biomass is promising and provide competitive means for producing value-added chemicals, in addition to the production of energy from renewable energy sources [33-36]. Despite the high energy consumption and damaging properties of thermochemical conversion in relation to biochemical conversions, it still possesses some merits. Some of which include zero pollution, simplicity of operation, economised water consumption rate, etc [28,37-38].

The pyrolysis of rice husk has attracted the attention of numerous researchers. The yield of biochar decreases as the pyrolysis temperature is increased [24,39-41]. However, the rice husk biochar yielded the highest at 350 °C (50.67 %) and the lowest at 650 °C (29.02 %) [30].

Claoston et al., stated that cracks and shrinkages were observed on the surface of the rice husk biochar where it was highly porous due to high pyrolysis temperature at 650 °C [30]. It was observed that the structures were fragile due to their thin walls. The study also indicated that the structure in the rice husk biochar was more ordered as a result of the rise of pyrolysis temperature due to decreased in the number of micropores, while the number of macropores increases.

Moreover, Alvarez et. al. observed that volatile matter and fixed carbon content in the rice husk biochar decreases at the rise of pyrolysis temperature due to the high ash content in the rice husk biochar [24]. In addition, they concluded that the total carbon content was lower with pyrolysis temperature increase. On the other hand, the calorific value of the rice husk biochar decreases with rise in pyrolysis temperature. Rice husk biochar was having low heating value, despite having good characteristics to produce activated carbon and amorphous silica.

This research presents the thermochemical characterisation of varieties of rice husks in Brunei Darussalam. We studied the thermal degradation behaviour in pyrolysis and combustion atmosphere of four varieties of rice husks in Brunei Darussalam, namely Laila (ARH), Pusu (BRH), Bandul Beminyak (CRH) and Sendakan (DRH). The biomass characterizations were conducted through proximate and elemental analysis and heating value, and thermogravimetric analysis (TGA). Biochar is produced by subjecting the rice husk mixtures to different temperatures (400 °C, 500 °C and 600 °C) through fixed bed pyrolysis process. The biochar was characterised using Scanning

Electron microscope (SEM) and Energy-dispersive X-ray spectroscopy (EDX).

2. Methodology

2.1 Material and Methods

Four samples of rice husk (i.e. ARH, BRH, CRH and DRH) were collected from Brunei Darussalam. All samples were chosen based on availability of the rice from a milling site. The physical appearance of the collected rice husks is presented in table 1 and picture of rice husks samples is in figure 1.

Table 1. The physical appearance of the rice husks.

Rice husk	Length (mm)	Width (mm)	Appearance
A	8.0 – 9.0	3.0– 3.5	Solid ^{LY}
B	7.5 – 8.5	2.0 – 3.0	Solid ^{MY}
C	8.8 – 10.2	2.8 – 3.2	Solid ^{LY}
D	8.2 – 10.0	2.8 – 3.2	Solid ^{MY}

^{LY} Light Yellowish
^{MY} Medium Yellowish

The samples were prepared according to the ASTM standard E 1757 – 01 to get uniform particle size prior to analyses. The samples were grounded and passed through a 60-mesh sieve to obtain particle size less than 250 µm. Also, the moisture content (MC) of the samples was calculated according to the ASTM standards E 871. At a high temperature of 105 °C, the samples were dried and weighed for a period of 3 hours. The volatile matter (VM) and ash content (AC) of the samples were collected using Thermogravimetric analyser. Fixed carbon (FC) was calculated by equation (1) below [42]:

$$\text{FC (wt. \%)} = 100 - (\text{MC} + \text{VM} + \text{AC}) \qquad (1)$$

In addition, elemental analyses of the samples were carried out using CE Instruments Flash EA 1112 Series CHNS-O analyser (Thermo Quest, Italy). Calorific values of all samples were determined using C200 Bomb Calorimeter (P.A. Hilton Ltd, United Kingdom) following the procedures as given in ASTM standard D 5468 – 02. The higher heating value (HHV) was obtained from the bomb calorimeter, and the low heating value (LHV) was calculated from the following equation (2) [37]:

$$\text{LHV} = \text{HHV} - 0.212\text{H} \qquad (2)$$

Where, H (hydrogen) is a dry-basis weight percentage in a sample. HHV and LHV are expressed in MJ/kg.

2.2 Thermal decomposition

Thermal degradation experiment by thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG) was carried out with TGA7 Perkin Elmer Thermogravimetric Analyzer. In TGA, the weight of the sample was plotted as a function of time and temperature as it was subjected to a controlled temperature program. All the samples were heated from 50 °C to 900 °C at the rate of 25 °C/min under nitrogen atmosphere for pyrolysis conditions and oxygen atmosphere for combustion conditions.

2.3 Pyrolysis experiment setup

The RH pyrolysis is carried out in a fixed bed reactor. Nitrogen flow rate of 10 L/min as purge gas is used. For all runs, we put 40g of RH powder (Equally divided for the four rice husk samples) (Size: < 35 mesh) inside reactor. Then, the reactor was tightly coupled with exception for an inert gas connection pipe which also has an exit outlet. The outlet pipe is linked to a condenser and a bio-oil collecting flask. Nitrogen is supplied inside the reactor to ensure the removal of air before each run. In this experiment, we used an electric furnace to regulate the temperature in the reactor for three consecutive runs for pyrolysis temperature of 400 °C, 500 °C and 600 °C. When the temperature reaches desired pyrolysis temperature in the reactor, the reactor begins to cool down to room temperature. The bio-oil was collected from the condenser after the volatiles generated from the reactor were run through. When the reactor was cooled down to room temperature, the remaining biochar was taken out, weighed, and recorded. The syngas yield was calculated by difference from mass balance for liquid bio-oil and solid biochar.

3. Results and Discussion

3.1 Biomass characterisation

Summary of the biomass analysis in Table 2, which includes the proximate analysis, elemental analysis and heating values of the samples on a dry basis. Thermochemical conversion process is easier when the moisture level of the feedstock is reduced, whilst biochemical conversion is more feasible when there is high moisture content in the feedstock [38]. The moisture content of ARH, BRH, CRH and DRH were 7.39, 8.33, 6.90, and 7.12 %, respectively. These values are low; hence, it can be concluded that the moisture content of the samples in this work are suitable for thermochemical conversion. Volatile matter was 65.11, 64.22, 55.98, and 55.32 % for ARH, BRH, CRH and DRH, respectively. The fixed carbon values were found in the range of 10.95 to 15.20 % with the lowest value in CRH and the highest in ARH. Ash content was found in the range of 12.30 to 26.17 % with the lowest in ARH and the largest value in CRH. The higher heating values were found between 14.88 to 17.39 MJ/kg.

Yusup et al. reported that the high content of O and H lessens the energy value of the fuel since energy contained in carbon-oxygen (C–O) and carbon-hydrogen (C–H) bonds are lower than energy in carbon–carbon (C–C) bonds [43]. Biomass with low O : C and H : C atomic ratios are usually favoured for thermochemical conversion of fuels as they contain more energy [43]. The percentage composition of hydrogen, carbon, nitrogen and oxygen in the varieties were measured experimentally, while oxygen was calculated by difference. Rice husks contains small amount of nitrogen and sulphur which indicated less pollutant in the form of nitrogen oxide (NOx) and sulphur oxide (SOx) during combustion [44].

Table 2. Characteristics of rice husk via proximate analysis, elemental analysis and heating value.

Characteristics	Rice husk samples			
	ARH	BRH	CRH	DRH
Proximate analysis (wt. %)				
MC	7.39	8.33	6.90	7.12
VM	65.11	64.22	55.98	55.32
FC	15.20	14.18	10.95	13.03
AC	12.30	13.27	26.17	24.53
Elemental analysis (wt. %)				
H	5.29	5.43	4.49	4.49
C	39.34	38.52	32.39	32.93
N	0.51	1.27	0.41	0.48
S	0.05	0.10	0.03	0.03
O[a]	54.82	54.68	62.68	62.06
O/C	1.05	1.06	1.45	1.41
H/C	1.61	1.69	1.66	1.64
Heating Values (MJ/kg)				
HHV	17.39	16.54	14.88	15.24
LHV[b]	16.27	15.39	13.93	14.29

[a] by difference.
[b] By Eq. (2).

3.2 Thermal degradation of rice-husks under nitrogen gas atmosphere

Thermogravimetric analysis under nitrogen gas atmosphere was observed from the changes in the mass of the rice husks as a role of temperature, and plotted in Fig. 2.

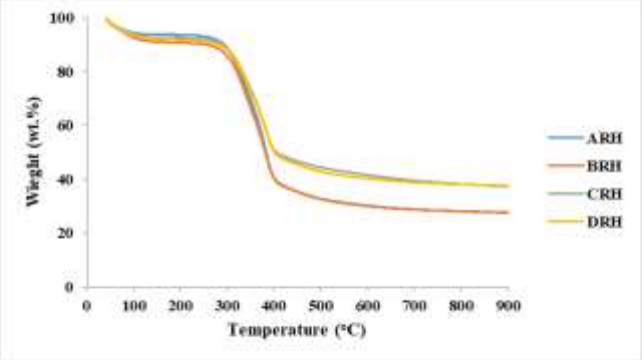


Figure 2 TGA of rice husks under a nitrogen atmosphere.

Fig. 2 illustrated that from 50 °C to 200 °C, the first mass loss of about 8.0 %, equivalent to the RH dehydration. A mass loss of around 55.0 % from 200 °C to 445 °C, which tallies with the concentrated decomposition of hemicellulose, cellulose, and lignin in the samples [45]. A gradual loss in mass of about ~15.0 % was recorded between 445 °C to 900 °C. Depending on the variety of RH, the final amount of residue corresponds to about 37 % of the first sample mass. Both CRH and DRH species, the mass loss degradation curves are comparable. In addition, ARH and BRH both have similar mass loss curves.

Williams and Besler observed that the thermal decomposition of hemicellulose initiates at about 250 °C, and the loss in mass occurs around 350 °C [46]. At about 325 °C, the cellulose in pure crystalline form started to disintegrate and the main area of mass loss arises from 350 °C to 430 °C. Between 200 °C and 900 °C, lignin shows a slow loss of mass, and illustrative about 55 wt. % of the final char residue (fig. 2). C. Quan, N. Gao and Q. Song [47] and Saldarriaga et al. [48] have mentioned that unlike hemicellulose, cellulose has higher thermal stability as well as a rigid structure that comprises of a branchless glucose chain.

Lignin decomposition is the hardest when compared with the other two components. Also the most thermally resistant component is lignin compared to hemicellulose and cellulose due to it has a more intricate chemical composition [49]. Thus, the essentially responsible factor for the volatile fraction of the product is the hemicellulose and cellulose components of the rice husks, whereas lignin contributes most to the char[46].

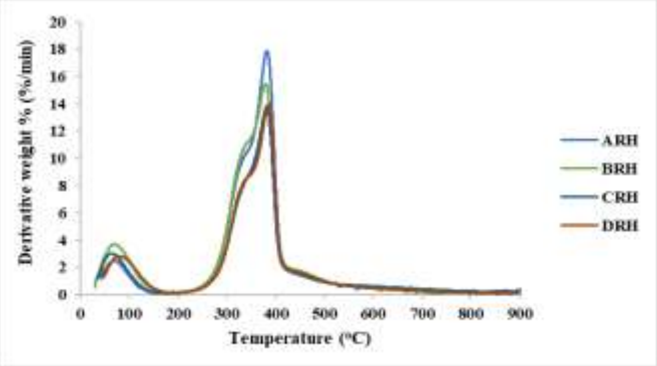


Figure 3 DTG of varieties rice husk under the nitrogen atmosphere.

Fig. 3 shows under N_2 atmosphere, the DTG curves as a function of the temperature for the samples. The largest intensity of the DTG was met around 380 °C at which instantaneous rate of thermal decomposition was maximum and the main reaction occurred [50]. The DTG curve for all the RH varieties decreases sharply from 380 °C to about 420 °C. The DTG peak for ARH, BRH, CRH and DRH were 382.0 °C, 379.0 °C, 381.0 °C and 384.7 °C; respectively. From 445 °C to 900 °C, the DTG value decreases slightly indicating low degradation rate. The intensity value of the DTG curve increases when using ARH and BRH, as shown in Fig. 3. In addition, the second and third stages of the inhibitive and accelerative interactions occurred from 250 °C to 400 °C, and 430 °C to 900 °C, respectively.

3.3 *Thermal degradation of the rice husks varieties under oxidation atmosphere*

Fig. 4 showed the role of temperature of the TGA under oxidative atmosphere. The progression of mass as a function of temperature under oxygen atmosphere is quite the same with the exception of BRH. BRH has a lower dehydration temperature and can decompose from 50 °C to 220 °C (contingent on the variety), the first mass loss of about 7 % corresponded to RH dehydration. Between 220 °C and 370 °C, a mass loss of ARH, BRH, CRH and DRH were 68.0, 63.2, 45.6, and 52.7 %, respectively. The mass loss range of 11.90 to 20.62 wt. % can be observed between 370 °C – 630 °C, with a minimum for ARH and maximum for CRH. From 630 °C to 900 °C, the slope of the curves becomes less significant than the earlier step which corresponds to the oxidation of the char and the mass loss which is negligible.

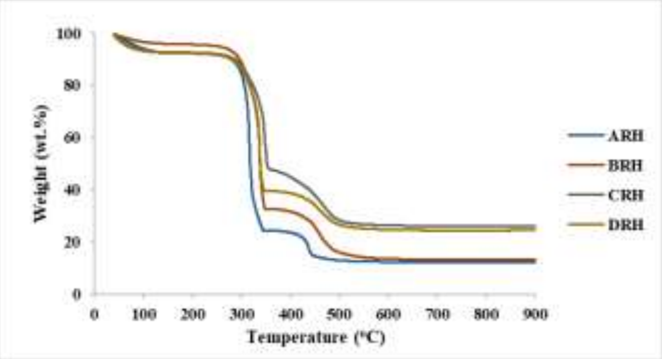


Figure 4 TGA of varieties rice husk under oxygen atmosphere.

The final residue corresponds around 25 % of sample mass for both CRH and DRH, and 12 % for both ARH and BRH. However, the evolutions of the DTG curves is required to get more understanding of the thermal decomposition of the varieties in all the steps under an oxidative atmosphere.

Fig. 5 shows the development of the DTG curves with respect to temperature in the presence of oxygen for the samples. Three global reaction steps represent the DTG of the samples. Drying and heating of the rice husks are included in the first step (around 50 °C – 250 °C). Then, the dehydrated rice husks are included in the second step to release volatile organic matter and create char (around 250 °C – 350 °C). Finally, oxidation of the char by oxygen occurs in the third step (around 350 °C – 900 °C).

The varieties of RH generated different DTG intensities. Also, DTG peaks illustrated are very comparative for all the varieties of RH through increase temperature. In addition, the ARH and BRH have shown a higher concentration of the main peaks in DTG.

The DTG peak for ARH, BRH, CRH and DRH were found at 317 °C, 326 °C, 338 °C and 330 °C, respectively due to hemicelluloses and cellulose decomposition. In addition, there were peaks observed on the DTG curve which indicates that the decomposition of hemicelluloses and cellulose is relatively slow around 450 °C while lignin decomposition occurred [26]. Similarly, the second and third stages of the inhibitive and accelerative interactions occurred with temperatures varying between 250 °C to 350 °C, and 350 °C to 900 °C, respectively.

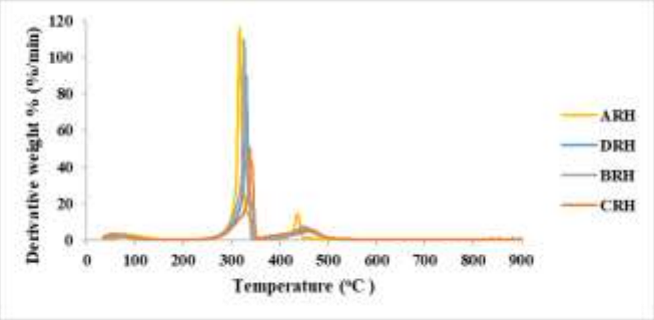


Figure 5 DTG of varieties rice husk under oxygen atmosphere.

3.4 *Comparison of the degradation atmospheres*

To show the effect of the oxygen and nitrogen atmospheres on the thermal degradation process of the rice husks, Fig. 3 and 5 illustrates the evolutions of the DTG for both the atmospheres. For the temperatures less than 200 °C, the mass curve forms are alike. The figures (i.e. 3 & 4) also depict the difference between the curves of mass loss under the two atmospheres, meaning that, between 200 °C and 420 °C, under oxygen atmosphere the curves of mass loss is higher than the curve of mass loss under nitrogen atmosphere. From 420 °C to 500 °C, among the curves are a more significant mass loss occurring under oxygen atmosphere than under nitrogen. Therefore, another DTG peak is present under oxygen atmosphere but not under a nitrogen atmosphere. This is because, the char oxidation is better under oxygen atmosphere [51]. Hence, the mass of the final residue was higher under nitrogen than oxygen

The rice husks through thermal decomposition seemed compatible for the two different atmospheres until 200 °C. The first stage of dehydration was observed under both nitrogen and oxygen when the temperature was between 50 °C to 200 °C. In addition, under nitrogen and oxygen atmospheres, two main stages of decomposition occur when the temperature was between 200 °C to 420 °C. Above 420 °C, it was the third stage for other reactions occurring in presence of oxygen atmosphere.

3.5 *Effect of pyrolysis temperatures on biochar yield and SEM-EDX analysis*

In each pyrolysis condition, dry weight of biochar was found compared to the dry weight of prototype rice husks. In the process, the biochar yield substantially decreased when temperature was increased. The biochar yield at a pyrolysis temperature of 400 °C was higher than 500 °C and 600 °C (see Fig. 6). At 400 °C, 500 °C and 600 °C, biochar yields were 49.5, 44.6 and 38.6 wt. %, respectively. A decrease in the yield was observed when the pyrolysis temperature increases.

Table 2 Total weight loss and largest weight loss rate of four rice husk samples under nitrogen and oxygen atmosphere.

Biomass Samples	Total weight loss (%)	Highest weight loss rate between start and end of active reaction (%/min)	Start of second stage of decomposition (°C)	End of second stage of decomposition (°C)
Nitrogen atmosphere				
ARH	72.40	55.74	260	425
BRH	72.47	53.13	243	425
CRH	62.76	44.68	255	440
DRH	62.38	45.56	249	445
Oxygen atmosphere				
ARH	87.70	67.45	250	348
BRH	86.70	61.84	260	350
CRH	73.80	44.50	250	360
DRH	75.47	52.09	250	350

This experiment is in line with literature in which the biochar yield decreased, while pyrolysis temperature increases from 400 °C to 600 °C [52]. At higher temperature, the decline of biochar yields occurs by both the disintegration of the biochar deposit and the high degradation of organic content of biomaterials to ash [53].

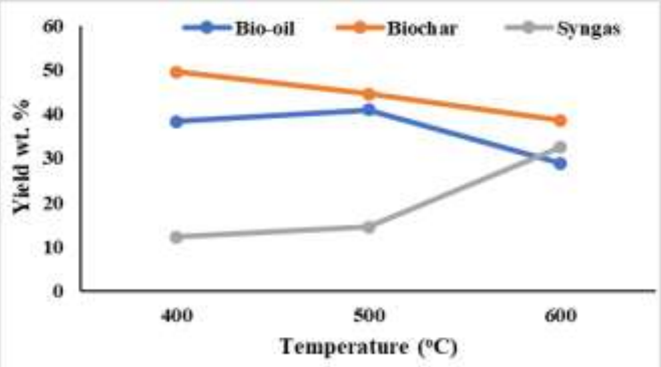


Figure 6 Yield of pyrolysis products at 400 °C, 500 °C and 600 °C.

Morphological images and EDX spectra of the RH biochar were taken for all four types together. Fig. 7 (a-c) showing the difference of SEM at 400 °C, 500 °C and 600 °C. The morphology showed that these types of RH in all cases are highly dense and well-connected microstructure even at a low temperature of 400 °C. SEM images of the RH biochar composites exposed that thin film structures extensively covered the sample surface as well as in RH biochar composites, which is the same as the EDX results [54].

According to Claoston et al. [30], at 500 °C the morphology of the rice husk biochar was honeycomb-like with cylinder-shaped holes that were well organised. The micrographs of rice husk biochar show multi-pores that was found on the surface that were properly arranged. In addition, at 650 °C, cracks and shrinkages could be seen on the surface. Similar results were seen in this research, which were observed at 400 °C, 500 °C, and 600 °C. In the case of Kizito et al., the rice husk biochar has a similar shape of amorphous and a less porous structure [55].

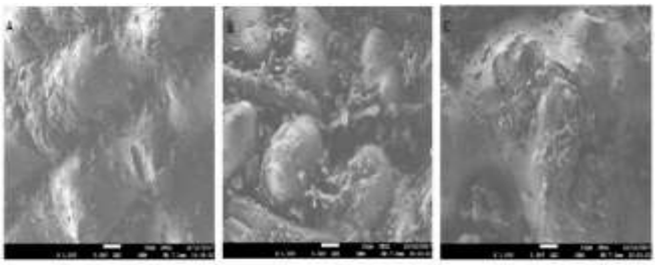


Figure 7 SEM of biochar at (A) 400 °C, (B) 500 °C and (C) 600 °C.

On the other hand, EDX investigations (see Fig. 8) showed high intensity peaks for silicon, oxygen and carbon for all mixed samples which are typical elemental composition of biochar. This finding is in accordance with Heo et al. [56], the rice husk biochar produce by fluidized bed reactor has carbon content 82 % at 450 °C higher than at 400 °C and 500 °C in this study, but oxygen content 14.1 % as lower. In their study, EDX was not used for collecting the carbon and oxygen content whereas in this study, EDX analyser was used. Table 4 shows the wt. % of the investigated elements (carbon, silicon, oxygen and some other elements).

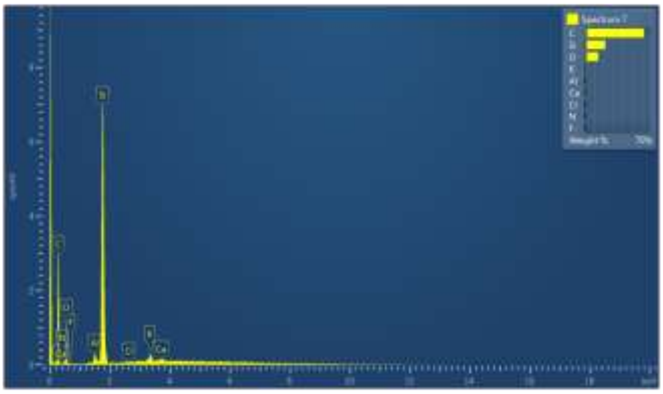


Figure 8 Graph of EDX for biochar showing a high peak of carbon and silica contents at 400 °C.

Table 4 EDX for biochar (A) at 400 °C, (B) at 500 °C and (C) at 600 °C.

Samples	Elements, wt. %						
	C	Si	O	Al	K	Ca	Cl
A	57.99	16.18	19.44	2.29	1.06	0.25	0.02
B	36.88	24.43	37.14	0.36	1.07	0.06	0.02
C	25.84	26.83	46.72	0.25	0.19	0.02	0.04

Conclusion

The thermochemical characterisations of different rice husks were investigated for their solid fuels characteristics. TGA and DTG studies were conducted on the rice husks Laila (ARH), Pusu (BRH), Bandul Beminyak (CRH) and Sendakan (DRH). Characterisations such as calorific value, proximate and elemental analysis measurements were performed. We demonstrate through experimental analysis that rice husks can be used as feedstock for pyrolysis process to produce bio-based products. The fixed bed pyrolysis of rice husks has been conducted focusing on the effect of pyrolysis temperature on biochar, bio-oil, and syngas yields. The yield of biochar was significantly reduced when the pyrolysis temperature increases. Pyrolysis and combustion of rice husks for biochar production is also suitable for agriculture and the reduction of GHG. The ASEAN region in general are leading producers of rice in the world including Brunei Darussalam, which plans to increase investment in rice production. In terms of biofuels and value-added material products from Brunei rice husks. The obtained results showed the potential in energy requirements of the country with neutral CO_2 emission to the environment.

Acknowledgements

The main author (Mujahid Alhinai) is thankful to the Ministry of Foreign Affairs and Trade (MOFAT) of Brunei Darussalam for postgraduate research scholarship for funding this research.

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