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ANALYSIS OF PHYSICAL AND CHEMICAL PROPERTIES FOR BIOCHAR FROM RICE HUSK BIOMASS

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Abstract

Biomass play significant roles in the production of eco-friendly biochar and as substitutes for renewable sources of energy. In this study, investigations were carried out on physical and chemical analysis of rice husk biochar (RHBs) from rice husk. The rice husk biomass was heated at 300˚C for 1h and sieved to the average particle size. The physical and chemical properties of RHBs were determined by direct measurements and calculations. Rice husk biochar (RHBs) were characterized for biochar yield, pH and Bulk Density. The biochar chemical analysis gave the following results; biochar yield of 73% for RHB-HT and 68% for RHB-PT, the pH values of 7.14% and 7.49%, Bulk density of 0.42% and 0.43% for RHBs. RHBs were characterized by analyzing the chemical composition mainly based on X-ray diffraction (XRD), Energy Dispersive X-ray Fluorescence (EDXRF), Fourier transform infrared spectroscopy (FTIR) and surface morphology from Field emission SEM (FE-SEM) and energy dispersive X-ray micro-analysis (EDXA). The XRD showed the carbon amorphous structure of rice husk biochar (RHBs), which consists of surface morphology from FE-SEM. The carbon (C), oxygen (O) and silica (Si) were observed by Energy Dispersive X-ray spectroscopy (EDX). Fourier transform infrared spectroscopy (FTIR) analysis showed the presence of a variety of functional groups for RHBs. The results of the physical and chemical properties of the rice husk biochar provide the prominent source of useful energy.

Keywords : Biomass, EDXRF, FE-SEM, FTIR, Rice Husk Biochar, XRD.

Introduction

Now a day, biomass is important one of a renewable energy resources. Biomass is a nonpolar and biodegradable organic material directly obtained from plants, animals or microorganisms [Demirbas, A.et.al (2009), Adilah Shariff et.al (2014)]. Biomass resources generally contained the various natural and artificial things such as woody and herbaceous species, wood wastes, sawdust, rice husk, bio solids, grass, waste from food processing, animal wastes, aquatic plants and algae [Adilah Shariff et.al (2014), Yaman, S. et.al (2004)]. Biomass can be transfer into liquid, solid and gaseous fuel via transformation of some physical, chemical and biological phenomenon. Rice husk is one of the biomass raw materials for thermochemical conversion. Although it can be widely used in pyrolysis and gratification, it is restricted for the application areas of biomass technology to measure some properties such as its high oxygen content, high moisture, low calorific value, large particle size, and grinding. Its advantages are observed to be the further development of biomass application technology [Van der Stele et.al (2014), Chen, D. Y. et.al (2012 a), Yin, R. Z. et.al (2012)].

Biochar is sustainably produced from biomass sources and it can be used in agricultural as non-oxidative application. Moreover, it can be also applied in the area of carbon sequestration [Lehman et.al (2015)]. Actually, biochar is almost the same with charcoal, the carbon chance into CO_2 by firing the biochar and may apply as fuel. In the past decade, potential attention increase

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the research concerned with biochar due to its novel application. The remarkable advantage outcome of the biochar is its used in carbon sequestration [S.P. Galinato et.al (2011), C.J. Barrow et.al (2012)]. Biochar can be employed in use for various for various applications and feedstock for many process according to its physicochemical characteristics [D.Ozcimen et.al (2010), X. Wang et.al (2009)]. In this research, rice husk collected from Ayewaddy Region in Myanmar conduct to carry out experiments as function of heating temperature and time. In the presence investigation, the main objective was to produce biochar from agricultural wastes namely rice husk biomass. The physicochemical characteristics of the resulting biochar was examined to evaluate biochar yield, pH and Bulk density. Crystal structucture and phase identification of biochar was characterized by X-ray diffraction (XRD). The rice husk biochar samples were also analyzed by Field Emission-Scanning Electron Microscope (FE-SEM) equipped with an energy dispersion X-ray Spectroscopy (EDX) and FRIR for identification of surface morphology and functional groups.

Materials And Methods

Biochar Preparation from Rice Husk

The rice husk used for the present work were obtained from Ayewaddy Region in Myanmar. Two different types of biochars derived from rice husk biomass were investigated. Biochar, rice husk from Hinthada, noted hereafter as RHB-HT and biochar, rice husk from Pathein, noted hereafter as RHB-PT.

Table 1 Sample label

Sir No.	Sample
1.	RHB-HT
2.	RHB-PT

20-g of rice husk were first washed with deionized (DI) water 1 L for 2 h at 90˚C. After successive washing, the wet rice husk was dried at room temperature for 48 h. Then rice husk was heated at 300˚C for 1 h. After heating, samples were sieved to the average particle size.

Biochar Chemical Analysis

Biochar Yield

The biochar yield was calculated as the propotion of the weight of biochar product to the original material. The percentage of biochar yield was calculated using the following equation. [Sadaka S et.al (2014), G.Stella Mary P.Sugumaran et.al (2016)]

$$\text{Yield}_{\text{biochar}} = \frac{\text{Mass of biochar (g)}}{\text{initial Mass of biomass (g)}} \times 100\%$$

Where, Yield $_{\text{biochar}}$ = mass yield of biochar %

pH Measurement

For pH determination, 1 g of bio-char was dispersed in 100 mL of deionized water with stirring. The mixture is heated at 90˚C with stirring for 20 min and then cooled at room temperature. The insoluble solute is separated from the solution by filtration with filter paper. The filtered solute is taken. The pH value was measured with pH meter.

Bulk Density Measurement

Bulk density was determined for rice husk biochar. The biochars were crushed and sieved through a mesh with 250 µm openings. An empty 25 mL graduated measuring cylinder was dried in a drying oven and weighed out. It was then filled up to a volume of 10 mL with the biochars. Then the biochar dried in oven at 80 C for 5h. With the filling of every 2 mL, the cylinder was tapped for 1-2 min onto a padded surface to compact the char and the bulk density was calculated by following formula:

$$\text{Bulk density (\%)} = \frac{\text{weight of dry material (g)}}{\text{Volume of packed dry materials (ml)}} \text{x100}$$

Results and Discussion

Biochar Chemical Analysis

The biochar yield of RHBs at 300˚C are summarized in Table. 2. Several studies indicate that the yield of biochar is highly dependent on the pyrolysis conditions such as temperature, heating rate and heating time [Tsai WT, Lee MK et.al (2007)] and is also greatly influenced by chemical, physical and biological properties of the biomass [Knoepp JD et.al (2005), Lehmann J et.al (2007), Basta AH et.al (2011)]. Biochar yield was found that low temperature produced a higher biocher yield and enhance volatile matter composition than the high temperature. The pH of biochars may change and either decrease or increase depending on type of feedstock. The pH of biochar may also change post-production depending on the environmental conditions. The pH of 7.14 for RHB-HT and 7.49 for RHB-PT (Table 2). In this study, the pH of RHBs were recorded alkaline pH. Bulk density of biochar, differing according to the raw materials used, is that of the material comprising multiple particles and includes the macro porosity each particle and the antiparticle voids. In this study, Bulk density of 0.42 for RHB-HT and 0.43 for RHB-PT.

Table 2 Physical and chemical characteristics of the rice husk biochar at 300˚C

Physical and chemical characteristics of RHBs from rice husk					
Sir No.	**Samples**	**Temperature (˚C)**	**Biochar Yield (%)**	**pH (%)**	**Bulk Density (g/mL) (%)**
1.	**RHB-HT**	**300˚C**	73	7.14	0.42
2.	**RHB-PT**	**300˚C**	68	7.49	0.43

X-ray Diffraction (XRD) Analysis

X-ray Diffraction (XRD) Analysis XRD is the most useful method to see the particle size and crystalline and amorphous materials. Figure 1. shows the X-ray diffraction (XRD) pattern of the rice husk biochars RHB-HT and RHB-PT at 300˚C respectively. Appearance of broad diffraction peak and the absence of a sharp peak reveals a predominantly amorphous structure. All the samples showed the same diffraction patterns, where the broad peak was observed between 15˚ and 30˚, the ranges are corresponding to the presence of amorphous carbon. Also, the XRD patterns revealed the absence of any ordered crystalline structure. It is desire to have amorphous carbon as the carbon fuel, however, we are unable to tell the percentage of amorphous carbon in the biochars RHB-HT and RHB-PT.

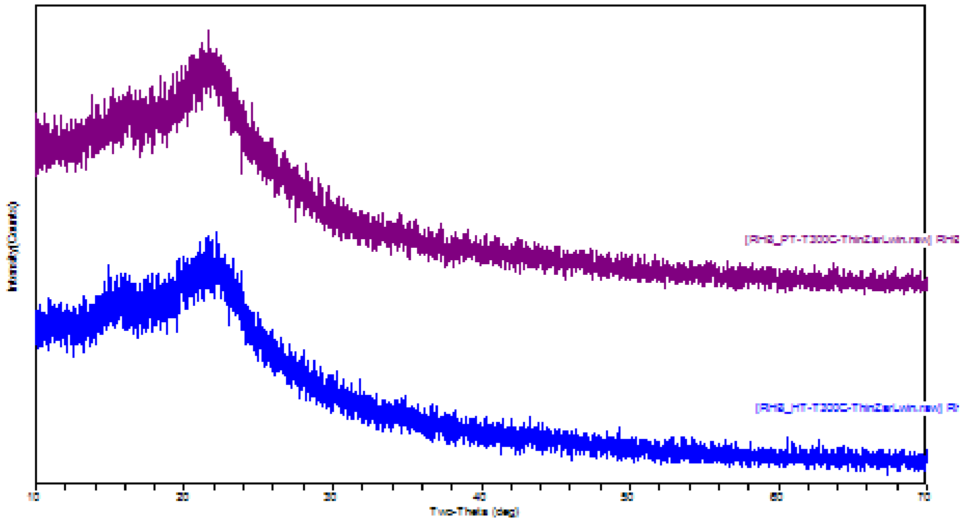

Figure 1 X-ray diffraction patterns of Rice Husk Biochar RHB-HT and RHB-PT at 300˚ C

Field Emission Scanning Electron Microscopy (FE-SEM) Analysis

Figures 2 (a-b). show the FE-SEM micrographs (100x) of the surface morphology of the RHB-HT and RHB-PT at 300˚C. The carbon is mainly localized in the tips of the domes, whereas a lower amount of carbon can be found in other regions of the RHBs (RHB-HT and RHB-PT). In addition, [Bidayatul Armynah et.al (2018)] have demonstrated from FE-SEM experiments, that the characteristic of surface morphology is dot may contain high carbon.

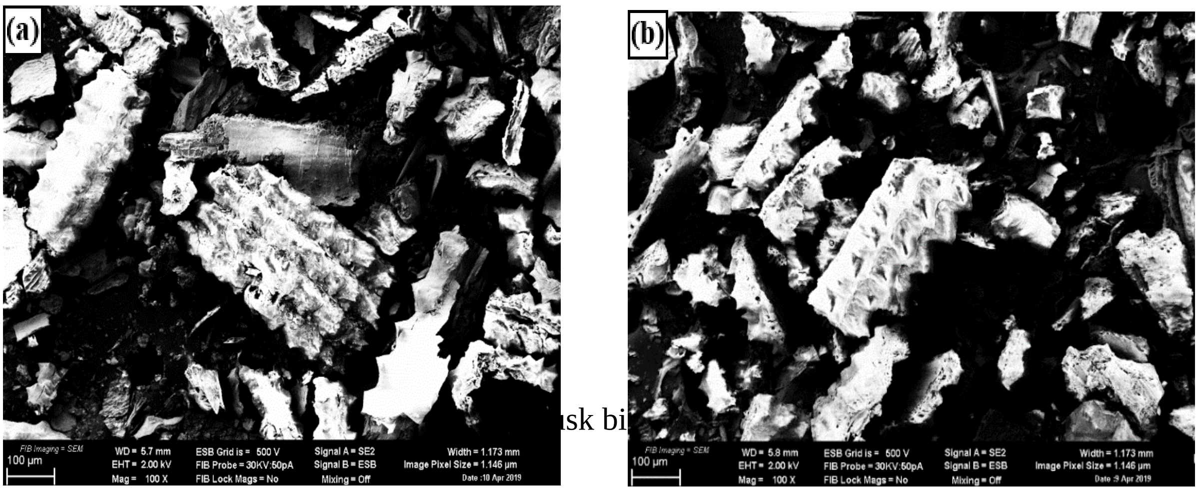

Figure 2 (a-b) FE-SEM micrograph of rice husk biochar RHB-HT and RHB-PT at 300˚C

Energy Dispersive X-ray (EDX) Analysis

The compositional analysis of the RHBs (RHB-HTT and RHB-PT) at 300˚C was analyzed by Energy Dispersive X-ray spectroscopy. The EDX results for RHBs samples obtained the elements of Carbon (C), Oxygen (O), Silicon (Si). Fig. 3(a-b) shows C, O and Si content in the RHBs at 300˚C and the corresponding weight percent of the elements present in the RHBs and result obtained further assures that no other impurity is present in the RHBs. Fig. 3(a) shows the EDX spectrum of RHB-HT at 300˚C. The strong peaks observed in the spectrum related to the Carbon and Oxygen. The elemental constitution of RHB-HT at 300˚C with one major peak and one minor peak were found to have weight percentage of 69.23 for Carbon and 30.77 for oxygen. The RHB-HT have atomic percentage of 70.22 for Carbon and 29.69 for Oxygen. Fig. 3(b) shows the EDX spectrum of RHB-PT at 300˚C. The elemental constitution of RHB-PT at 300˚C with two major peaks and one minor peak were found to have weight percentage of 65.42 for Carbon, 34.55 for oxygen and 0.03 for Silica. The RHB-PT have atomic percentage of 58.69 for Carbon, 41.29 for Oxygen and 0.01 for Silica (Fig. 3-b). From these result, the RHB-HT at 300˚C sample have 79.23% (wt %) and 70.22% (At %) of carbon elements which is the higher percentage than RHB-PT at 300˚C. It contains low fractions and absent of various elements such as S, Cl, K, Zn and Mg. This is important to study the influence of these impurities on the fuel source, high Carbon (C) content and low Sulphur content were required. Elemental and atomic content for RHBs-HT and RHB-PT at 300˚C expressed in percentage by EDX analysis are summarized in Table. 3.

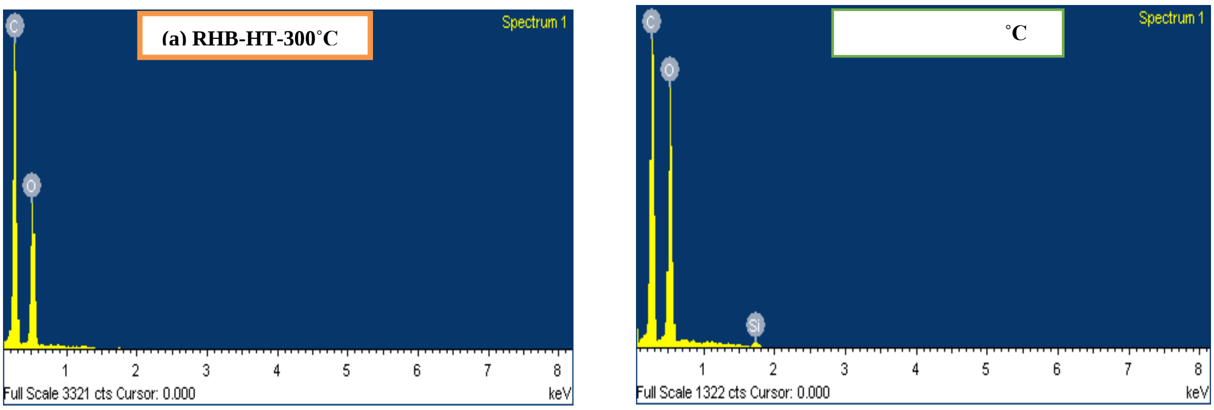

Figure 3 (a-b) EDX spectrum of RHB-HT and RHB-PT at 300˚C

Table 3 Element content for RHB-HT and RHB-PT at 300˚C

Component	RHB-HT 300˚C		RHB-PT 300˚C	
	Weight%	Atom%	Weight%	Atom%
Carbon	69.23	70.22	65.42	58.69
Oxygen	30.74	29.78	34.55	41.29
Silicon	0.03	0.01	0.03	0.01

Energy Dispersive X-ray Fluorescence (EDXRF) Analysis

Table 4 indicates that elemental concentration in RHB-HT and RHB-PT at 300˚C. Eleven elements namely C, K, Si, Ca, S, Fe, Cr, Cu, Mn, Ti and Ni were detected in the rice husk biochar sample by EDXRF technique. Carbon (C) was found to be present as the major element, whereas K, Si, Ca, S, Fe, Cr, Cu, Mn, Ti and Ni were present as minor element. Therefore, RHB-

HT and RHB-PT at 300˚C were quantified by EDXRF. From the result, RHB-HT contains 96.912 % of Carbon (C) which is higher than RHB-PT at 300˚C. This results are good agreed with FE-SEM result.

Table 4 Chemical composition for RHB-HT and RHB-PT were measured by using EDXRF

Elemental Analysis of RHBs												
Sir. No	Sample	Content Weight%										
		C	K	Si	Ca	S	Fe	Cr	Cu	Mn	Ti	Ni
1.	**RHB-HT**	96.912	2.019	1.025	0.004	0.001	0.002	0.020	0.035	0.004	0.051	0.000
2.	**RHB-PT**	95.902	3.004	0.035	0.000	0.003	0.001	0.010	0.018	0.002	0.025	0.010

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR results of RHB-HTT and RHB-PT for 300˚C were shown in Fig. 4 (a-b). As it was explained in the introduction, the organic part of the rice husk mainly composed of cellulose, hemicellulose, lignin, and waxes, which most likely consist of alkene, esters, aromatics, ketones, and alcohols. At 300˚C, FTIR spectra contain four peaks at 3344.65 cm^{-1}, 2345.79 cm^{-1}, 1617 cm^{-1}, 1050.76 cm^{-1} and 788.36 cm^{-1} for RHB-HT and 3330.35 cm^{-1}, 2361.12 cm^{-1}, 1602.51 cm^{-1}, 1051.14 cm^{-1}, 793.02 cm^{-1} for RHB-PT.

i) O-H stretching

The peaks obtained for RHBs at 300˚C were 3344.65 cm^{-1} for RHB-HT and33330.35cm^{-1} for RHB-PT respectively. The peaks indicate O-H stretching (3650 to 3200 cm^{-1}) carboxylic group. [Demirbas, A. (2000), De Rosa, I. M. et.al (2010), Socrates, G. et.al (1994).]

ii) C-O stretching

The band visible at 2360 cm^{-1} – 2365 cm^{-1} corresponds to the stretching vibration of the C-O for carbon monoxide or carbon dioxide derivatives in RHB-HT and RHB-PT [H.P.S. Abdul Khalil et.al (2013)].

iii) C=C stretching

The peaks recoded for RHBs (300˚C) were at 1599.55 cm^{-1} for RHB-HT and 1602.51cm^{-1} for RHB-PT respectively. These were indicating C=C stretching vibration consists of aromatic groups in lignin [G. Stella Mary (2016)].

iv) C-O stretching

The presence of peaks 1050.76 cm^{-1} for RHB-HT and 1051.14cm^{-1} for RHB-PT at 300˚C were indicative of C-O secondary alcohol stretching for cellulose, hemicellulose, and lignin [Bidayatul Armynah et.al (2018)].

v) C-H bending

The RHBs at 300˚C showed four strong peaks; the peaks recoded at 788.36 cm^{-1} for RHB-HT and 793.02 cm^{-1} for RHB-PT respectively, which reveals alkynes with C-H bending is present [Bidayatul Armynah et.al (2018)].

The presence of functional groups such as the carboxylic group, alcohol aromatics and alkynes groups suggest that these RHBs could be affected in fuel source. The functional groups identified in FTIR analysis for RHBs are tabulated in Table 5.

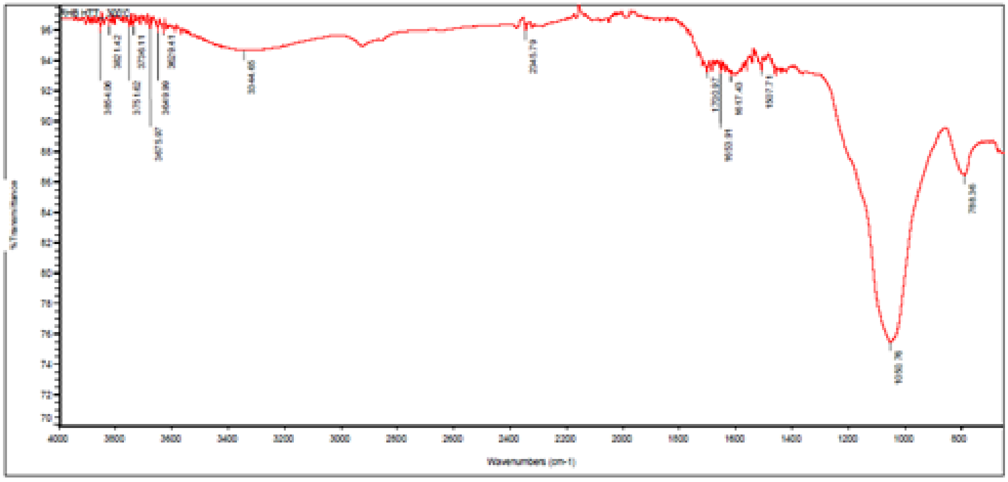

Figure 4(a) FTIR spectrum of RHB-HT at 300˚C

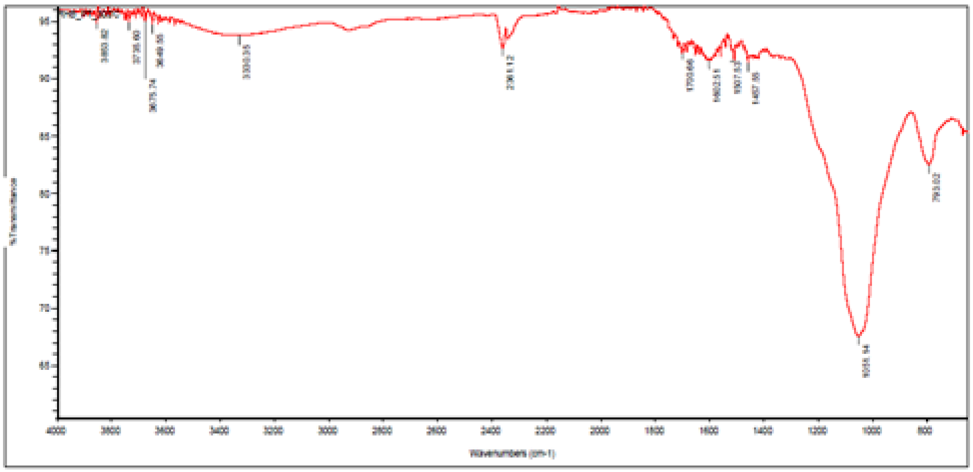

Figure 4(b) FTIR spectrum of RHB-PT at 300˚C

Table 5 Functional groups identified in FTIR analysis of RHBs at 300˚C

Sr No.	Chimical bond	RHB-HT	RHB-PT
		300˚C Peak position (cm^{-1})	300˚C Peak position (cm^{-1})
1.	O-H	3344.65	3330.35
2.	C-O	2345.79	2361.12
3.	C=C	1617.00	1602.51
4.	C-O	1050.76	1051.14
5.	C-H	788.36	793.02

Conclusion

RHBs were produced from rice husk biomass heating at low temperature. The properties of biochars have different chemical structures and carbon content depend on agricultural conditions and types of raw material. The XRD shows the same diffaration pattern and amorphous carbon structure. Surface morphology from FE-SEM indicated high carbon content. The RHBs were quantified by EDXRF spectrometer. Carbon (C) was found that the highest concentration in the RHBs (RHB-HT and RHB-PT). But RHB-HT is higher carbon content than RHB-PT. FTIR shows aromatic compound group corresponds to carbon atoms C=C at (1602-1617 cm^{-1}). These results of physical and chemical composition characteristic, structural properties, surface morphology and bonding formation of RHB-HT show that the prominent source of useful energy can replace as the fuel.

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