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Analysis of Chemical and Physical Properties of Biochar from Rice Husk Biomass

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Abstract: Chemical and physical properties of Rice Husk as a potential energy resource were analyzed by means Fourier transform infrared (FTIR), x-ray diffraction (XRD), scanning electron microscope (SEM), and energy disperse spectroscopy (EDS). Rice husk is heated with varied temperature of 250°C, 350°C, 450°C and 30, 60, 90 minutes respectively combine with time variation. The results show that the calorific value decreases whenever the temperature and time increase. The heating time of 30 minutes at 250°C of temperature gives calorific value of 10.4 MJ/Kg. While at the 450°C of temperature, the calorific value decrease to 4.7 MJ/Kg. The EDS shows that the time of heating is an important parameter where carbon and nitrogen were decreasing with the increment of the heating time while the oxygen increase when the heating time increase. The XRD shows that the broad (002) reflections between 20^o and 30^o indicate carbon disordered with small domains of coherent and parallel stacking of the graphene sheets, which consists of surface morphology from SEM. FTIR shows that the O-H stretching pronounced at around 3452 cm^{-1} and 3412 cm^{-1} and pronounced clearly at the highest temperature. The aromatic group from lignin gives rise to C=C asymmetric stretching at cm^{-1} as a G band corresponds to the sp2-hybradization bonding of carbon atoms and C-H bending modes at 2927 at 796 cm^{-1}. This results of the characteristic of chemical and physical properties of the rice husk examination provide the prominent source of useful energy that can eventually replace the fossil fuel.

1. Introduction

Biomass is the third largest primary energy resource in the world, after coal and oil. Biomass is a major source of energy in developing countries, where it provides 35% of all the energy requirements. The use of biomass is importance as concerns global warming since biomass combustion has the potential to be CO2 neutral [1]. Biomass materials agricultural from residues such as straw, bagasse, and groundnut shell, coffee husks and rice husks as well as residues from forest-related activities such as wood chips, sawdust and bark having high energy potential. Residues from forest-related activities account for 65% of the biomass energy potential whereas 33% comes from residues of agricultural crops [2].

Among the proven combustion technologies (grate-fired, suspension-fired and fluidized bed systems), the fluidized bed technology is reported to be the most efficient and suitable for converting agricultural and wood residues into energy [1,3]. Extensive experimental investigation has been carried out to date on the feasibility and performance of the fluidized bed combustion of different alternative fuels. CO and NOx (generally, as NO) are also the major harmful pollutants emitted from biomass combustion in fluidized bed systems [4]. For a selected fuel, CO emission (strongly affecting the combustion efficiency) is a function of operating variables, such as excess of combustion air as well as combustor load, and can be effectively controlled by the air supply [5].

Today, various methods have been tried to obtain energy from biomass. Typically, all of those methods can be grouped into two categories, thermochemical process (e.g. combustion, gasification and pyrolysis) and biochemical process (e.g. fermentation and anaerobic digestion), which have been carefully summarized in previous report [6] .This research of Rice Husk from South Sulawesi Indonesia conduct to carry out experiments as function of heating temperature and time.

This research analyze carbon, oxygen, and Hydrogen content from the EDS the results while the Chemical bonding and structural properties of biochar analyzed by using Fourier transform infra-red (FTIR) spectroscopy and X-ray diffraction spectroscopy (XRD), respectively.

2. Research Methods

This research uses the carbonization method to examine the rice husk. The carbonization processes examine the burned biomass in the absence of oxygen. But due to the insufficient equipment conditions, this research is not capable to remove the oxygen in the feeding chamber. This research observes the temperature and time parameter which conducts with the variations temperature of 250°C, 350°C, and 450°C with the time variations of 30 minutes, 60 minutes, and 90 minutes for each temperature.

Infrared spectroscopy was carried out on IR Prestige-21 FT-IR spectrometer (Shimadzu Corp) equipped with a bright ceramic light source, a KBr beamsplitter, and a deuterated triglycine sulfate doped with L-alanine (DLATGS) detector. The measurements of the sample were collected over the range of 4000-600 cm^{-1} and 16 co-added scans. All samples were ground into powders prior to the spectral acquisition. All spectra were in Transmittance units.

The X-ray diffraction (XRD) pattern was collected on an X-ray diffraction (XRD) spectroscopy (Shimadzu 7000) with Cu Kα radiation (λ = 1.5405 Å) over the angular range $15° \leq 2\theta \leq 80°$, operating at 30 kV and 10 mA. It was performed to examine the structural properties of the samples.

Morphological and chemical characterization (composition of carbon, oxygen, nitrogen and another element in the samples) of particles has been performed by a Scanning electron microscope-energy-dispersive X-ray spectrometry (SEM-EDS) (JEOL JSM-IT- 300) with acceleration voltage 10 kV, beam current 7.475 nA, and the lowest vacuum is 50 pa.

3. Results and Discussion

Formation Charcoal Rice Husk with temperature and time varied

Table 1. Chemical composition for Rice Husk as a function of temperature and time of heating were measured by using energy disperses spectroscopy (EDS) from Experiment

Element	250°C /Time (Min.)			350°C /Time (Min.)			450°C /Time (Min.)		
(%)	30	60	90	30	60	90	30	60	90
C	18.62	16.91	22.33	14.27	14.77	7.72	18.29	5.68	6.15
N									
O	43.11	43.95	41.22	45.44	45.05	48.79	42.02	49.82	49.04
Na	0.01		0.04		0.01	0.05			
Mg	0.05	0.05	0.04	0.07	0.1	0.14	0.12	0.15	0.23
Al	0.39	2	0.41	0.17	0.96	0.25	5.67	0.87	0.47
Si	37.43	36.95	35.8	39.61	38.65	42.4	32.24	42.84	42.14
K	0.33	0.07	0.15	0.31	0.43	0.54	0.79	0.64	1.65
Ca	0.06	0.06	0.01	0.13	0.02	0.11			0.33

Table 1 shows EDS from experiment biomass from Rice Husk with the heating temperature from 250^0C, 350^0C, 450^0C and time varied for 30, 60, and 90 minutes respectively. The ash composition shows that the temperatures of 350^{O}C and 450^{O}C increase when the heating time increment as the release of CO_2 and CH_4 gasses from hemicelluloses, cellulose, and lignin decomposition [6]. The temperature of 450ºC for the heated time of 90 minutes shows a discoloration in the samples from black to gray which indicates the high content of ash. Some study which stated that fixed carbon increase with the increment of heating temperature, is not found in this research since the mass density of Rice Husk is lower than another source of biomass[6,7,8,]. As showed in Table 1, the element composition also show N, Na, Mg, Si, AL, K, and Ca with dominant content are C, O, and S for bio-chart from Rice Husk.

Based on the X-ray fluorescence spectroscopy (XRF), this research shows that Si, Ca, K, and P are the most abundant ash element in Rice Husk biomass with composition of 87%, 2%, 9%, and 0.9 %, respectively.

Table 2. Chemical composition for Rice Husk as a function of temperature and time of heating were measured by using ultimate analysis

NO	ELEMENT (%)				REF.
	C	N	O	H	
1	37.65	1.63	55.5	5.13	[9]
2	34.6	0.47	31.7	4.23	[10]
3	38.30	0.30		5.60	[11]
4	37.9	0	57.2	4.9	[12]
5	35	0.32	37	5	[13]

The detailed of elemental detected by ultimate analysis from biomass reference from Rice Husk are showed in Table 2. Table 2 shows that the element of C, N, O, and H with dominant content for bio-chart from Rice Husk is C, O, and H.

The heating time affects the produced calories. The results show that the calories decrease when the heating time increase. The heating time of 30 minutes for every temperature provides the highest calories as shown in Figure 1.

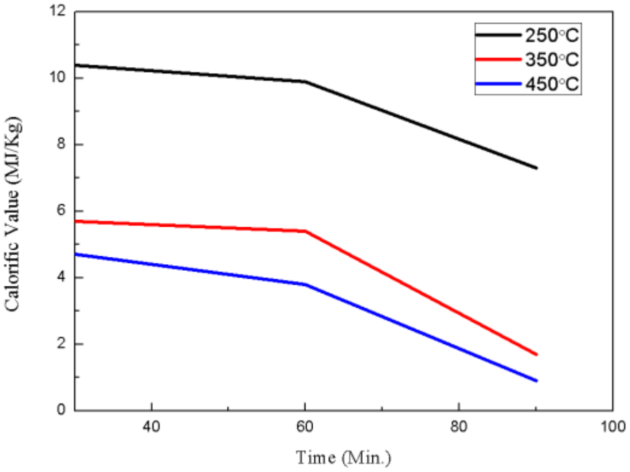
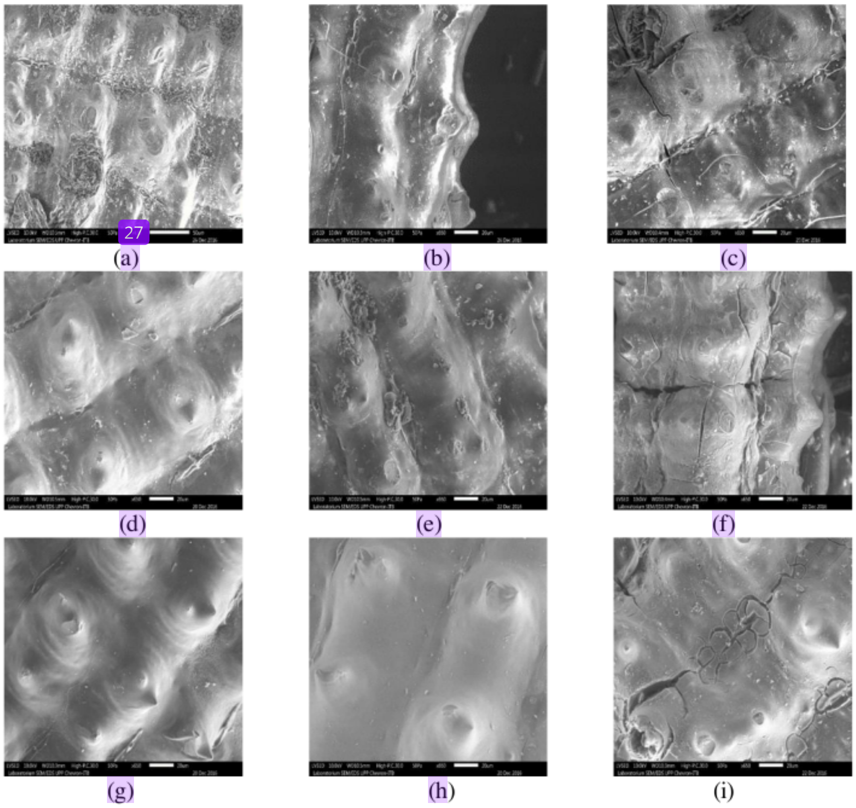


Figure 1. Calorific value determined of biochar from Rice husk by bomb calorimeter



(a) (b) (c)
(d) (e) (f)
(g) (h) (i)

Figure 2. Scanning electron microscopy (SEM) image as a function of temperature and time of heating in free oxygen environments. From the left to right, rows 1, 250°C, row 2, 350°C, and row 3, 450°C for [(a), (d), (g)] 30 minutes, [(b), (e), (h)] 60 minutes, [(c), (f), (i)] 90 minutes.

Figure 2 shows the SEM image as a function of temperature and time of heating in free oxygen environments. From the left to right, rows 1, 250°C, row 2, 350°C, and row 3, 450°C for [(a), (d), (g)] 30 minutes, [(b), (e), (h)] 60 minutes, [(c), (f), (i)] 90 minutes. The characteristic of surface morphology are dot may contain high carbon in the form of nanocarbon.

Table 3. Peak position and intensity of chemical bond for Rice Husk as a function of temperature and time of heating were measured by using Fourier transform infra-red (FTIR).

Chemical bond	250^{O}C Peak Position and Intensity (Time (minutes))	350^{O}C Peak Position and Intensity (Time (minutes))	450^{O}C Peak Position and Intensity (Time)
O-H stretching (Lignin, Hemicellulose, Cellulose)	3412 cm^{-1}, 27.21 (30), 38.26 (60), 48.67 (90)	3423 cm^{-1}, 40.68 (30), 48.19(60) 55.58(90)	3452cm^{-1} 30.64(30), 47.89(60), 64.25 (90)
C=O Stretching (Lignin)	1710cm^{-1}, 54.39(30), 66.11(60), 66.32(90)	1705 cm^{-1} 61.386(30), 58.85(60) 83.57(90)	1697cm^{-1} 0(30), 84.03(60) 95.21(90)
C=C Asymmetric Stretching (Lignin)	1612 cm^{-1}, 49.82(30), 65.11(60), 68.47(90)	1620 cm^{-1} 48.55(30), 65.43(60) 72.69(90)	1627 cm^{-1} 55.62(30), 70.57(60) 84.47(90)
C-O Stretching (Hemicellulose)	1383 cm^{-1}, 85.03(30), 87.05(60), 85.93(90)	1382 cm^{-1} 89.24(30), 86.93(60) 84.94(90)	1382cm^{-1} 89.41(30), 95.88(60) 92.62(90)
C-C Stretching (Hemicellulose)	1421cm^{-1} 66.63(30), no observed(60) no observed(90)	Peak no observed	1423cm^{-1} no observed(30), no observed(60) 97.47(90)
C-O Stretching C-OH Bending (Hemicellulose, Cellulose)	1095 cm^{-1} 2.56(30), 4.83(60) 11.59(90)	1087 cm^{-1} 14.26(30), 6.007(60) 9.654(90)	1095 cm^{-1} 0.50(30), 1.05(60) 5.93(90)
C-H Bending (Lignin)	792 cm^{-1} 55.81(30), 59.97(60) 70.15(90)	796cm^{-1} 51.05(30), 59.40(60) 61.42(90)	798 cm^{-1} 36.91(30), 41.81(60) 45.47(90)

Fourier Transform Infrared (FTIR) spectroscopy used to analyses the effect of temperature and time of heating to the surface functional groups of biochar. Besides the porosity, adsorption behavior is influenced by the chemical reactivity of the surface especially in the form of chemisorbed oxygen in various forms of functional groups. The temperature effect on biochar was successfully investigated using FTIR but not a significant change in intensity with time varied. Figure 3 shows the FTIR spectra as a function of temperature and time of heating in free oxygen environments. Detailed of chemical bonding, peak position, and intensity of Fig. 3 are shown in Table 3. Commonly, most FTIR gives features from organic functional groups for the examination of the organic components of biochar. The peak at 3452 cm^{-1} expected from organic O-H stretching with contribution from any water might retained in the sample or other mineral derived from hydroxyl group. The intensity of the hydroxyl peak which decrease for the temperature increment from 250^{O}C to 450^{O}C indicates that the loss of hydrogen and oxygen atoms due to the breaking bond from hydroxyl group. The aromatic group from lignin give rise to C=C asymmetric stretching at 1612 cm^{-1} indicated as a G band in Refs. [8], corresponds to the sp2-hybradization bonding of carbon atoms. C-H bending modes at 872 cm^{-1} decreases and produced CH_4 as a gas with increasing temperature from 250^{O}C to 450^{O}C .

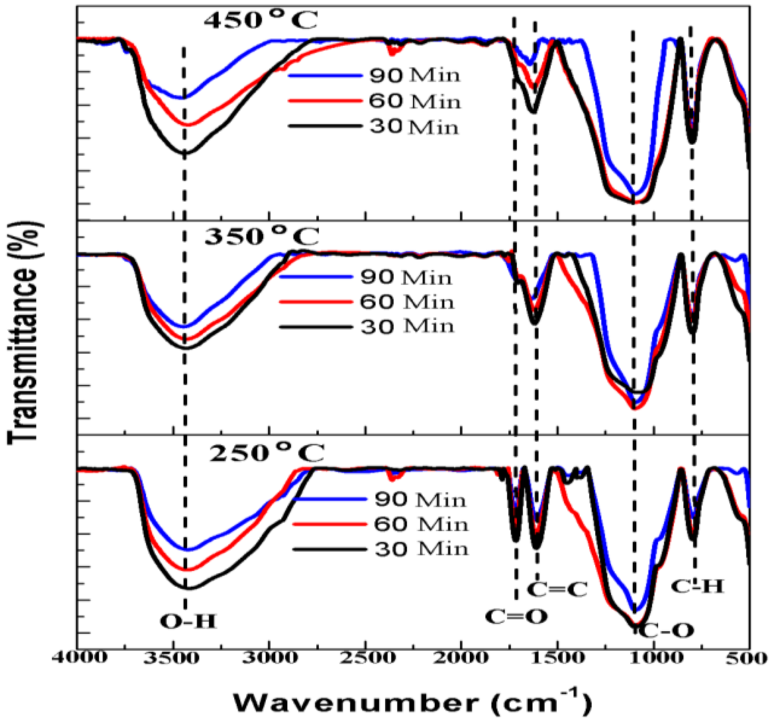


Figure 3. Fourier transform infra-red spectra (FTIR) spectra for biochar from Rice husk as a function of wave number for different temperature and time of heating (see Table 3 for the detail chemical bonding and intensity detected by FTIR).

As shown in Fig. 3 and Table 3, the vibration of C=O stretching of a cyclic and acid anhydride provide the high intensity at 1716 cm^{-1} for temperature 250^{O}C. As the temperature increase the intensity decreases and disappear at the temperature of 450 ^{O}C due to thermal degradation with substantial loss of oxygen atoms and produced CO_2 gas. This is consistent with the result of hydrogen and oxygen content from EDS data in Figure 1. As the heating temperature increase to 350 ^{O}C, the breaking bond of C=O may be also increased.

The transmittance at 1087 cm^{-1} is due to the sp3-hybradization bonding of carbon atoms [8-10], and 1098 cm^{-1} is symmetric C-O stretching for cellulose, hemicellulose, and lignin. The transmission peak of 1421 cm^{-1} recommends the occurrence of aromatic with C-C stretching (ester and phenol). On the other hand, the peak observed at 792 cm^{-1} reveals alkynes with C-H bending is present. The FTIR was confirmed as all cellulose, hemicellulose and some lignin content in bamboo leaf biomass assigned peaks increase with increasing the heating temperature produced gas CO_2 and CH_4.

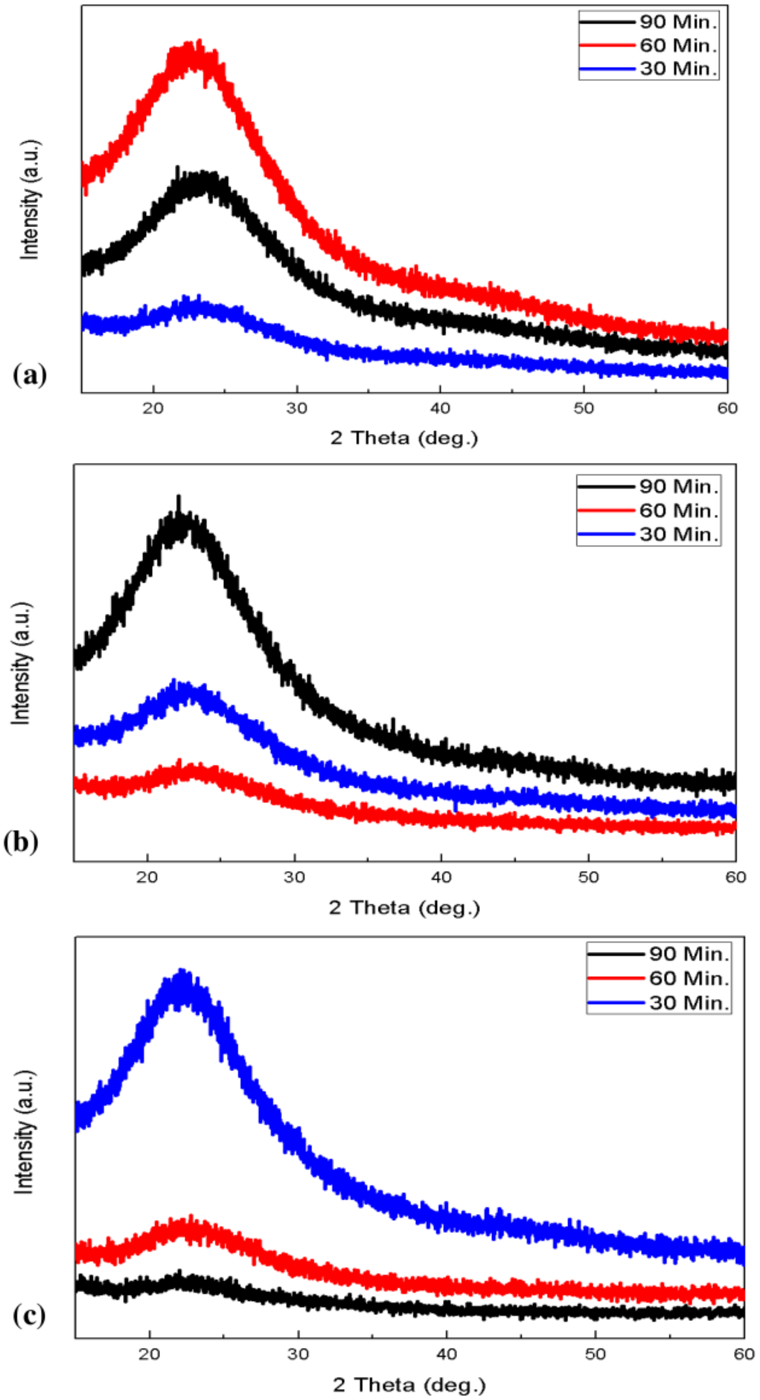


Figure 4. X-ray diffraction (XRD) patterns of the biochar from rice husk for heating time 30 and 90 minutes with temperature (a) 250 ºC, (b) 350ºC, and (c) 450ºC

Figure 4 shows the XRD patterns of the biochar for heating time of 30 and 90 minutes with the temperature of 250 ^{o}C, 350^{o}C, and 450^{o}C. The rice husk samples were burnt in a furnace for the temperatures of 250^{0}C, 350^{0}C, 450^{0}C, continue with the XRD plot observation (Fig. 4). The XRD analysis performs for the selected samples to identify the formation differences of amorphous or crystalline silica for different combustion temperatures. A qualitative assessment of the crystallinity of the samples can be obtained from the intensity of the narrow reflections with the comparation to the broad band around 22^{0}(2theta) for Rice husk burned at 450^{0}C. The intense broad peak observed for the Rice Husk at 450^{0}C samples indicates the amorphous nature of silica (Fig. 4). The start of crystallization of all Rice Husk samples burnt below 450^{0}C shows the amorphous nature. They are typically disordered carbons with high carbon content as can be seen clearly by the broad (002) reflections between 20^{0} and 30^{0} [8,14,15] which is consistent with morphology surface in Fig. 2 from SEM image.

4. Conclusion

Chemical and physical properties of Rice Husk were analyzed by means, SEM-EDS, FTIR, and XRD. The EDS analysis shows that the ash composition for temperatures 350^{o}C and 450^{o}C increases as the heating time increase due to the release of CO_2 and CH_4 gasses from hemicelluloses, cellulose, and lignin decomposition. Carbon and nitrogen content determined by the EDS shows that the time of heating play an important role as a parameter. It shows that the carbon and nitrogen decrease when the heating time increase. While on the contrary, the oxygen increase when the heating time increase. The XRD shows broad (002) reflections between 20^{o} and 30^{o} and surface morphology from SEM indicated high carbon content. FTIR shows aromatic group corresponds to the sp2-hybradization bonding of carbon atoms C=C asymmetric stretching at 1612 cm^{-1}. These results of the chemical composition characteristic, calorific value, bonding formation and structural properties of the Rice husk shows that the prominent source of useful energy can eventually replace the fossil fuel.

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