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Physical and chemical characterizations of biochars derived from different agricultural residues

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Abstract

Biochar has received large attention as a strategy to tackle against carbon emission. Not only carbon fixation has been carried out but also other merits for agricultural application due to unique physical and chemical character such as absorption of contaminated compounds in soil, trapping ammonia and methane emission from compost, and enhancement of fertilizer quality. In our study, different local waste feed stocks (rice husk, rice straw, wood chips of apple tree (*Malus Pumila*) and oak tree (*Quercus serrata*)), in Aomori, Japan, were utilized for creating biochar with different temperature (400–800 °C). Concerning to the biochar production, the pyrolysis of lower temperature had more biochar yield than higher temperature pyrolysis process. On the contrary, surface areas and adsorption characters have been increased as increasing temperature. The proportions of carbon content in the biochars also increased together with increased temperatures. Infrared-Fourier spectra (FT-IR) and ^{13}C-NMR were used to understand carbon chemical compositions in our biochars, and it was observed that the numbers of the shoulders representing aromatic groups, considered as stable carbon structure appeared as the temperature came closer to 600 °C, as well as in FT-IR. In rice materials, the peak assigned to SiO_2, was observed in all biochars (400–800 °C) in FT-IR. We suppose that the pyrolysis at 600 °C creates the most recalcitrant character for carbon sequestration, meanwhile the pyrolysis at 400 °C produces the superior properties as a fertilizer by retaining volatile and easily labile compounds which promotes soil microbial activities.

1 Introduction

The interests in biochar utilization as a strategy for mitigation of global warming steadily are increasing. Besides the growing attention to biochar utilization for the carbon sequestration, a number of works are reported on a variety of purposes such as the improvements of soil fertility, plant growth and decontamination of pollutants such as pes-

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ticides, heavy metals, and hydrocarbons (Beesley et al., 2011; Cabrera et al., 2011). Since the diverse applications of biochar should be in accordance with the adequate ability and property each biochar has, the information on production scheme, which mostly determine fundamental biochar character, is a key factor to understand them. Pyrolysis is the thermo-chemical process of heating biomass under low oxygen presence to produce pyrogenic material. Heating durations, temperatures, and types of feedstock materials are main components to determine the property of final product. The biochar derived from low temperature pyrolysis contained easily decomposable substrates, which contributed the plant growth as an additional nutrition (Lammirato et al., 2011). On the other hand, the structure of the biochar derived from the high temperature was refined to more selected compounds, formed larger surface area and also showed higher aromatic carbon contents, and finally this physico-chemical property promotes the adsorption capacity for bioremediation as well as the recalcitrant character for carbon sequestration (Lehmann, 2007). Also, it has been considered that the yields by-products such as fuels and gases generated during the pyrolyisis were influenced by pylolysis temperature and duration (Grierson et al., 2009; Mahinpey et al., 2009; Gell et al., 2011).

The type of feedstock material is also important factor to guide the proper orientation of application. The cation exchange capacity is enhanced more by manure-based biochars than wood (*Eucaryptus*) biochar (Singh et al., 2010), meanwhile the treatment of the soil with woodchip biochar resulted in higher saturated hydraulic conductivities than the manure biochar treatment (Lei and Zhang, 2012) The aim of our study is to evaluate the thermo-chemical properties of biochars derived by different feedstock with different temperature (400–800 °C). Rice husk (RH) and rice straw (RS) are used in our work as original materials, since the global amounts of residues from rice crop (*Oryza sativa* L.) are 0.9 Gt for a year which constitutes the 25 % of the total amount of whole agricultural residues in the world (Knoblauch et al., 2011). Also, the research on characteristic of biochars derived from orchard residues, pruning woodchips of apple tree

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(AB) (*Malus pumila*) are conducted as well as the other tree wood chip from oak tree (OB), (*Quercus serrata*).

2 Materials and methods

2.1 Biochar preparation from agricultural residues

Biochars used in this work were made from two rice residues (*Oryza sativa* L.): straw and husk, and two woody materials: a kind of broad-leaved trees (*Quercus serrate* Murray) and apple wood chips (*Malus pumila*). All materials were first dried in air and then cut into small pieces to put into a ceramic vessel (370 cm^3) for a commercial electric furnace (SOMO-01 Isuzu, Japan), and were charred for 10 h at different temperatures (from 400 to 800 °C) with 10 °C min^{-1} heating rate.

2.2 Chemical analysis of biochar

After the pyrolysis process, all samples were grounded to be homogenized, and sieved to less than 0.5 mm in diameter. The biochar yield was calculated by the proportion of the weight of pyrolysis product to the original material. The analysis of volatile matter was conducted according to the American Society for Testing and Materials (ASTM) D1752-84, which is recommended in the biochar international organization (http://www.biochar-international.org): the volatile matter was determined by measurement of weight loss following combustion of about 1 g of charcoal in a crucible at 950 °C. The pH of each mixture pH at 1 : 10 (w/v) ratio was measured with a MP220 pH. Micro- and meso-porosity was evaluated by the iodine (I_2) and methylene blue (MB) adsorption capacity, respectively, following the methodology used by Gaspard et al. (2007). The specific surface area was determined using N_2 sorption isotherms run on automated surface area. The specific surface areas distribution reading were taken from adsorption isotherms, using the equation of Brunauer–Emmett–Teller (BET) surface area (Zang et al., 2011).

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2.2.1 Elemental composition

The elemental composition of carbon (C), hydrogen (H), and nitrogen (N) was determined using an Elemental analyzer (Thermo Finnigan EA-1112, Thermo Fisher Scientific Inc., Massachusetts, America), and the oxygen (O) content was determined by Vario EL cube, Elementar Analysensysteme GmbH Co.

2.2.2 Infrared spectra

Infrared spectra were recorded on a Varian 670-IR (Agilent Technologies Inc., CA) using the pellet technique by mixing 1 mg of dried biochar with 300 mg of pre-dried and pulverized spectroscopic-grade KBr (from Merck & Co., Whitehouse Station, NJ). The following broad band assignation was used (Chen and Chen, 2009; Haslinawati et al., 2011; Novak et al., 2010; Peng et al., 2011; Yuan et al., 2011; Wu et al., 2013): 3400 to 3410 cm^{-1}, H-bonded O–H stretching vibrations of hydroxyl groups from alcohols, phenols, and organic acids; 2850 to 2950 cm^{-1}, C–H stretching of alkyl structures; 1620–1650 cm^{-1}, aromatic and olefinic C=C vibrations, C=O in amide (I), ketone, and quinone groups; 1580 to 1590 cm^{-1}, COO- asymmetric stretching; 1460 cm^{-1}, C–H deformation of CH_3 group; 1280–1270 cm^{-1}, O-H stretching of phenolic compounds; and band around 1000–1100 cm^{-1}, bending of Si-O stretching.

2.2.3 Solid-state nucleic resonance spectroscopy (NMR)

Cross-polarisation magic angle spinning (CPMAS) ^{13}C nuclear magnetic resonance (^{13}C-NMR) spectra were acquired in the solid samples with a Varian 300, equipped with a 4 mm-wide bore MAS probe, operating at a ^{13}C resonating frequency of 75.47 MHz. The assignation of the following peaks and broads was used (Brewer et al., 2009; Calvero et al., 2011): The peak around 30 ppm, is considered as content of methylenic chains and/or CH_2 groups deriving from various lipid compounds, plant waxes and plant biopolyester; two peaks at 55 ppm and 70 ppm, assigned to methoxy and O-

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alkyl groups characteristic of the relatively-easily-biodegradable compounds such as remaining cellulose, and hemicellulose; the broad band around 130 ppm, related to alkyl substitutions in the p-hydroxy phenyl ring of cinnamic and p-coumaric units of both lignin and suberin biopolymers, as well as to both partially-degraded lignin structures and condensed aromatic and olefinic carbons; the sharp peak at 170 ppm, assigned to a large content of carboxyl groups in aliphatic acids of plant and microbial origin and/or amide groups in amino acid moieties. The spectra were integrated in the chemical shift (ppm) resonance intervals of 0–45 ppm (paraffinic carbons), 46–65 ppm (methoxy C from OCH_3, and complex aliphatic carbons), 66–90 ppm (O-aliphatic C, such as alcohols and ethers), 91–145 ppm (aromatic carbon), 145–160 ppm (phenolic carbons), 160 –185 ppm (carboxyl, amides and ester) and 185–220 ppm (carbonyls) (Wang et al., 2007; Zhang et al., 2011).

3 Result and discussion

3.1 Physicochemical character of biochar

The characteristics of biochars originated from different materials are shown in Table 1. Lower temperature pyrolysis process, has higher biochar yield and more enriched volatile matter inside biochar product. The volatile compounds are easily degradable compounds, which stimulates the activity of microorganisms in soil as substrate and ends up supplying plant nutrition (Steinbess et al., 2009; Zimmerman, 2012). The biochar yields and volatile contents gradually reduced by increasing pyrolysis temperatures. Not only the range of pyrolysis temperature, the type of feedstock also affected biochar yields and the volatile matter. Among different biochar types, two types of wood biochars (AB and OB) showed low yields and low volatile contents compared to other two kinds of biochar from rice residues (RS and RH). Those results might be due to the differences in compositions of the original agricultural material since rice plants are enriched with Si while recalcitrant carbons such as lignin content are predominantly

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contained in woody plants (Liu and Zhang, 2009; Spokas et al., 2009; Joseph et al., 2013).

The pH value increased by higher temperature, probably resulted from the concentration of non-pyrolyised inorganic elements in the original feedstocks (Novak et al., 2009). The porosity and surface area represent physical properties of biochar, and connect strongly with adsorption, and water retention abilities those has potential to improve soil property (Kalderis et al., 2008), and in particular, taking the advantage of enhanced these properties, the number of works on the application of biochar derived from the rice husk have been reported (Kalderis et al., 2008; Liu and Zhang, 2009; Lei and Zhang, 2013). As shown in Table 1, the biochar production with higher temperature generally enlarged methylene blue number, I_2 absorption, and surface area than the production with lower temperature ($p < 0.001$), and these results are in accordance with other previous work (Gaskin et al., 2008; Liu and Zhang, 2009; Yu et al., 2011). Regarding of comparison among the variety of origins, the difference in micro-porosity (I_2) of biochars between from wood feedstock (AB and OB) and from rice residues (RH and RK) was gradually enlarged as increasing pyrolysis temperature from 500 °C to 800 °C. In contrast, an apparent difference in surface area was observed. The surface areas of RH and RK reduced at 800 °C, while those of AB and OB still had been extending. These decreases in surface areas of RH and RK will be attributed to ash content in biochar, that high content of inorganic ash in biochar filled or blocked access to micropores, resulting in relatively low surface area (Mackay and Roberts, 1982; Song and Guo, 2012).

3.2 Analytic elements

Analytical elements and both ratios of H/C and O/C are also indicators to evaluate the characters of biochars (Nguyen and Lehmann, 2009). As shown Table 2, it is observed that the increasing temperature results in loss of hydrogen and oxygen than carbon. Dehydrogenation of CH_3 on biochar properties as consequence of thermal induction is the indicative of changes in biochar recalcitrance (Harvey et al., 2012). Concerned with oxygen loss, the biomass material normally comprises of labile and recalcitrant O fractions; a labile O fraction that is quickly lost upon initial heating, and a recalcitrant O fraction which retained in the char of final product (Rutherford et al., 2013).

Regarding with the H/C and O/C, Table 2 shows the redaction in these ratios with charring temperature, being attributed to the dehydration and decarboxylation reactions. The change of O/C ratio in the range from 400 °C to 500 °C was remarkably observed in the order of RS > RK > AB > OB. It is reported (Yang et al., 2007; Khodadad et al., 2011) that biochar made from wood and at higher temperatures are less biologically labile, containing relatively higher proportions of more aromatic organic matter, compared to other original materials of agricultural residues and at lower temperature. As shown in the van Krevelen diagram (Fig. 1), the constancy of the reduction in the H/C and O/C ratios as increasing temperature are observed, reflecting the loss of easily degradable carbon compounds such as volatile matters. At the parallel, lower C/O ratio in biochar with higher temperature indicate the arrangement of aromatic rings (Spokas et al., 2010), forming crystal graphite-like structure with high stability (Wu et al., 2012; Dong et al., 2013).

3.3 Chemical composition with spectra parameters (FT-IR and NMR)

The FT-IR is a great tool to shed on the light to understand the shift change of chemical composition. The aliphatic loss process is represented by the band of FT-IR with aliphatic C–H stretching (2950–2850 cm^{-1}) at increasing temperature from 400 °C to 600 °C (Fig. 2), meanwhile a couples of representative peaks for aromatic carbon appeared more clearly such as C–H stretching (750–900 cm^{-1} and 3050–3000 cm^{-1}), C=C (1380–1450 cm^{-1}), C–C and C–O stretching (1580–1700 cm^{-1}). As shown by the infrared spectra, charring temperature modifies the functional group, and thus aliphatic C groups decrease but aromatic C increases (Lee et al., 2010). Since longevity of biochar is a matter of debate in relation with the biochar production (Nguyen and Leehman, 2009; Peng et al., 2011), the pyrolysis process of 600 °C, which creates more recalcitrant character by increasing aromatic compounds, has suitable method in

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terms of the carbon sequestration. However, when the charring temperature increase over 700 °C and 800 °C, the intensity of bands decline gradually such as hydroxyl group (3200–3400 cm^{-1}) and even aromatic bands (1580–1600 cm^{-1} and 3050–3000 cm^{-1}). Other works (Yuan et al., 2011) report that the number of bounds representing functional groups are present at the lower-temperature biochars (300 °C and 500 °C), and are absent in the biochar-derived by 700 °C.

The characteristic of feedstock type of biochar has been reflected by the presence of bound 1040–1100 cm^{-1}, assigned to SiO_2, and this band was shown in all biochars originated from rice materials (RH and RS), showing in the Fig. 3a. The SiO_2 is a major component in chemical structure of rice material, and is typical of recalcitrant property.

The ^{13}C NMR spectra of different biochars are shown in Fig. 4a and b with similar shape for all biochar materials, dominated by the presence of condensed aromatic signal at 127 ppm. Furthermore, the signal was observed more clearly in all different biochars which are produced at 600 °C, and the sharpness of this peak was gradually weaken as temperature goes away from 600 °C. Same trend was shown in the shoulder of methoxyl carbons of lignin and carboxylic carbon, at 57 ppm and 190 ppm, respectively. Especially, the biochars, made from rice straw at 800 °C (Fig. 3b) demonstrated the decomposition of shoulder of these ranges. From this result as well as shown in FT-IR figure, it worth of noting that there is no much need to produce biochar with very high temperature (700 °C and 800 °C) for preserving stability character,based on the predominance of aromatic structure and keeping higher chemical property with functional groups.

Concerning with lower temperature, the shoulder at around 20 ppm, assigned to easily degradable carbon, appeared in the 400 °C-produced biochars, and this shoulder was eliminated in the biochar produced at 500 °C. This accords with other's work (McBeath et al., 2013) that biochar, produced at 300–400 °C, exhibited broad alkyl signals and carbohydrate band, meanwhile these bands were not detectable over 400 °C. As shown in previous section, the biochar, produced at lower temperature, has higher volatile carbon contents. The type of easily degradable compounds contributes to mi-

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croorganisms in soil as substitute (Khodadad et al., 2011). In this sense, lower temperature is suitable for the biochar utilization for soil fertility. On the contrary, higher temperature pyrolysis selects functional groups and provides aromatic predominant presence in chemical composition, consequently resulting to the formation of recalcitrant structure.

4 Conclusions

In conclusion, the temperature of pyrolysis process and the type of feedstock change the physic-chemical properties of biochar. In our study, high temperature pyrolysis process produces more recalcitrant character of functional group and adsorption property due to large surface area and porosity. By contrast, lower temperature pyrolysis has volatile carbon compounds retained inside biochar structure, which can induce microbial activities in soil.

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Table 1. Physical and chemical characteristics of the biochars from different feedstocks; AB (apple tree branch), OB (oak tree), RH (rice husk), and RS (rice straw).

Samples	Temperature (°C)	Biochar Yield (%)	Volatile Content (%)	pH (H_2O)	Methylene Blue (mg g^{-1} DW)	I_2 adsorption (mg g^{-1} DW)	BET Surface Area (m^2 g^{-1})
AB	400	28.3	32.36 ± 0.05	7.02 ± 0.08	4.36 ± 0.21	44.96 ± 2.61	11.90
	500	16.7	18.27 ± 0.28	9.64 ± 0.07	12.04 ± 0.38	97.87 ± 2.65	58.60
	600	16.6	11.07 ± 0.20	10.04 ± 0.02	5.65 ± 0.39	122.05 ± 1.53	208.69
	700	15.8	7.72 ± 0.10	10.03 ± 0.02	10.63 ± 0.67	208.28 ± 1.50	418.66
	800	15.5	6.82 ± 0.07	10.02 ± 0.02	51.79 ± 0.58	298.51 ± 1.70	545.43
OB	400	35.8	32.06 ± 0.05	6.43 ± 0.04	3.90 ± 0.28	38.66 ± 0.04	5.60
	500	28.6	19.42 ± 0.27	8.10 ± 0.12	5.59 ± 0.47	91.74 ± 0.06	103.17
	600	22.0	12.30 ± 0.01	8.85 ± 0.07	5.51 ± 0.39	131.27 ± 1.47	288.58
	700	20.0	8.28 ± 0.07	9.54 ± 0.00	17.06 ± 0.16	212.79 ± 0.09	335.61
	800	19.1	7.87 ± 0.06	9.68 ± 0.03	29.38 ± 0.47	250.28 ± 1.40	398.15
RH	400	48.6	22.00 ± 0.13	6.84 ± 0.03	2.91 ± 0.75	44.07 ± 1.55	193.70
	500	42.4	10.56 ± 0.11	8.99 ± 0.04	9.72 ± 0.37	75.38 ± 1.58	262.00
	600	37.3	6.02 ± 0.27	9.41 ± 0.00	13.50 ± 0.21	68.95 ± 3.13	243.00
	700	32.8	3.85 ± 0.11	9.52 ± 0.02	13.88 ± 0.99	121.02 ± 1.58	256.00
	800	32.0	3.17 ± 0.19	9.62 ± 0.01	34.06 ± 0.34	174.40 ± 3.07	295.57
RS	400	39.3	22.42 ± 0.09	8.62 ± 0.03	29.32 ± 2.40	74.66 ± 2.64	46.60
	500	32.6	12.80 ± 0.11	9.82 ± 0.01	29.58 ± 2.81	95.94 ± 1.52	59.91
	600	23.4	8.36 ± 0.03	10.19 ± 0.01	33.65 ± 4.93	85.57 ± 1.58	129.00
	700	18.4	5.33 ± 0.13	10.39 ± 0.03	40.45 ± 3.02	100.55 ± 1.52	149.00
	800	18.3	4.47 ± 0.15	10.47 ± 0.04	82.61 ± 2.02	190.21 ± 1.31	256.96

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Table 2. The elemental compositions of biochars from different feedstocks; AB (apple tree branch), OB (oak tree), RH (rice husk), and RS (rice straw).

Samples	Temperature (°C)	C* (%)	H* (%)	N* (%)	O (%)	O/C	H/C
AB	400	70.18 ± 0.15	4.13 ± 0.01	0.76 ± 0.00	20.56 ± 0.12	0.22	0.71
	500	79.12 ± 0.00	2.65 ± 0.09	0.34 ± 0.01	11.98 ± 0.10	0.11	0.40
	600	81.46 ± 0.05	1.96 ± 0.02	0.46 ± 0.00	13.63. ± 0.26	0.12	0.29
	700	82.26 ± 1.39	1.21 ± 0.05	0.41 ± 0.02	16.34 ± 0.68	0.15	0.18
	800	84.84 ± 0.08	0.60 ± 0.01	0.34 ± 0.01	5.81±0.03	0.05	0.08
OB	400	70.52 ± 0.21	3.70 ± 0.02	0.69 ± 0.02	21.47 ± 0.15	0.23	0.63
	500	77.57 ± 0.31	2.51 ± 0.16	0.51 ± 0.03	17.73 ± 0.45	0.17	0.39
	600	81.22 ± 0.48	1.92 ± 0.01	0.48 ± 0.02	15.96 ± 0.15	0.15	0.28
	700	83.22 ± 0.23	1.16 ± 0.06	0.31 ± 0.00	14.97 ± 0.07	0.13	0.17
	800	82.85 ± 0.42	0.69 ± 0.06	0.32 ± 0.00	17.29 ± 0.12	0.16	0.10
RH	400	44.59 ± 0.26	2.50 ± 0.00	0.69 ± 0.02	16.32 ± 0.23	0.27	0.67
	500	45.15 ± 0.33	1.27 ± 0.03	0.47 ± 0.02	7.12 ± 0.16	0.12	0.34
	600	40.35 ± 0.74	0.85 ± 0.05	0.37 ± 0.01	9.23 ± 0.31	0.17	0.25
	700	38.81 ± 0.46	0.46 ± 0.04	0.26 ± 0.02	12.69 ± 0.20	0.25	0.14
	800	40.41 ± 0.68	0.28 ± 0.01	0.22 ± 0.00	2.69 ± 0.01	0.05	0.08
RS	400	49.92 ± 0.15	2.80 ± 0.12	1.22 ± 0.01	12.02 ± 0.06	0.18	0.67
	500	37.48 ± 0.22	0.93 ± 0.03	0.61 ± 0.01	8.64 ± 0.33	0.17	0.30
	600	33.78 ± 1.02	0.60 ± 0.07	0.41 ± 0.04	13.68 ± 0.32	0.30	0.21
	700	36.26 ± 0.79	0.51 ± 0.06	0.34 ± 0.02	17.38 ± 0.94	0.36	0.17
	800	29.17 ± 0.37	0.25 ± 0.02	0.25 ± 0.01	3.71. ± 0.01	0.10	0.10

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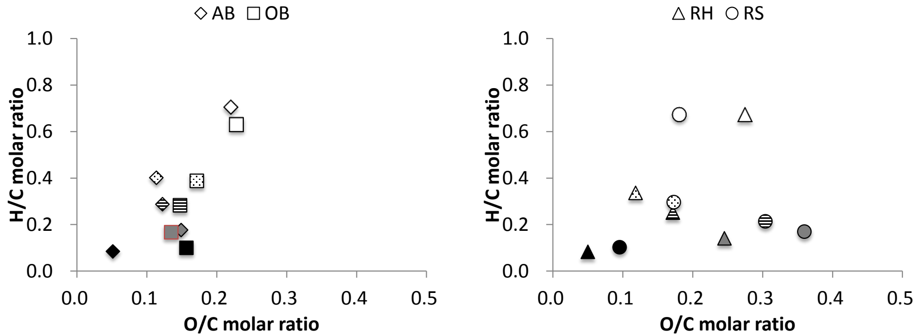

Figure 1. Van Krevelen diagram of biochar originated from different feedstock; AB (apple tree branch), OB (oak tree), RH (rice husk), and RS (rice straw). Each symbol shows pyrolysis temperature as follows; Black = 800 °C, Gray = 700 °C, Line = 600 °C Dot = 500 °C, and White = 400 °C.

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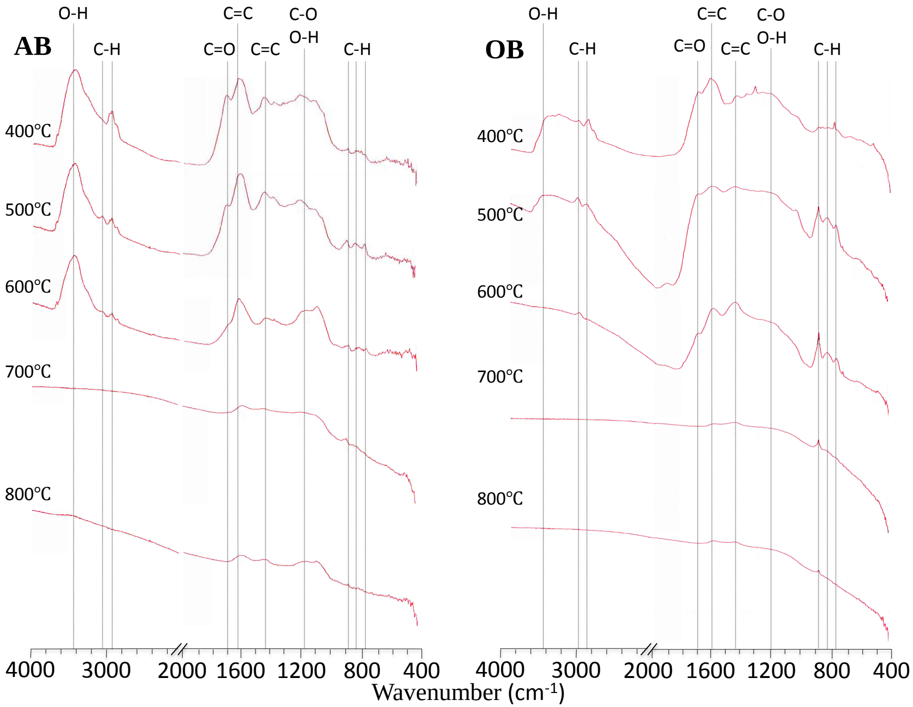

Figure 2a. FT-IR spectra of biochars of wood materialss; AB (apple tree) and OB (oak).

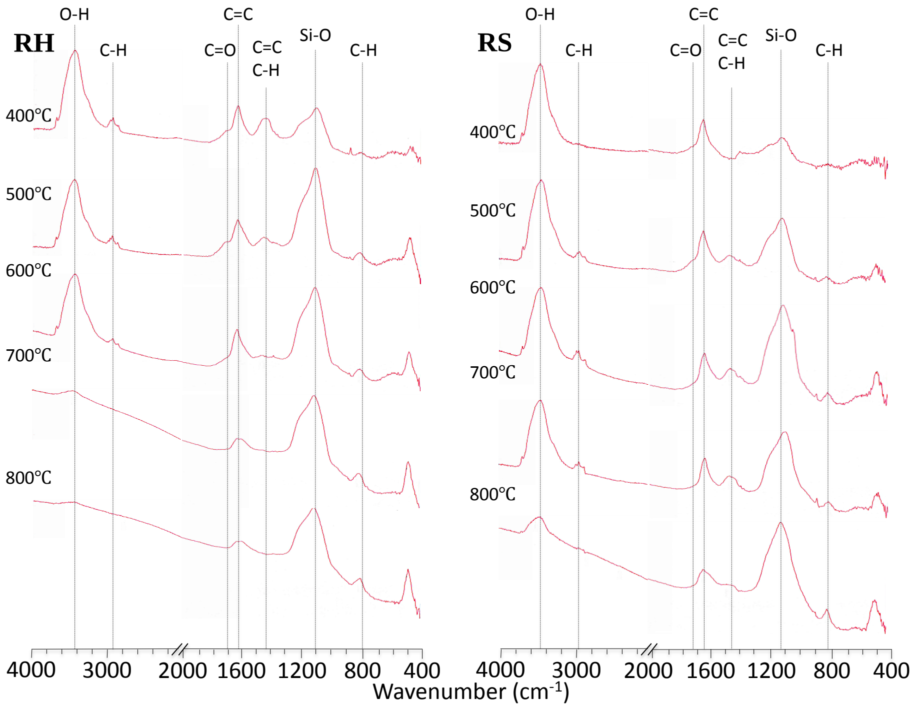

Figure 2b. FT-IR spectra of biochars of rice residues; RH (rice husk) and RS (rice straw).

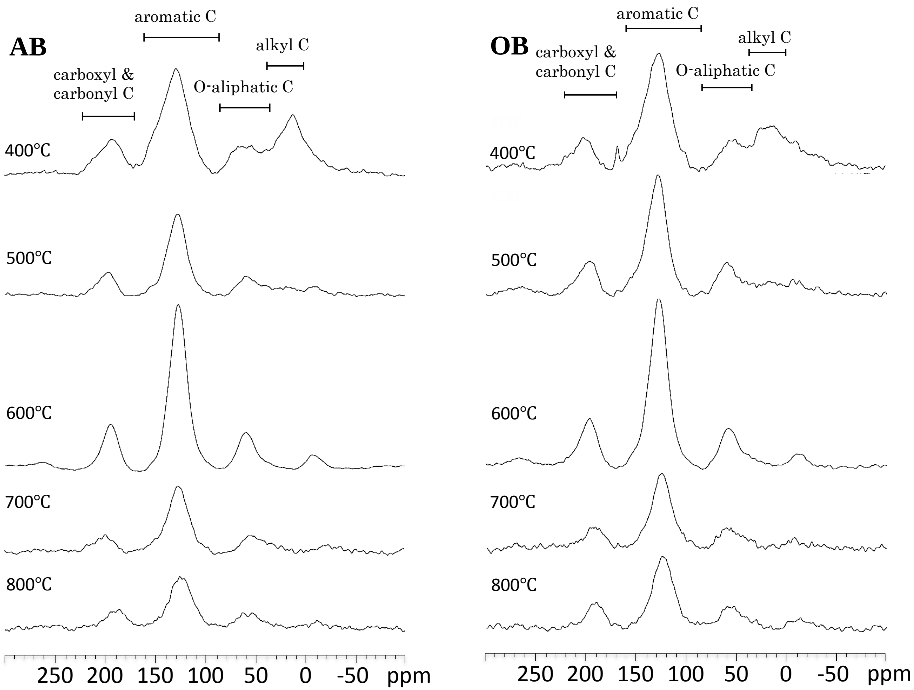

Figure 3a. ^{13}C CPMAS-NMR of biochars of wood materials; AB (apple tree) and OB (oak tree).

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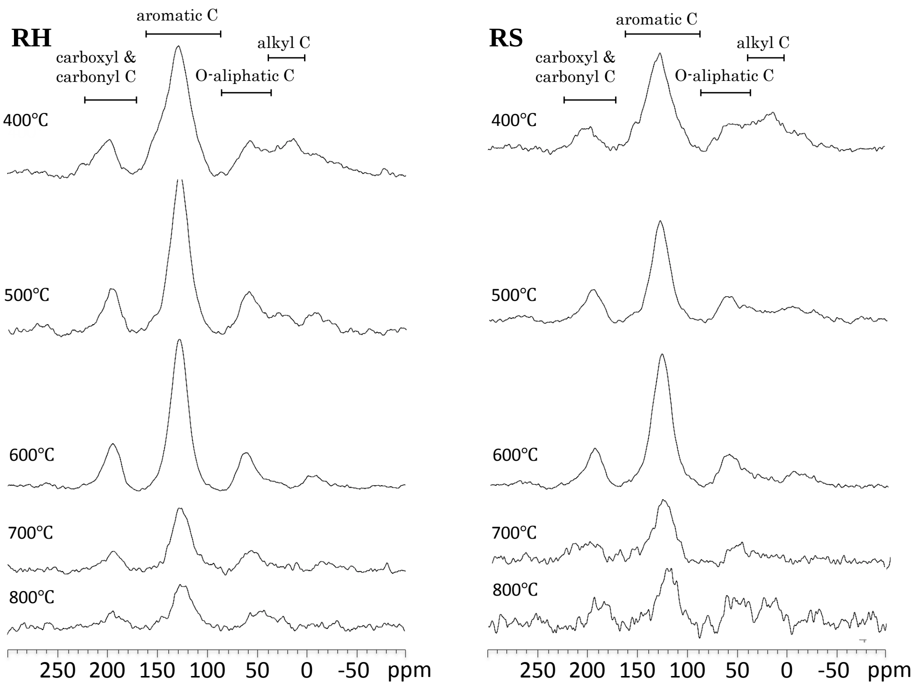

Figure 3b. ^{13}C CPMAS-NMR of biochars of rice residues; RH (rice husk) and RS (rice straw).

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