Development of Technologies for the Utilization of Agricultural and Forestry Wastes: Preparation of Biochar from Rice Residues

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ABSTRACT

In this study biochar was prepared by pyrolysis of rice husk and rice straw. The effect of pyrolysis temperature (350, 450 and 550 °C) and the heating rate (10 °C/min or 50 °C/min) on the yield and properties of the biochar were investigated. The results indicated that the char yields decreased with increasing charring temperature and heating rate, ranging from 44-38% with RH char and from 43-34% with RS char, respectively. The effect of heating rate on the biochar yield and proximate analysis was more important at low temperature. Ash content, fixed carbon content and the pH of the char increased while volatile matter content and CEC decreased with the increase of pyrolysis temperature. Elemental analysis showed that the carbon content of the char increased while H and O decreased with raising temperature, resulting in the progressive decrease of H/C and O/C ratios. The volatiles released during the pyrolysis process creating pores in the carbonaceous materials which was revealed with the scanning electron microscope images. The results of pore volume from N_2 adsorption and Hg porosimeter showed that the most of biochar pores were macropores (≥50 nm). Although the RH char had higher specific surface area, micro- and mesopore volume, but the water adsorption capacity of the RH biochar was much lower than the RS biochar. Thus the macropore volumes may have a significant effect on the water adsorption capacity of the biochar. The main components in the pyrolysis gas detected were CO_2, CO, CH_4 and H_2. Bio-oils from RH and RS were composed of a complex mixture of aromatic hydrocarbons.

KEYWORDS

Agricultural Waste, Biochar, Pyrolysis, Rice Husk, Rice Straw.

INTRODUCTION

Pyrolysis is a thermo-chemical decomposition process in which organic material is converted to liquid (bio-oil), gaseous, and solid (char) fractions in the absence of oxygen (Demerbas, 2001). The proportion and composition of pyrolysis products (oil, liquid and char) depends strongly on the pyrolysis technique used and the reaction parameters. Typically, pyrolysis is divided into two modes as: fast and slow. Slow pyrolysis employs a lower heating rate and longer residence time and it is used to maximize solid product yields which are known as char or biochar (Maggi and Delmon, 1994; Overend, 2004).

Biochar includes high C content, which mainly composed of aromatic compounds. As a soil amendment material, biochar is believed to store carbon in the soil for hundreds to thousands of years. The relative stability of biochar determines the length of its contribution to the mitigation of green house gas (GHG) emissions. Moreover, its presence in the soil is practical due to improvement of soil properties and crop yields. Thus, biochar may offer a win-win technology to mitigate global warming effect and food security (Lehmann, 2007).

In recent years, many studies have been carried out to deal with the characterization of various agricultural wastes such as: safflower seed press cake (Angın, 2013), oil-palm-shell (Lua et al., 2006), soybeen stover, peanut shells (Ahmad et al., 2012), rubber wood sawdust (Ghani et al., 2013), bagasse, cow biosolids (Shinogi and Kanri, 2003), swine manure (Tsai et al., 2012) and wheat straw (Bruun et al., 2011). However, biochar`s properties, including stability, depend not only on feedstock type but also processing conditions such as; temperature, heating rate, pressure, reaction residence time, reaction vessel, pre-treatment and particle size. Although all of these parameters contribute to the final biochar structure, the pyrolysis temperature is expected to be the most important of the factors studied (Lehmann et al., 2009). Thus, many studies characterized the effects of pyrolysis temperature on biochar`s properties.

Rice husk and straw are most abundant agricultural wastes in South-East Asia, therefore many researchers were interested in characterizing rice husk and rice straw biochar (Maiti et al., 2006; Tsai et al., 2007; Fu et al., 2012 and Wu et al., 2012). There are many studies reported on the influence of pyrolysis temperature on rice residue, however, a few study deals with the effect of heating rate on the properties of biochar. Therefore, the objective of this study is to characterize the biochar prepared from rice husk and rice straw at different temperatures (350, 450 and 550 ^{o}C) and heating rates (10 and 50 ^{o}C/min). In addition, the pyrolysis gas and oil compositions were also determined.

MATERIALS AND METHODS

3.1. Raw materials

The raw biomasses materials were rice husk and rice straw which were collected from Japan and Vietnam. The feedstocks were firstly sundried and kept in well-sealed plastic containers for long-time room-temperature storage. The raw materials were characterized for the proximate and ultimate analyses by the methods similar to those for biochar as described in Section 3.3. In addition, moisture content was determined by calculating the weight loss after heating the biomass at 105 ^{o}C until a constant weight was obtained.

3.2. Pyrolysis

The biochar was prepared by pyrolysis of the feedstock in a tubular electric furnace at constant nitrogen flow. In a typical run, a bundle (7 cm long) of dried feedstock was loaded in a stainless steel mesh basket and the basket was placed inside a tubular ceramic reactor. The reactor was purged with dry N_2 (99.99%) gas flow for 1 hour to remove any oxygen remaining in the reactor. Then the reactor was heated at a heating rate of 10 or 50 °C/min from room temperature to a predetermined pyrolysis temperature (350, 450 and 550 °C) at a nitrogen flow of 1 L/min and kept at final temperature for 1 hour. Then the reactor was cooled down to the room temperature over a period of 2-3 hours. Finally the biochar sample was removed from the furnace and weighed immediately.

In this study, char samples were denoted as RHXXX-YY and RSXXX-YY, where RH is rice husk, RS is rice straw, XXX is pyrolysis temperature (^{o}C) and YY is heating rate (^{o}C/min).

3.3. Biochar characterization

3.3.1. The yield

The yield of a biochar was determined from the initial weight of the dry feedstock and the final weight of the biochar sample.

Yield (%) = $(W_f / W_b) \times 100$,

where W_f and W_b are weights of the dry feedstock and the biochar sample, respectively.

3.3.2. Proximate analysis

The proximate analysis was performed to measure volatiles and ash content. Ash content was determined by combustion of biochar sample in a ceramic crucible at 700 °C for 6 hours without ceramic cap. Volatile matter content was determined as weight loss after combustion in a ceramic crucible with a loose ceramic cap at 850-900 ^{o}C for 6 minutes (Mukherjee, 2011). The fixed carbon was calculated by difference.

3.3.3. Ultimate analysis

Ultimate analysis describes the elemental composition of the biomass. Carbon, nitrogen and hydrogen contents of the biochar samples were determined by dry combustion using a Perkin Elmer Elemental Analyzer 2400II with routine analytical uncertainty better than ± 5%. Oxygen was estimated as follows: O = 100 – (C + H + N + ash) (all wt.%) (Calvelo Pereira et al., 2011).

3.3.4. pH measurement

The char sample was soaked in distilled water in a weight ratio of 1:100 and stirred for 3 hours at room temperature. The pH was observed using a digital pH meter (Mettler-Toledo AG).

3.3.5. Cation exchange capacity

Cation exchange capacity (CEC) was estimated for the biochar samples by using NH_4^+ exchange method (Schollenberger and Simon, 1945), where 0.5 g of biochar was rinsed twice with 25 ml of 1 M CH_3COONH_4 (pH 7) to saturate exchange sites. Excess solution was removed from biochar samples by washing the biochar with 10 ml of 95% ethyl alcohol. The adsorbed NH_4^+ was extracted with 25 ml of 0.1 M KCl twice and titrated with calibrated 0.05 M NaOH in the presence of 5 ml of formalin and phenolphthalein.

3.3.6. The porous characteristics of biochar

The porous characteristics of biochar were determined by N_2-adsorption analyzer (Micromeritics Gemini 2375) and Hg porosimeter (Micromeritics Autopore III).

Specific surface area and micropore volume were determined by using nitrogen absorption analyzer at 77 K. The BET standard method, applied in a relative pressure ranged from 0.05 to 0.30, was used to determine the surface areas (S_{BET}) of the samples. Total pore volume of the biochar was divided into micropores (size <2 nm), mesopores (2-50 nm) and macropores (>50 nm) (Rouquerol et al., 1999). The total pore volume $V_t(N_2)$ was calculated by converting the amount of nitrogen gas adsorbed at a relative pressure of 0.96 into the volume of liquid adsorbate. The micropore volume, $V_{micro}(N_2)$ was obtained by applying the Dubinin-Radushkevich (DR) equation (Dubinin, 1989; Fu et al., 2012). The mesopore volume, $V_{meso}(N_2)$, was obtained by subtraction of the volume of nitrogen adsorbed at a relative pressure 0.10 and the total volume $V_t(N_2)$. The average pore size D_{av} was calculated by using the data of S_{BET} and $V_t\ (N_2)$, $D_{av} = 4V_t\ (N_2) / \mathrm{S_{BET}}$ (Tsai et al., 2012). Pore size distribution was estimated by using the Barrett-Joyner-Halenda (BJH) method.

The total volume $V_t(Hg)$ and macropore volume $V_{mac}(Hg)$ were assessed using a mercury porosimeter (Micromeritics Autopore III) with the software Win9400 Series V200. The macropore volume was regarded as being equal to the cumulative pore volume of the pore diameter greater than 50 nm. The combination of N_2 adsorption and mercury intrusion data enables to estimate the complete pore volume distribution of the samples.

3.3.7. Scanning Electron Microscopy

Surface morphologies were visualized by scanning electron microscopy (Hitachi/S-3500N) at accelerating voltages from 0.3 to 20 kV. Samples for cross-sectional images were prepared by placing them into epoxy resins and polishing for high image quality. Before visualization, the samples were coated with platinum by an ion sputter (Hitachi/E-1030 Ion sputter) to provide electrical conduction.

3.3.8. Water Adsorption Capacity

Water adsorption capacity of biochar sample was determined gravimetrically by adsorbing saturated water vapor at 25 °C in desiccators until a constant weigh was obtained. The adsorption-desorption isotherms of water vapor were determined at 25 ^{o}C by using Belsorp 28SA (Bel Japan, Inc.) instrument.

3.4. Gas and oil analysis

3.4.1. Gas analysis

The by-product (gas and oil) from pyrolysis of biomass were collected at 450 ^{o}C for the sample at a heating rate of 50 ^{o}C/min. Gas samples were collected upon reaching the temperature at 200 ^{o}C in plastic bags. The product gas composition was determined by using whole the gas sample collected for 20 min. in a typical pyrolysis experiment. The chemical composition of the gaseous products was analyzed by gas chromatograph. H_2, CO, CO_2, CH_4 and C_2H_4 were determined by using GC with TCD detector and Ar and He as a carrier gas. In addition, GC-FID was used for C_2H_6, C_3, C_4 and C_5 analyses.

3.4.2. Oil analysis

The oil from RH and RS pyrolyzed at 450 ^{o}C and with a heating rate of 50 ^{o}C/min were extracted by acetone and subsequently analyzed by a GC-Mass spectrometer Shimadzu GCMS-QP2010. The analyses were carried out by using a 30 m (L)× 0.32 mm (i.d.) capillary column coated with a 0.25 μm film thickness under helium (99.999%) carrier. The GC oven temperature programme was as follows: held at 60 ^{o}C for 5 min., heated from 60 to 240 ^{o}C with a heating rate of 10 ^{o}C/min and finally held for 50 min. at 240 ^{o}C. All injections (0.5 μL) were performed by a universal injector in the splitless mode by using a 10 μL gas tight syringe. GC-MS spectrum was identified based on the NIST mass spectra library.

RESULTS AND DISCUSSION

4.1. Feedstock characteristics

According to the proximate and ultimate analyses, the composition of the rice husk was found as: 14% moisture, 15% ash content, 41%, 4.76%, 0.34%, 38.9 % of C, H, N, O content, respectively. Rice straw characteristics were 9% of moisture, 10% of ash content, 42.27%, 5.03%, 0.71%, 42.99% of C, H, N, O content, respectively.

4.2. Biochar characterization

4.2.1. The yield of biochar

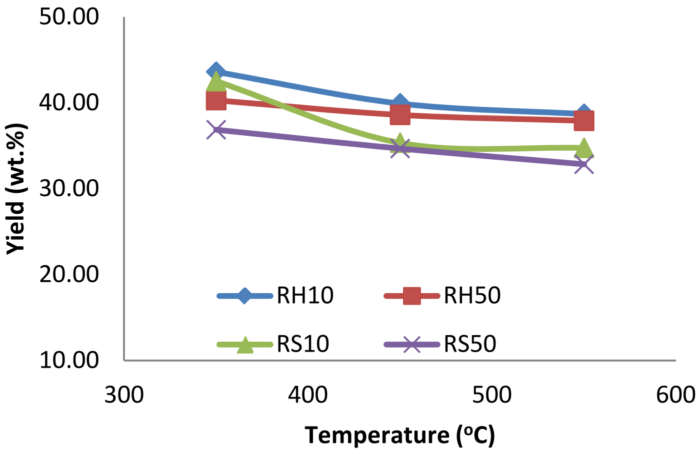


Figure 1. Effect of pyrolysis temperature and heating rate on rice husk and rice straw biochar yields.

As can be seen in Fig. 1, char yields were depended on both pyrolysis temperature and heating rate. The yield decreased with increasing charring temperature (350-550 ^{o}C) and heating rates (10-50 ^{o}C/min), ranging from 44 to 38% with RH chars and from 43 to 34% for RS chars, respectively. The results are in good agreement with earlier studies, Li et al. (2008), Peng et al. (2011), Bruun et al. (2011), Ahmad et al. (2012) that investigated the influence of temperature on the properties of coconut shell, rice straw, wheat straw, soybean stover and peanut shell biochar; and Fu et al. (2012), Angın (2013) that reported the influence of heating rate on the characteristics of rice straw and safflower seed press cake biochar. The decrease in char yield could be due to the thermal decomposition of organic materials in biomass into the bio-oil and gas fractions (Maiti et al., 2006). In addition, high heating rate tends to reduce the secondary reaction of char residues and the further degradation of the earlier formed products that leads to a decrease in the yields of biochar (Rei et al., 1986; Scott et al., 1988). According to the recent study (Angın, 2013), the effect of pyrolysis heating rate becomes more important at low temperatures and our results are in agreement with this study.

4.2.2. Proximate analysis

Table 1. Properties of biochar

Samples	Proximate analysis (wt.%)			Ultimate analysis (wt.% db)				Ratios			pH	CEC (cmol.kg^{-1})
	VM	Ash	FC	C	H	N	O	H/C	O/C	C/N		
RH350-10	24.65	35.34	40.01	47.83	2.84	0.29	13.70	0.059	0.29	164.93	9.86	45.63
RH450-10	19.48	39.44	41.08	48.69	2.19	0.33	9.35	0.045	0.19	147.55	9.90	41.92
RH550-10	14.16	40.51	45.33	51.15	1.64	0.34	6.36	0.032	0.12	150.44	10.36	34.58
RH350-50	20.72	38.58	40.70	46.02	2.41	0.71	12.28	0.052	0.27	64.82	9.92	45.58
RH450-50	17.72	38.95	43.33	46.23	1.87	0.5	12.45	0.040	0.27	92.46	9.53	39.79
RH550-50	14.29	41.15	44.56	50.89	1.64	0.36	5.96	0.032	0.12	141.36	10.06	35.68
RS350-10	41.79	23.39	34.82	52.52	3.01	1.2	19.88	0.057	0.38	43.77	10.47	68.64
RS450-10	32.15	30.84	37.01	52.1	2.25	0.96	13.85	0.043	0.27	54.27	10.42	56.00
RS550-10	25.69	33.47	40.84	58.01	1.84	0.74	5.94	0.032	0.10	78.39	11.68	49.95
RS350-50	34.81	26.85	38.34	52.2	2.83	1.06	17.06	0.054	0.33	49.25	10.42	62.00
RS450-50	30.54	30.59	38.87	50.07	2.05	1.16	16.13	0.041	0.32	43.16	10.90	53.69
RS550-50	26.76	32.83	40.41	62.97	2.1	1.12	0.98	0.033	0.02	56.22	11.70	45.56

The results of proximate analyses, ultimate analyses, pH measurements and cation exchange capacity (CEC) of biochar obtained at different pyrolysis temperatures and heating rates are given in table 1. The results of the proximate analysis indicated that the ash content of the biochar increased with raising charring temperature, and also with heating rate at 350 ^{o}C. The ash content of RH biochar increased from 35.34 to 40.51% for the heating rate of 10 ^{o}C/min, and increased from 38.58% to 41.15% for the heating rate of 50 ^{o}C/min as the pyrolysis temperature rose from 350 to 550 ^{o}C. The ash content of RS biochar at a heating rate of 10 and 50 ^{o}C/min were 23.39% to 33.47% and 26.85% to 32.83%, respectively. In line with Peng et al. (2011), Maiti et al. (2006) and Wu et al. (2012), the ash contents of rice husk and rice straw biochar were quite higher and it increased with increasing pyrolysis temperature. The ash contents of RH and RS biochar were much higher than that derived from grass (<20%) and woody feedstock (typically lies between 2 and 8%) (Keiluweit et al., 2010). High ash content of RH and RS biochar can attribute to high Si and alkali content of the RH and RS compared to wood or grass (Raveendran et al., 1995).

Volatile matter (VM) and fixed carbon (FC) contents give relative measures of the labile and stable components of char, respectively (Keiluweit et al., 2010). As the pyrolysis temperature increased from 350 to 550 ^{o}C, volatile matter contents of RH and RS biochar decreased from 24.65 to 14.16% and 41.79 to 25.69%, respectively; while the decrease with heating rate was clear at low temperatures. On the other hand, the fixed carbon content of the biochar increased with raising temperatures from 40.01 to 45.33% and 30.82 to 40.84%, for RH and RS biochar respectively. The highest fixed carbon contents were achieved at 550 ^{o}C in this study.

4.2.3. Ultimate analysis

The results of the elemental analysis of biochar indicated that the temperature had a significant effect on the elemental composition of the biochar than the heating rate. Carbon (C) content generally increased, while hydrogen (H) and oxygen (O) content decreased with increasing temperatures. As the temperature increased, H/C and O/C atomic ratios gradually decreased, indicating that the biochar became increasingly more aromatic and carbonaceous (Argin, 2013).

Nitrogen (N) content of biochar strongly depends on starting material. According to former studies N content was concentrated in rice straw biochar as charring temperature from 250-450 ^{o}C (Peng et al., 2011). However, it decreased with soybeen stover, peanut shells biochar produced at 700

^{o}C than those produced at 300 ^{o}C (Ahmad et al., 2012) and rubber-wood-sawdust biochar as increasing temperature from 450-850 ^{o}C (Ghani et al., 2013). On the other hand it remained rather constant with wheat straw biochar with raising temperature from 475-575 ^{o}C (Bruun et al., 2011). According to our experimental results N content could be constant or decreased depending on the pyrolysis conditions (temperature and heating rate). The maximum N content of RH char was obtained at 350 ^{o}C and with a heating rate of 50 ^{o}C/min, and at 350 ^{o}C and with a heating rate of 10 ^{o}C/min for RS char. N content in RS char was higher than in RH char, this result was corresponded with N content in the feedstocks.

4.2.4. pH and CEC

The results of pH measurement showed that a slight increase in pH value occurred when the temperature increased from 350 to 550 ^{o}C, that can be explained due to a separation of organic (carbon) and inorganic components (alkali metal salts) in the husk. According to Abe (1988); Shinogi and Kanri (2003), at higher temperature more than 350 ^{o}C, the alkali salts began to separate from the organic matrix and reasonably the pH was increased. Finally, the pH became constant at around 600 ^{o}C when all the alkali salts were leached from the pyrolysis structure. Li et al. (2002) reported that the reduction of O at high temperature resulted the removal of various acidic functional groups, which cause the surfaces of the biochar to become more basic. It is reported that wood charcoal produced at a high pyrolysis temperature is alkaline and it can consume protons (H^+) from the soil, which enables the charcoal to improve soil acidity (Abe, 1988).

Cation exchange capacity (CEC), a measure of the negative charge of a material that can be neutralized by exchangeable cations, enhances soil's ability to hold and exchange nutrients such as ammonium, calcium and potassium (Brady and Weil, 1984). CEC of biochar at pH=7 decreased with the increasing temperature due to the decrease of volatile organic material which carries the majority of cation exchange capacity on biochar surfaces (Mukherjee, 2011). The average CECs of RH and RS biochar tested in this study (40 and 56 cmol.kg^{-1}, respectively) are at least 2 times larger than that of most soils (3-20 cmol.kg^{-1}, except Histosols) (Brady and Weil, 1984). The high CEC of biochar leads its potential in applying into soil to enhance soil fertility.

4.2.5. Effects of temperature and heating rates on micropore and surface area of biochar

The results in table 2 showed that the BET surface area, total pore volume and micropore volume from N_2 adsorption of the char increased, while the average diameter decreased with increasing pyrolysis temperatures and heating rates, indicating that the higher temperature and heating rate enhanced the pore structure of the char. However, the effect of temperature on pore structure is more dominant than the heating rate. With increasing charring temperature, devolatilization of bigger molecules occurred and size of the volatile molecules decreased gradually, while the release of smaller molecules would enhance the micropores in the sample giving rise to increase in the BET surface area. Those results were in accordance with Li et al. (2008) who reported the micropore volume increased with the increase of temperature; however, micropore volume decreased above 500 ^{o}C for safflower seed press cake char (Angın, 2013) and eucalyptus char (Pastor-Villegas et al., 2007); at 600 ^{o}C and heating rate of 10 ^{o}C/min for oil-palm-shell char (Lua et al., 2010); and at 900 ^{o}C and heating rate of 300 ^{o}C/s for rice straw char due to the collapse of the micropore structures and the sintering effect (Fu et al., 2012). Thus, the micropore development depends not only on the pyrolysis parameters (temperature and heating rate) but also starting biomass. Fig. 2 showed the micropore and mesopore distribution curves of the RH and RS char. It was observed that the micropores sharply range from 0.7 to 1.5 nm and mesopores at 4 nm.

Table 2. Porous properties of the char prepared at different pyrolysis temperature and heating rates.

Samples	From nitrogen adsorption					From Hg porosimetry	
	S_{BET} (m^2/g)	V_t (N_2) (cm^3/g)	V_{micro} (N_2) (cm^3/g)	V_{meso}(N_2) (cm^3/g)	D (nm)	V_t(Hg) (ml/g)	V_{macro}(Hg) (ml/g)
RH350-10	33.5	0.035	0.016	0.019	4.173	1.4376	1.2343
RH450-10	58.1	0.057	0.027	0.030	3.924	2.4163	2.1541
RH550-10	216.2	0.139	0.110	0.029	2.572	1.5627	1.4351
RH350-50	55.6	0.054	0.027	0.027	3.881	3.3064	3.1121
RH450-50	112.1	0.086	0.053	0.033	3.068	3.8209	3.5983
RH550-50	232.7	0.151	0.116	0.035	2.595	2.7748	2.5299
RS350-10	6.7	0.011	-	-	6.591	5.4123	5.1445
RS450-10	17.0	0.023	0.008	0.015	5.411	5.3991	5.1657
RS550-10	68.2	0.045	0.034	0.011	2.640	5.6424	5.3662
RS350-50	13.2	0.021	0.006	0.015	6.342	6.1244	5.8611
RS450-50	50.7	0.048	0.024	0.024	3.786	5.6814	5.4301
RS550-50	84.6	0.059	0.043	0.016	2.789	5.3783	5.0255

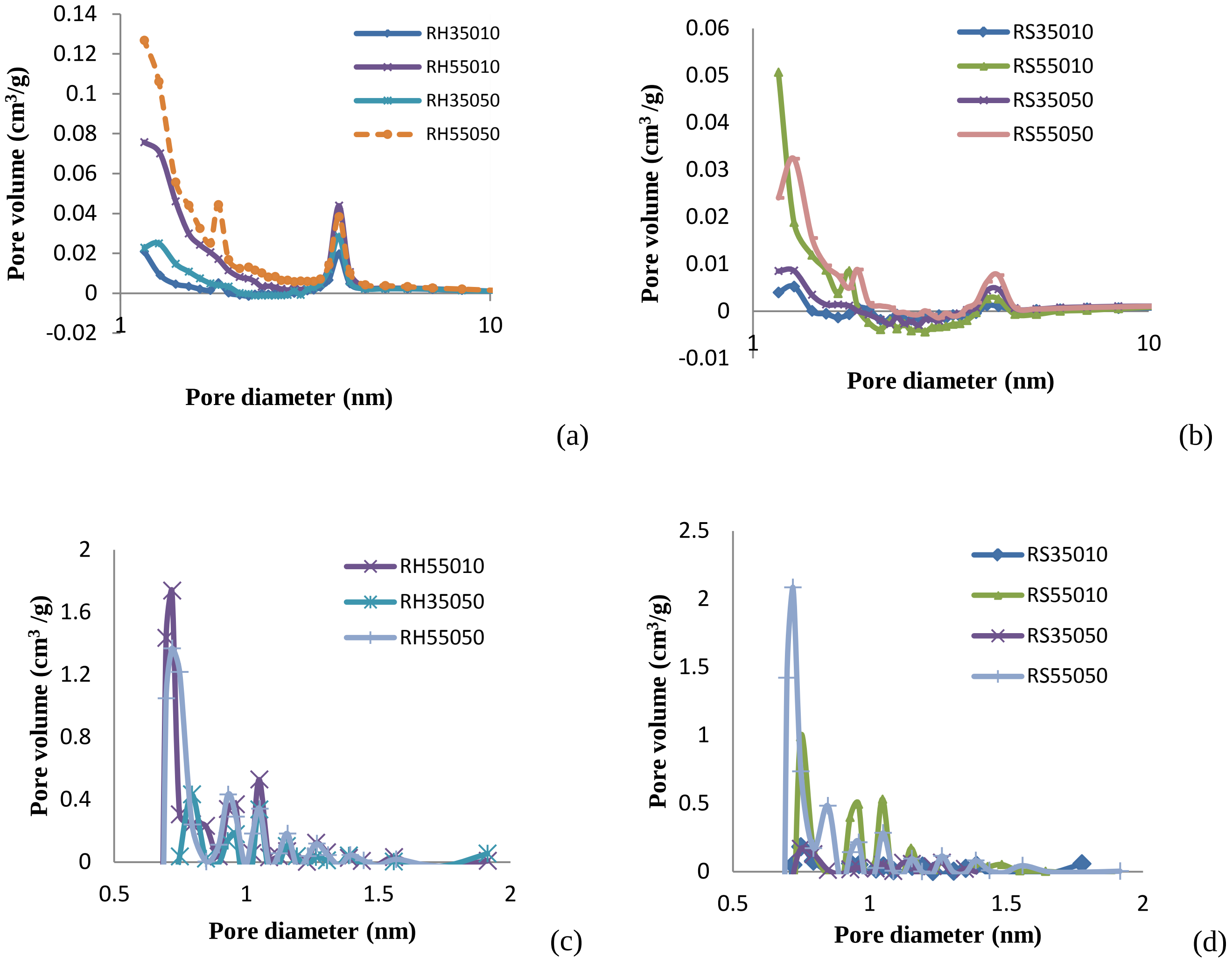
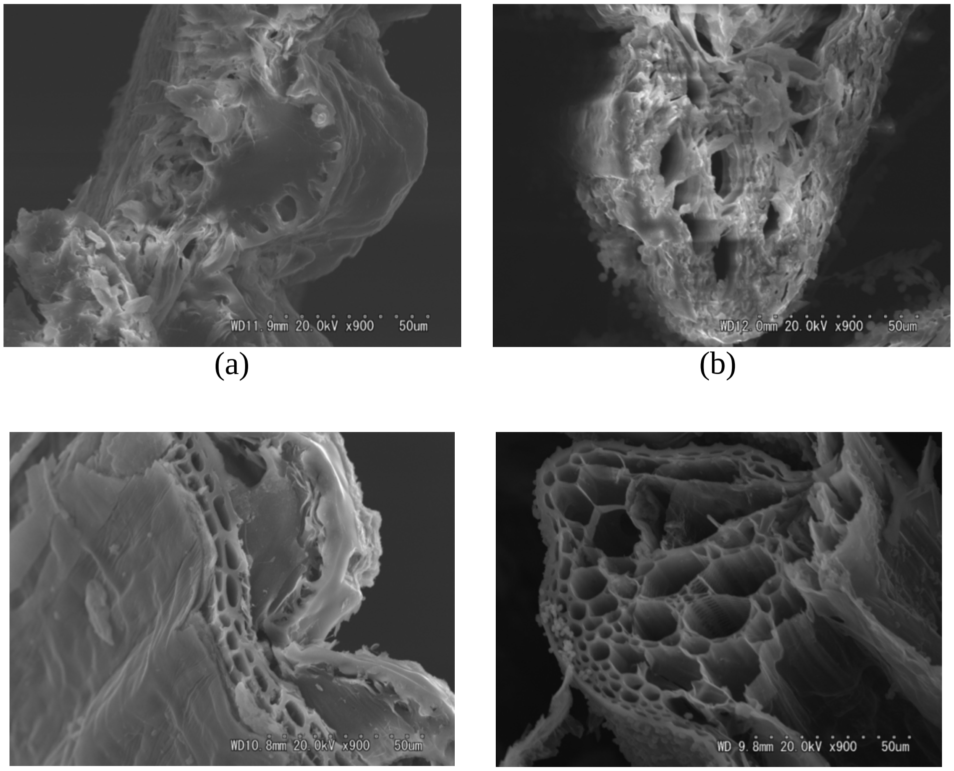


Figure 2. Meso and micro pore size distribution obtained by using the Barrett-Joyner-Halenda (BJH) method at different pyrolysis temperatures and heating rates for RH char (a and c) and RS char (b and d), respectively.

4.2.6. Effects of temperature and heating rates on macropore of biochar

In the past, when biochar and activated carbon were assessed mainly for their role as adsorbents, macropores were considered to be only important as a passage for the adsorbate to the meso- and micro-pores (Wildman and Derbyshire, 1991). However, macropores are very relevant to vital soil functions such as aeration, hydrology (Troeh and Thompson, 2005) and microorganism’s habitat (Lehmann et al., 2009), therefore we determined the biochar`s macropore volume by using Hg porosimeter. The results (Table 2) showed that RH and RS biochar contain high proportions of macropore occupied more than 90% of total pore volume (including micropore and mesopore volume). Macropore volumes of RS char were about 2 times greater than the RH char. The maximum value of macropore volumes RH char occurred at 450 ^{o}C (Fig. 4). For RS char, there was almost negligible change in macropore volume for heating rate of 10 ^{o}C/min, however, a significant decrease was occurred for heating rate of 50 ^{o}C upon increasing temperatures.

SEM images of materials before and after pyrolysis (Fig. 3) indicated that the volatile compounds released during the pyrolysis process, created pores in the pyrolysis product. The SEM image supported the results of higher macropore volume in RS chars than the RH char.



(a) (b)

(c) (d)

Figure 3. Scanning electron micrograph (SEM) of (a) rice husk, (b) rice straw (c) rice husk biochar and (d) rice straw biochar.

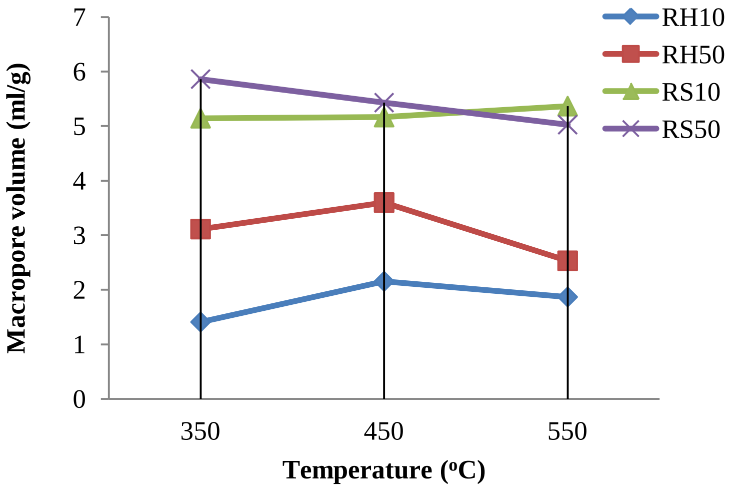


Figure 4. Macropore volume of RH and RS char obtained at different temperatures and heating rates determined by Hg porosimeter

4.2.7. Effects of temperature and heating rates on water adsorption capacity

Table 3 showed water adsorption capacity of RH and RS char prepared under various pyrolysis conditions. Water adsorption capacity of RH and RS char were obtained from the incubator had maximum amount at 48 hours and 96 hours, respectively. Although, RH char had higher micropore and mesopore volume than RS char about 3 times, however water adsorption of RS char was greater approximate 6 times than RH char. These results indicated that the macropore volume of char may have a significant effect on the water adsorption of the biochar in our study, because of the two main reasons: Firstly, macropore volume in biochar accounted more than 90% of total pore volume of biochars; Secondly, macropore volume in RS chars was about 2 times higher than the RH char. However, the adsorbed water only occupied about 8 and 20% of total macropore volume in RH char and RS char, respectively.

Table 3. Water adsorption capacity determined by adsorbing saturated water vapor at 25°C in a desiccator inside an incubator (g H_2O/g char)

Sample	Charring temperature (°C)		
	350	450	550
RH10	0.147	0.12	0.247
RH50	0.171	0.206	0.219
RS10	0.815	1.138	1.332
RS50	1.034	0.827	1.284

Adsorption-desorption isotherms of water vapors (Fig. 5) supported the results of water adsorption from the incubator that water adsorption of RH chars had negligible change while those of RS char fluctuated with the increase of pyrolysis temperatures and heating rates.

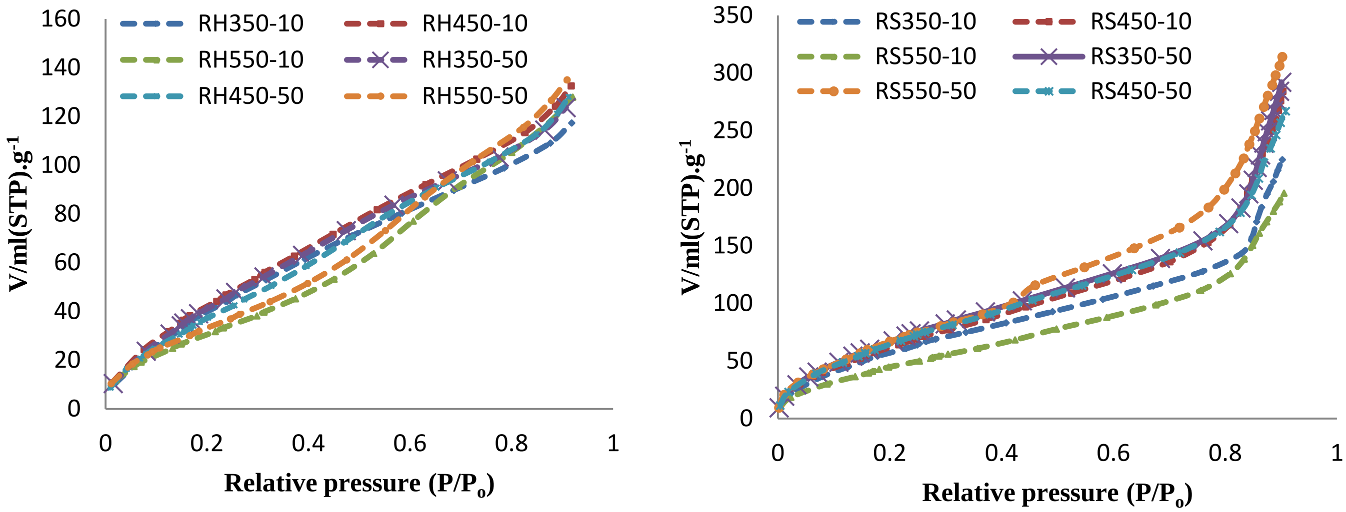


Figure 5. H_2O vapor adsorption-desorption isotherms (at 25 ^{o}C) for rice husk and rice straw char.

4.3. Composition of the volatile matters (gas and oil)

4.3.1. Gas analysis

Table 4. Composition of the gaseous products determined by GC (cc/1 gram of dried biomass)

Sample	CO_2	CO	CH_4	H_2	C_2H_4	C_2H_6	C_3	C_4	C_5
RH450-50	46.41	36.64	10.11	2.85	0.86	1.45	0.75	1.95	0.54
RS450-50	63.34	37.48	10.54	4.62	0.95	1.55	0.53	0.05	0.02

The gas compositions were analyzed by gas chromatograph and the results were calculated by volumes based on 1 gram of dried biomass. The detected gas components included H_2, CH_4, CO, CO_2, C_2H_4, C_2H_6, C_3, C_4 and C_5. Table 4 showed that main compositions of gaseous product were CO_2, CO, CH_4 and H_2 which accounted about 94 and 97% of total gas volume evolved during pyrolysis of RH450-50 and RS450-50, respectively. As carbon content in gaseous product was determined completely, therefore we calculated the proportion of carbon in the feedstock distributed in gas, char and oil as shown in figure 6. It was found that carbon distribution of RH and RS products were quite similar in which majority of carbon distributed in char and oil (about 85%).

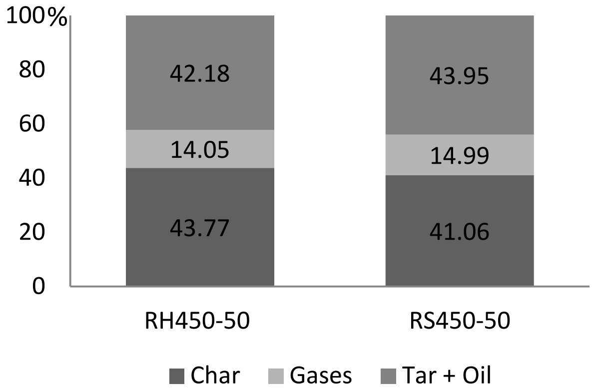


Figure 6. Carbon distribution of pyrolysis products.

4.3.2. Oil analysis

Identification of the GC/MS peaks was based on comparison with the NIST 147 spectrum library. The separation of all the peaks was not possible due to the complex composition of the pyrolysis vapours. Only those separated products were quantitatively evaluated which arose in considerable amounts (Table 5). Bio-oils from rice husk and rice straw are composed of complex mixtures of phenols, benzenediols, furans, ketones, alkenes, aldehydes, esters, alcohols, acids and various aromatics.

Table 5. Chemical composition of the rice husk oil detected by GC-MS

Compound	***%***	***Formula***
Phenol	***32.85***	
Phenol	1.96	C_6H_6O
Phenol, 2-methoxy-	3.3	$C_7H_8O_2$
Phenol, 2-methoxy-3-(2-propenyl)-	0.68	$C_{12}H_{14}O_3$
Phenol, 2-methoxy-4-(2-propenyl)-, acetate	3.47	$C_{12}H_{14}O_3$
Phenol, 2-methoxy-4-propyl-	0.84	$C_{10}H_{14}O_2$
Phenol, 2-methoxy-4-methyl-	0.18	$C_8H_{10}O_2$
Phenol, 2-methoxy-4-methyl-	1.72	$C_8H_{10}O_2$
Phenol, 2-methyl-	1.71	C_7H_8O
Phenol, 2,3-dimethyl-	0.21	$C_8H_{10}O$
Phenol, 2,4-dimethyl-	0.81	$C_8H_{10}O$
Phenol, 2,6-dimethoxy-	2.54	$C_8H_{10}O_3$
Phenol, 2,6-dimethoxy-4-(2-propenyl)-	0.65	$C_{11}H_{14}O_3$
Phenol, 2,3,6-trimethyl-	0.35	$C_9H_{12}O$
Phenol, 3-ethyl-	0.3	$C_8H_{10}O$
Phenol, 3,4-dimethyl-	0.71	$C_8H_{10}O$

Table 5. *(Continued)*

Compound	***%***	***Formula***
Phenol, 3,5-dimethyl-	0.45	$C_8H_{10}O$
Phenol, 4-ethyl-	2.36	$C_8H_{10}O$
Phenol, 4-ethyl-2-methoxy-	2.16	$C_9H_{12}O_2$
Phenol, 4-methyl-	3.53	C_7H_8O
Phenol, 4-methoxy-3-(methoxymethyl)-	0.88	$C_9H_{12}O_3$
Phenol, 4-(1-methylethyl)-	0.62	$C_9H_{12}O$
2-Methoxy-4-vinylphenol	3.42	$C_9H_{10}O_2$
Benzenediol	***14.06***	
Benzenediol, 3-methyl-	2.36	$C_7H_8O_2$
Benzenediol, 4-methyl-	2.32	$C_7H_8O_2$
1,2-Benzenediol	6.14	$C_6H_6O_2$
1,2-Benzenediol, 3-methoxy-	0.92	$C_7H_8O_3$
1,3-Benzenediol, 4-ethyl-	1.53	$C_8H_{10}O_2$
1,4-Bezenediol, 2-methyl-	0.79	$C_7H_8O_2$
Furan	***8.29***	
Benzofuran, 2,3-dihydro	7.13	C_8H_8O
Furan, 2-butyltetrahydro-	0.51	$C_8H_{16}O$
5-Hydroxymethyldihydrofuran-2-one	0.65	$C_5H_8O_3$
Ketone	***6.84***	
Bicyclo[3.3.1]non-3-ene-2,6-dione	0.22	$C_9H_{10}O_2$
Hydroquinone	1.66	$C_6H_6O_2$
1,2-Cyclopentanedione	0.53	$C_5H_6O_2$
1,2-Cyclopentanedione, 3-methyl-	1.12	$C_6H_8O_2$
1,4:3,6-Dianhydro-.alpha.-d-glucopyranose	0.5	$C_6H_8O_4$
2-Cyclopenten-1-one, 2,3-dimethyl-	0.15	$C_7H_{10}O$
2-Cyclopenten-1-one,3-ethyl-2-hydroxy-	0.56	$C_7H_{10}O_2$
2-Propanone,1-(4-hydroxy-3-methoxyphenyl)-	1.27	$C_{10}H_{12}O_3$
2,3-Dimethylhydroquinone	0.43	$C_8H_{10}O_2$
5-Hydroxy-1-tetralone	0.4	$C_{10}H_{10}O_2$
Alkane	***1.75***	
Bicyclo[2.2.1]heptane-2,3-diol,1,7,7-trimethyl-,(2-3xo,3-endo)-	0.54	$C_{10}H_{18}O_2$
Trans-1,2-Diethoxycyclohexane	0.26	$C_{10}H_{20}O_2$
1,4-Dimethoxy-2-phenylbutane	0.59	$C_{12}H_{18}O_2$
1,5-Hexadiene-3,4-diol, 2,5-dimethyl-	0.36	$C_8H_{14}O_2$
Aldehyde	***2.21***	
Benzaldehyde, 3-hydroxy-	0.37	$C_7H_6O_2$
2-[2-(4-Nitro-phenoxy)-ethoxy]-benzaldehyde	0.74	$C_{15}H_{13}NO_5$
4-Hydroxy-2-methoxycinnamaldehyde	0.67	$C_{10}H_{10}O_3$
5-Isopropenyl-2-methylcyclopent-1-enecarboxaldehyde	0.43	$C_{10}H_{14}O$

Table 5. *(Continued)*

Compound	***%***	***Formula***
Ester	***2.28***	
Acetic acid, (2-isopropenylcyclopentylidene)-, methyl ester	0.91	$C_{11}H_{16}O_2$
Acetic acid, 7-hydroxy-1,3,4,5,6,7-hexahydro-2H-naphthalen-4a-ylmethyl ester	0.29	$C_{13}H_{20}O_3$
Benzenepropanoic acid, 4-hydroxy-,methyl ester	0.53	$C_{10}H_{12}O_3$
Chloroacetic acid, 2-tetradecyl ester	0.13	$C_{16}H_{31}ClO_2$
2-Propenoic acid, 3-(4-hydroxyphenyl)-, methyl ester	0.42	$C_{10}H_{10}O_3$
Alcohol	***0.69***	
Cyclohexanol, 2-(1-methylethyl)-	0.14	$C_9H_{18}O$
2-methoxybenzyl alcohol	0.43	$C_8H_{10}O_2$
5-Hepten-3-yn-2ol,6-methyl-5-(1-methylethyl)-	0.12	$C_{11}H_{18}O$
Aromatics	***3.02***	
Adenosine, N(6)-4-methoxybenzyl-4`-N-ethylcarbamoyl-4`-dehydroxymethyl-	0.5	$C_{20}H_{24}N_6O_5$
Pseduosarsasapogenin-5,20-dien	0.29	$C_{27}H_{42}O_3$
Vanillin	0.93	$C_8H_8O_3$
1H-Indene-1,5 (6H)-dione,2,3,7,7a-tetrahydro-7a-methyl-	0.74	$C_{10}H_{12}O_2$
1,4-Dihydrothujopsene-(I1)	0.56	$C_{15}H_{26}$
Benzen	***0.45***	
Benzene, 1,2,3-trimethoxy-5-methyl-	0.45	$C_{10}H_{14}O_3$

CONCLUSIONS

In this study, the effect of pyrolysis temperature (400, 450 and 550 °C) and the heating rate (10 and 50 °C/min) on the yield and properties of rice husk and rice straw char were investigated. The results indicated that the chemical, physical, morphological and textural properties of biochar are influenced by charring temperature and heating rate as well. However, the temperature had significant effect on biochar than the heating rate. Increasing temperature and heating rate decreases the biochar yield and volatile content, but increases ash content and fixed carbon. Increasing charring temperature reduces O, H content but concentrates aromatic C. The results of pH and CEC of the biochar indicated that these biochar have potential for application into soil to improve soil acidity or enhance soil fertility. In addition, high porosity of biochar leads to potential in the increase of water adsorption capacity for soil. The results of by-product (gas and oil) obtained at temperature of 450 ^{o}C and heating rate of 50 ^{o}C/min showed that main components of pyrolysis gas detected were CO_2, CO, CH_4 and H_2. Bio-oils from RH and RS were composed of a complex mixture of aromatic hydrocarbons.

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Preparation and Characterization of Biochar from Agricultural Residues

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Aim and outcome of this project

■ This project is aimed at the development of methods to prepare biochar.

■ It is part of a broader project on the development of technologies for the utilization of agricultural waste.

♦ The biochar was prepared by the pyrolysis of agricultural residues such as rice husk, rice straw, wheat straw, corn stover, and coconut husk at various temperatures and heating rates.

♦ The biochar prepared in this project demonstrated excellent properties, including high surface area, large pore volume, high cation exchange capacity, high pH, etc., which may qualify it to be a soil additive to enhance soil quality.
Biochar may also be suitable for carbon sequestration.

Outline of this presentation

1. Introduction
2. Materials and Methods
3. Results and Discussion
4. Conclusions

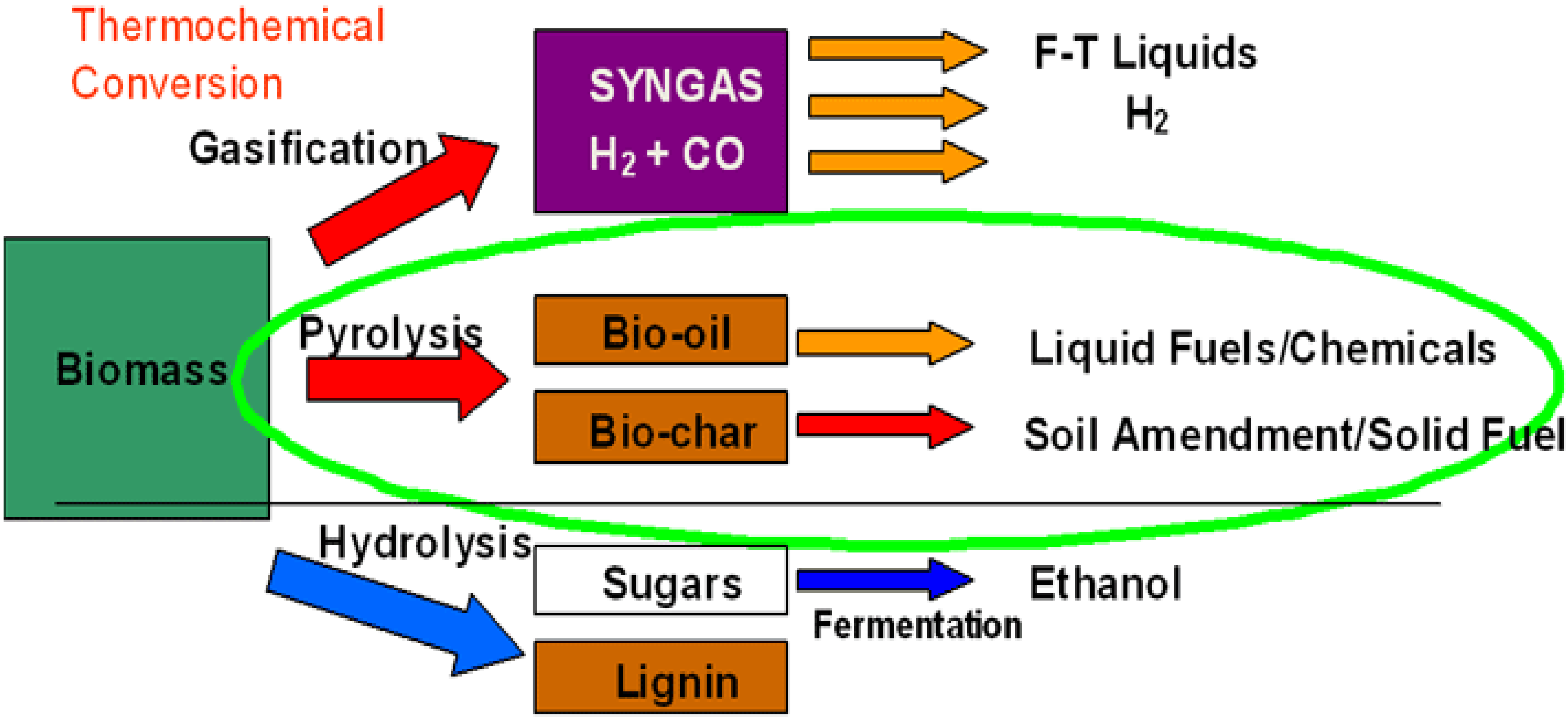
1. Introduction

Why biochar?

Biochar is a fine-grained charcoal high in organic carbon and largely resistant to decomposition. It is produced from pyrolysis of plant and waste feedstocks.

As a soil amendment, biochar creates a recalcitrant soil carbon pool that is carbon-negative, serving as a net withdrawal of atmospheric carbon dioxide stored in highly recalcitrant soil carbon stocks.

Thermochemical conversion of Biomass



What are the essential properties of biochar as soil amendment materials?

- Porosity
- Cation Exchange Capacity
- Water adsorption capacity
- pH (Alkalinity and Acidity)
- Stability (Thermal and Biochemical)
- Non-polluting (Leaching of harmful elements)

Our previous study

Study of preparation of biochar from wheat straw, corn stover and coconut husk

- Yield of wheat straw, corn stover and coconut husk biochar decreased with increasing pyrolysis temperature

- pH of the biochar was in the range of 8-11:

  Coconut husk char>Corn stover char> Wheat straw char

- Specific surface area of the biochar increased with the increase of pyrolysis temperature.

- Water adsorption capacity of various biochar was in the following order:

  Coconut husk char > Corn stover char >> Wheat straw char

- Phosphorous eluted from the biochar treated with water was in the following order:

  Corn stover char >> Wheat straw char >Coconut husk char

The objectives of this study

Preparation and characterization of biochar from rice husk and rice straw at different temperatures (350, 450 and 550^{o}C) and heating rates (10 and 50^{o} C/m).

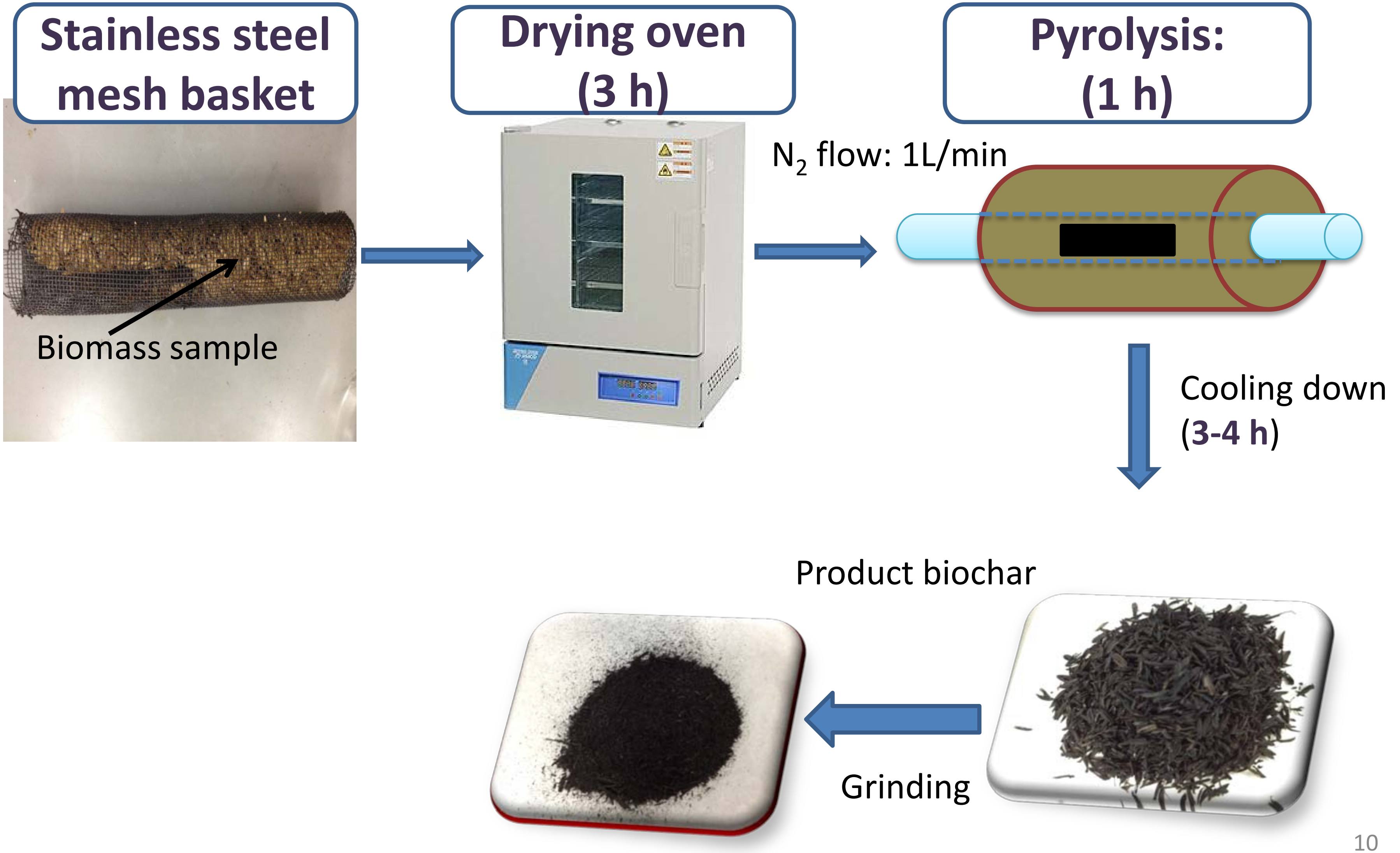
Determination of compositions of volatile matters (gas and oil).

Materials

Rice husk

Rice straw

Preparation of biochar



Stainless steel mesh basket

Biomass sample

Drying oven (3 h)

Pyrolysis: (1 h)

N_2 flow: 1L/min

Cooling down (**3-4 h**)

Product biochar

Grinding

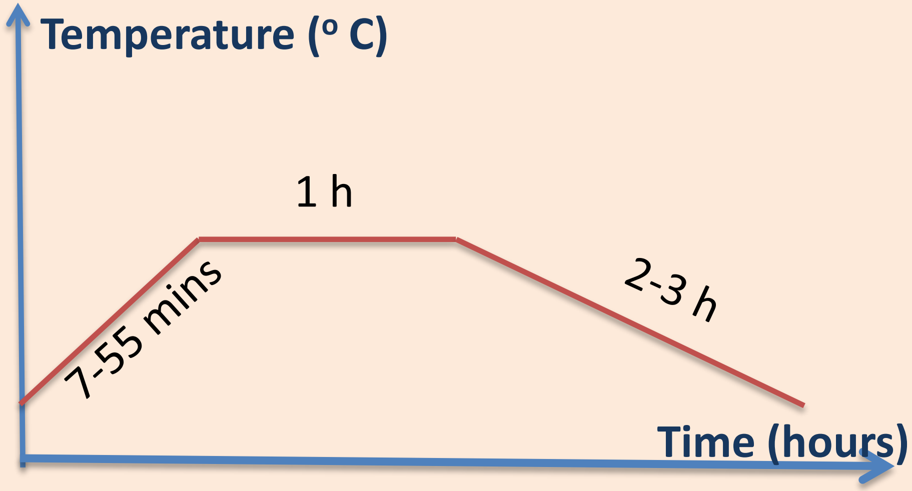
PYROLYSIS

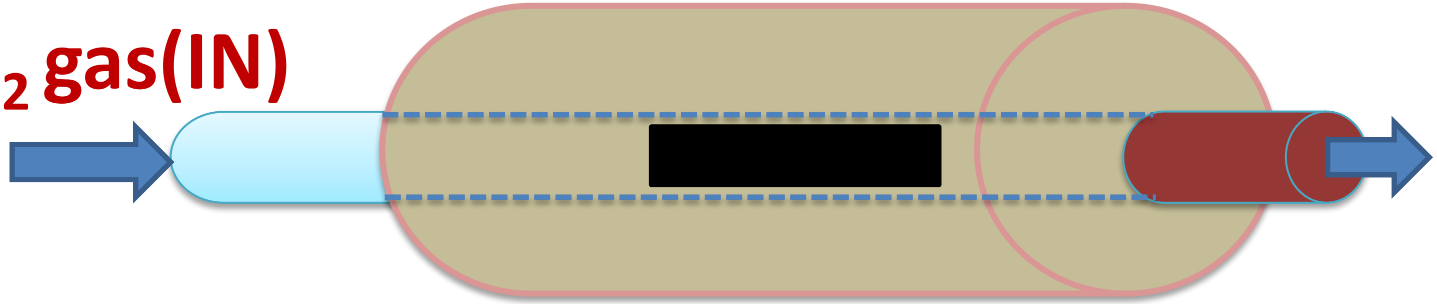
Parameters

- N_2 gas flow: 1l/m
- Residence time: 1h
- Heating rate: 10, 50°C/m
- Temperature (°C): 350 450 and 550

Processes

- The reactor was purged with N_2 gas flow for 1h.
- Then, the reactor was heated according to the following program.

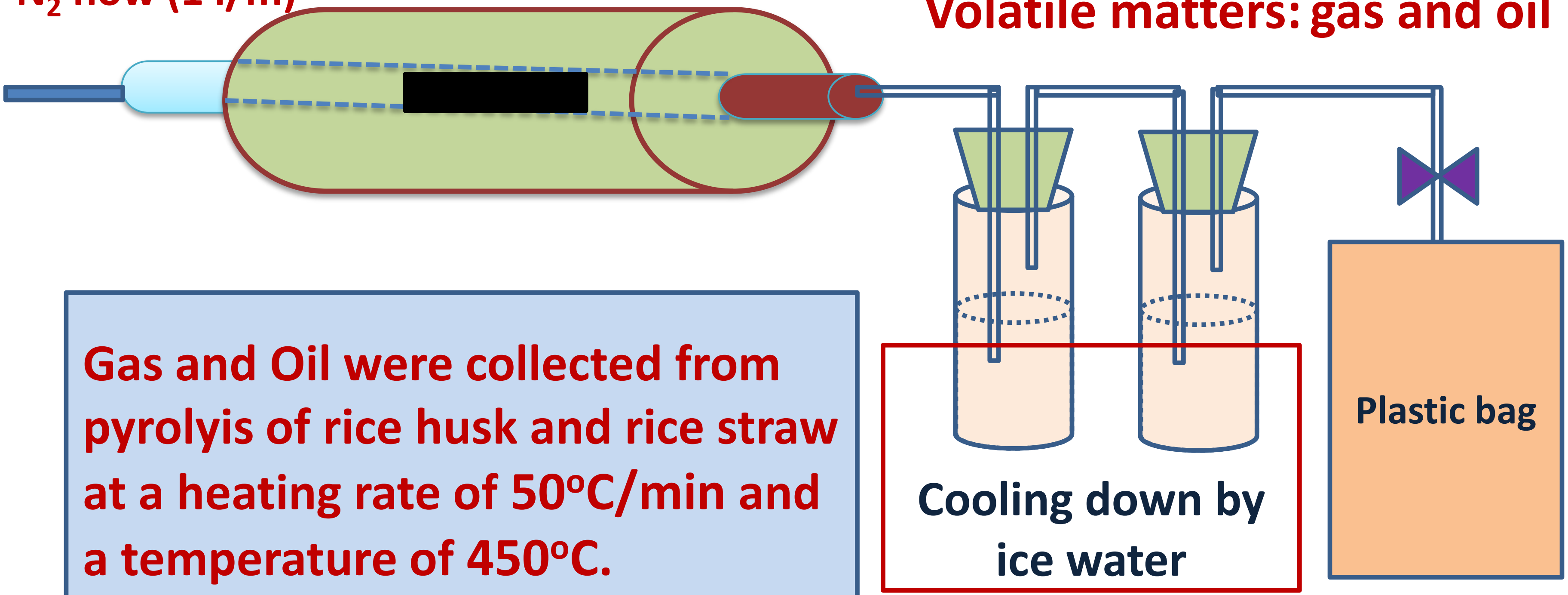




N_2 gas(IN)

Volatile matters: gas and oil(OUT)

PYROLYSIS



Gas and Oil were collected from pyrolyis of rice husk and rice straw at a heating rate of 50°C/min and a temperature of 450°C.

Collection of oil

Oil was extracted by solvents:

Acetone is the best solvent to extract pyrolysis oil.

2. Methods

Biochar obtained by pyrolysis was characterized:

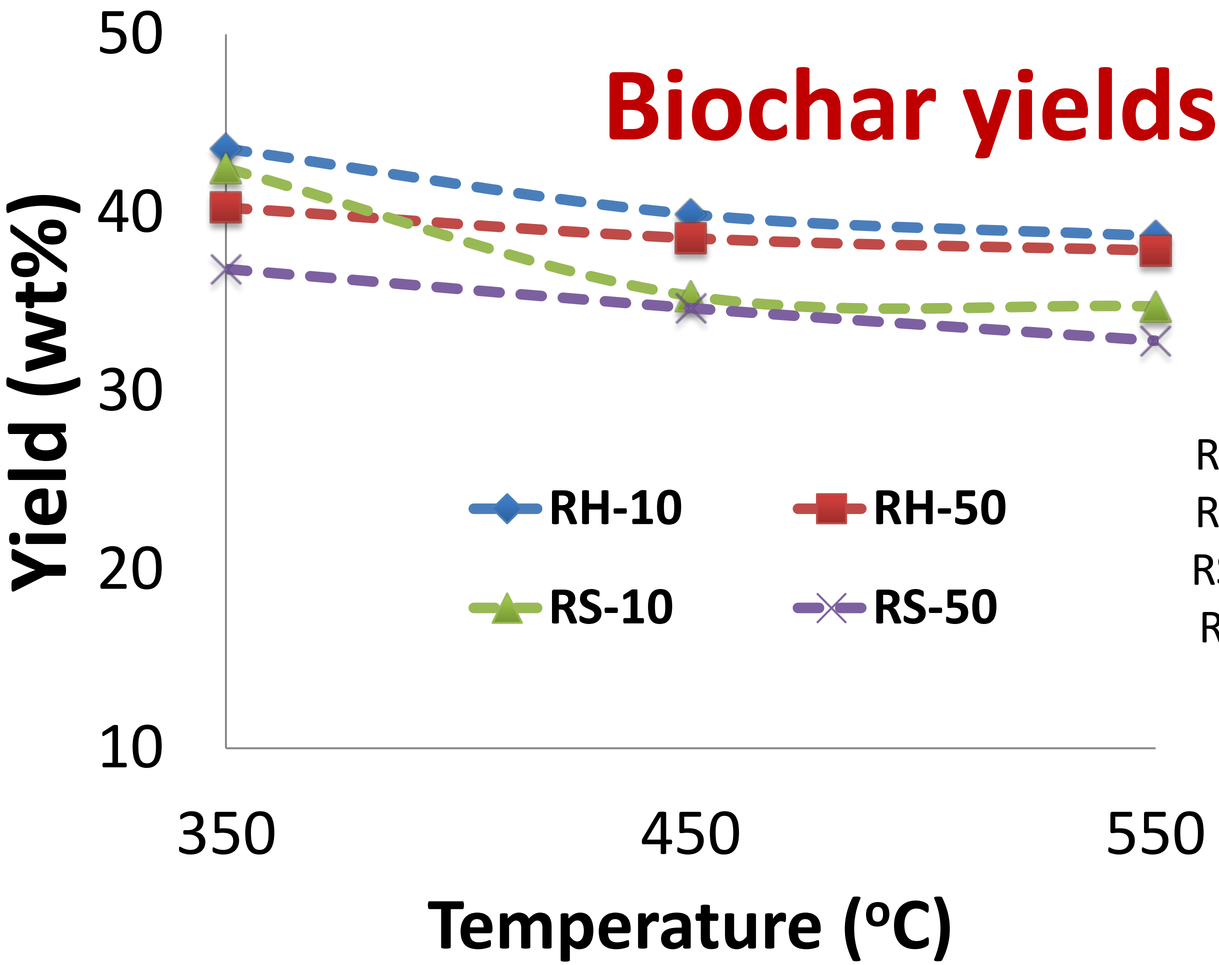
- ***Char yield***
- ***Proximate analysis:*** ash content, volatile matter content and fixed carbon
- ***Elemental anlysis***: C, H, O and N
- ***Porous and morphological characteristics*** were determined by N_2 adsorption analyzer, Hg porosimeter and scanning electron microscopy (SEM).
- ***Others***: Water adsorption capacity, pH and CEC
- **Chemical composition of pyrolysis gas and oil** were determined by gas chromatography technique with TCD, FID and MS detection systems.

3. Results and discussion

Table 1. Properties of the biomass

	Rice husk	Rice straw
Ash, wt% (on db)	15	10
Moisture, wt%	13	10
Elemental analysis, wt % (on db)		
C	41	42.27
H	4.76	5.03
N	0.34	0.71

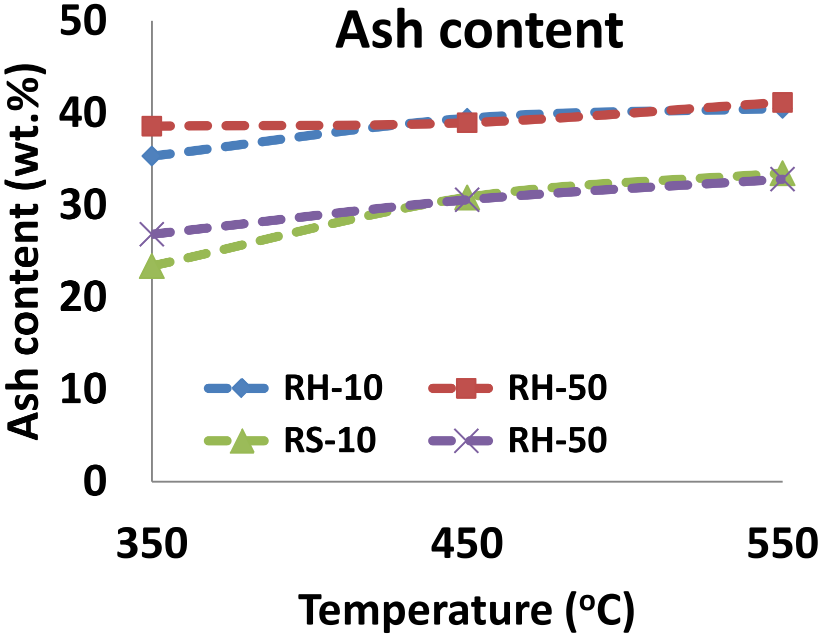
Biochar yields

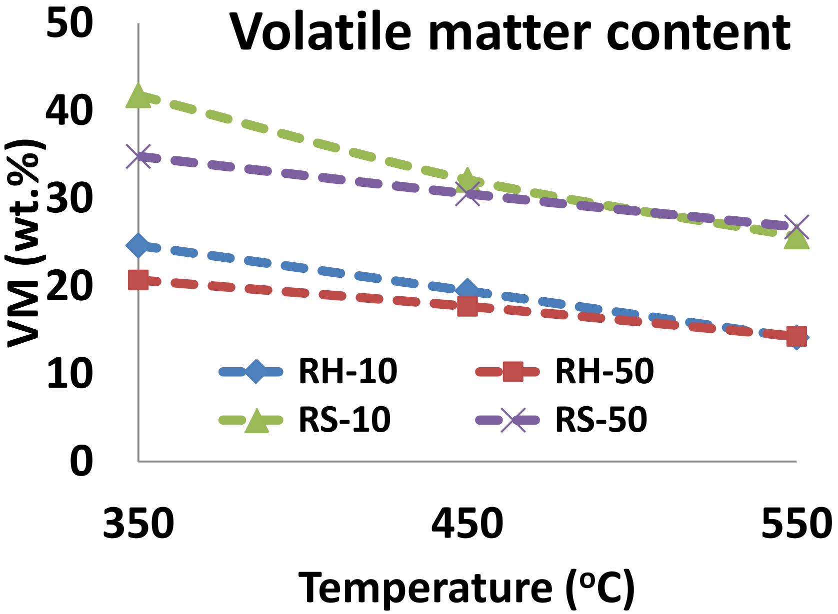


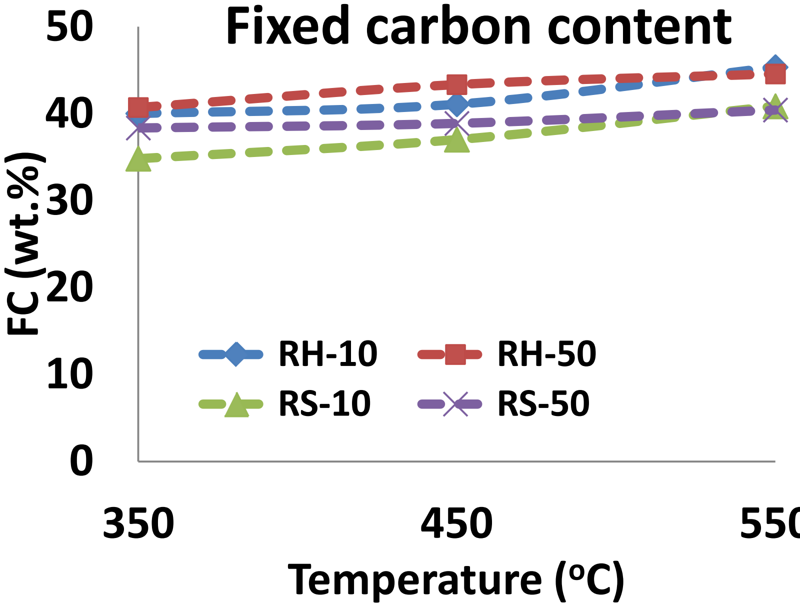
RH-10: rice husk at 10° C/min
RH-50: rice husk at 50° C/min
RS-10: rice straw at 10° C/min
RS-10: rice husk at 10° C/min

- **❑ Char yield ranged from 34-44%, decreased with increasing charring temperature and heating rates.**
- **❑ The effect of pyrolysis heating rate became more important at low temperatures.**

Proximate Analysis







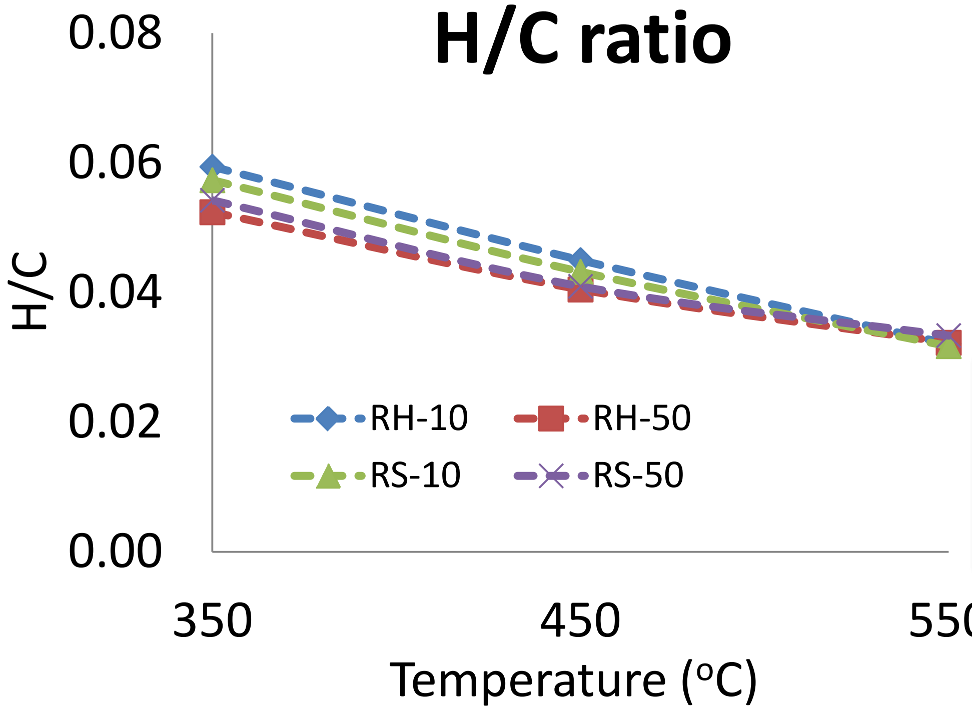
- ❖ Ash content and fixed carbon content increased but volatile matter content decreased with temperature.
- ❖ The influence of heating rate becomes more significant at low temperatures.

Elemental compositions (wt%) of biochar obtained at heating rate 10 °C/min

Element	C		H		O	
Sample	RH biochar	RS biochar	RH biochar	RS biochar	RH biochar	RS biochar
350	47.83	52.52	2.84	3.01	13.70	19.88
450	48.69	52.1	2.19	2.25	9.35	13.85
550	51.15	58.01	1.64	1.84	6.36	5.94

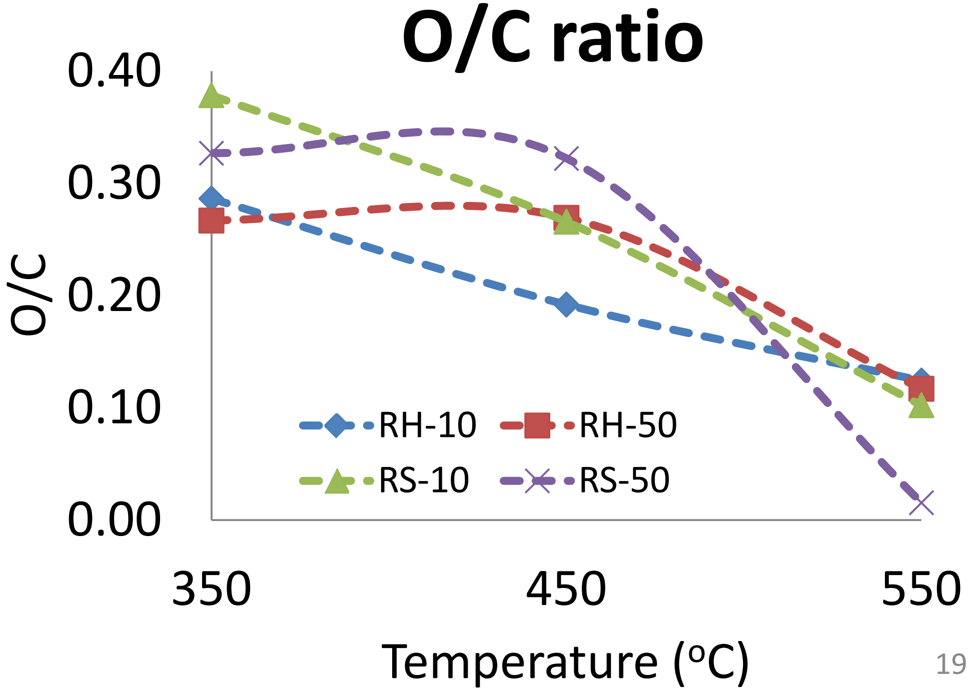
RH: Rice husk; RS: Rice straw

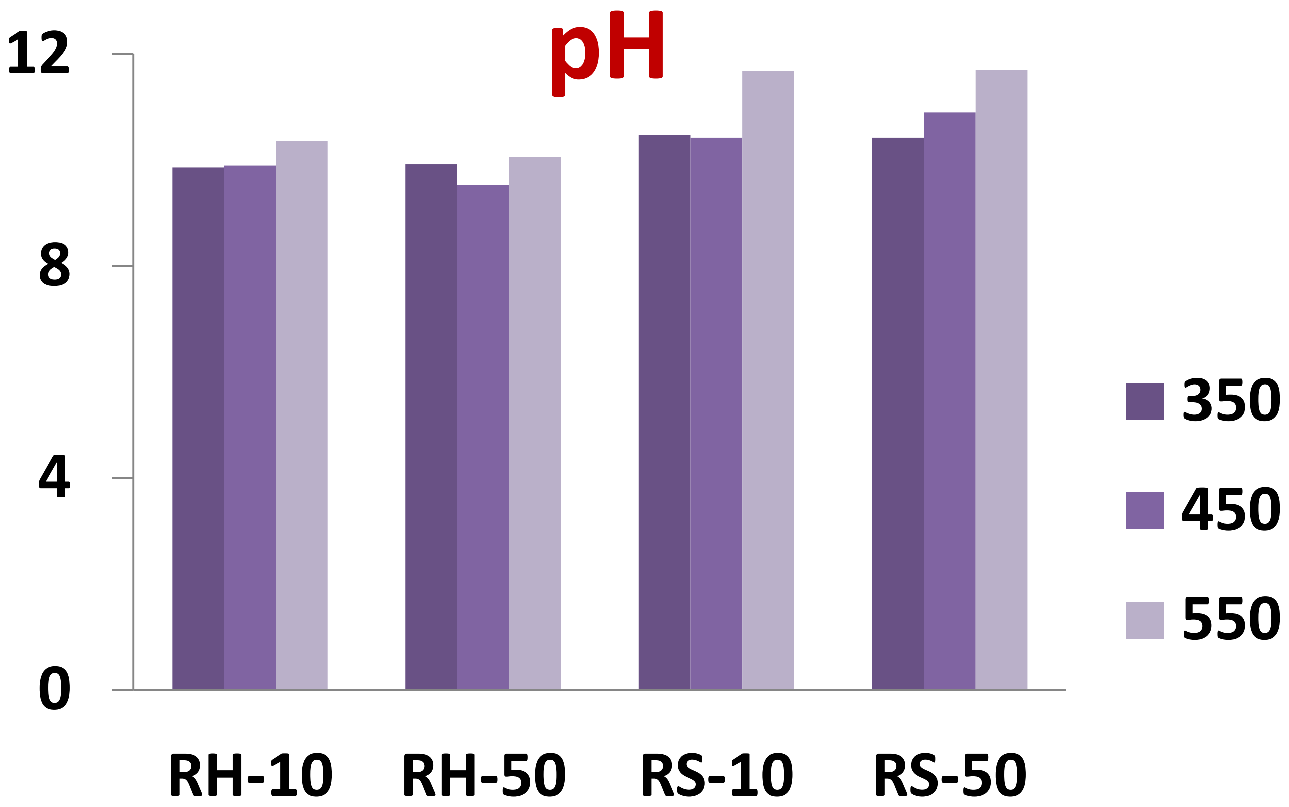
- ➢The carbon content increased with the raising temperature while the H and O content decreased.
- ➢Pyrolysis temperature had a significant effect on the C,H,O contents of the biochar than the heating rate.



Temperature ↑ → H/C and O/C ↓
→ Aromatization ↑

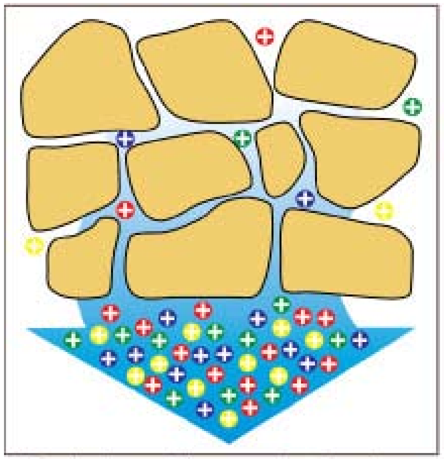
Aromatization led to increase in stability of chars



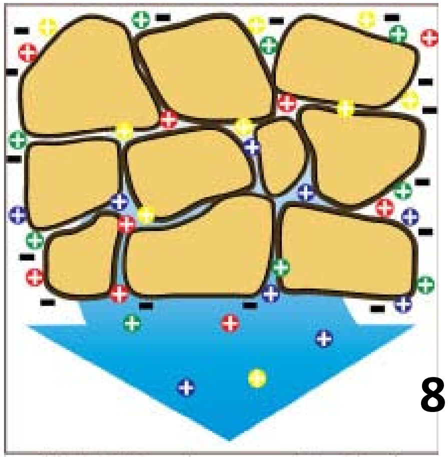


- ❖ pH slightly increased with the charring temperatures
- ❖ The high alkalinity of RS and RH biochar would enable them to improve the soil acidity.

Cation Exchange Capacity (CEC)



Sandy soils poor in humus can't retain nutrients.



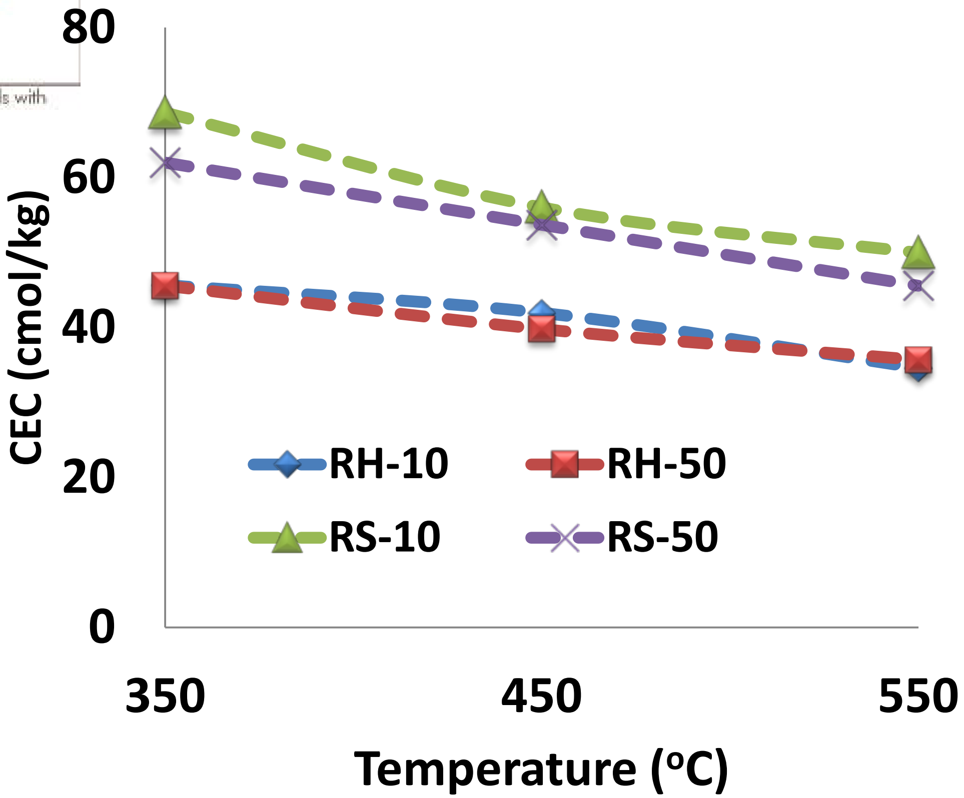
Effect of cation exchange capacity to soils with the addition of humic acid.

CEC of soils:

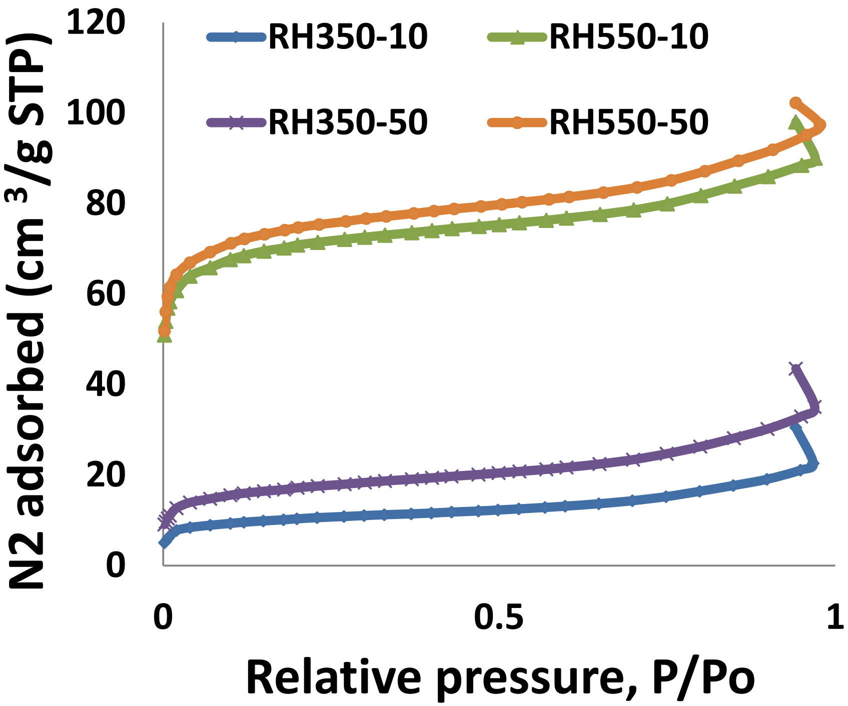
3-20 $\mathrm{cmol_c\ kg^{-1}}$

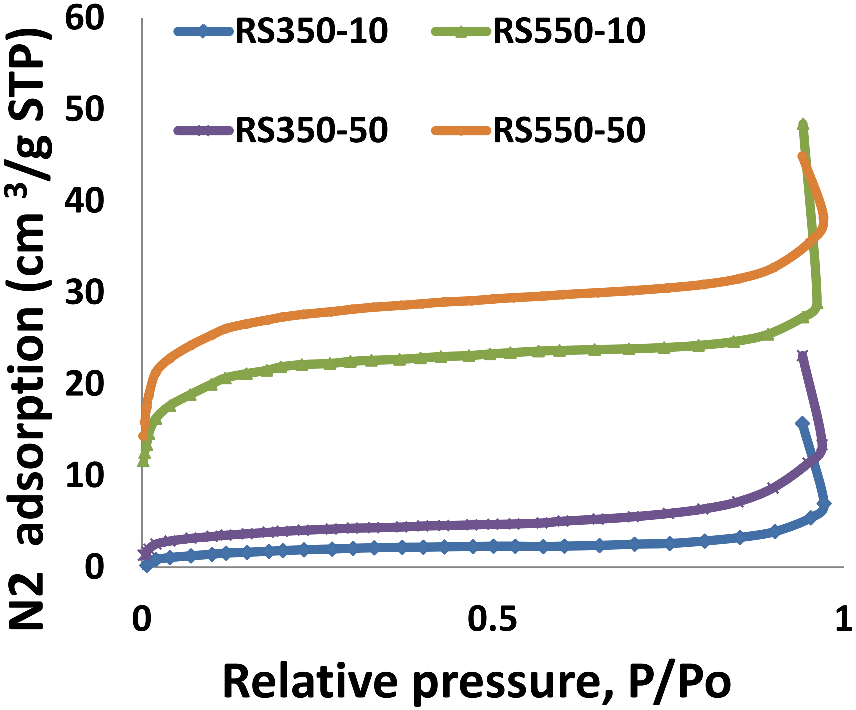
(Brady and Weil, 1984)

The average CEC of the biochar of this study are least 2 times greater than most soils CEC, which may qualify them to be used as soil additive to enhance soil quality.



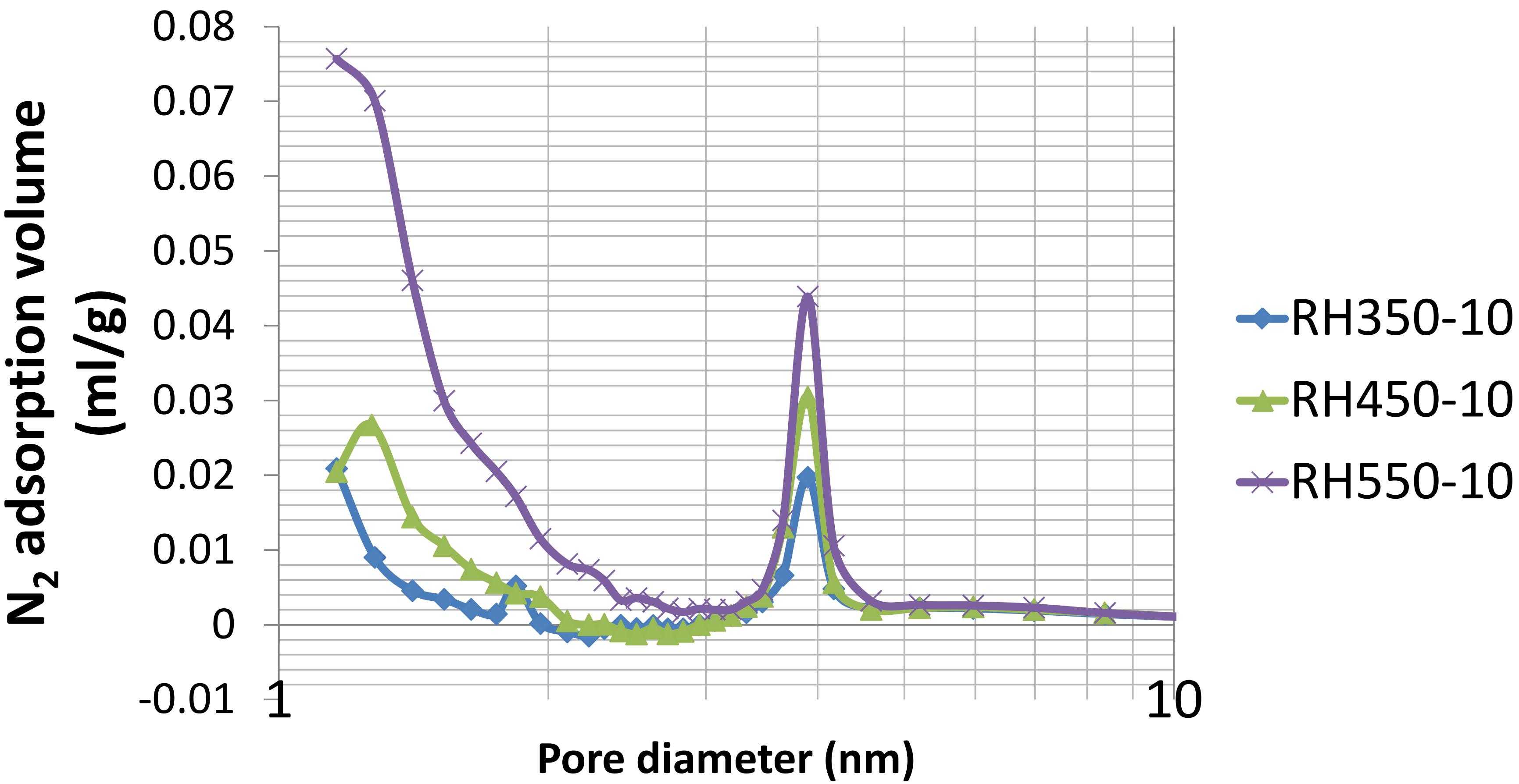
Micropore & Mesopore





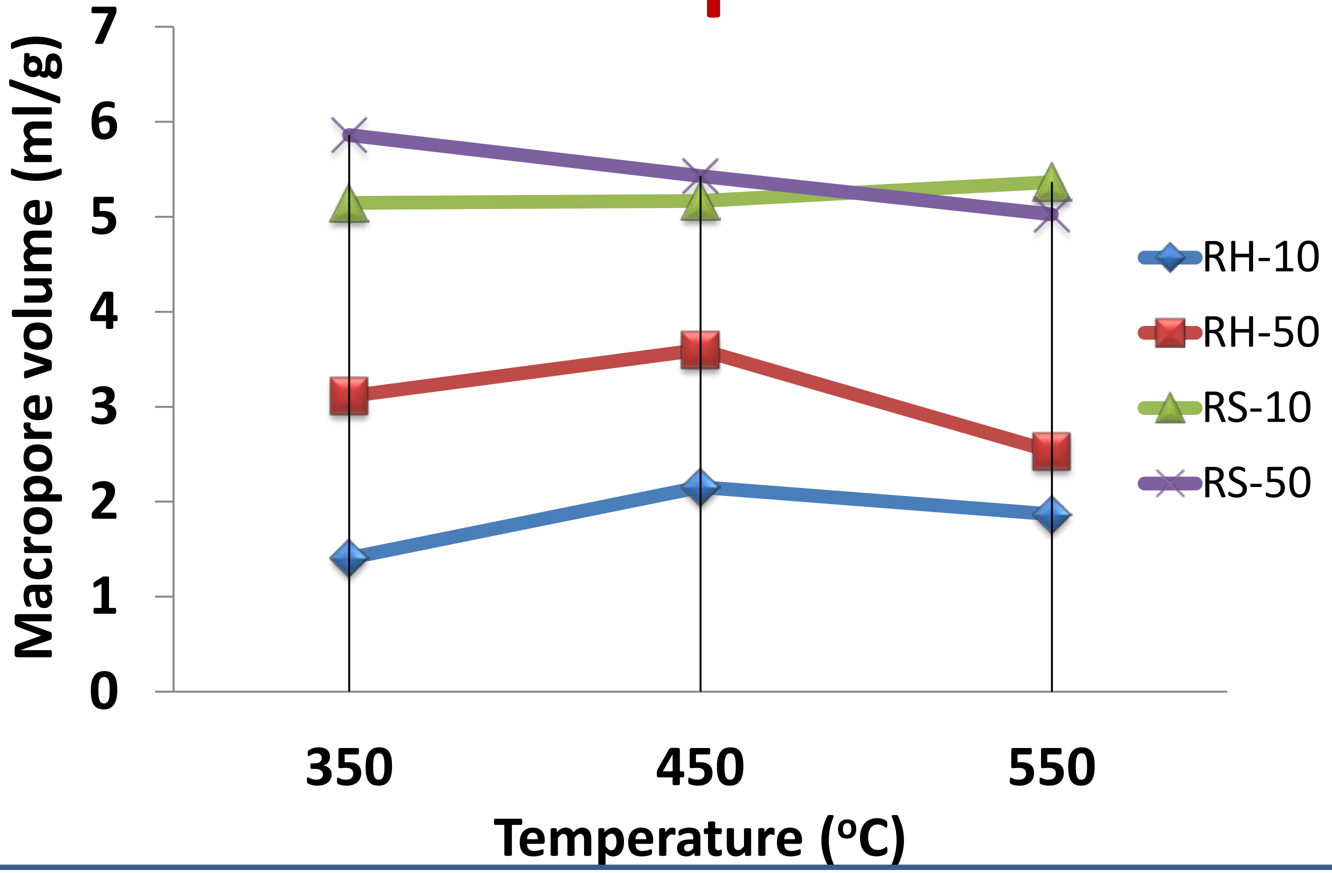
The N_2 adsorption isotherms of biochar suggest that the higher temperature and higher heating rate enhanced the micro and mesopore volume.

Micro & mesopore size distribution



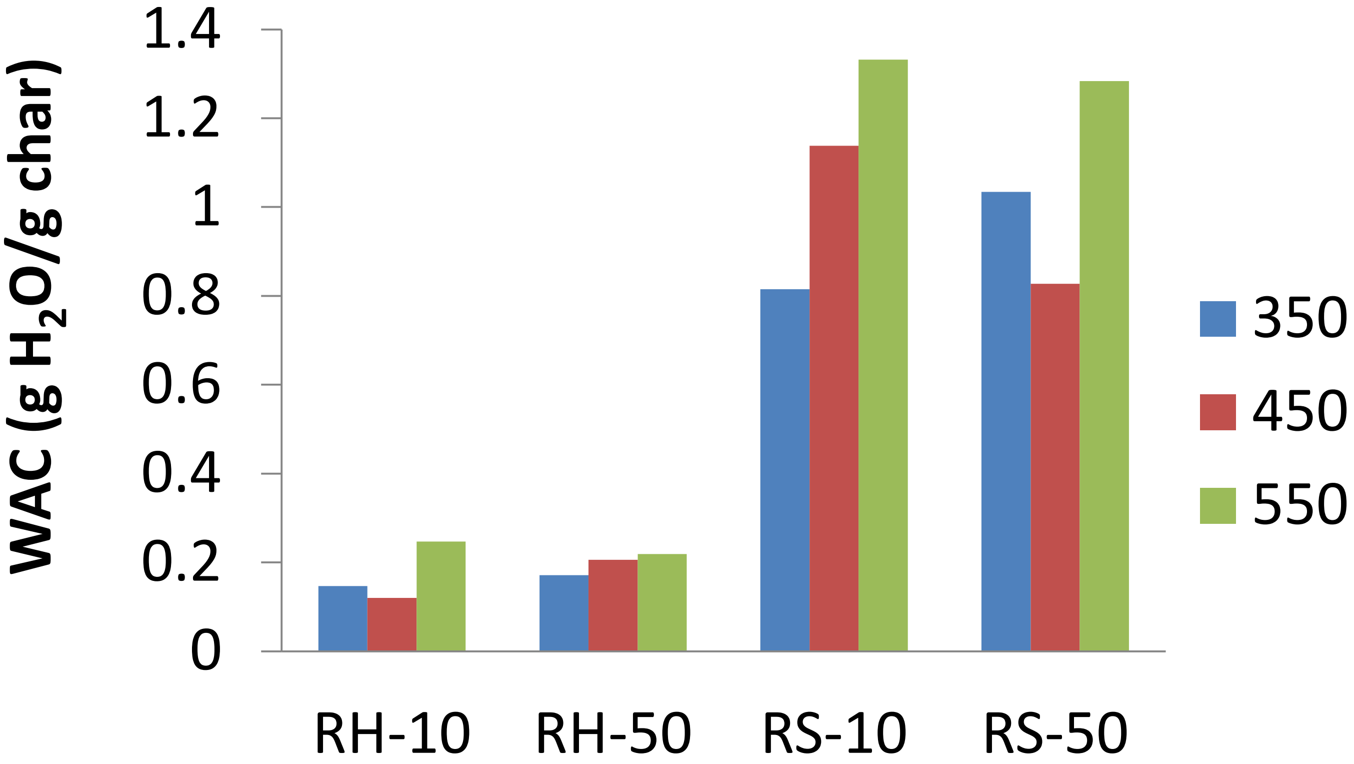
Micropores are sharply ranged from 0.7 to 1.5 nm and mesopores at 4 nm.

Macropore



- ❑ Macropore volume of RS char was about 2 times larger than that of the RH char.
- ❑ The maximum macropore volume of RH char occurred at 450°C.
- ❑ Macropore volume of RS char at 10°C/min changes a little above 450°C, but significantly decreases at 50°C/min for the RH biochar.

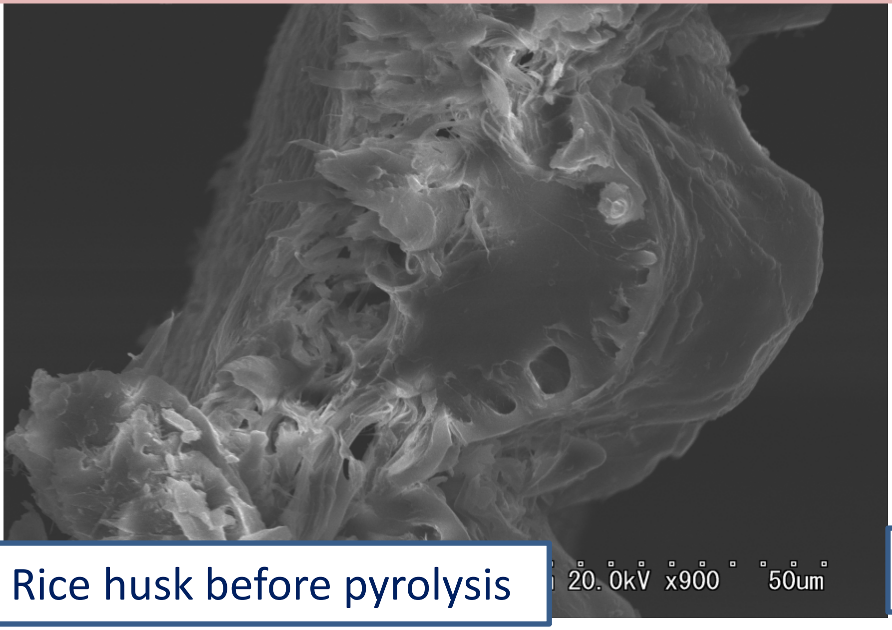
Water adsorption capacity (WAC) of RH and RS biochar at 25°C



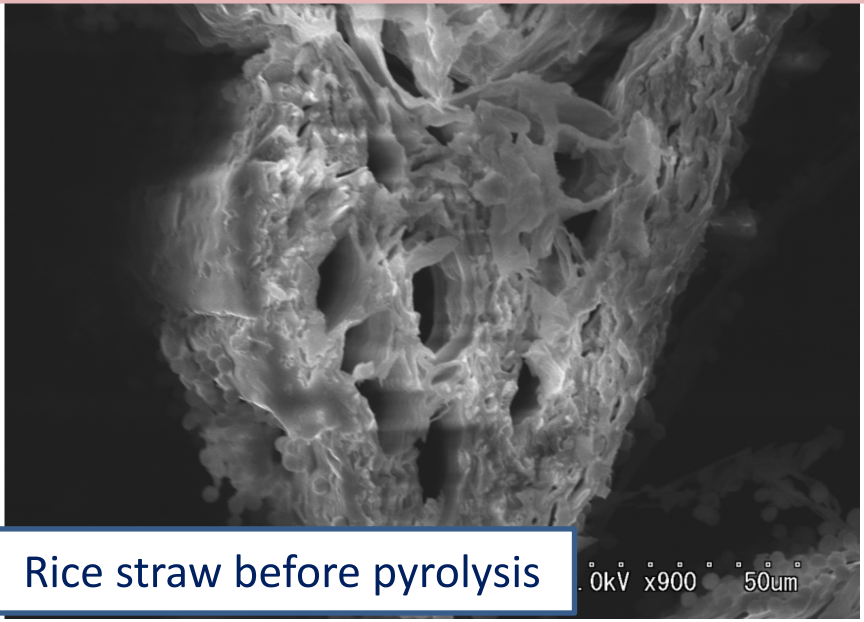
◆ **Although, the RH biochar have higher micro and mesopore volume than the RS char (about 3 times), however the water adsorption capacity of RS char was about 6 times higher than the RH char.**

◆ **Macropre volume of the char may have a significant effect on the water adsorption capacity.**

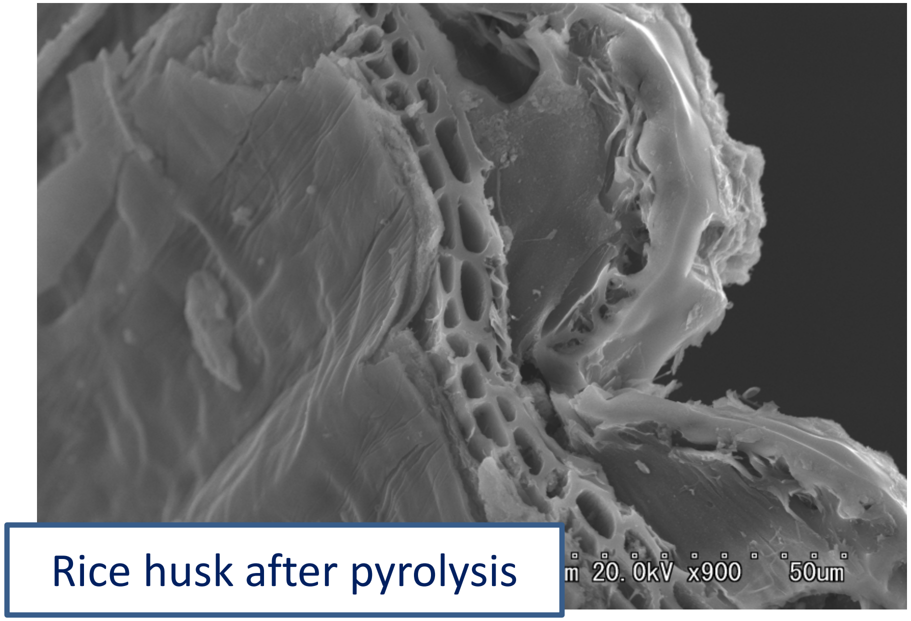
Biochar under electron microscope



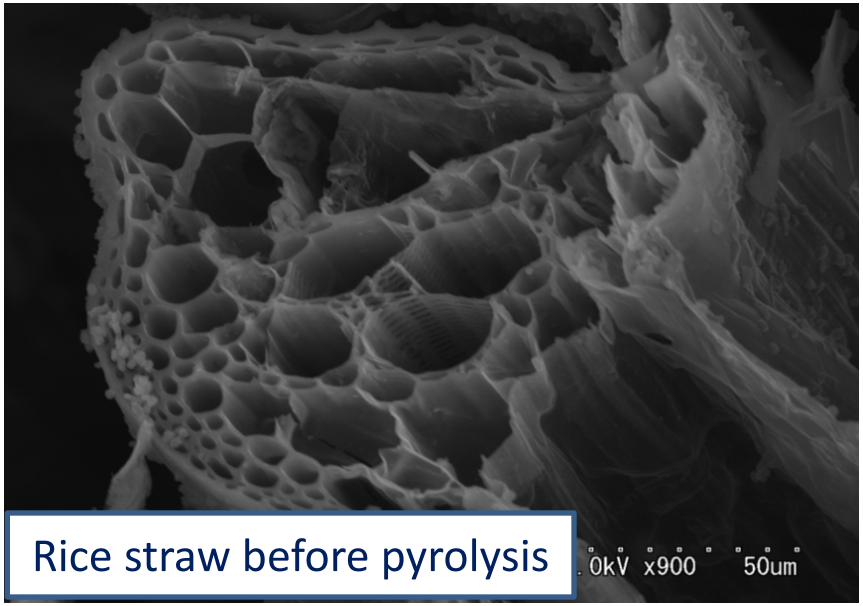
Rice husk before pyrolysis



Rice straw before pyrolysis

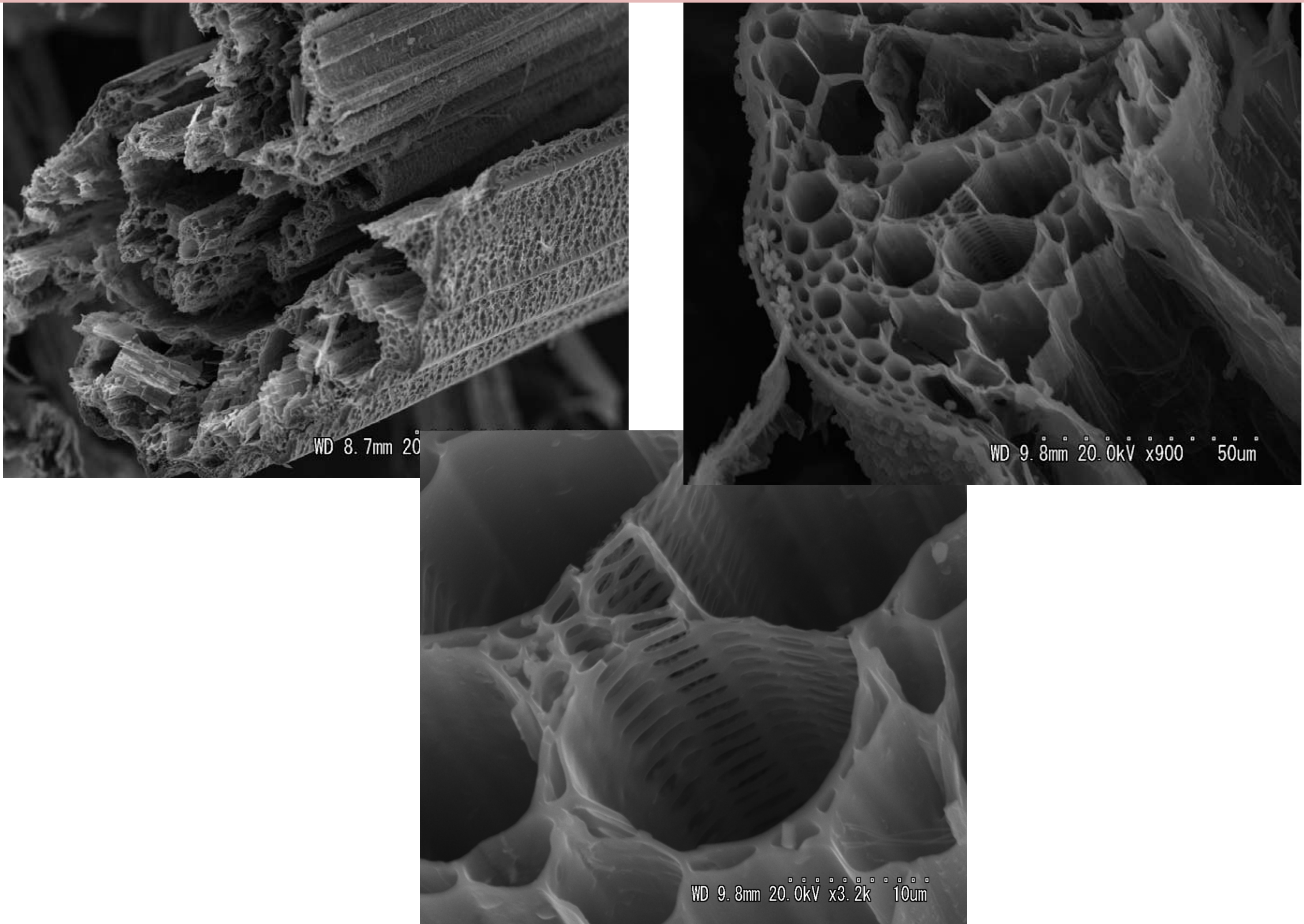


Rice husk after pyrolysis

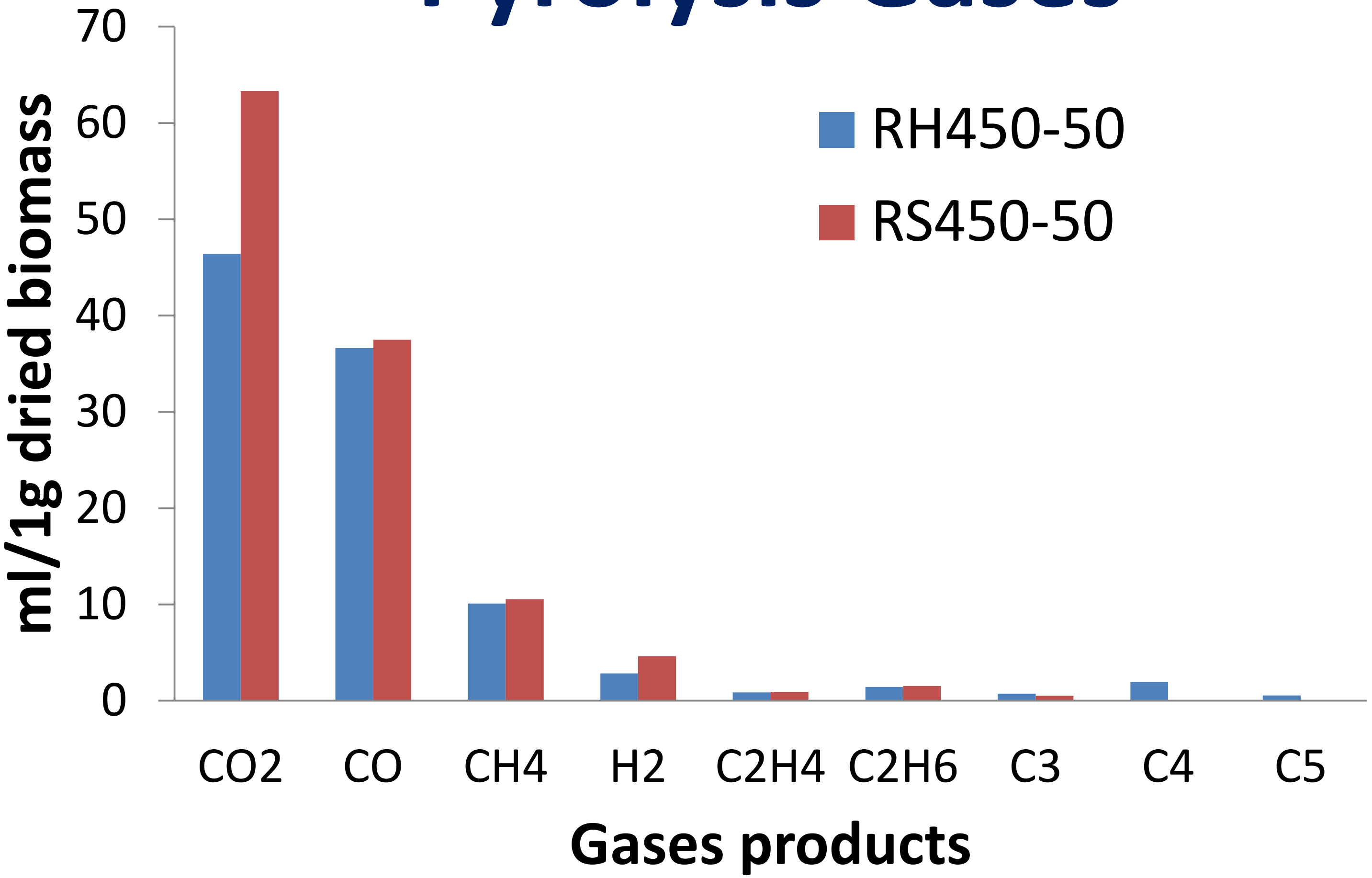


Rice straw before pyrolysis

Rice straw char under electron microscope

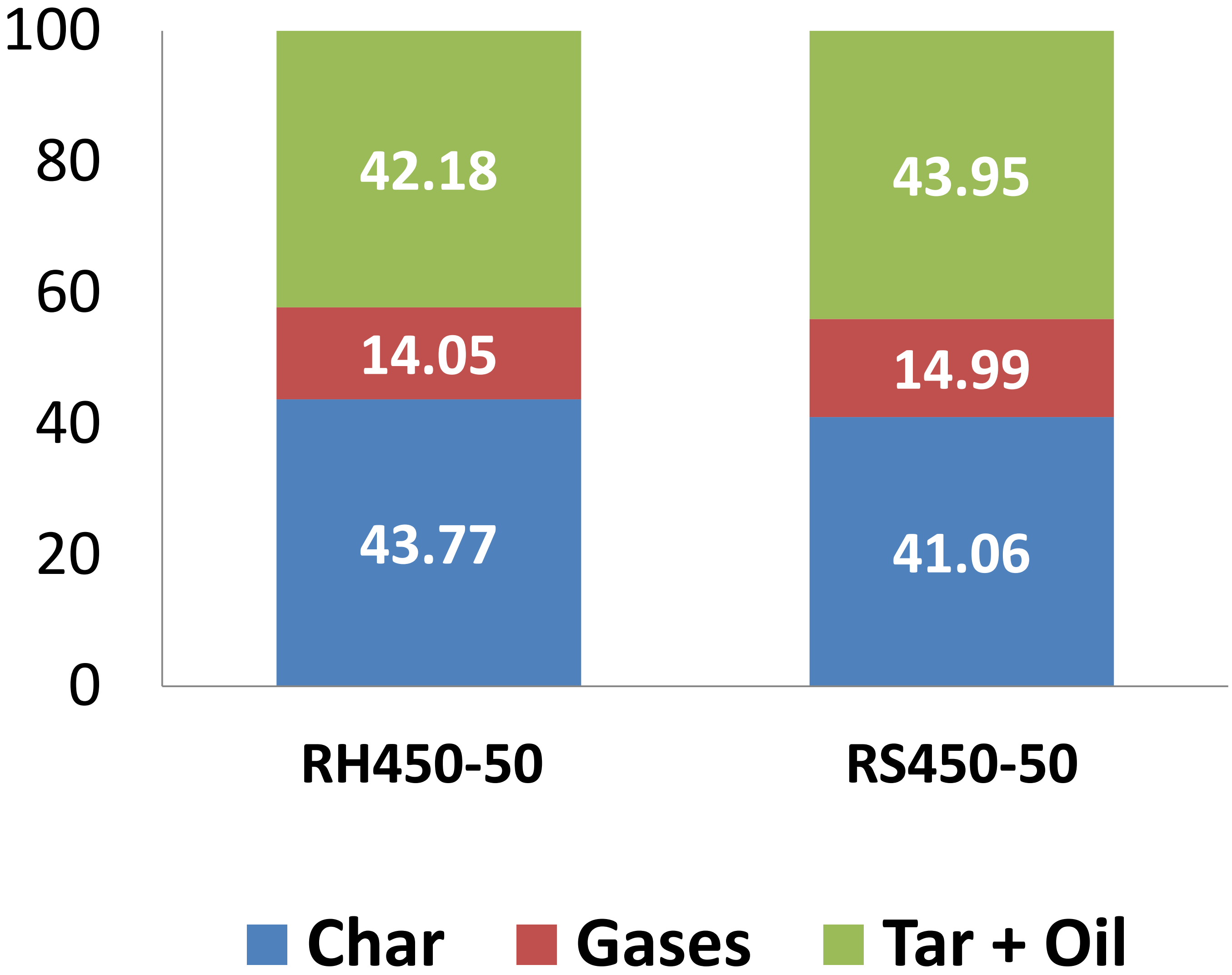


Pyrolysis Gases

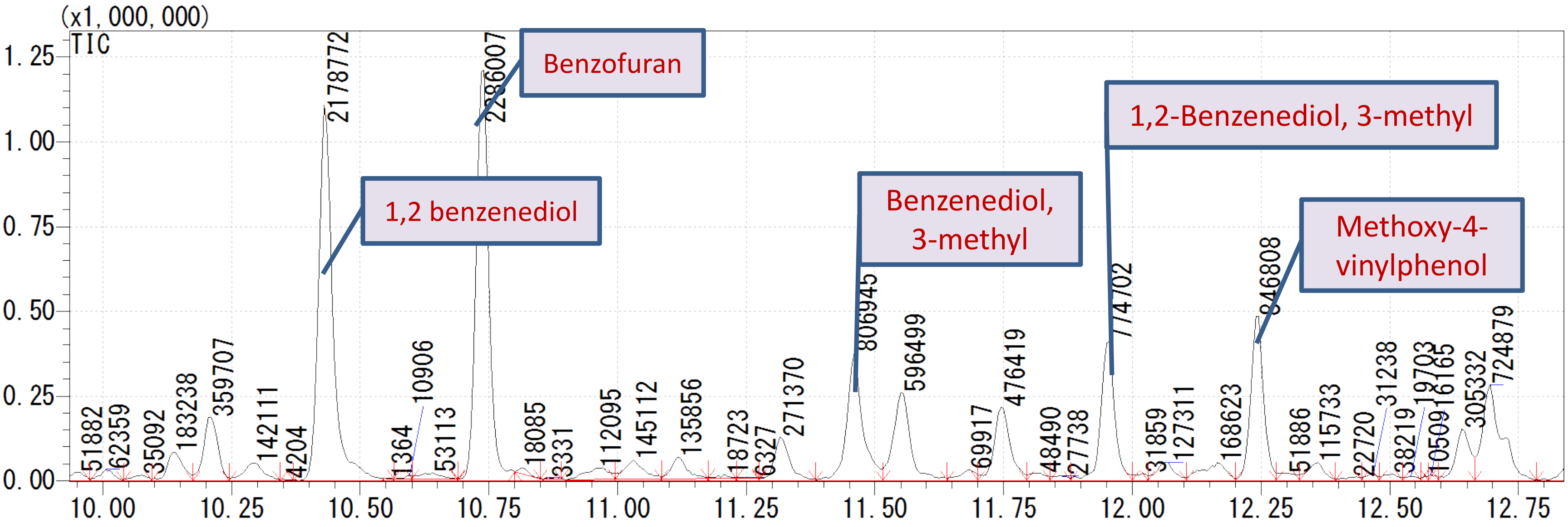


The main gases released during the pyrolysis of rice husk and rice straw were CO_2, CO, CH_4 and H_2 which accounted for about 94-97% of the total gases.

Proportion of the biomass carbon (C%) distributed in the pyrolysis products



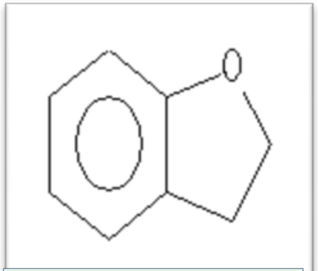
Pyrolysis oil of RH450-50 extracted by acetone

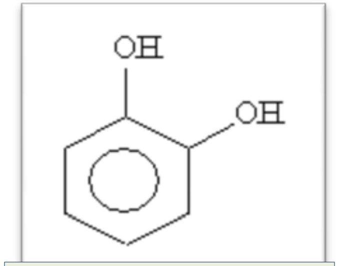


Portion of the GC-MS spectra of rice husk pyrolysis oil

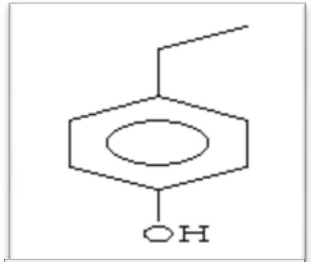
- **Several hundred peaks (compounds) were detected in the GC-MS spectra but the main components were oxygenated aromatic hydrocarbons**

Main compounds in the pyrolysis oils of RH450-50 extracted by acetone

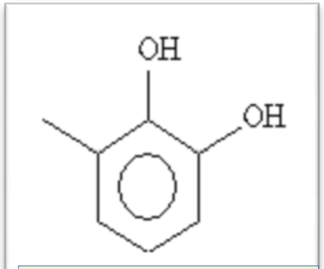

Benzofuran



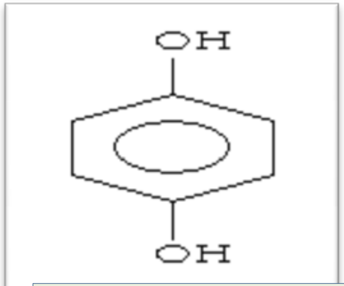
1,2 benzenediol



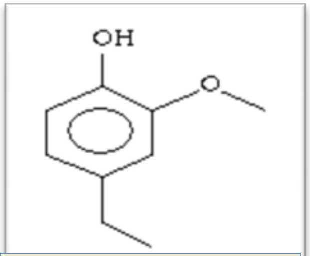
Phenol, 4-ethyl



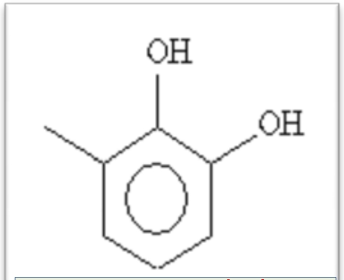
Benzenediol, 3-methyl



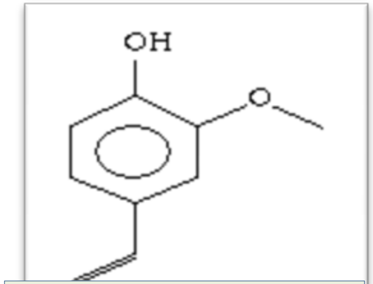
1,4-Benzenediol



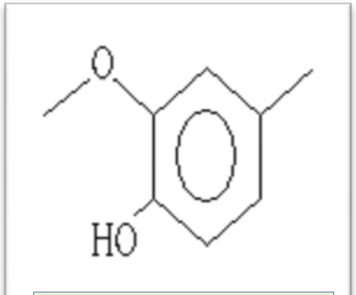
Phenol 4-ethyl-2-methoxy-



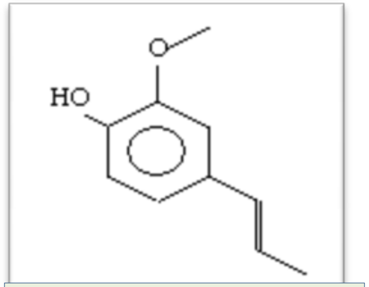
1,2-Benzenediol, 3-methyl



methoxy-4-vinylphenol



Phenol, 2-methoxy-4-methyl



Phenol, 2-mothoxy-4-(1-propenyl)

Biomass pyrolysis Oil composition (area %)*

	Rice Husk	Rice Straw
Phenol	32.85	23.33
Benzenediol	14.06	7.04
Furan	8.29	5.62
Ketone	6.84	7.27
Alkane	1.75	2.67
Aldehyde	2.21	1.87
Ester	0.91	-
Alcohol	0.69	3.87
Benzene	0.45	-
Aromatics	3.02	6.11
Acid	-	0.28
Total, %	72.44	60.98

*** Determined from the area percent of GC-MS peak for each group of compounds**

Making biochar with rice husk in the field

4. Conclusions

- Yield of all biochar decreased with increasing pyrolysis temperature.

- At a low pyrolysis temperature and heating rate, a high char yield was obtained, but, with low aromaticity.

- Biochar obtained from both rice husk (RH) and rice straw (RS) are alkaline, so they can be used to neutralize the soil acidity.

- Amendment of soil with biochar prepared at lower temperatures may enhance the soil CEC.

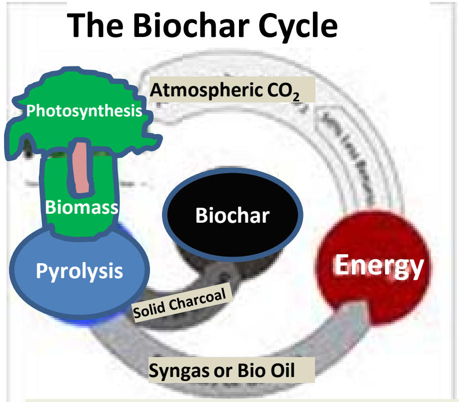
- Macropore volume of RS biochar was much higher than that of RH biochar and the larger macropore volume of RS biochar led to its higher water adsorption capacity.

4. Conclusions

- The byproducts (gas and oil) obtained at 450 ^{o}C and 50 ^{o}C/min showed that the
  - ▪ main components of pyrolysis gas detected were CO_2, CO, CH_4 and H_2,
  - ▪ bio-oils from RH and RS were composed of a complex mixture of aromatic hydrocarbons.

- The pyrolysis byproducts can be used as a source of energy to prepare biochar

Thank you very much for your attention!


The Biochar Cycle

Up to half of the carbon is sequestered

A better world with biochar