

Comparison between Properties of Biochar Produced by Traditional and Controlled Pyrolysis

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ABSTRACT

Biochar is usually produced from crop residues (feedstock) by pyrolysis process under controlled conditions in specialized reactors. The characterization of biochar properties produced under field condition is limited. The objective of this study was to compare the physicochemical properties of biochar produced from two feedstocks; sugarcane bagasse feedstock (SCBF) and rice husk feedstock (RHF), under traditional field conditions (primitive) and controlled conditions. The temperature of traditional pyrolysis process was kept around 500 °C. However pyrolysis temperature under controlled conditions was repeated at 450 °C and 550 °C. In general, the results showed that increasing pyrolysis temperature decreased biochar yield and increased volatile matter, total surface area and total pore volume which were higher in RHBs than in SCBBs, the pHs of both biochars were to the alkaline range, ash and Si percentages of RHBs were higher than that of SCBBs. Furthermore, the C percentage which was higher in SCBB than in RHB. The FR-IR spectral showed that the presence of free OH group and C-H stretch in both feedstocks disappeared in produced biochars under different pyrolysis conditions. The SEM images showed the presence of longitudinal pores in SCBF while SCBB (550 °C) contained longitudinal structure and fewer micropores, while the images of the RHF showed disturbed order of elongated pores which have been changed to developed pores in RHB (550 °C and traditional). The data of XRD analysis of SCBF indicated the presence of cellulose and disappeared in its three biochars while, XRD data of RHF and RHBs indicate the presence of amorphous silica and quartz. Both biochars produced in the field using traditional pyrolysis unit showed approximately biochar properties as those produced under controlled condition (550 °C).

Key words: Feedstock, Sugarcane bagasse, Rice husk, Pyrolysis, Biochar.

INTRODUCTION

About 30 million tons of agriculture biomass or crop residues in Egypt is produced annually (Gomaa et al., 2011 and El-Haggar et al., 2004). Traditional uses of

agricultural residues in Egypt are animal feeding, composting and 50 % of biomass is used as a fuel in low efficiency traditional furnaces or direct combustion in the agricultural field. The traditional furnaces in rare areas are primitive mud stoves which cause air pollution and are extremely energy inefficient (El-Haggar et al., 2004). Due to the wide availability of its raw materials (feedstock), which makes the production of biochar much less expensive, as compared to activated carbon. Biochar is a carbon-rich highly porous material produced from various types of biomass under thermal decomposition and under limited or no supply of oxygen. Recently biochar has received great attention by many researcher because of its promising potentials in many environmental applications, including enhance soil properties, water treatment as a filtration media, and environmental remediation as absorbing and adsorbing agent to reduce organic and inorganic pollutants from wastewater as well as carbon sequestration and mitigate greenhouse gas (GHG) emissions (Novotny et al., 2015, Trakal et al., 2011). The potential uses of biochar as soil improvement by enhancing physicochemical and microbial properties including decrease bulk density, improve soil aggregate stability and water holding capacity in the coarse-textured soil, facilitate drainage in the poorly drained soil, improve soil erosion potential via macroaggregates formation, increase soil pH, cation exchange capacity, base cation percentage, enhance soil fertility, biomass carbon and microbial activity (Burrell et al., 2016, Novotny et al., 2015, Herath et al., 2013, Jien and Wang, 2013).

The thermal process of biochar production is called pyrolysis. There are several types of pyrolysis processes, i.e. slow pyrolysis (carbonization), fast pyrolysis and gasification. During pyrolysis, heating the raw biomass increases its local temperature and consequently its water is evaporated (drying stage), and then pyrolytic volatiles are progressively released (primary pyrolysis stage) from biomass. Variations in the pyrolysis process

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Received July 20, 2017, Accepted August 11, 2017

and its conditions greatly affect the quality and properties of biochar including temperature and residence time, which are considered the most important parameters. The properties of the final product are also dependent upon the nature of the feedstock. The higher pyrolysis temperature tends to increase the volatile matter content, pH value, C %, mineral and ash contents, biochar aromaticity, porosity and specific surface area, and therefore increase the sorptive capacity of biochar (Wu et al., 2012 ; Zhao et al., 2013, Zhang et al., 2015 and Novotny et al., 2015).

The type of feedstock usually affects several biochar's properties such as ash content, the H/C ratio, pH, surface area, cation exchange capacity and oxygen-rich function groups (Mukome et al., 2013 and Břendová et al., 2012). Thus, biomass pyrolysis generates a complex combinations of products from the individual pyrolysis of cellulose, hemicellulose, lignin and extractives (Novotny et al., 2015). It has been reported that lignin-rich biomass is considered to produce a higher yield with better biochar properties (Lee et al., 2013 and Novotny et al., 2015). Thus, different types of plants produced their specific types of biochar. Biomass with higher cellulose content pyrolyzes faster than biomass with higher lignin content (Gani and Naruse, 2007). The overall pyrolysis stage is completed at temperature around 500 °C (Novotny et al., 2015). Pyrolysis at relatively high temperature (above 500 °C) produced biochar of high aromaticity, relatively low O/C atomic ratio. This O/C ratio is an important indicator of stability and functionality of biochar (Spokas, 2010). Many researchers studied biochar properties which produced under controlled conditions (temperature and time), while the properties of the traditional biochar which produced under field condition still limited.

Farmers can use the simple methods to produce traditional biochar in the field to improve soil properties, so this study aimed to compare the physical and chemical properties of biochars produced from sugarcane bagasse (SCB) and rice husk (RH) feedstocks under field and controlled thermal conditions.

MATERIALS AND METHODS

1- Pyrolysis Unit Design:

A traditional pyrolysis technique was used to produce biochar from agricultural wastes. A prototype of pyrolysis unit was manufactured in a workshop located in New Borg Al-Arab City. The design of pyrolysis unit as showed in Fig. (1), consists of double jacket reactor which made from iron. The height and diameter of the external barrel reactor are 100 and 60 cm respectively, this barrel has six holes in the bottom the diameter of each is 10 cm to allow air moving inside

and burn the wood to reach the desired temperature. The cover of this barrel has a chimney with 100 cm in height. The internal barrel has 80 cm and 40 cm in height and diameter respectively, its cover has many small holes (0.5 cm diameter) to allow gases to escape away during carbonization process. A stainless steel net inside the reactor is ready to contain the desired amount of feedstock which needed to be converted to biochar.

2- Feedstock and Biochar preparation:

Sugarcane bagasse feedstock (SCBF) and Rice husk feedstock (RHF) as cellulosic and lignin-cellulosic biomass, respectively, were selected to be used as feedstocks for biochar preparation. SCBF was collected from a variety of local markets located in New Borg Al-Arab City, Alexandria Governorate. SCBF was washed by tap water, dried in an open field and divided into smaller particle sizes (Fig. 2). RHF was collected from private sector rice mill located in small village called Ezbet Elaw, Sidi Salem, Kafr El-Sheikh Governorate.

The raw material was put in a stainless steel net then inside well-sealed barrel of the pyrolysis unit, and the raw material was burnt under partially absence of oxygen. During this process, the temperature was kept around 500 °C for 2 for SCBF and 5 hrs for RHF. The weight of the produced material (biochar) was measured after cooling to 25 °C. Both SCB biochar (SCBB) and RH biochar (RHB) samples were ground in a mortar and passed through 0.5 mm sieve. The biochar produced by this process is defined as Traditional (Primitive) Biochar (Fig. 2). Other biochars were produced from SCBF and RHF under controlled conditions in a muffle furnace (VULCANE A-550 model) at two temperatures; 450 and 550 °C and for the same time periods (2 hrs for SCB and 5 hrs for RH) which have been used for traditional biochar production (Saleh et al., 2012).

The biochar yield was calculated according to Lynch and Joseph (2010), on air-dry weight basis of the raw material, as follows:

$$\text{Biochar yield (\%)} = (W_1/W_0) * 100(1),$$

Where: W_0 is the weight of the raw feedstock on an air-dried basis (g), and W_1 is the weight of the produced biochar (g).

3- Characterization of Physicochemical properties:

Some specific physicochemical parameters were determined in feedstocks and biochars as follow:

Structural Constituent: cellulose, hemicellulose and lignin contents in both feedstocks were determined according to the methods described by Van Soest et al., 1991.

Ash content: the ash content was determined by the dry combustion for 1.00 g of the different feedstock or

biochar samples at 700 °C for 12 hrs in an open porcelain crucible (Samsuri et al., 2014). The crucible was cooled in a desiccator until reaching the room temperature, the ash weight was determined. The percentage of the ash content was calculated according to Lynch and Joseph (2010) as follows:

$$\text{Ash content (\%)} = (W_{\text{ash}} / W_{\text{sample}}) \times 100 \quad (2),$$

Where: W_{ash} is the weight of ash (g) and W_{sample} is the weight of feedstock or biochar (g).

Moisture content: five grams of biochar were put in a porcelain crucible and dried in an oven at 80°C for 24

hours. After cooling in a desiccator, the sample was re-weighed and the moisture content of the biochars was calculated. This process was repeated several times until a constant weight of biochar sample was obtained (Samsuri et al., 2014) as follows:

$$\text{Moisture content (\%)} = (W_2 - W_3 / W_2 - W_1) \times 100 \quad (3)$$

Where: W_1 is the weight of crucible (g), W_2 is the initial weight of crucible plus biochar sample (g), W_3 is the final weight of crucible with biochar sample (g).

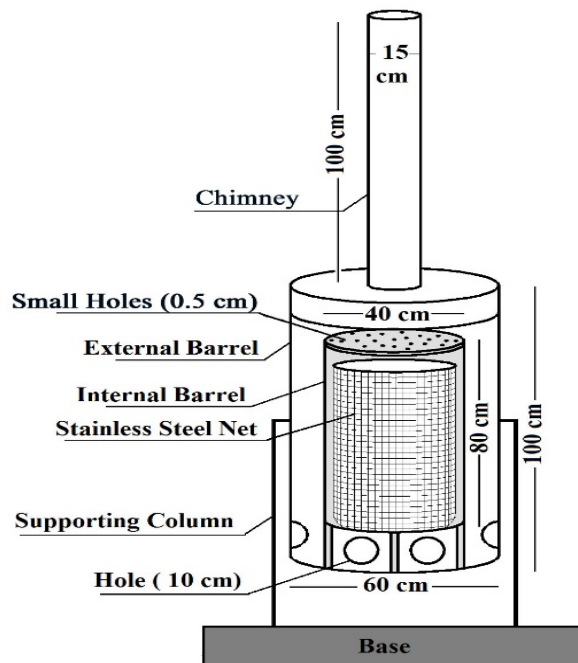


Fig. 1. Schematic layout of Prototype of Pyrolysis Unit (Front View)



Fig. 2. Photographs of biochar produced from (A) Sugarcane Bagasse and (B) Rice Husk

Surface area: the surface area of biochar was measured using N₂ sorption isotherms run on BELSORP-mini II and the Brunauer-Emmett-Teller (BET) measured at 273 °K and interpreted using Grand Canonical Monte Carlo simulations of the non-local density functional theory for micropore-enclosed (<1.5 nm) surfaces.

Scanning Electron Microscopy (SEM): the morphology of the feedstocks and biochars was studied using a JEOL, Model JSM 6360 LA, Japan. In order to avoid the build-up of local electrical charges, the samples were coated with gold using sputtering coater (model: S150B, Edwards High Vacuum Ltd., UK) before investigation.

Fourier transform infrared (FTIR) spectral analysis: this technique was used to study the compositional properties for biomass and biochar determining the functional groups by scanning these samples with infrared rays in the range 400 – 4000 cm⁻¹ using SHEMATZU infra-red spectrophotometer model FT/IR-5300, JASCO Corporation, Japan.

X-ray diffraction (XRD) diffractometry: it was studied to characterize the crystalline structure and identify the main components in the feedstocks and biochars. XRD was performed on the ground samples using a SHIMADZU XRD-7000, X-ray diffractometer with Cu-K α radiation operated at 30 kV and 30 mA and scanning from 5 to 90° 2 θ , using a step size of 0.02° 2 θ and at a scanning rate of 4°/min.

pH: one gram biochar was mixed with 100 ml of de-ionized water. The obtained suspension heated to about 90 °C with stirring for 20 min. After cooling to room temperature, the pH of the suspension was measured by pH-meter model, Accumet Research AR50 (Masulili *et al.*, 2010).

Cation exchange capacity (CEC): a modified ammonium- acetate compulsory displacement method (Sumner and Miller, 1996) was used for measuring the CEC as described by Gaskin *et al.* (2008). 1.0 g biochar sample was placed in a 50-ml polyethylene centrifuge tube, leached five times by deionized water, then 20 ml of deionized water were added and shaken using an orbital shaker for 5 min. at 180 rpm. This process was repeated five times, then the sample was centrifuged and the suspension was saved for further analysis. Ten ml of neutral Na- acetate (1M) was added to the sample with shaking for 10 min. This process was repeated three times. The Na-biochar samples were then washed three times by ethanol, the adsorbed Na⁺ ions were displaced by 30 ml of one normal neutral NH₄. acetate for three times in 100 ml volumetric flask and the concentration of Na⁺ was measured by flame photometer.

Elemental analysis: carbon, hydrogen, nitrogen, and sulphur contents of the feedstocks and biochars were determined by using a CHNS Elemental Analyzer (Vario type, EI, elemental analyzer). The oxygen content was obtained by subtracting [100% – (C % + H %)] according to El-Sherif and Fathy, (2011). Total nutrients in the feedstocks and biochars were extracted by adding 10 mL concentrated HNO₃: HCl (1:3) “Aqua Regia” to the ash (dry combustion of 1.00 g of biomaterial at 500 °C for 6 hrs). After cooling to room temperature, the digested solution was filtered into a 100-mL volumetric flask and brought to volume with de-ionized water. Total concentrations of Cu, Fe, Mn, and Zn were measured in the extract of the dry-ashing by atomic absorption spectrophotometer, model, Varian, Spectra (AA-220). The concentration of total P in this extract was measured by ammonium paramolybdate-vanadate method and the color intensity was measured at 420-nm wavelength by T80 UV/VIS Spectrophotometer, PG Instruments Ltd (Olsen and Sommers, 1982). The concentration of K and Na, in the same extract, were measured by flame photometer, FP902, PG Instruments. The concentration of dissolved organic carbon (DOC) was determined in the filtrate (0.45 μ m membrane filter) of 5% W/V solid/distilled water extract (after shaken the suspension for 24 hrs at 150 rpm and centrifuged in at 5000 rpm for 20 min) by using the TOC analyzer, multi-N/C UV3100, Analytikjena product, Germany (Wong *et al.*, 2007). The electric conductivity (EC) was measured in the DOC extraction using a conductivity meter WTW inoLab Cond 720 (WTW GmbH, Weilheim, Germany).

RERSULTS AND DISCUSSION

1- Feed stock Properties

The Structure constituents for both SCBF and RHF were illustrated in Table 1. The contents of cellulose and hemicellulose as well as moisture content of SCBF were higher than that of RHF. It has been reported that the typical amount of cellulose in biomass ranges from 40 to 60 % and that of hemicellulose from 20 to 40 % (Zhang *et al.*, 2010). On the other hand, RHF had higher contents of lignin and ash than that of SCBF. It is common that lignin accounts a range from 18 to 40 % of several biomass materials (Novotny *et al.*, 2015). These variations can be attributed to the presence of different organic constituents in the two feedstocks that indicated higher lignin in RHF which is amorphous and hydrophobic polymer with high molecular weight and numerous functional groups of aromatic substructure (Lee *et al.*, 2013).

Table 1. The selected physiochemical properties of the (SCB) and (RH) air-dried feedstock

Parameter	Feedstock	
	SCB	RH
Chemical Parameter		
pH	3.39	6.76
EC	dS/m	0.97
DOC	%	6.00
Total Elemental Analysis		
C	%	42.82
N	%	0.50
H	%	1.54
O	%	53.90
S	%	0.42
H/C	atomic ratio	0.430
O/C	atomic ratio	0.944
(O+N)/C	atomic ratio	0.954
Si	%	1.74
P	%	0.10
K	%	1.03
Na	%	0.19
Fe	mg/kg	253.81
Mn	mg/kg	12.53
Zn	mg/kg	171.35
Cu	mg/kg	17.83

* DOC dissolved organic carbon

The % of cellulose, hemicellulose and lignin for both biomaterials are similar with those in the previous studies (Ludueña et al., 2011, Rezende et al., 2011, Kumar et al., 2013, Guilherme et al., 2015, Wang et al., 2016).

Where, cellulose and hemicellulose are consisting of simple sugar monomers they decompose at a temperature lower than 450 °C. These two compounds have lower molecular weight than lignin and easily released as pyrolytic vapors (Lee et al., 2013), while lignin is very resistant to thermal degradation.

The higher ash content in RHF than in SCBF indicates the presence of higher element contents in RHF than in SCBF. Several studies reported that SCBF contains low ash content as compared with that of RHF (Saleh et al., 2013, and Jindo et al., 2014), this could be due to the higher amount of Si in RHF (Table 1). Si is a major component in the chemical structure of rice (Jindo et al., 2014). However, the higher amount of DOC for SCBF may be attributed to the high content of cellulose and hemicellulose (Bottino et al., 2016).

As shown in Table 1 the results indicated lower pH value for SCBF than that of RHF. This indicated the presence of more acidic groups in SCBF compared with RHF.

It is clear from Table 1 that there is no marked variation in the electrical conductivity (EC) value of both SCB and RH feedstocks even it seems relatively higher in SCB than in RH. Thus there are slight variations in the percentages of N, H, and O between both feedstocks (Table 1). On the other hand, the percentages of C and S were markedly higher in SCBF than in RHF while that of Si % was higher in RHF than in SCBF. The concentration of S, P, K, and Na were relatively higher in SCBF than that in RHF. However, the concentrations of Fe and Zn were approximately close, while Mn was markedly higher in RHF than in SCBF and Cu was markedly higher in SCBF than in RHF.

Biochar Properties

Physical properties: Table 2 shows that the biochar yield of RHBs produced under different pyrolysis condition was higher than that of SCBB. However, it was observed that the yield of both SCBB and RHB was decreased with increasing pyrolysis temperature from 450 to 550 °C, this could be due to thermal decomposition and destructive of reaction of cellulose (Novak *et al.*, 2009, Jindo *et al.*, 2014 and Claoston *et al.*, 2014). It is clear from Table 2 that the yield of traditional biochar of both SCBB and RHB were very close to their yield when produced under a controlled condition at 550 °C for both biochars.

The percentage of volatile matter released from SCBB were higher than from RHB at different pyrolysis conditions, and increased with increasing pyrolysis temperature from 450 to 550 °C. It is clear that percentage of volatile matter in traditional SCBB was

higher than in that of traditional RHB and both two traditional biochars have volatile matter close to those produced at 550 °C pyrolysis temperature.

It is clear from these data that the type of feedstock had marked effect on the percentage of volatile matter. The percentages of moisture content of SCBBs were higher in SCBBs than in RHBs and these percentages were approximately very close at the two pyrolysis temperatures (450 and 550 °C) and also with that of traditional biochars of both SCB and RH (Table 2). The moisture content in biochar is related to feedstock rather than to pyrolysis temperature.

Ash percentage in both biochars has increased with increasing pyrolysis temperature from 450 to 550 °C and this percentage was higher in the RHBs than SCBBs (Table 2).

Table 2. The physiochemical properties of the SCB and RH biochars

Parameter	SCBB			RHB			
	Traditional	450 °C	550 °C	Traditional	450 °C	550 °C	
Physical Parameters							
Biochar yield	%	32.06	41.71	34.93	40.31	46.17	39.92
Volatile matter	%	67.94	58.29	65.07	59.69	53.83	60.08
Ash	%	19.07	17.69	21.43	40.19	43.43	49.51
Moisture content	%	9.32	9.51	8.80	5.03	4.89	5.15
Total Surface Area	m ² /g	185.60	107.87	124.11	154.67	98.01	212.99
Total Pore Volume p/p ⁰	cm ³ /g	0.11	0.06	0.08	0.10	0.07	0.12
Mean Pore Diameter	nm	2.31	2.28	2.41	2.55	2.66	2.31
Chemical Parameters							
pH		8.63	8.40	9.00	8.85	8.33	9.44
EC	dS/m	0.70	0.63	0.62	0.46	0.58	0.45
CEC	meq/100gm	31.89	33.85	33.00	26.15	24.85	27.13
DOC	%	0.02	0.03	0.02	0.05	0.03	0.02
Total Elements Analysis							
N	%	0.76	0.86	0.82	0.68	0.58	0.85
C	%	51.37	54.84	67.92	38.50	41.31	42.82
H	%	0.80	0.46	0.64	0.47	0.58	0.45
O	%	43.88	40.78	26.70	33.73	28.61	23.10
S	%	0.74	0.70	0.36	0.16	0.38	0.13
H/C	atomic ratio	0.19	0.10	0.11	0.15	0.17	0.13
O/C	atomic ratio	0.64	0.56	0.30	0.66	0.52	0.41
(O+N)/C	atomic ratio	0.65	0.57	0.31	0.67	0.53	0.42
Si	%	3.96	3.92	4.74	27.30	29.50	33.63
P	%	0.29	0.27	0.21	0.01	0.02	0.02
K	%	0.50	0.44	0.37	0.54	0.61	0.63
Na	%	0.39	0.34	0.43	0.29	0.28	0.29
Fe	mg/kg	1914	2344	2688	968	246	322
Mn	mg/kg	54.35	67.64	54.74	57.85	55.33	54.61
Zn	mg/kg	158.10	146.31	127.85	143.51	131.16	140.49
Cu	mg/kg	12.87	15.06	14.07	10.21	8.49	9.71

The high percentage of ash content in RHB at different pyrolysis temperature, relative to that of SCBB is related to chemical composition of rice (Wu et al., 2012 and Jindo et al., 2014).

Table 2 shows wide variations in the values of total surface area due to both the type of feedstock and the pyrolysis temperature. Low pyrolysis temperature (450 °C) produced the least total surface area of both SCBB and RHB relative to that of high temperature (550 °C) or to that of traditional biochar. Similar trend can be observed with respect to total pore volume (Table 2), low pyrolysis temperature (450 °C) produced the least total pore volume for both SCBB and RHB which were approximately very close. Similarly, traditional SCBB and RHB have approximately close values of total pore volume values. The increase of total surface area of biochar with increasing pyrolysis temperature is attributed to the formation of micropores (García-Jaramillo et al., 2015). Claoston et al. (2014) found that the surface area of the rice husk biochar increased with increasing temperature and this could be due to the removed of volatile material and resulting in increasing micropore volume (Ahmad et al., 2012).

Fourier Transform-Infrared (FT-IR) spectra: As presented in Fig. 3, FTIR data of SCB feedstock and its biochars were less different from those of RH with respect to the intensity. The peaks around 3813.4 and 3786.39 cm⁻¹ indicates the existence of free hydroxyl functional group (O-H stretching) in SCBF and RHF, due to the chemically absorbed water and also due to the surface hydroxyl groups (Saleh et al., 2013). These peaks could be disappeared in the different biochars, except for RHB produced at 450 °C which appeared at

3740.10 and 3840.40 cm⁻¹. The FTIR spectra of the feedstocks and biochars for SCB and RH, indicated that the band in the range 3410.26 to 3452.7 cm⁻¹ is depicting the stretching of hydroxyl group and H-bonds groups from alcohols, phenols, and organic acids (Jindo et al., 2014). In addition, silanol group (Si-OH) was found in rice husk. These bands were more intense in the feedstock than in the biochar, which is produced under different pyrolysis conditions (Daffalla et al., 2010). Throughout the increasing pyrolysis temperature, the stretching band of hydrogen-bonded hydroxyl groups started to diminish (Fig. 3). This was may be due to higher mass loss during thermal decomposition and gas evolution (Claoston et al., 2014).

The peaks at 2921.61 and 2928.04 cm⁻¹ in SCBF and RHF, respectively, is assigned to saturated C-H stretching vibration (aliphatic C-H) which indicates the presence of alkane functional group. This has been observed for the two feedstocks and disappeared in the biochars as a result of dehydration of cellulosic and ligneous components (Zhao et al., 2013 and Guilherme et al., 2015) and dehydrogenation of methylene groups, which yielded increasingly condensed structures (R-CH₂-R → R=CH-R → R=C=R), controlled biochar recalcitrance (Harvey et al., 2012 and Zhao et al., 2013). Similar observation has been found in the band at 1055.10 cm⁻¹ in SCBF only (alcohol C-O stretch and aliphatic amines C-N). Due to the high content of silicon in rice husk (Mahmoud et al., 2011), the different functional groups in which silicon exists (e.g., siloxane Si-O-Si and silanol Si-OH) were clearly observed in FT-IR spectra (Fig. 3-b).

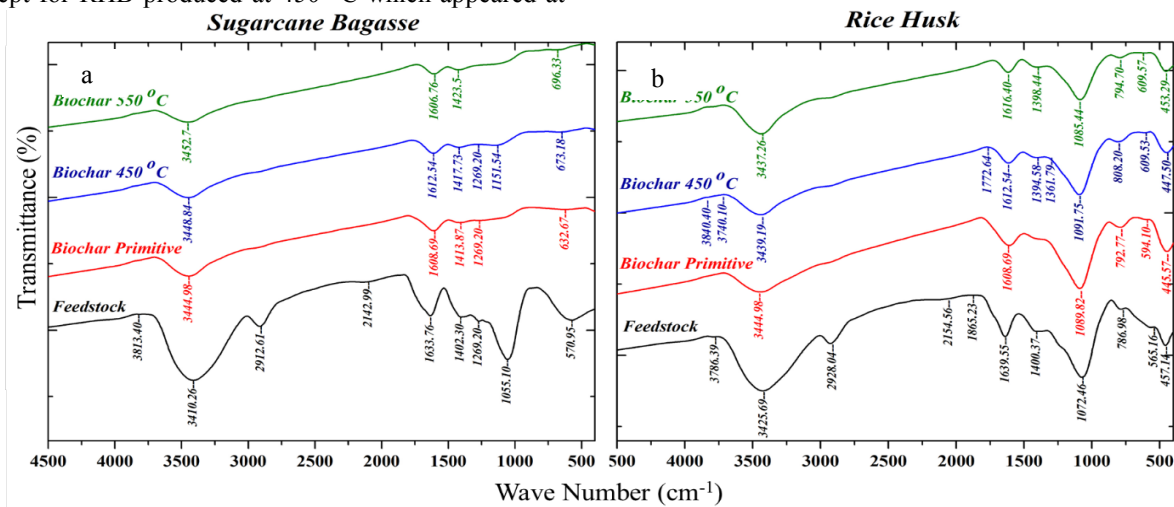


Fig. 3. FT-IR Spectra of Feedstocks and biochars of Sugarcane Bagasse (a) and Rice Husk (b) feedstock

The FT-IR spectra showed typical bonds of Si–O–Si stretching at 1072.46–1091.75 cm^{-1} and the bands at Si–O at 786.98–808.20 cm^{-1} and 445.57–457.14 cm^{-1} for RH and RHB. Similar results were obtained by Saleh *et al.* (2013). The N–H bend and C=C stretching vibrations between 1606.76–1639.55 cm^{-1} in SCB and RH are indicative of amines, alkenes and aromatic functional groups, respectively (Claoston1 *et al.*, 2014 and Daffalla *et al.*, 2010). The presence of amide groups in both SCBB and RHB, are indicated by the high N concentrations (Table 2) (Claoston1 *et al.*, 2014). The intensity of C=O stretching of aromatic rings (1606.79–1639.55 cm^{-1}) was higher in the feedstocks than biochars and decreased with temperature rise in biochars and seemed similar in SCB and RH feedstocks (Zhao *et al.*, 2013). Claoston *et al.* (2014) mentioned that peaks around 1700 cm^{-1} indicate the existence of holocellulose and lignin, while the peak around 1400 cm^{-1} may be attributed to the aromatic CH and carboxyl-carbonate structures and silanol groups. The silanol groups are in the form of silicon dioxide structure (–Si–O–Si–OH). This structure is similar to the silanol groups of silicic acid (Srivastava *et al.* 2006). Aromatic C=C ring stretching were observed between 1400 and 1600 cm^{-1} and the peaks at 1260 cm^{-1} , corresponding to aromatic CO- stretching, were observed for all biochars. Aromatic C=C peaks are an indication of benzene-like rings. These aromatic molecules have extra stability caused by the nature of their structure. The bands around 565.16 and 609.57 indicate the presence of alkyl halides (C–Br stretch), in contrast another study showed that it may be due to the stretching variation of both inorganic compounds such as KCl and CaCl₂ (Claoston *et al.* 2014).

Comparing the surface chemistry of feedstock and biochars produced at traditional, 450 °C, and 550 °C showed that raw feedstocks and biochars had similar surface functional groups and in spite of that some of these peaks had disappeared. The disappearance of these peaks was an indication of decreases in water and aliphatic compounds. Also, a new peak around 1400 cm^{-1} , corresponding to aromatic C=C stretching, appeared on the biochar surface. This indicates an increase in the aromaticity during pyrolysis as temperature increased

SEM micrographs: The images showed a significant changes to the surface morphology of the both biomaterials (SCB and RH) before and after pyrolysis process. The SEM images showing fibrous channels in both biochars. The biochars' structure consisted of a highly complex network of pores. The SEM images of SCBF showed differences as compared with those of

different SCB biochars which produced at different conditions of pyrolysis process (traditional, 450 °C and 550 °C). At 2000 magnification the presence of longitudinal pores in SCBF is originated from the vascular structure of this biomass (Fig. 4). On the other hand, the micrograph of the external surface of traditional SCBB (X 2000) is full of cavities of porous structures. These pores are of different size and shapes and different than those of SCBF (Fig.4). These pores can be devolved from the thermal decomposition of cellulose and hemicellulose and left the cell walls which contain large proportions of lignin (Novotny *et al.*, 2015). The mean pore diameter of SCBBs are 2.31, 2.28 and 2.41 nm for traditional, 450 °C and 550 °C, respectively. SEM micrograph of SCBB produced at 550 °C and at magnification of 2000 contained longitudinal structure. The surface of this biochar contains slit-shaped pores containing particulates of different size. The wall thickness of traditional SCBB was less than biochar produced at 450 °C and higher than that produced at 550 °C which became very thin due to the effect of high temperature. It means that thermal degradation of cellulose and hemicellulose as the pyrolysis temperature increases.

The SEM images of RHF showed, at 2000 magnification, disturbed order of elongated pores. On the other hand, RHB produced at traditional, 450 °C and 550 °C showed developed pores (X 2000) as a result of thermal decomposition of RH structure and converted it to small particles. It seems that the walls of pores contain materials that disturb its regular order. Increasing pyrolysis temperature to 550 °C produced RHB of well-developed pores which are observed clearly at 2000 magnification. The SEM micrograph of traditional and 550 °C RHB exhibited a variety of shapes and sizes of micropores and macropores. Similar observations have been reported by Daffalla *et al.*, 2013. The regular pattern of pores was more observed in SCBB produced at traditional °C and in RHB produced at 550 °C. This variation could be due to the difference in the specific structure of raw SCB and RH feedstocks. Thus, by increasing pyrolysis temperature (from 450 °C – 550 °C), the structure of RHB became more ordered than SCBB and the pore diameter decreased with increasing pyrolysis temperature of RHB as follows: 2.66 nm (450 °C), 2.41 nm (traditional) and 2.31 nm (550 °C). Claoston *et al.* (2014) suggested that with increasing pyrolysis temperature, the number of micropores of RH biochar had decreased and that of macropores has increased. They also reported that the properly arranged pore structure possess high BET surface area and adsorptive capacity.

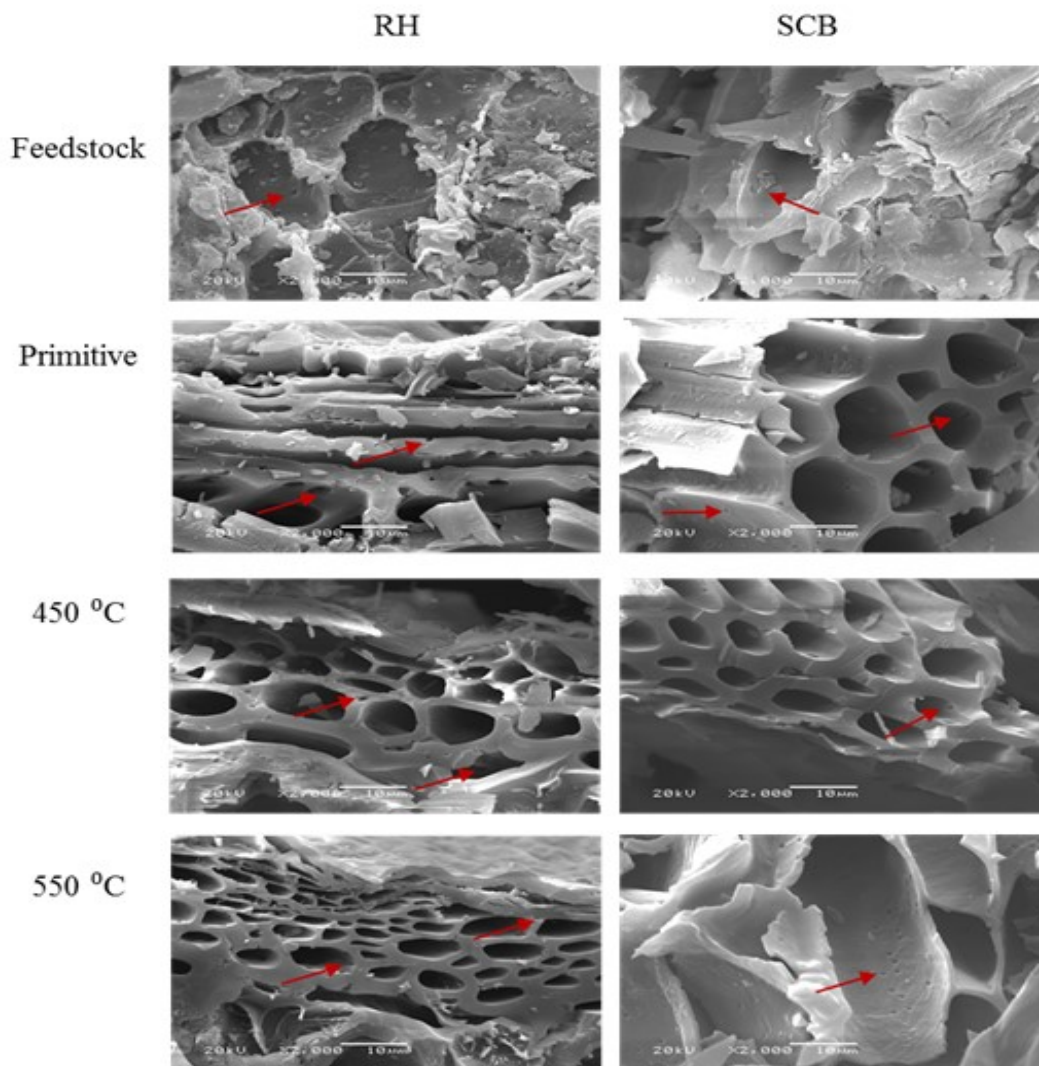


Fig 4. The SEM images of RH and SCB feedstocks and different biochars (traditional “primitive”, 450 °C, and 550 °C) representing the changes and voids formation on the surface of biochar samples

X-Ray Diffraction: The X-ray patterns for SCB feedstock and biochars (Fig. 5) showed a remarkable differences among both of them which indicate that phase transformation has been taken. The peaks at 2θ between 22° and 28° indicate the presence of cellulose which has a broad peak. The intense broad peak of cellulose in feedstock has been disappeared as a result of the thermal treatments. This may be due to the thermal degradation of cellulose in biochars. According to Govindarajan and Jayalakshmi (2011) carbon compounds present in the SCB are considerably removed at high temperature. The other peak at $2\theta \sim 26^\circ$, which indicates the presence of silica, is more sharp in biochars especially in traditional one. This may be

due to the recrystallization of amorphous silica with increasing temperatures (Tantawy et al., 2014).

The results of X-ray diffraction patterns of RH feedstock and its derived biochars made at (traditional, and of 450 °C, and 550 °C) did not show well-defined peaks. In all biomaterials, a peak is observed within the ranges from 15° to 30° , indicating disordered structure. XRD analysis of the feedstock and biochars confirmed the presence of amorphous silica as indicated by the heap at $2\theta \sim 22.5^\circ$ due to amorphous silica and quartz amorphous silica, which is a major constituent of these biomaterials, can be present in form of disordered cristobalite (SiO_2) (Singh et al., 2008 and Srivastava et al., 2006).

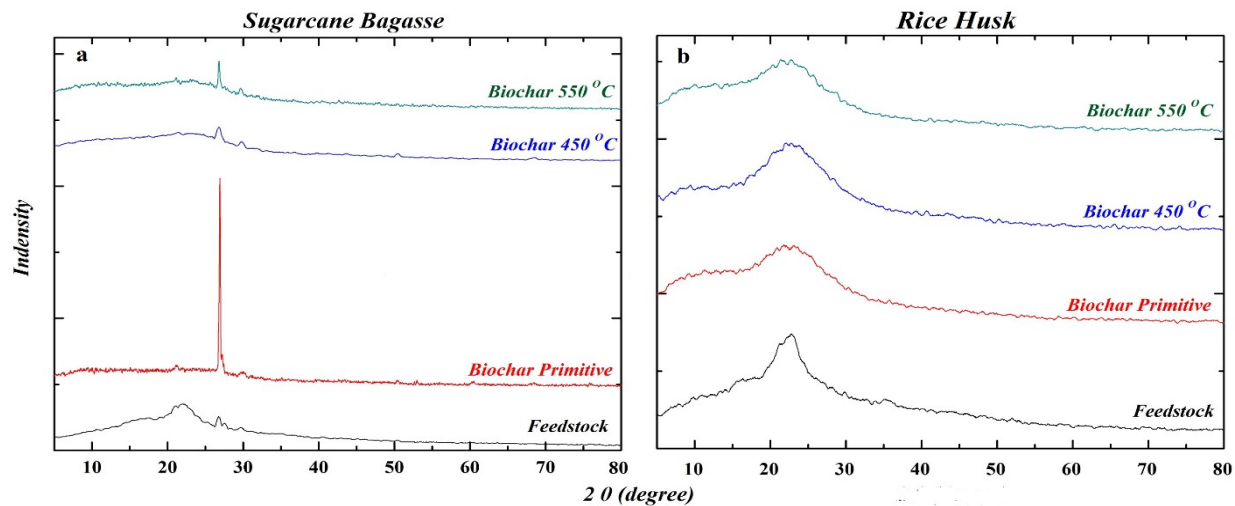


Fig. 5. XRD of feedstock and biochar produced at different pyrolysis conditions for Sugarcane Bagasse (a) and Rice Husk (b)

Chemical properties: Table 2 shows that, on the average, the pH value of SCBBs lower than in the RHBs. The value of pH of both biochars has increased with increasing pyrolysis temperature and both are with in alkaline range. The pH values of traditional SCBB and traditional RHB were higher than those produced at 450 °C and lower those that produced at 550 °C for both biochars. This result could be due to the high ash content in RHB than in SCBB (Novake *et al.*, 2009 and García-Jaramillo *et al.*, 2015). Increase the pH value of biochars due to increasing temperature could be attributed to the concentration of non-pyrolyzed inorganic elements (Novake *et al.*, 2009 and Jindo *et al.*, 2014) and also to decomposition of organic matrix and consequently of minerals such as alkali metals (ash) which are main cause of high pH (Ahmad *et al.*, 2012; Tsai *et al.*, 2012; and Al-Wabel *et al.*, 2013 and Claoston *et al.*, 2014). García-Jaramillo *et al.* (2015) reported that high pyrolysis temperature reduces the amount of carboxylic group, and the acidic groups and de-protonated to the conjugate basic nature, resulting in more alkaline pH of the biochar. On the average, the EC values of all biochars were less than unity and represent non-saline biochars. In general, EC was higher, in SCBBs than in the RHBs. With respect to the levels of DOC, there were no marked variations in the concentration of DOC in both biochars (Table 2). Due to low DOC content, indicate that biochar degraded very slowly in soil.

Table 2 shows that, on the average, SCBBs has higher values of CEC than those of RHBs. The pyrolysis temperature (traditional, 450 °C and 550 °C) did not affect markedly the value of CEC of SCBBs which

decreased in biochar produced at traditional than at 450 °C and 550 °C. While CEC of RHB produced at traditional and 550 °C was increased as compared to 450 °C with a value of relative 5.23 % and 9.17 %, respectively. However, Claoston *et al.* (2014) obtained different and opposite results with RHB who found that cation exchange capacity of RHB was decreased with increasing temperature.

Elemental Analysis: The N content in SCBB did not change markedly due to increasing of pyrolysis temperature (from 450 and 550 °C) but increased in the case of RHB. The N percentage in traditional biochars was higher in SCBB (0.76 %) than in RHB (0.68 %) and both were lower than N percentage in both biochars produced at 550 °C. However, high pyrolysis temperature (550 °C) produced biochars rich in N contents in both SCBB and RHB. On the other hand, increasing pyrolysis temperature from 450 to 550 °C decreased H percentage in RHB, while there was an increase in H content in SCBB with increasing temperature from 0.46 % to 0.64 % at 450 and 550 °C, respectively. As shown in Table 2. The percentage of H in traditional RHB (0.47 %) was lower than that of SCBB (0.80 %). The C contents in SCBB increased from 54.84 % to 67.92 % with temperature increase from 450 to 550 °C while, this trend was less observed for RHB. Traditional biochar of SCB contained higher C (51.37 %) than that of RHB (38.50 %). However, these two traditional SCBB and RHB contained the lowest C % as compared with C contents in both biochars of produced under controlled conditions at 450 and 550 °C (Table 2). It was observed an opposite trend in the O contents relative to C contents in both biochars.

Jindo et al. (2014) found that an increase in pyrolysis temperature led to in a large loss of O and H as compared to that of C which increases. The highest H/C atomic ratio was found in traditional SCBB (0.19) higher than those produced under controlled conditions at 450 °C (0.10) and 550 °C (0.11). This ratio was lower in traditional RHB (0.15) than in that of RHB of 450 °C (0.17) and was higher than that of RHB of 550 °C (0.13). These data showed that increasing pyrolysis temperature (from 450 °C to 550 °C) had increased H/C atomic ratio of SCBB (Table 2). This may be due to the loss of easily degradable C compounds such as volatile matter (Jindo et al., 2014). The O/C atomic ratio of traditional biochar had the highest ratio for SCB (0.64) and for RH (0.66) as compared with those of SCBB (0.56 and 0.30) and of RHB (0.52 and 0.41) which are produced at controlled pyrolysis conditions of 450 °C and 550 °C, respectively. Also, the low atomic ratio of O/C at the highest pyrolysis temperature can be attributed to a structural arrangement of the aromatic rings (Spokas et al., 2010) which form very stable graphite-like structure (Wu et al., 2012). And thus, the atomic ratio (O+N)/C has the same trend for both biochars produced under different pyrolysis conditions. The occurrence of high ratio of both H/C, O/C and (O+N)/C in traditional SCBB and RHB and relative to those produced under controlled conditions. These data indicate that these ratios are more interactive with polar compounds. This also indicates that traditional SCBB and RHB and those produced at low temperature (450 °C) are most polar at lower temperature than high temperature (Novake et al., 2009).

The percentage of S in SCBBs was almost higher than in RHBs (Table 2) and this percentage decreased with the increase of pyrolysis temperature from 450 °C to 550 °C in both biochars, while that of traditional SCBB was the highest (0.74 %). Silicon (Si) contents in biochars produced from SCBB at different condition were extremely lower than those produce from RHB (Table 2). The highest Si percentage, in SCBB or RHB, was produced at high pyrolysis temperature (550 °C). The Si percentage in traditional biochars were markedly close to those produced at 450 °C for both biochars (Table 2). These data confirm positive relation between ash content and Si content in biochars and that both properties are influenced by pyrolysis temperature since both are increased markedly with increasing pyrolysis temperature especially with respect RHB rather than SCBB (Table 2).

The P percentage in SCBB decreased with increasing pyrolysis temperature from 450 to 550 °C while, those of RHBs were approximately close at the two temperatures (450 and 550 °C). Table 2 showed

higher P percentage in traditional SCBB than in RHB and also in those of controlled SCBB and RHB. On the other hand, K contents decreased in SCBB with increasing temperature while it increased in RHB with increasing temperature. Traditional SCB and RH biochars contained approximately close K percentage. The percentage of Na in SCBB increased with increasing temperature while this percentage was approximately close in RH biochar. The traditional SCBB contained more Na (0.39 %) than traditional RHB (0.29 %).

Fe percentage of SCBB and RHB increased with increasing pyrolysis temperature, and they were higher in SCBB than in RHB produced under different pyrolysis conditions. However traditional RHB contained higher level of Fe than in those produced at 450 °C and 550 °C. On the other hand the content of Mn was higher value in SCBB at 450 °C than at 550 °C. The contents of Mn in RHB produced at these two temperatures were very close. The traditional SCBB and RHB contained approximately close Mn contents (54.35 and 57.85 mg/Kg, respectively). The content of Zn in SCBB decreased with increasing temperature and was higher at 450 °C than at 550 °C while the opposite was recorded for RHB which increased from 131.2 to 140.5 mg/Kg, at 450 and 550 °C, respectively. Traditional SCBB contained higher Zn than that of RHB. The lowest contents of micronutrients was Cu which showed no remarkable variations due to increase of pyrolysis temperature in both SCBBs and RHBs. It is clear that the percentage of Cu contents was higher in SCBBs than in RHBs which produced under different conditions. The nutrients in feedstocks (P, K, S, Na and Fe) were concentrated in the biochar and were higher in the biochars produced at traditional and/or 550°C.

CONCLUSION

The variation and heterogeneous in biochar properties based on the pyrolysis conditions and biomass type. The biochar produced in the field from air-dried feedstock using traditional pyrolysis unit under partially absence of the oxygen showed approximately biochar yield (from SCB or RH feedstocks) as those produced under controlled condition (550 °C). In general, the physical properties (ash, moisture content, total pore volume and mean pore diameters) were approximately close of those SCB and RH biochars produced under controlled condition. Furthermore, pH, EC, DOC, CEC, N and C content were approximately close in traditional biochar of SCB and RH with those biochars produced under controlled conditions. However, O content, H/C ratio, O/C ratio and (O+N)/C were higher in traditional biochar than in biochars of SCB and RH produced under controlled condition.

In general, the effect of pyrolysis conditions on biochar properties in this study followed the trend 450 °C > traditional > 550 °C. In general, depending on feedstock, traditional biochars have positive impact as nutrient sources as well as sequester C.

These results confirm the reliability of producing traditional biochar using pyrolysis unit in the field rather than producing under controlled conditions.

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الملخص العربي

مقارنة بين خصائص الفحم الحيوي المنتج تحت ظروف التحطم الحراري التقليدي والمتحكم فيه

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RHB. وأظهرت نتائج تحليل FT-IR وجود مجموعة OH الحرة ومجاميع C-H المشبعة في كل من SCBF و RHF والتي اختفت في الفحم الحيوي المنتج تحت ظروف التحطم الحراري المختلفة. كما أظهرت صور الميكروسكوب الإلكتروني SEM وجود المسام الطولية في المادة الخام لمصاصة القصب (SCBF) بينما يحتوي الفحم الحيوي له على بنية طولية وعدد أقل من المسام الدقيقة و كانت أكثر وضوحاً عند درجة حرارة ٥٥٠ °م، في حين أظهرت صور RHF تغير في نظام المسام الطولية حيث تغيرت إلى مسام أكثر تطوراً في RHB سواء عند التحكم في درجة الحرارة ٥٥٠ °م أوفى الظروف التقليدية. وأشارت بيانات تحليل XRD للمادة الخام لمصاصة القصب إلى وجود السليلوز والذي اختفى تماماً في الفحم الحيوي المنتج تحت الظروف المختلفة في حين تشير بيانات XRD للسرسة سواء للمادة الخام أو الفحم المنتج تحت الظروف المختلفة إلى وجود السيليكا غير المتبلورة والكوارتز. عموماً أظهرت النتائج أن خصائص الفحم الحيوي المنتج في الحقل تحت الظروف التقليدية باستخدام وحدة التحطم الحراري البدائية لا تختلف كثيراً عن خصائص الفحم الحيوي المنتج تحت الظروف المتحكم فيها (٥٥٠ درجة مئوية).

عادة ما يتم دراسة خصائص الفحم الحيوي الناتج من عملية التحطم الحراري لمخلفات المحاصيل الزراعية تحت ظروف متحكم فيها في أفران ذات طابع خاص يتيح القدرة للتحكم في درجة الحرارة والوقت. أما دراسة وتوصيف خصائص الفحم الحيوي الناتج تحت ظروف الحقل التقليدية فهي محدودة ولا يمكن الإعتماد عليها في التوصيف الدقيق للفحم المنتج. لذلك فإن الهدف من هذه الدراسة هو مقارنة الخصائص الفيزيائية والكيميائية للفحم الحيوي لأثنين من المخلفات الزراعية المختلفة هما مصاصة قصب السكر (SCBF) وغلاف حبة الأرز ("السرسة" (RHF)) المنتج تحت الظروف الحقلية التقليدية والظروف المتحكم فيها (درجات حرارة الإنحلال الحراري ٤٥٠ و ٥٥٠ °م). أما درجة حرارة عملية التحطم الحراري باستخدام وحدة بدائية الصنع كانت حوالي ٥٠٠ °م. وبصفة عامة أظهرت النتائج أن زيادة درجة حرارة الإنحلال الحراري أدت إلى انخفاض نسبة الفحم الناتج وزيادة نسبة المواد المتطايرة ومساحة السطح الكلية وحجم المسام الكلي التي كانت أعلى في السرسة (RHB) عن مصاصة القصب (SCBB) المنتج تحت الظروف المختلفة، وكانت قيمة pH في كلاهما في المدى القلوي، ونسبة الرماد والسيليكون أعلى في RHB عن SCBB، أما نسبة C% كانت أعلى في SCBB عن