



Change in nutrient composition of biochar from rice husk and sugarcane bagasse at varying pyrolytic temperatures

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Abstract

Purpose Waste management is one of the significant challenges facing Nigeria's agricultural sector. To manage this problem, the conversion of agricultural wastes to biochar is a practical solution.

Methods In this work, the chemical composition of biochar from rice husk and sugarcane bagasse, two predominant agricultural wastes, commonly generated in Nigeria was studied. These wastes were pyrolyzed at 300–700 and 350–700 °C for rice husk biochar (RHB) and sugarcane bagasse biochar (SBB), respectively.

Results It was found that biochar yield and total nitrogen decreased with increasing pyrolysis temperature while ash content, pH, EC, total carbon, extractable Ca, Mg, Na, available phosphorus and silica were increased. In RHB, extractable K increased with increasing pyrolysis temperature, while in SBB it decreased with its maximum observed in RHB.

Conclusions Rice husk pyrolyzed at high temperature may produce nutrient-rich biochar with high nutrient contents and these could ameliorate acidic soils. Finally, SBB could give high available silicon with acid pre-treatment.

Keywords Rice husk · Sugarcane bagasse · Pyrolysis temperature · Nutrient content · Biochar

Introduction

In Nigeria, human population has been increasing rapidly. In 2016, Nigeria's population was estimated to about 188.9 million (UNDESA/PD 2017). To sustain the food requirement for the teeming population, there is need for increase in food production. Food production increase means an increase in agricultural system and with increase in agricultural system a significant amount of waste will be generated (Walsh et al. 2000). It has been reported that about 140 billion metric tonnes of waste biomass are solely generated globally from agricultural activities every year (UNEP 2009). Rice husk is

one of the most widely available agricultural wastes in many rice producing countries of the world and produces about 20–23% by weight of the entire paddy processed (Kumar et al. 2013; Giddel and Jivan 2007). In Nigeria, for example, an estimated amount of about 1,032,993.6 metric tonnes of rice husk are generated annually (NAERLS/PCU 2004).

Sugarcane bagasse, a fibrous residual material derived from sugarcane after extracting its juice, is also a common agricultural waste. Traditionally, in the sugar mill bagasse is used as fuel in steam production for the milling process and electricity generation. In Nigeria, from the recent record, an estimate of 1.06 million tonnes of sugarcane were produced out of which about 30% of bagasse residue was generated by sugarcane processed in the mill (FOASTAT 2014). Not all bagasse produced are used; a substantial amount is left unused for the remainder of the year. The conversion of these wastes into biochar can result in advantages, such as sustainable waste recycling and carbon sequestration.

Biochar, a carbon rich and porous substance produced by thermal decomposition of biomass under oxygen-limited and relatively low temperature (< 700 °C) conditions, is receiving great research attention (Lehmann and Joseph 2009). This is due to its potential importance in agronomy

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and environment. Application of biochar on soil has been shown to have a clear effect on improving soil fertility, crop production and on decontamination of pollutants such as pesticides, heavy metals and hydrocarbon (Beesley et al. 2011; Cabrera et al. 2011). According to Lehmann et al. (2011), physico-chemical properties of biochar can cause changes in the soil nutrient and C availability and also provide physical protection to microorganisms against predators and desiccation. The type of biomass from which biochar is produced is another important factor. This is because its properties are affected by the nature of the original material. For instance, biochar from manure source possess higher soil cation exchange capacity than that from wood source (Singh et al. 2010). Again, treating soil with woodchip biochar results in higher saturated hydraulic conductivities than with manure-based biochar (Lei and Zhang 2013).

There has been a growing interest in the creation of recycling society in which wastes are steadily reduced. The technology that is used to produce biochar from waste started in antiquity, but recently there is a growing interest on pyrolysis. This is because pyrolysis is seen as an effective process for converting biomass into resources. The process of pyrolysis concentrates plant nutrients such as phosphorus, potassium, calcium, magnesium and sodium as heating temperature increases (Camps-Arbestain et al. 2015). Consequently, research has proven that even with the concentration of nutrients, percentage retention of alkaline earth metals is higher as opposed to the alkali metals. However, the release and retention of divalent alkaline earth metals are of great importance when biochar acts as a soil amendment (Okuno et al. 2005). Nitrogen is the most important nutrient for plant, but some parts (forms) of N are lost by pyrolysis. N containing organic materials when exposed to high temperature lead to the enrichment of the heterocyclic form of N such as pyrrole—type N with a decrease in amide N (Knicker and Hatcher 1997). According to Almendros et al. (2003), the microbial decomposition of the enriched N compounds is relatively recalcitrant which limit the conversion of these compounds to plant available form of N. Hongguang et al. (2017) also explained the importance of these enriched heterocyclic N compounds when he observed significant decrease on nitrogen loss from 109.6 mg kg^{-1} (0.01%) to 53.3 mg kg^{-1} (0.005%) in relation to increase in temperature on soils amended with biochar pyrolyzed at 250, 350, 450 and 550 °C.

However, not much research attention has been given on the extent to which different sources of biomass and pyrolysis temperature could increase the efficiency of applied biochar in soil sustainability and crop productivity. The use of biochar to ameliorate the soil physical properties for instance porosity and chemical properties for instance chelating nutrients is a function of biomass and pyrolysis condition as well as quantity (Kammann et al. 2011; Van Zwieten et al.

2009; Kudayarova 2007). Therefore, there is need to study the changes in chemical composition of biochar in relation to pyrolysis temperature. In this study, we tend to control the chemical composition of biochar by investigating different pyrolysis temperatures and agricultural wastes used as biomass. To achieve this aim, the chemical properties of biochar of rice husk at different temperature (300–700 °C) using continuous and plant-size carbonizer and sugarcane bagasse at different temperature (350–700 °C) using batch type carbonizer were evaluated.

Materials and methods

Biochar production

The biochar used in this work was obtained from the two agricultural waste residues: rice husk (*Oryza sativa* L.) and sugarcane bagasse (*Saccharum officinarum* L.). All the materials were first air dried and the sugarcane bagasse was cut into smaller pieces (less than 4–5 cm). Then, 10 kg of samples was inserted into a pilot batch type carbonizer (model ECO500, Meiwa Co. Ltd. Kanazawa) and pyrolyzed for 10 min after the inside chamber reached the target temperature.

Biochar chemical composition characterization

Prior to pyrolysis, the original biomass was analyzed for its chemical composition (Table 1). All samples were ground to less than 0.5 mm in diameter using manual agate

Table 1 Selected chemical properties of the original biomass prior to pyrolysis

Analyzed parameters	Biomass	
	Rice husk	Sugarcane bagasse
pH	6.5	5.1
Electrical conductivity (dS m^{-1})	0.33	0.32
Ash content (%)	11.5	4.0
Available phosphorus ($\text{P}_2\text{O}_5 \text{ mg kg}^{-1}$)	605	1167
Available silica ($\text{SiO}_2 \text{ mg kg}^{-1}$)		
0.1 M citric acid	295	398
0.5 M HCl	109	145
0.1 M CaCl_2	1004	753
Total carbon (g kg^{-1})	410.9	460.5
Total nitrogen (g kg^{-1})	2.7	2.7
Total calcium (mg kg^{-1})	509	1300
Total magnesium (mg kg^{-1})	222	518
Total potassium (mg kg^{-1})	2397	1939
Total sodium (mg kg^{-1})	636	512



mortar and pestle after the pyrolysis process. The biochar yield was calculated as the proportion of the weight of product to the original material. Ash content determination was conducted according to the American Society for Testing and Materials D1752-84. The ash content was thus determined by measuring the weight loss that follows the combustion of about 1 g of biochar in a crucible at 750 °C after 3 h. The pH and EC of biochar were measured in 1:20 w/v biochar–water extracts using pH and EC meters (Models D-15 and D-24 Horiba, Kyoto Japan, respectively) (modification in ratio from Ahmedna et al. 1997). Total carbon and nitrogen were determined by dry combustion method using automatic high sensitive NC analyzer (Model Sumigraph NC-22 Analyzer, Tokyo Japan). Total base cations: Ca, K, Mg and Na were determined after digestion of 0.05 g of biochar with HNO₃ at 160 °C for 5 h using Teflon vessel and measured by Inductively Coupled Plasma Spectroscopy (Model ICPE-9000, Shimadzu Kyoto Japan). Available P was extracted by NaHCO₃ and was determined by molybdenum blue method according to Olsen and Sommers (1982).

Extractable Ca, K, Mg and Na, which consist of water soluble and ionically exchangeable fractions, were extracted by shaking for 1 h 0.2 g of biochar in 20 ml of neutral CH₃COONH₄ and quantified on Inductively Coupled Plasma Spectroscopy (Model ICPE-9000, Shimadzu Kyoto Japan). Available silica from the biochar was extracted using 0.01 M CaCl₂ (1:30 ratio, continuous shaking for 16 h, filter) (modification in ratio from Haysom and Chapman 1975), 0.5 M HCl (1:50 ratio, shake for 1 h, filter) (modification in ratio from Savant et al. 1999) and 0.1 M Citric acid (1:50 ratio, shake for 2 h, allow to stay overnight then shake for 1 h, filter) (Acquaye and Tinsley 1964). Silicon concentrations from the supernatant were determined by molybdenum blue method and measured

with spectrophotometer (Model UV 1800, Shimadzu Kyoto Japan) at a wavelength of 810 nm.

Statistical analysis

The data were subjected to analysis of variance (ANOVA) using IBM package SPSS 20, while significant differences among the means were tested using Duncan's multiple range test ($P < 0.05$). The available P graphs were plotted using Microsoft excel 2007.

Results and discussion

Yield, chemical and elemental properties

Table 2 summarizes the chemical and elemental properties of RHB and SBB. As pyrolysis temperature increases, the yield of biochar from both rice husk and sugarcane bagasse decreased from 55 to 35% and from 27 to 22%, respectively. This was as a result of further disintegration of pyrolytic volatiles into low molecular weight organics and gases as heat passes through the biomass (Thangalazhy-Gopakumar et al. 2010). The relative ash content derived from different temperature ranges in the two biomass increases significantly ($P < 0.05$) as the pyrolysis temperature increased from 21.8 to 35.6% and 11.0 to 12.8% respectively. The ash content was higher in RHB. This was as a result of rich silica content found in rice plants, which relates strongly with the ash content in the biochar (Mukome et al. 2013). The increase could also be attributed to the progressive concentration of minerals and destructive volatilization of lingo-cellulosic matters as heat passes through the biomass (Tsai et al. 2012; Cao and Harris 2010).

The pH values of RHB and SBB ranged from 6.8 to 10.7 and 7.8 to 9.7, respectively (Table 2). The pH of the two

Table 2 The chemical and elemental properties of the biomass at different pyrolysis temperature

Biomass	Temperature (°C)	Biochar yield (%)	Ash content	pH (H ₂ O)	EC (dS m ⁻¹)	Total C (g kg ⁻¹)	Total N
Rice husk	300	55	21.8 ± 0.2e	6.8 ± 0.4c	0.09 ± 0e	512.9	4.5
	400	44	27.5 ± 0.3d	8.6 ± 0.2b	0.13 ± 0d	541.1	4.9
	500	36	32.8 ± 0.2c	10.4 ± 0.4a	0.23 ± 0c	546.7	4.7
	600	35	33.9 ± 0b	10.6 ± 0.1a	0.31 ± 0b	560.6	4.3
	700	35	35.6 ± 0a	10.7 ± 0.1a	0.40 ± 0a	545.0	3.6
Sugarcane bagasse	350	27	11.0 ± 0.6c	7.8 ± 0.6bc	0.11 ± 0e	693.4	6.0
	400	24	11.0 ± 0.4bc	8.3 ± 0.1b	0.12 ± 0d	707.3	5.7
	500	24	11.5 ± 0.5bc	9.2 ± 0.7a	0.16 ± 0c	729.5	5.3
	600	22	12.4 ± 0.1ab	9.6 ± 0.2a	0.17 ± 0b	702.2	4.7
	700	22	12.8 ± 0.2a	9.7 ± 0a	0.21 ± 0a	696.3	3.8

Values followed by the same letter within RHB or SBB columns were not significantly different at $P < 0.05$ according to Duncan's Multiple Range Test. Values represent mean ± standard deviation of ($n = 3$)

biochars increased significantly ($P < 0.05$) with increasing pyrolysis temperature and the maximum (10.7) was observed in RHB at 700 °C. This could be attributed to higher ash content at higher temperature and also due to the hydrolysis of salts of Ca, K and Mg as the pyrolysis temperature increases (Gaskin et al. 2008). The electrical conductivity (EC) which estimates the amount of total dissolved salts or the total amount of dissolved ions in the biochar varied from 0.09 to 0.40 and 0.11 to 0.21 dS m⁻¹ in RHB and SBB, respectively (Table 2). The EC of the RHB and SBB increased significantly ($P < 0.05$) with increasing pyrolysis temperature and the highest EC (0.40 dS m⁻¹) was observed in RHB at 700 °C. The increase was likely due to the loss of volatile materials as temperature increases while promoting the relative concentrations of salts in the ash fraction (Gai et al. 2014; Ding et al. 2010).

The elemental composition of the RHB and SBB is also presented in Table 2. The carbon content ranged from 512.9 to 560.6 g kg⁻¹ and from 693.4 to 729.5 g kg⁻¹ in RHB and SBB, respectively. The maximum (729.5 g kg⁻¹) was observed in SBB at 500 °C. The increased biochars' carbon content with increasing pyrolysis temperature indicates that pyrolysis temperature promotes carbonization (Chun et al. 2004; Chen et al. 2012). This promotion was due to high degree of polymerization leading to more condensed carbon structure in the biochar (Lehmann and Joseph 2009). The increase in carbon content could also be associated with H and O loss from biochar as the pyrolysis temperature increases (Antal and Gronli Antal and Grønli 2003). Total nitrogen content was found to decrease with increased pyrolysis temperature from 4.9 to 3.6 g kg⁻¹ and from 6.0 to 3.8 g kg⁻¹ in RHB and SBB, respectively (Table 2). This is because when plant biomass is subjected to pyrolysis, their N containing structures, i.e., amino sugars, amino acids and amines, which are part of nitrogen transformed into

heterocyclic N aromatic structures (Cao and Harris 2010; Koutcheiko et al. 2006). This implies that nitrogen will not be available to plants on the immediate but will be released with passage of time.

Base nutrient composition

Though there was an increase in the total concentration of base elements, there was no specific trend with increase in pyrolysis temperature (Table 3). They ranged from K (3105 to 4211 and 3753 to 4711 mg kg⁻¹), Ca (1012 to 1619 and 1922 to 3844 mg kg⁻¹), Mg (522 to 864 and 905 to 1711 mg kg⁻¹) and Na (509 to 1629 and 1203 to 2660 mg kg⁻¹) in RHB and SBB, respectively. The overall maximum K (4711 mg kg⁻¹ at 350 °C), Ca (3844 mg kg⁻¹ at 400 °C), Mg (1711 mg kg⁻¹ at 350 °C) and Na (2660 mg kg⁻¹ at 350 °C) were all observed in SBB. The increase found in the total base elements after pyrolysis of the original biomass is an indication that the relevant chemical components were concentrated in biochar as heat passed through the crop residues (Yuan et al. 2011). The loss of volatile compounds of the original materials after thermal degradation yielded the remaining nutrient content in the biochars (Amonette and Joseph 2009; Chan and Xu 2009).

Pyrolysis significantly altered the extractable nutrient elements in both RHB and SBB at $P < 0.05$. Although, the extractable Na in SBB showed no significant difference, a change in its composition was observed with increasing pyrolysis temperature (Table 3). The extractable K and Mg of RHB increased with increasing pyrolysis temperature while extractable Ca and Na showed no specific trend. They ranged from K (165 to 684 mg kg⁻¹), Ca (67 to 103 mg kg⁻¹), Mg (16 to 29 mg kg⁻¹) and Na (21 to 38 mg kg⁻¹). In SBB, the extractable K (Table 3) decreased significantly with increasing pyrolysis temperature from 396

Table 3 The base nutrient composition of the biomass at different pyrolysis temperature

Biomass	Temperature (°C)	CH ₃ COONH ₄ extractable elements				Total elements			
		K	Ca	Mg	Na	K	Ca	Mg	Na
		mg kg ⁻¹							
Rice husk	300	165 ± 2e	67 ± 7d	16 ± 4c	21 ± 1c	3357	1411	716	1596
	400	380 ± 4d	103 ± 6a	19 ± 3bc	32 ± 8a	4211	1619	864	1629
	500	537 ± 3c	90 ± 7bc	21 ± 1bc	26 ± 0bc	3628	1240	631	700
	600	608 ± 7b	84 ± 4c	23 ± 1b	29 ± 5ab	3122	1012	522	603
	700	684 ± 6a	101 ± 1ab	29 ± 0a	38 ± 1a	3105	1027	585	509
Sugarcane bagasse	350	396 ± 3a	170 ± 2b	18 ± 2a	26 ± 4a	4711	3721	1711	2660
	400	378 ± 4b	167 ± 2b	16 ± 2a	25 ± 5a	4417	3844	1656	2028
	500	332 ± 6c	153 ± 5c	13 ± 0b	23 ± 2a	3753	1922	905	1368
	600	304 ± 9d	137 ± 4d	13 ± 0b	22 ± 2a	4287	2053	986	1590
	700	251 ± 1e	180 ± 7a	16 ± 1a	24 ± 3a	3951	2253	949	1203

Values followed by the same letter within RHB or SBB columns were not significantly different at $P < 0.05$ according to Duncan's Multiple Range Test. Values represent mean ± standard deviation of ($n = 3$)



to 251 mg kg⁻¹ while extractable Mg, Ca and Na decreased from 350 °C (18, 170 and 26 mg kg⁻¹) to 600 °C (13, 137 and 22 mg kg⁻¹) and afterwards an increase was observed at 700 °C (16, 180 and 24 mg kg⁻¹). The overall maximum Ca (180 mg kg⁻¹) was observed in SBB at 700 °C while maximum K (684 mg kg⁻¹), Mg (29.0 mg kg⁻¹) and Na (38 mg kg⁻¹) were observed in RHB at 700 °C. Extractable K data obtained indicate that for K release, higher temperature is required. This is because as heat passes through the biomass during combustion, they tend to induce higher effective reaction temperature. This leads to an increase in vapor pressure and the convective flux of KCl (Johansen et al. 2011). At higher temperature in RHB, sublimated KCl was not volatilized but rather combines with silicate to form potassium silicate (Muthadhi et al. 2007). In SBB, a decrease in extractable K was observed with increasing pyrolysis temperature, which could be as a result of higher diffusional resistance during devolatilization due to still intact organic matrix as heat passes through the biomass. (Van Lith et al. 2008).

Available phosphorus

In RHB, phosphorus availability significantly increased (225 to 1842 mg kg⁻¹) with increasing pyrolysis temperature at $P < 0.05$ (Fig. 1). Though SBB is statistically significant at $P < 0.05$, it was found to have irregular trend with increasing pyrolysis temperature (Fig. 2). In biochar, its P content is a function of the type of biomass and pyrolysis condition (Downie et al. 2009). Generally, charring enhances P availability from plant tissue. This is because with combustion, there is disproportionate volatilization of carbon which leads to cleavage of organic phosphorus bonds and hence yields biochar with high soluble phosphorus salts (Knoepf et al. 2005). This was in line with the continuous increase of available P observed in RHB with increasing pyrolysis temperature. In sugarcane bagasse biochar (SBB), the trend of

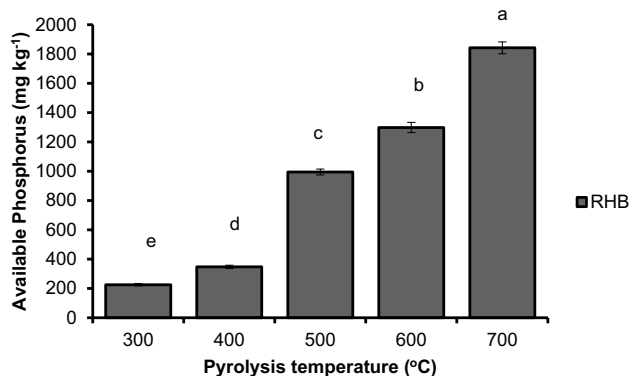


Fig. 1 Available phosphorus (Olsen P) concentration of rice husk biochar (RHB)

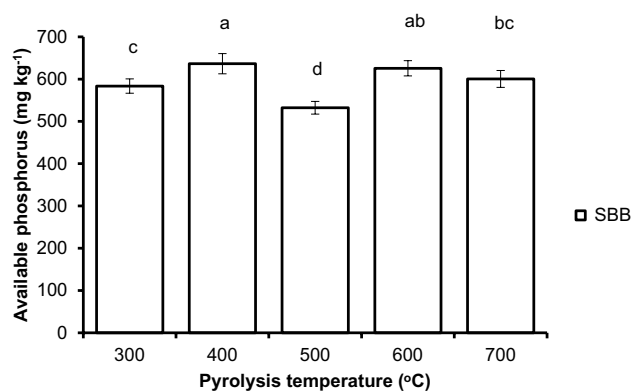


Fig. 2 Available phosphorus (Olsen P) concentration of sugarcane bagasse biochar (SBB)

the phosphorus bioavailability was not specific. Part of the reason might be because cation-phosphate complexes which exist in biochar strongly influence phosphorus reactivity and bioavailability (Hunger et al. 2008; Güngör et al. 2007). In this study, data obtained suggest that there is every possibility that Ca/Mg-P complexes dominated P availability. Again, the change might be because diverse forms of organic phosphorus (OP) have diverse levels of mobility. For instance, the OP bond to organic matter containing abundance of *O*-alkyl groups (e.g., phytic acid) is more labile than those containing ample refractory alkyl and aromatic carbon groups (e.g., nucleic acid and phospholipid) (Hamdan et al. 2012). Hence, the production of biochar can alter the fate of P by changing the form of P in agricultural residues.

Available silicon

Available silicon of the biomass at $P < 0.05$ significantly increased with increasing pyrolysis temperature in both RHB and SBB (Table 4). In RHB, 0.01 M CaCl₂ extracted silicon ranged from 61 to 3124 mg kg⁻¹ with its maximum (3124 mg kg⁻¹) at 700 °C. In SBB, the trend of increase continued from 350 to 500 °C (266 to 343 mg kg⁻¹); afterwards, there was a decrease at 600 °C (275 mg kg⁻¹). In 0.5 M HCl and 0.1 M citric acid extracted silicon, the range found in RHB was (86 to 475 and 16 to 418 mg kg⁻¹) and SBB was (236 to 7262 and 207 to 9313 mg kg⁻¹), respectively. The maximum of 0.5 M HCl and 0.1 M citric acid was observed in SBB (7262 and 9313 mg kg⁻¹), respectively, at 700 °C. The increase obtained with increasing pyrolysis temperature was as a result of change in the form of silica upon heating. It is reported that the amorphous form silica changes into crystalline form when heat is applied (Todkar et al. 2016; Parry and Smithson 1964). This form change generally reduces the availability of silicon. However, our results showed reverse trend. It is probably due to the short heating



Table 4 The available silicon of the biomass at different pyrolysis temperature under different extraction methods

Biomass	Temperature (°C)	0.1 M CaCl ₂	0.5 M HCl (SiO ₂ mg kg ⁻¹)	0.1 M citric acid
Rice husk	300	61 ± 3e	86 ± 4e	16 ± 2e
	400	1306 ± 12d	117 ± 3d	55 ± 4d
	500	2952 ± 51c	190 ± 10c	157 ± 6c
	600	2800 ± 12b	263 ± 11b	214 ± 5b
	700	3124 ± 77a	475 ± 30a	418 ± 21a
Sugarcane bagasse	350	266 ± 10b	236 ± 4d	207 ± 7d
	400	333 ± 8a	259 ± 7d	306 ± 5d
	500	343 ± 5a	2074 ± 20c	2946 ± 40c
	600	275 ± 3b	4686 ± 117b	5391 ± 145b
	700	279 ± 4b	7262 ± 135a	9313 ± 137a

Values followed by the same letter within RHB or SBB columns were not significantly different at $P < 0.05$ according to Duncan's Multiple range test. Values represent mean ± standard deviation of ($n = 3$)

time, i.e., 10 min. The differences observed with the different extraction methods might be a function of the chemical composition of the biomass. The amount of available silicon extracted from RHB with acidic extraction method was low when compared with SBB. Part of the reason might be because rice husk upon charring produces about 70–80% reactive SiO₂ while sugarcane is about 40–50% (Ganesan et al. 2007). According to Patcharin et al. (2009), introduction of acid can increase the amount of silica extracted from bagasse by the removal of its alkali and alkaline metals. It is viewed that CaCl₂ extraction method extracts only the soluble fraction of silica (Berthelsen et al. 2001), while that with citric acid or diluted acid (example H₂SO₄) extract a combination of both soluble and adsorbed silica fractions (Beckwith and Reeve 1964). This is because acidic ions compete not only with silicic acid in the sorption sites but also formed complexes with metallic ions that are known to bind silicic acid. In the present study, we could not find out the reason why available silicon in RHB was not extracted by acid solutions (HCl and citric acid).

Conclusion

The data indicated that both the pyrolysis temperature and biomass significantly affect the chemical compositions of the biochar. In all, an increase in temperature leads to the generation of biochar with high nutrient content. Developing an innovative process, which can generate high temperature, would be a key priority for the local communities to improve the productivity and quality of the produced biochar. RHB at high temperature may produce nutrient-rich biochar, with high pH, EC, ash content, available silicon, phosphorus, extractable base cations for acidic soil amendment. Finally, SBB could give high available silicon with acid pre-treatment.

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