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Exploring the Quality of Biochar derived from Simarouba Seed Coat

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ABSTRACT: The study was conducted to investigate the influence of pyrolysis temperature on quality parameters and recovery of biochar from Simarouba seed coat biomass waste. Fixed-bed (batch) slow pyrolysis reactor was used to produce biochars at three different pyrolysis temperatures- 300°C (BC_{300}), 400°C (BC_{400}), and 500°C (BC_{500}) with 4 hours residence time per cycle. The biochar yield exhibited a significant decline, ranging from 78.15% at 300°C to 39.23% at 500°C. To unravel quality variations among the resulting biochars, proximate and nutrient analyses were conducted, coupled with an assessment of physiochemical properties such as bulk density (BD), maximum water holding capacity (MWHC), pH, and electrical conductivity (EC). With rising pyrolysis temperature, pH, EC, elemental carbon, and fixed carbon content showed consistent increase. BC_{500} significantly shows higher pH (8.67), EC (1.53 dS m^{-1}), total carbon (70.46%) and fixed carbon content (81.47%) compared to BC_{300} and BC_{400}. Whereas, BC_{500} showed the higher MWHC (270.79%) and lower BD (0.28 Mg m^{-3}) indicative of enhanced porosity. BC_{500} also exhibited enriched concentrations of potassium (K) and calcium (Ca) (4.58% and 1.33% respectively), while nitrogen (N) concentration was lowest (1.12%). The high-temperature biochars with elevated pH, MWHC, and K-content could be beneficial for remediating acidic soils, enhancing water retention in sandy soils, and ameliorating K-deficient soils.

Keywords: Biochar recovery, Quality parameters, Pyrolysis temperature, Simarouba biomass-waste, Nutrient content.

INTRODUCTION

Simarouba (*Simarouba glauca* DC.) belonging to family Simaroubaceae, is a multipurpose evergreen tree. The tree thrives in warm, humid tropical climates with temperatures ranging from 10 to 40°C and annual rainfall ranging from 500 to 2200 mm. It can grow at elevations from mean sea level to 1000 m above mean sea level in all types of well-drained soil with pH range between 5.5-8.0 and minimum soil depth of 1 m. The tree can grow well even in the degraded soils. The tree grows to a height of 12 to 15 m tall and has a crown spread of 7.6 to 9.1 m wide. The average yield of fruit from a hectare of a 10-year-old plantation of Simarouba is about 6000 to 8000 kg (Joshi and Hiremath 2001). Simarouba seed consists of 71 per cent woody seed coat and 29 per cent kernel. The seed kernel has higher non-edible oil content (61.04%) which makes it an appropriate candidate for biodiesel production (Dash *et al.*, 2008). After recovery of kernels for biodiesel production, seed shells (containing no oil) are obtained in huge quantity which are considered as waste. Eco-friendly technologies like combustion, torrefaction, gasification, and pyrolysis, convert crop residues into biochar (Basu, 2018). The most popular, easy-to-use, and efficient method among the several treatments is pyrolysis that produces three products: solid-biochar, liquid-bio-oil, and gas-syngas.

Biochar is a carbon-rich substance produced by pyrolysis of agricultural or forest biomass characterized by its porous structure, higher stability, and capacity to enhance soil quality and sequester carbon. By using biochar, agriculture can shift from being a net carbon emitter to a way of restoring carbon stocks in the soil. This is because biochar has a durable and robust structure that makes it more resistant to biochemical processes. Consequently, it can persist in the soil for extended durations, spanning for several years. Biochar improves soil pH, available nutrients, and soil organic matter content. Due to greater cation exchange capacity (CEC), biochar efficiently prevents nutrient leaching and retains most of the essential nutrients. Biochar itself contains various nutrients and trace elements essential for crop growth. Biochar due to its physio-chemical properties stimulates root development of plants, leading to improved nutrient and water uptake, which ultimately contributes to improved crop growth and yield.

Quality of the biochar with respect to its physical parameters and nutrient content depends on many factors such as feedstock type, feedstock moisture content, pyrolysis temperature, pyrolysis time, heating rate, particle size and other pyrolysis process parameters which play important role in deciding recovery and quality of biochar. Temperature during pyrolysis was found to have a substantial impact on stability and physicochemical properties of biochar, however heating duration during biochar synthesis did not have any significant effect on the properties of biochar (Zhang *et al.*, 2015). Changes in the structure and physicochemical characteristics of biochar are closely linked to the temperature at which pyrolysis takes place (Jindo *et al.*, 2014). Pyrolysis temperature can decide a wide range of pH, electrical conductivity, maximum water holding capacity, bulk density, available nutrients, and total carbon content of biochar (Chan *et al.*, 2008). So, the study aimed to understand the relationship between pyrolysis temperature and biochar quality with respect to its agricultural application which is essential for optimizing biochar production processes.

MATERIAL AND METHODS

Feed-stock collection and preparation. Simarouba seed coat *i.e.*, feedstock for pyrolysis was collected from Biofuel Park, Agricultural Research Station, Madenur, Hassan, Karnataka, India. The feedstock was shade dried until moisture content of the coats was reduced up to 6.28 per cent (*i.e.*, below 10 per cent, is ideal for pyrolysis). Then the seed coats were shredded to 0.2-2 mm size using mechanical biomass shredder and used for biochar production.

Biochar production. In a fixed-bed (batch) slow pyrolysis reactor, slow pyrolysis of Simarouba seed coat was performed at three different pyrolysis temperatures ranging from 300 to 500°C with a constant heating rate, 10°C min^{-1}. The residence time was fixed for four hours and the material was run six times at three different temperatures that made up to a total of 18 runs. The treatment details were given by producing biochars at three different temperatures- 300, 400 and 500 °C and named as BC_{300}, BC_{400}, and BC_{500} respectively.

Biochar recovery analysis. The biochars obtained were weighed using an electronic weighing scale and their yield percentages were calculated using the formula (Sahoo *et al.,* 2021),

Biochar yield (%) = Mass of biochar (kg) / Mass of the raw material (kg) × 100

Bio-oil yield was measured in terms of litres and converted into kilograms by multiplying the value by its density (1.2 kg m^{-3}). The syngas yield was calculated by using formula (Irfan *et al.*, 2016)

Syngas yield (kg) = 100 – [Biochar yield (kg) + bio-oil yield (kg)]

Biochar quality analysis. Biochars produced at different temperatures were ground and passed through a 0.2 mm sieve and used to analyse their physiochemical properties, including bulk density (BD), maximum water holding capacity (MWHC), pH, electrical conductivity (EC), and nutrient analysis. Standard procedures used for the analyses are given below.

Proximate analysis of biochars. Proximate analyses of biochars for estimation of moisture content, volatile matter, ash content and fixed carbon was carried out as per ASTM D1762-84 method suggested by International Biochar Initiative (IBI), (Igalavithana *et al.*, 2017).

Physiochemical analysis of biochars. For determination of pH and EC of biochar samples, method suggested by IBI was employed (Igalavithana *et al.*, 2017). While for determination of biochar bulk density and maximum water holding capacity, Keen's cup method (Piper, 1966) was used.

Nutrient analysis of biochars. To determine the total carbon content of the biochars, the dry combustion method (CHNS, LECO) was used (Page *et al.*, 1982). The biochar samples were tested for nitrogen (N) content by using Kjeldahl digestion & distillation method, for phosphorus (P) content by following diacid digestion and Vanadomolybdate phosphoric yellow colour method and for potassium (K) content by diacid digestion and flame photometer method (Jackson, 1973).

The Versenate titration method, as outlined by Jackson (1973), was used to determine the calcium (Ca) and magnesium (Mg) content of the di-acid digested biochar samples. As described by Piper (1966), the turbidometry method was used to detect the sulphur (S) level at a wavelength of 420 nm using a spectrophotometer.

Statistical analysis. The data on various parameters recorded during the investigation were tabulated and subjected to statistical analysis using one-way ANOVA. The test of significance (‘F’ test) and critical difference (CD) were read at 0.05 probability (Panse and Sukhatme, 1967). Wherever ‘F’ test was found significant, the ‘t’ test was performed to estimate critical differences among different treatments.

RESULTS AND DISCUSSION

Biochar recovery. The feedstock was slow pyrolyzed at three different temperatures (300, 400, and 500ºC) in a fixed-bed (batch) pyrolysis reactor. The biomass underwent pyrolysis, producing three distinct products: solid biochar, liquid bio-oil and a gas called syngas. The change in pyrolysis temperature showed a significant impact on yield of all the three pyrolysis products. The yield of these three products were measured in terms of kilograms (kg) and the data pertaining to biochar recovery is given in Table 1. By taking reference of obtained quantities of these products and feedstock used for pyrolysis cycle, assessed recovery percentage are given in Fig. 1.

The yield of biochar was significantly reduced as the pyrolysis temperature increased. Biochar production through slow pyrolysis yielded 10.16 kg of biochar at 300 °C from 13 kg of feedstock (Table 1), which was equivalent to 78.15 per cent of the feedstock weight (Fig. 1). At temperature 400°C, the biochar yield was decreased to 7.54 kg (58%), and at 500°C, it further decreased to 5.10 kg (39.23%). The results of current findings were in accordance with Elnour *et al.* (2019), reported that the yield of biochar from date palms decreased from 43 to 32 per cent with rise in pyrolysis temperature from 300°C to 700°C. Similar trend was reported by Suliman *et al.* (2016); Kim *et al.* (2012).

Table 1: Quantity of pyrolysis products obtained from Simarouba seed coats at different temperatures through slow pyrolysis.

Feedstock per pyrolysis cycle (kg)	Pyrolysis temperature (°C)	Heating rate (°C min^{-1})	Residence time per cycle (h)	Biochar yield (kg)	Bio-oil yield (kg)	Syngas yield (kg)
13	300	10	4	10.16	1.71	1.13
13	400	10	4	7.54	4.00	1.46
13	500	10	4	5.10	6.00	1.90
S. Em±				0.07	0.05	0.06
C.D @ 5%				0.21	0.14	0.18
C.V (%)				2.28	2.98	2.12

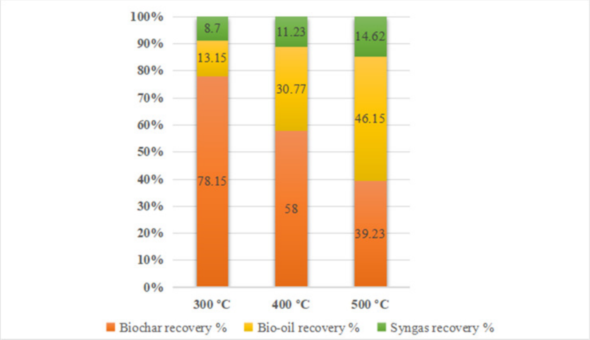

Fig. 1. Recovery percentage of pyrolysis products obtained from Simarouba seed coats at different temperatures.

Unlike the biochar yield, there was a substantial increase observed in yield of bio-oil and syngas. Specifically, at 300 °C, 1.71 kg of bio-oil, equivalent to 13.15 per cent (by weight), was obtained. This yield increased to 4.00 kg (30.77%) at 400 °C and further to 6.00 kg (46.15%) at 500 °C. While the syngas yield was 1.13 kg (8.7%) at 300 °C, 1.46 kg (11.23%) at 400°C, and 1.90 kg (14.62%) at 500 °C, showing a significant rise with increasing temperature. The thermal breakdown of cellulose, hemicellulose, and lignin into syngas and bio-oil at higher pyrolysis temperatures results in the reduction of biochar yield and increase in bio-oil and syngas yield (Yue *et al.,* 2016). Al Arni (2018) pyrolyzed sugarcane bagasse at three different temperatures (480, 580 and 680 °C) and concluded that, by increasing temperature, yield of syngas increased for slow pyrolysis.

Proximate analysis of biochars. Proximate analysis includes determining the amount of moisture, volatile matter, fixed carbon, and ash in biochar. The proximate analysis found all these parameters statistically significant for the three biochars (Table 2).

Moisture content in biochars ranged from 1.24 to 4.14 per cent, least of which was observed in BC$_{500}$ (1.24%) and maximum in BC$_{300}$ (4.14%). Further, Higher volatile matter content was observed in BC$_{300}$, reaching 23.06 per cent. Conversely, lower volatile matter content was noted in BC$_{500}$, measuring 8.64 per cent. There was a consistent decrease in volatile matter content with temperature increase, suggesting a gradual loss of volatile matter during the charring process. Zhang *et al.* (2015), reported that as pyrolysis temperature increased, biochar yield and volatile matter decreased.

Higher ash content (8.65%) was recorded in the BC$_{500}$. In contrast, lower ash content (5.12%) was recorded in BC$_{300}$. While in BC$_{400}$, the ash content was intermediate (6.34%) to the two temperatures. The ash content of biochar serves as an indicator for the components of biochar that are neither volatile nor combustible, as reported by Angin and Sensoz (2014). With the elevation in temperature, it can be expected that the mineral concentration would rise, and there would be an increase in the detrimental volatilization of lignocellulosic substances. Consequently, this would lead to a higher ash content, as suggested by Tsaia *et al.* (2012).

As the pyrolysis temperature rose, the fixed carbon content of the biochar increased significantly. It varied from 67.68 to 81.47 per cent. Higher fixed carbon was found in BC$_{500}$ (81.47%), intermediate in BC$_{400}$ (77.16%) and lower in BC$_{300}$ (67.68%). Noor *et al.* (2019) found that when the temperature was elevated from 350°C to 600°C at a constant heating rate of 5°C min^{-1}, the fixed carbon content increased from 45.20 to 79.09 per cent.

Table 2: Proximate analysis of biochars produced at different temperatures through slow pyrolysis.

Treatments	Moisture content (%)	Volatile matter (%)	Ash (%)	Fixed Carbon (%)
BC$_{300}$	4.14	23.06	5.12	67.68
BC$_{400}$	2.02	14.48	6.34	77.16
BC$_{500}$	1.24	8.64	8.65	81.47
S. Em±	0.06	0.30	0.17	0.35
C.D @ 5%	0.17	0.91	0.50	1.07
C.V (%)	3.64	2.82	3.02	1.15

Physicochemical analysis of biochars. pH. Table 3 shows that there was a significant increase in pH of biochar with rise in pyrolysis temperature. The pH of the biochar samples ranged from acidic to alkaline (6.64 to 8.67). BC_{300} showed lower and acidic pH of 6.64. In contrast, BC_{500} showed higher and alkaline pH of 8.67. While BC_{400} had nearly neutral pH (7.27). The alkalinity of biochar is primarily caused by rising pyrolysis temperatures that decreases acidic functional groups and increases alkali salts and functional groups (Mukherjee *et al.*, 2011). Wan *et al.* (2014) showed that pH, carbonate content, base cation, and biochar alkalinity increased with temperature rise in pyrolysis. Biochars elevated pH was due to hydrolysis of the Ca, Mg, and K salts. An additional element leading to the elevation of pH with rise in pyrolysis temperatures was the proportional rise in ash content present within the biochar. Several studies have demonstrated a correlation between increased ash content and higher pH levels as the temperature of pyrolysis rises (Nwajiaku *et al.*, 2018; Wakamiya *et al.*, 2022; Novak *et al.*, 2009).

Electrical conductivity (EC). With rise in pyrolysis temperature, EC of biochar increased significantly from 0.97 (BC_{300}) to 1.53 dS m^{-1} (BC_{500}) (Table 3). Higher EC was seen in BC_{500} (1.53 dS m^{-1}). The increase in EC of biochar was attributed to greater production of ash with rise in temperature and higher salt concentration of the produced ash (Nwajiaku *et al.*, 2018). Similarly, Al-wabel *et al.* (2013) noted that the EC of biochar rose with an increase in pyrolysis temperature, as evidenced by EC values of 0.76 dS m^{-1} and 1.34 dS m^{-1} for biochar produced at 200 ºC and 400 ºC, respectively.

Bulk Density (BD). Pyrolysis temperature showed a significant influence on biochar bulk density (Table 3). With rise in temperature, there was a significant reduction in bulk density indicating increased porosity of biochar. The MWHC of low-temperature pyrolyzed biochar types was reduced because of their lower pores, lower interconnectivity pores, and residual tar components that clog the biochar pores (Useviciute and Baltrenaite 2021). BC_{300} showed highest bulk density of 0.36 Mg m^{-3}, BC_{400} showed lesser bulk density of 0.30 Mg m^{-3} and the lowest bulk density of 0.28 Mg m^{-3} was found in BC_{500}. Dhar *et al.* (2022) observed that the bulk density exhibited a decline from 0.55 to 0.39 Mg m^{-3} with rise in pyrolysis temperatures from 350 °C to 600 °C.

Maximum water holding capacity (MWHC). All the three biochars produced at different temperatures showed higher value of MWHC ranging from 192.67 to 270.79 per cent (Table 3). There was a significant increase in water holding capacity of biochar with rise in pyrolysis temperature. Higher water holding capacity was seen in BC_{500} (270.79%) and lowest in BC_{300} (192.67%). Useviciute and Baltrenaite (2021); Yabi Gadi *et al.* (2023) reported that, biochars produced at lower temperature showed lower pores, inter-connectivity pores and more residual tar components blocking biochar pores which ultimately reduces its MWHC.

Table 3: Physicochemical properties of biochars produced at different temperatures.

Treatments	pH	EC (dS m^{-1})	BD (Mg m^{-3})	MWHC (%)
BC_{300}	6.64	0.97	0.36	192.67
BC_{400}	7.27	1.33	0.30	227.93
BC_{500}	8.67	1.53	0.28	270.79
S. Em±	0.04	0.01	0.006	2.43
C.D @ 5%	0.12	0.03	0.019	7.38
C.V (%)	1.28	2.05	1.81	2.56

Nutrient analysis of biochars

Total carbon (%). There was a significant increase in total carbon content of biochar with rise in pyrolysis temperature (Table 4). It ranged from 52.39 to 70.46 per cent. The BC_{500} showed higher carbon content (70.46%), followed by BC_{400} (62.82%), and lower in BC_{300} (50.39%). Zheng *et al.* (2013), noted that, when the temperature rose from 300 °C to 500 °C, the total carbon content of the biochar made from giant seeds increased from 65.26 to 78.61 per cent. This increase was attributed to increase in degree of structural modification and carbon stability due to carbonization reactions in biochar with increasing pyrolysis temperature.

Primary nutrient content of biochars. The biochar primary nutrient content showed significant variation with increase in pyrolysis temperature (Table 4). Biochar N content reduced from 1.25 (BC_{300}) to 1.12 per cent (BC_{500}) with rise in pyrolysis temperature. In BC_{400} the N content was 1.14 per cent. This might be because nitrogen starts to volatilize at relatively low temperatures, around 200 °C due to its association with many organic molecules. (DeLuca *et al.*, 2015). Similar results were found by many studies (Zheng *et al.*, 2013; Zhang *et al.,* 2015; Yuan *et al.,* 2016; Elnour *et al.*, 2019; Zhang *et al.,* 2020). Biochar P content showed no significant variation among the three biochars, in contrast to N.

Biochar potassium (K) content increased significantly with rise in pyrolysis temperature. The K content of biochar was 3.61, 3.81 and 4.58 per cent at temperature 300, 400 and 500 °C, respectively. These results of present study are in accordance with DeLuca *et al.* (2015); Manish *et al.* (2022). Who observed that, K concentration of the biochar rose as the pyrolysis temperature rose, and this could be because K is associated with various inorganic minerals, which require higher temperature (700 °C to 800 °C) to get volatilized. Further, Zheng *et al.* (2013) also reported that, K content of giant seeds biochar increased significantly as the temperature rose from 300 to 600 °C.

Table 4: Total carbon and primary nutrient analysis of biochars produced at different temperatures.

Treatments	Total Carbon (%)	N (%)	P (%)	K (%)
BC_{300}	52.39	1.25	0.048	3.61
BC_{400}	62.82	1.14	0.049	3.81
BC_{500}	70.46	1.12	0.050	4.58
S. Em±	0.49	0.01	0.001	0.04
C.D @ 5%	1.50	0.03	NS	0.11
C.V (%)	1.95	1.80	1.302	2.20

Table 5: Secondary nutrient analysis of biochars produced at different temperatures.

Treatments	Calcium (%)	Magnesium (%)	Sulphur (%)
BC_{300}	0.95	0.28	0.23
BC_{400}	1.00	0.23	0.23
BC_{500}	1.33	0.22	0.21
S. Em±	0.06	0.05	0.01
C.D @ 5%	0.18	NS	NS
C.V (%)	3.07	4.46	3.26

Secondary nutrient content of biochars. With rise in pyrolysis temperature, the calcium content increased from 0.95 (BC_{300}) to 1.33 (BC_{500}) percent (Table 5). Zheng *et al*. (2013) reported that, as the temperature rose from 300 to 600 °C, the Ca content of the biochar made from giant seeds significantly increased since Ca needs a very high temperature (above 1000 °C) to volatilize (DeLuca *et al.*, 2015). However, as the pyrolysis temperature increased, the magnesium (Mg) and sulphur (S) content of the biochar did not change significantly.

Many studies have reported that with increase in pyrolysis temperature, plant nutrients in biochar become less available to plants (Tag *et al.*, 2016; Yuan *et. al.*, 2016 Zornoza *et al.*, 2016). This could be the result of dehydration and decarboxylation decreasing the amount of ion exchange functional groups (Glaser *et al.*, 2002; Shenbagavalli *et al.*, 2023). Therefore, when compared to biochars produced at higher temperatures, biochars produced at lower pyrolysis temperatures can more effectively improve crop growth and yield.

CONCLUSIONS

The study shows that, there is significant reduction in the yield of Simarouba seed coat biochar with rise in pyrolysis temperature. Also, temperature change had a significant impact on the biochar quality parameters. When compared to biochar produced at lower temperature, biochar produced at higher temperature showed higher pH, maximum water holding capacity, and potassium content. These high temperature pyrolyzed biochars can be useful in acidic soil remediation, improving water holding capacity of sandy soils and for potassium deficient soils as a natural source of fertilizer.

FUTURE SCOPE

Present study demonstrate to convert bulk agri-biomass waste as a resource. Generally these agri biomass was burnt in rural area causing environmental pollution and also resulted in reduced incorporation of organic carbon to soil. Pyrolysis technology converts biomass into biochar in the absence of oxygen with bi-products of bio-oil and producer gas as renewable eco-friendly energy sources. Biochar has wide application in soil stabilization by providing congenial condition in the soil for biochemical reaction to elevate soil health for better microbial activities with better soil physical conditioning for higher productive system.

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