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PRODUCTION AND CHARACTERIZATION OF TOBACCO STALK BIOCHAR

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Pyrolysis temperature and time are known to have profound influence on biochar yield. The optimum parameters of pyrolysis for preparation of biochar from the tobacco stalk biomass are not known. An attempt has therefore been made to find out the optimum pyrolysis process parameters for preparation of biochar from tobacco stalks. The representative tobacco stalk biomass was subjected to thermo-chemical conversion at different temperatures (350 – 500^{0}C) and holding time (60-90 min). Tobacco stalk biochar yield decreased with an increase in temperature and holding time. System operating conditions of 500^{0}C temperature and 90 min holding time were optimum to ensure the complete charring of the biomass and maximum biochar yield. The biochar yield at optimized pyrolysis conditions was around 40% and had 74.9 % fixed carbon, 17.5% ash and remaining 7.6% as volatile matter. Total C and N content of the tobacco stalk biochar was 79 and 1.23%, respectively. While, the total C, N, P, K, Ca, Mg and micronutrient contents were higher in tobacco stalk biochar than in raw biomass. The amount of C conserved in the biochar was 70%. The recovery of total N was the lowest at 55%, while that of other major nutrients remained more or less same at about 80%. Our results indicate the optimum conditions for better biochar recovery from tobacco stalks along with nutrients.

Key words: Biochar, Crop residue, Pyrolysis, Tobacco stalk

INTRODUCTION

Pyrogenic carbonaceous material (PCM) is defined as any carbonaceous residue resulting from pyrolysis. Char is the PCM produced from natural fires. Charcoal is PCM produced from pyrolysis of animal or plant biomass in kilns for use in cooking or heating, including industrial applications such as smelting. Biochar is carbonaceous material produced specifically for application to soil for agronomic or environmental management (Lehmann and Joseph, 2015). International Biochar Initiative, the IBI (2013) defines biochar as a solid material obtained from thermo-chemical conversion of biomass in an oxygen-limited environment. Biochar can potentially play a major role in the long-term storage of carbon in soil, *i.e.*, C sequestration and green house gases (GHG) mitigation. The efficiency of biochar depends upon the material from which it is prepared. Biochar in soils persists against biological and chemical degradation over much longer periods of time because of its recalcitrant nature. The objective of this work is to produce and characterize biochar from the tobacco stalk biomass, an agri-waste having no known economic value.

MATERIALS AND METHODS

Tobacco stalk biomass was manually cut to appropriate size (average 30 cm in length and 1.0 to 1.5 cm in diameter). Fresh samples were stored and left to sundry naturally to moisture content below 10%. Dry bioresidue is a prerequisite to hasten satisfactory and quicker conversion. Representative tobacco biomass samples were tested for chemical composition.

Five experiments with varying operating conditions of temperature (350 – 500^{0}C) and holding time (60-90 min) were conducted at ICAR-CIAE, Bhopal, in order to optimize the annual core biochar reactor operating conditions for producing tobacco stalk biochar. The details of experiment are given in (Table 1).

Table 1: Optimization of tobacco stalk biochar production parameters

S. No	Temperature	Time (min)	Recovery (%)	Condition
Exp. 1	350^{0}C	60	66.0	Torrified
Exp. 2	400^{0}C	60	66.0	Torrified
Exp. 3	450^{0}C	60	60.0	Torrified
Exp. 4	450^{0}C	90	53.3	80% charred
Exp. 5	500^{0}C	90	40.0	Completely charred

The dry biochar was homogenized thoroughly, manually ground to pass through 2 mm sieve prior to analyses. The biochar samples were oven dried at 105^{0}C for 24 h. The pH of the biochar in 1:20 suspension (w/v) was measured using a pH meter (Systronics pH system 362). The electrical conductivity (EC) of biochars was measured at room temperature after suspending biochar in deionised water for 24 h (1:10 biochar to deionised water) using a EC meter. Cation exchange capacity of the biochar was determined by saturating the biochar exchange complex with 1N sodium acetate solution (pH 8.2). One g of biochar sample was leached with sodium acetate solution (pH 8.2) for replacement of exchangeable cations by Na^+ ions. The excess salts were washed down by ethanol and the adsorbed Na^+ ions were released by NH_4^+ ions, using 1N ammonium acetate (pH 7.0) solution. The Na^+ ions so released from the exchange spots were measured by using flame photometer. Total organic carbon (C) content was determined directly by dry combustion on TOC analyzer (Elementar, Germany). Total Nitrogen concentration was estimated using N distillation unit. Concentrations of total P, K, Ca, Mg, Fe, Cu, Zn and Mn in biochar were determined by digesting 0.5 g of each biochar sample in a di-acid mixture (HNO_3:$HClO_4$ in 3:1 ratio).

Biochar yield from the tobacco stalk biomass was calculated by the following equation (Antal and Groni, 2003).

Biochar yield (%) = $(M_{biochar} / M_{biomass}) \times 100$ eq-1

Where, $M_{biochar}$ is the mass of biochar obtained after conversion and $M_{biomass}$ is the dry mass of the original tobacco stalk biomass loaded into the reactor.

Proximate analyses were conducted for biochar to estimate the percentage volatile matter (VM), ash content and fixed C, on an oven dry-weight basis. The percentages of VM, ash and fixed C were estimated by measurement of weight loss/mass balance from a sequential muffle procedure. The VM content of the biochar was determined by heating the biochar in a covered ceramic crucible to 700 C ignition for 10 min using laboratory muffle furnace. The samples were withdrawn, weighed and measured weight loss and it was defined as volatile matter (VM), and the residual solid was carbonized biochar. Ash content was determined by heating the carbonized biochar residue of the VM determination in an open crucible via combusting at 700 C for 2 h. The percentage fixed carbon, volatile matter (VM) and ash content of the biochar were calculated using the following equations (Antal and Groni, 2003).

Volatile matter (%) = $(M_{biochar} - M_{cc}) / M_{biochar} \times 100$ eq-2

Where, $M_{biochar}$ was the initial dry mass of biochar, M_{cc} was dry mass of the carbonized biochar that remained after heating.

Ash content (%) = $(M_{ash} / M_{biochar}) \times 100$.... eq-3

Where, M_{ash} was the dry mass of ash remained after combustion of the carbonized biochar, $M_{biochar}$ was the initial dry mass of biochar.

Fixed carbon (%) = 100 – VM (%) – Ash (%)....eq-4

Where, VM is the volatile matter content of biochar.

All the chemical analyses for different parameters were performed in triplicate and the results presented as mean ± standard errors

RESULTS AND DISCUSSION

Production of tobacco stalks biochar and optimization of biochar production parameters

Results on the biochar yield from tobacco stalk biomass at different process variables (temperatures and holding time) of the biochar reactor were presented in Table 1. At low temperatures and small holding time charring of the biomass was not complete while, with an increase in temperature and holding time the material was transformed from torrified to charred condition. The optimum conditions for complete charring of tobacco stalk biomass were attained at a temperature of 500 ^{0}C and holding time of 90 minutes with the yield recovery of 40%. Biochar yield tended to decrease with increase in reactor temperature and holding time.

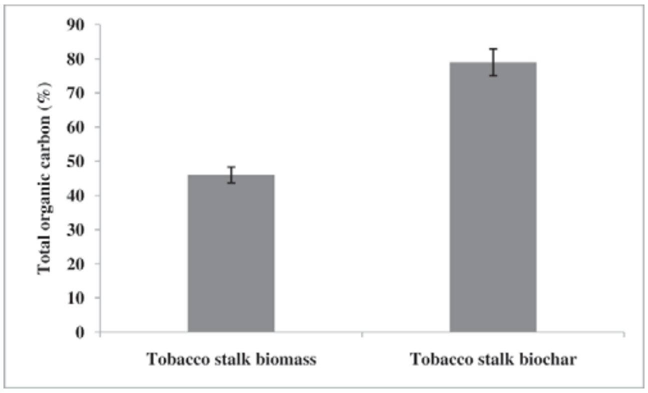

Figure 1: Total organic carbon content of tobacco stalk biomass and tobacco stalk biochar

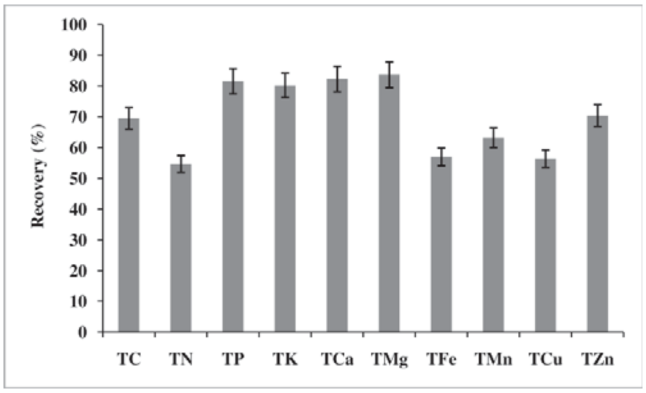

Figure 2: Per cent recovery of nutrients in tobacco stalk biochar (T = total)

Carbon and nutrient composition of tobacco stalk biomass and biochar

Tobacco stalk biomass contained 45.5% total organic C, 0.9% nitrogen, 0.25% P, 1.9% K, 1.02% Ca and 0.43% Mg (Table 2). The concentration of micronutrients in the biomass varied from 3.8 ppm (Mn) to 82.5 ppm (Fe). The pyrolysis process giving 40% biochar yield lead to enrichment of the carbon and nutrients in the end product (biochar) owing to mass reduction by 2.5 times. This is evident from the higher concentration of C and other nutrients in biochar than in original tobacco biomass (Table 2). The tobacco stalk biochar contained 79% total organic carbon, 1.23% N, 0.51% P, 3.81% K, 2.1% Ca and 0.9% Mg. The concentration of micronutrients in the biochar

Table 2: Average nutrient concentrations of feedstock (tobacco stalk) and biochar (tobacco stalk biochar)

Parameters	Tobacco stalk biomass	Tobacco stalk biochar
Total organic carbon	45.5 ± 1.86	79 ± 3.03
Nitrogen (%)	0.90 ± 0.01	1.23 ± 0.01
C:N Ratio	51:1	67:1
Phosphorus (%)	0.25 ± 0.02	0.51 ± 0.01
Potassium (%)	1.90 ± 0.04	3.81 ± 0.09
Calcium (%)	1.02 ± 0.03	2.10 ± 0.01
Magnesium (%)	0.43 ± 0.03	0.90 ± 0.04
Iron (ppm)	82.50 ± 2.46	117.50 ± 4.49
Manganese (ppm)	3.80 ± 0.10	6.00 ± 0.16
Zinc (ppm)	11.00 ± 0.49	15.50 ± 0.68
Copper (ppm)	4.55 ± 0.18	8.00 ± 0.54

varied from 6 ppm (Mn) to 117.5 ppm (Fe). Quality of feedstock source influences end-product characteristics. In general, most plant based biochar contain elevated C content and the same was also observed in the case of tobacco stalk biochar. The increase in total organic carbon (Fig.1) content of the biochar could be attributed to pyrolysis process where in several chemical reactions takes place resulting in creation of thermally stable fixed C structures (Spokas *et al.*, 2012).

It was clear from the results depicted in Fig. 2, that the amount of carbon conserved from biomass to biochar (% recovery) was 70%. The loss of C under higher temperatures of pyrolysis may be attributed to volatilization of carbon bonded to volatile chemical constituents as reported earlier by Kloss *et al.* (2011). The per cent recovery of nitrogen in the biochar was the lowest (55%) compared to that of all other nutrient elements. The lower N recovery may be attributed to the loss of N compounds during pyrolysis process at higher temperatures. Novak *et al.* (2009) suggested that the decrease in N during higher production temperature can be attributed to aromatization. Further, Gaskin *et al.* (2008) showed that the amount of total N conserved from biomass to biochar ranged from 27.4 to 89.6% in poultry litter and pine chip biochars, respectively. Per cent recovery values in respect of P, K, Ca, Mg (80%) and other micronutrients (60-65%) in the biochar, though relatively greater than that of N, still indicate a substantial loss of nutrients during the process of pyrolysis. During the pyrolysis or oxidation process that generates biochar, heating causes some nutrients to volatilize, especially at the surface of the material, while other nutrients become concentrated in the remaining biochar. Therefore, biochar could act as a soil conditioner and could propel nutrient transformations than serving as a primary source of nutrients (Glasser *et al.*, 2002; Lehmann *et al.*, 2003).

The other important characteristic of biochar was its C: N ratio. The C: N ratio of tobacco stalk biochar produced in the present study was 67:1. In contrast, the C:N ratio of the raw tobacco stalk biomass was 51:1. The C: N ratio of biochar depends not only on relative quantities of the total C and N, but also on relative degree of their loss during the pyrolysis. The wider C: N ratio in the tobacco stalk biochar as compared to that of its biomass indicates preferential volatilization of nitrogen over carbon. This is in agreement with the findings of Shanbagavalli (2012) and Venkatesh *et al.* (2013), who reported wider C/N ratios in maize stover biochar (90:1), groundnut shell biochar (70:1), coir waste biochar (89:1), prosopis wood biochar (83:1) and cotton stalk biochar (40:1 to 62:1).

Physico-chemical characteristics of tobacco stalk biochar

The data on other physical characteristics (Table 3) indicate that the tobacco stalk biochar was alkaline in reaction, with pH of 9.42. The pH of tobacco stalk biochar was greater than that of other reported biochars such as groundnut shell biochar (9.3), prosopis wood biochar (7.57), cotton stalk biochar (8.9 – 9.3) *etc.* (Shenbagavalli, 2012; Venkatesh *et al.*, 2012). The higher pyrolysis temperature was known to have an impact on biochar pH. Higher pyrolysis temperature (500^{0}C) in the present study may have made tobacco stalk biochar more basic. The higher pyrolysis temperatures are reported to remove acidic functional groups and elevate ash content thereby causing biochar to be more basic (Novak *et al.*, 2009). Similar result was observed in the present study where tobacco stalk biochar has recorded alkaline pH and an ash content of 17.47%. Higher temperature during the conversion process had the strongest influence on the biochar pH suggesting that higher temperature might have resulted in higher degree of volatilization, decomposition of surface oxygen groups and dehydroxylation contributing to increased ash residue portion in the biochar (Hass *et al.*, 2012).

Table 3: Characteristics of tobacco stalk biochar

Parameter	Tobacco stalk biochar
pH (1:20)	9.42
EC (dS/m) (1:10)	0.11
CEC (C mol (+)/kg	30.0
Ash (%)	17.47
Volatile matter (VM) (%)	7.61
Fixed carbon (%)	74.92

Cation exchange capacity is also an important characteristic of biochar. Similar to soils, biochar cation exchange capacity (CEC) represents its ability to electrostatically sorb or attract cations. Tobacco stalk biochar had a CEC of 30 C mol (p+) / kg and was well within the range of CEC values reported by Venkatesh *et al.* (2012) for cotton stalk biochar (11.7 to 51.3 C mol (p+) / kg).

The biochar produced from tobacco stalks had 74.9 % fixed carbon, 17.5% ash and remaining 7.6% as volatile matter. Contents of these components in the tobacco stalk biochar appear to be more or less similar in cotton stalk biochar with C 58.7-71, ash 18.1 to 30.2 and 7.9-15.3% as volatile matter. High fixed carbon in tobacco stalk biochar suggests that it is more beneficial as soil amendment, particularly to improve soil physical environment.

The tobacco stalk biochar (having completely charred residue) can be optimally prepared by the pyrolysis at a process temperature of 500^0C and a holding time of 90 min. The biochar yield obtained was about 40% of the dry tobacco stalk biomass. Conversion of tobacco stalk biomass to biochar resulted in recovery of carbon and nutrients to the extent of more than 60%, with the exception of nitrogen for which the recovery was 55%. The biochar produced from tobacco stalks was alkaline in nature and had C:N ratio of 67:1. Tobacco stalk biochar also had relatively high proportion of fixed carbon (74.9%) and thereby indicating its suitability as soil amendment for improving the soil physical environment.

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