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Biochar production through drum method and characterization for soil amendment qualities

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Abstract
Biochar preparation using portable drum technology is an affordable strategy for efficient use of unused and excess crop residues. The aim of this study was to prepare biochar from pigeonpea and cotton residues at farm level and characterising their soil amendment qualities. The fabricated drum for the preparation of biochar suits the needs of small farmer. The slow pyrolysis (350 to 400 ºC temp. range) process yields 24.5 and 22.6% of biochar, correspondingly 16.2 and 18.5% ash content from pigeonpea and cotton residues respectively. The physico-chemical properties of both biochars were compared for assessing soil amendment qualities. The pH of pigeonpea and cotton biochar is 9.86 and 9.82 respectively, which indicates the suitability of both biochar for soil acidity amelioration. Bulk density of pigeonpea and cotton biochar is 0.26 and 0.29 g/cm^{-3} respectively. the lower BD indicating more pore space, which leads to better soil aeration and more water holding capacity. The carbon recovery of pigeonpea (27.6%) and cotton (29.0%) biochar after thermo-chemical conversion indicates the carbon sequestration potential of both biochar in the background of climate change. Besides, both biochar material contains small concentration of major plant nutrients which improve soil fertility. Therefore, production of biochar under modified drum method suits the small farmer. Besides the properties of both pigeonpea and cotton biochar has greatest soil amendment qualities for soil application.

Keywords: Biochar production, drum method, pigeonpea, cotton, soil amendment

Introduction
The total amount of biomass produced annually in India is about 550 mt, of which 141 mt were estimated to be surplus after accounting for multiple uses (MOA, 2012) [10]. Most of the surplus residues are subjected to on farm burning in India (MNRE, 2009; Jain *et al.*, 2014) [9, 6]. Among different crops, oilseeds (29 mt), pulses (13 mt) pigeonpea (57 mt) and cotton (53 mt) generate maximum crop residues in India, which are advertently niche crops for rainfed areas. The surplus crop residues of pigeonpea and cotton are estimated to be 9.0 and 11.8 mt, respectively. These residues, when left unattended, often disrupt land preparation and crop establishment. Therefore, burned on-farm, which causes environmental problems and substantial nutrient losses. For effective management of these crop residues, their conversion into biochar through thermo-chemical process (pyrolysis) is gaining importance as novel alternative technology. Much of the stimulus for this interest has come from research on soils of the Amazon basin known as Terra Preta soils, related to high content of organic carbon in the form of char (Glaser *et al.*, 2001) [4].
In this scenario, biochar offers a significant, multidimensional opportunity to convert excess agricultural waste into valuable soil amendment (Bera *et al.*, 2018) [3]. Use of biochar in agricultural systems is a viable option which enhance natural rates of carbon sequestration, reduce the hitherto excess crop residues into useful material for enhancing soil health and crop productivity (Srinivasarao *et al.*, 2013) [16]. Many researchers have characterised the biochar derived from different crop residues in India. Purakaystha *et al.*, (2015) [13] reported that, maize and rice straw biochar has nutrient value and carbon stability, hence, can be utilized for enhancing soil fertility and long term carbon sequestration. Similarly, Venkatesh *et al.*, (2013a and b) [21] reported, operational process for preparation of castor and cotton biochar and their characterization for use as soil amendment. However, in Rayalseema region of southern Andhra Pradesh, considerable quantity of pigeonpea and cotton residues are available; as these crops are major rainfed crops in the region. Using locally available crop residue for preparation of biochar provides unique opportunity to smallholding farmers of rainfed region to improve soil health.

Biochar plays a potential role in rainfed agriculture for improvement of soil physical properties, improved retention of nutrients and soil moisture, consequently enhanced crop yields (Venkatesh *et al.*, 2018) [22]. Every biochar has its own characteristic which ultimately influences the crop growth after field application. But, little published information is available for the preparation of biochar from pigeonpea and cotton crop residue at the local farmer level by using locally available resources. Thus the objective of this study is to standardise the preparation process at farmer level by using local resources and characterise the derived biochar for its soil amendment qualities.

Materials and Methods

The experiment was conducted during the year 2018-19 at Agricultural Research Station, (14º 41' E Latitude; and 77º 40’ N Longitude) Anantapur, Acharya N.G. Ranga Agricultural University. Andhra Pradesh.

Biochar production by Drum method (slow pyrolysis)

In the present study, the portable and locally available drum was fabricated (Two drums used; one for pigeon pea and another drum for cotton stalks) for the thermo-conversion process (Fig 1). The cylindrical metallic drum of approximately 200 litre capacity was selected and made perforated base with intact bottom. The top section was with openable vent for loading the biomass into drum kiln. The top portion was welded with 3 iron sticks for holding top cover while closing the drum. The size of the kiln or drum was about85 cm height and 55 cm radius. The general view of the production process was presented in Fig 1 and 2.

Fig 1: Field operational process of biochar production under drum kiln**:** (A) General view of biochar drum Kilm, (B) Welded iron sticks at top vent, (C) Bottom view of drum with holes, (D) Drum loaded with feedstock, (E) Drum ready for pyrolysis, (F) Drum kiln under combustion process.

Pre configuration and seasoning of crop residues

Freshly harvested pigeonpea (Cajanas cajan (L.) Millsp.) stalks collected from Agricultural Research Station, Anantapur and cotton (Gossypium spp) stalks were collected from local farmer’s field. Both the pigeon pea and cotton stalks were manually chapped separately and screened to appropriate size (average 20-25 cm length and 10-15 mm diameter). This was done to load the biochar kiln with crop residue of pigeon pea and cotton stalks in uniform manner and also for the uniform heat transfer between the crop residues during the thermal conversion process (Fig 2). The stalks were sundried separately to bring the moisture content below 10%. The sundried stalks are prerequisite to hasten satisfactory and quicker thermal conversion. Simultaneously, representative pigeon pea and cotton stalks were stored for nutrient content analysis.

Thermo-chemical process (Pyrolysis)

The sundried crop residues were loaded into the drum kiln chamber for combustion process. Screened stalks of pigeonpea and cotton stalks were manually packed separately in the drum kiln. Stalks were packed parallel to the bottom of the drum in as much as possible quantity by gentle shaking. The weights of empty and loaded drums filled with pigeon pea and cotton stalks were taken separately. Then the loaded kiln was lifted and placed over the hearth of three flat stones of about 30 cm height to facilitate the air flow through the vents created at bottom side of the drum (Fig 1). Sufficient quantity of the locally available dry twigs was used for combustion at the bottom firing point of the drum. This has to be done very carefully to raise the temperature for spontaneous ignition followed by continues thermo-conversion process. The pyrolysis process attained the temperature about 350 to 400 ºC. the process continues for 1.2 to 1.3 hrs. The end stage of the bio carbonization was indicated by distinctive thin grey and blue coloured gases will leaked out from the top vent. At this stage, the top cover edges have to be sealed with clay mixture to restrict the air flow in to the drum/combustion chamber for significant biochar yield recovery. Then the partial combustion of crop residues continues in the inner side of the drum kiln. Later the drum kiln was transferred to levelled surface to cease the primary air ingression and also cut-off partial combustion process. Then allow the drum kiln to cool for nearly four hours in the open atmosphere. After cooling, the sealed clay mixture was removed and biochar produced inside the drum kiln was taken out, weighed and stored (Fig 2). The process was conducted in similar manner for both pigeonpea and cotton stalks separately. The entire process is one run for the biochar production under locally prepared drum kiln and repeat the process for enough quantity of biochar.

Fig 2: Biochar produced using Drum kiln**:** (A) Seasoned pigeonpea stalk, (B) Pigeonpea stalk biochar (whole), (C) Pigeonpea stalk biochar (pulverised), (D) Seasoned cotton stalk, (E) Cotton stalk biochar (whole), (F) Cotton stalk biochar (pulverised).

Analysis of Biochar

The biochar produced from pigeonpea and cotton stalks was homogenized and manually ground to pass through 2 mm sieve. The samples collected for laboratory analysis were oven dried at 105ºC for 24 hr. The pH of biochar in water was determined at 1:20 (W/V) ratio after continuous shaking over 1 hr, using pH meter. The EC of the biochar was determined using electrical conductivity meter. Total Carbon and Nitrogen content of biochar was determined by dry combustion on CN analyser (Vario EL Cube, Elementar). The concentrations of total phoshprus and potassium was analysed with an inductively coupled plasma spectrometer. The bulk density of biochar was measured by core method (Veihmeyer and Hedrickson, 1948) [19]. Particle density of biochar was determined by pycnometer method (Hernandez-Mena *et al.*, 2014) [5]. The biochar recovery (%) from the resulting carbonized products were calculated by taking the difference of masses before and after conversion using the equation: Biochar Yield (%) = ($m_{biochar}$ / m_{straw}) x 100. Where, $m_{biochar}$ is the mass of biochar obtained after thermos conversion process and m_{straw} was the dry mass of the raw crop residue loaded into the drum kiln (Antal and Groni, 2003) [1]. The proximate analyses were conducted for biochar. The Ash content was determined as out lined by Yuan *et al.*, (2011) [23] Ash (%) = (Wt. ash / Wt. biochar) x 100.

Results and discussion

Production of Biochar by drum (kiln) method

Biochar was produced from two different biomasses *viz*., pigeonpea and cotton stalks using the same pyrolysis condition (Fig 1 and 2). A portable low cost drum was fabricated for the production of biochar with available local resources for easy operation of local farmers. Two separate drums were used for pigeonpea and cotton stalks. Before loading into drum kiln, biomass was pre-configured and seasoned to facilitate uniform combustion process (Srinivasrao, *et al.*, 2013; Venkatesh *et al.*, 2018) [22]. Colour changes in the exhaust was considered for approximation of kiln temperature (350 to 400 ºC) by using the correlation given in USAID, (1984) and Tillman *et al.*, (1981) required for the slow pyrolysis.

Biochar yield derived from pigeon pea and cotton residues

The biochar yield from feedstock and corresponding ash content, volatile matter and fixed carbon are the proximate analysis of biochar produced. Proximate analysis results are presented in Table 1 and graphically depicted in Fig 3. The results revealed that, yield of biochar from pigeonpea stalks (24.5%) is higher than cotton stalks (22.6%). The varying yield among two biochars obtained may be attributed to feedstock type used and the pyrolysis condition (Shaon Kumar Das *et al.*, 2018) [15]. Previous studies also described that, the varied proportions of cellulose, hemicellulose and lignin content in the feedstock influences the conversion of residue into biochar yield. In addition, the load of the kiln and pyrolysis temperature strongly influences the biochar yield (Srinivasrao, 2013; Venkatesh *et al.*, 2013a) [21].

The ash content is critical in the production process, as its quantity alters the product distribution in terms of yield of gas, char and bio-oil (Patwardhan *et al.*, 2010). The cotton biochar (18.5% wt.) having higher ash content than pigeonpea biochar (16.2% wt.). However, in the present study (Table 1), the ash content in both biochars is in the acceptable range of biochars produced from non woody feedstock for use as soil amendment (Ronsse *et al.*, 2013). The moisture content of pigeonpea biochar is 4.48% compared to 3.91% of cotton biochar is due to residue type used. Bera *et al.*, (2018) [3] reported in their study that, ash content less than 20% under low temperature influences the volatile matter. The similar results were obtained in present study as indicated by volatile matter content of pigeon pea (5.85%) and cotton (6.90%). According to Bera *et al.*, (2018) [3] under the slow pyrolysis process of temperature 350 to 400 ºC, polyaromatic graphene sheets with greater carbon concentration begins to increase at the expense of amorphous carbon pool in the process. However, the fixed carbon content of pigeonpea (63.84%) and cotton (62.69%) does not change much but it may increase slightly as per the temperature range (Table 1). Hence, under the slow pyrolysis process a series of devolatilization reactions occurs and leads to more condensed carbonaceous matrix in the resulted biochar (Bera *et al.*, 2018; Ronsse *et al.*, 2013) [3, 14].

Table 1: Biochar yield and ash content under thermo - chemical conversion process

Biochar properties	Pigeonpea Biochar	Cotton Biochar
Biochar yield (%)	24.5 ± 0.06	22.6 ± 0.15
Ash (%)	16.2 ± 0.04	18.5 ± 0.02
Moisture content (%)	4.48 ± 0.08	3.91 ± 0.05

Values are given as mean ± standard deviation for triplicate measurements

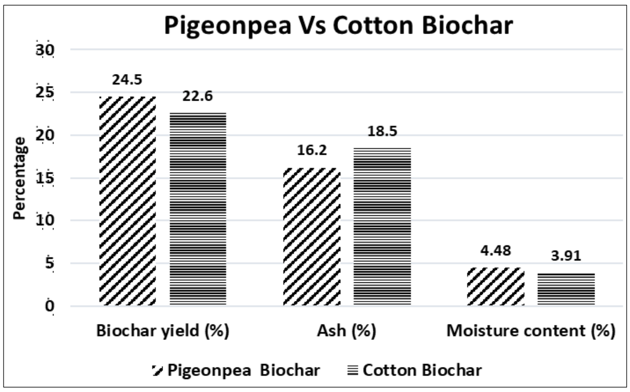


Fig 3: Yield, ash and moisture content of pigeonpea and cotton biochar

Physico-chemical properties of pigeonpea and cotton Biochar

Several studies reported that biochar application improved the soil reaction similar lime application, (Bera *et al.*, 2018) [3]. In our study (Table 2), pH of pigeonpea biochar is 9.86 and cotton biochar 9.82. This differences in pH values attributed to the feedstock type. In addition, during the pyrolysis process, the carboxylic functional groups reduce in biochars. Further, the deprotonation of acidic groups results in conjugation of bases and leads to alkaline pH of biochar (Ronsse *et al.*, 2013) [14]. The EC values also higher in both pigeonpea (2.04 d Sm^{-1}) and cotton (2.53 d Sm^{-1}) biochar. This higher EC values may create a problem if it is applied to soil as amendment in higher application rates (20-40 tones ha^{-1}) as reported by Lehman and Joseph (2009) [8]. The major contributing factor for alkaline pH and higher electrical conductivity is the relative increase of ash content in two biochars produced (Bera *et al.*, 2014) [2]. The alkaline nature of the biochars produced revealed that, pigeon pea and cotton biochar can be effectively utilized as soil amendment for ameliorating soil acidity.

Table 2: Physico-chemical properties of biochar

Biochar properties	Pigeonpea Biochar	Cotton Biochar
pH	9.86 ± 0.03	9.82 ± 0.04
EC (d Sm^{-1})	2.04 ± 0.04	2.53 ± 0.01
Bulk Density (g/m^{-3})	0.26 ± 0.01	0.29 ± 0.01
Particle Density (g/m^{-3})	0.54 ± 0.01	0.53 ± 0.01

Values are given as mean ± standard deviation for triplicate measurements

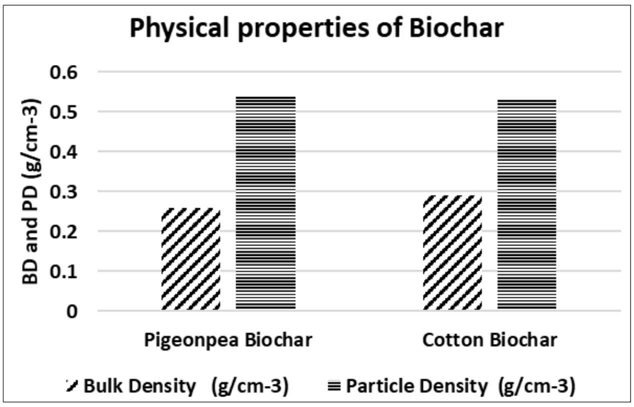


Fig 4: Physical properties of pigeonpea and cotton biochar

The physical properties of biochar derived from pigeonpea and cotton was measured to determine the effect of feedstock type. The results are presented in Table 2 and graphically depicted in Fig 4. The Bulk density of pigeonpea is 0.26 (g/cm^{-3}) and cotton biochar is 0.29 g/cm^{-3}. The residues used in the present study are semi woody in nature and biochar prepared under drum kiln in the temperature range of 350 to 400 ºC. In contrast to the findings of Shaon Kumar Das *et al.*, (2018) [15] bulk density increased with increasing ash content of biochar. The amount of total carbon and ash content in biochar along with range of temperature influences the bulk density. Similar bulk density results were also reported by Bera *et al.*, (2018) [3]. However, in our study the pigeonpea biochar had lower BD as compared to cotton revealed that, lower the BD of pigeonpea biochar as soil amendment is greater benefit for soil. Lower BD indicates more porosity, which enhances the potential for better soil aeration and increased water holding capacity (Shaon Kumar Das *et al.*, (2018) [15]. The particle density of pigeonpea biochar is 0.54 (g/cm^{-3}) and cotton biochar is 0.53 g/cm^{-3}. The ash content has major contribution towards particle density similar results reported by Bera *et al.*, (2015).

Changes in carbon and nitrogen levels during thermo-chemical conversion to biochar

The recovery of total carbon and nitrogen in pigeonpea and cotton biochar is presented in Table 3. The biochar yield and recovery of carbon and nitrogen during the conversion to biocar is depicted under Fig 5. The amount of carbon conserved or recovered in pigeonpea biochar is 27.6% and in cotton biochar the recovery is 29.0%. This recovery of carbon is inversely proportional to the ash content of the corresponding biochars produced at the temperature range of 350 to 400 ºC under slow Pyrolysis. During carbonization process the volatilization of compounds which are in conjunction with volatile matter occurs. The loss of carbon also might have occurred but largely the carbon conserves in increasing temperature as recalcitrant carbon in biochar as indicated by carbon recovery (%). which was in accordance with the findings of venkatesh *et al.*, (2013) [21]; Kloss *et al.*, (2011) [7]. Similarly, the recovery of total N in the pigeonpea and cotton biochar is 20.3% and 18.8% respectively. Previous studies revealed that, N recovery slightly decreases or stabilizes as the temperature increases under Pyrolysis process. The present study results are in accordance with findings of Venkatesh *et al.*, (2013ab) [21]. The reasons for decreased N recovery in present study attributed to the increase of temperature during Pyrolysis leads to aromatization and condensation of N - containing structures in biochar into a recalcitrant heterocyclic N rather than more bioavailable amoniacal nitrogen as explained by Novak *et al.*, (2009) [11].

Table 3: Recovery of carbon and nitrogen under thermo-chemical conversion

Particulars	Pigeonpea Biochar	Cotton Biochar
Stalk load (kg)	25.0	25.0
Biochar yield (kg)	6.12	5.65
Total C in stalk (kg)	10.05	8.23
Total C in Biochar (kg)	2.78	2.39
Total N in stalk (kg)	0.21	0.16
Total N in Biochar (Kg)	0.04	0.03

Values are given as mean ± standard deviation for triplicate measurements

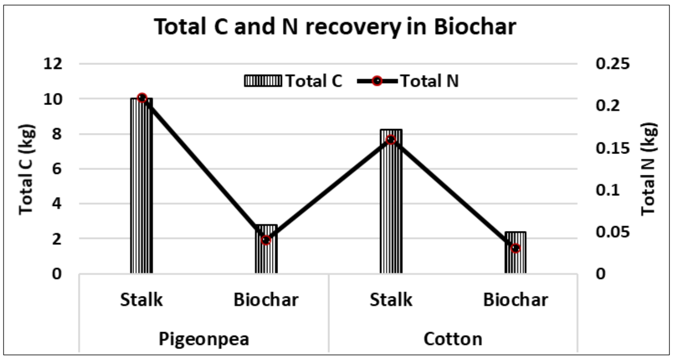


Fig 5: Carbon and Nitrogen recovery in pigeonpea and cotton biochar

Nutrient composition of pigeon pea and cotton residues and their derived biochar

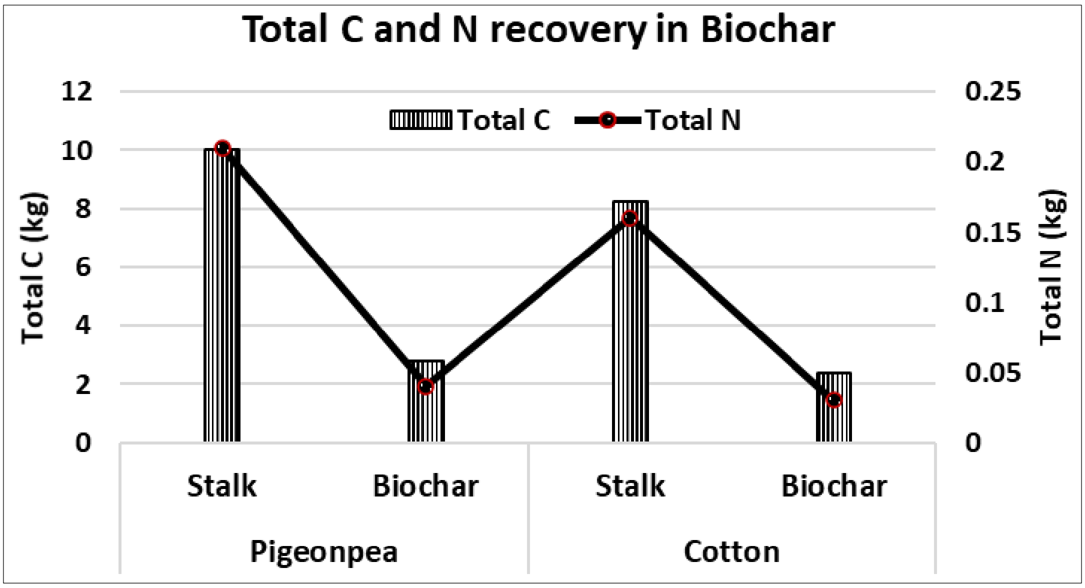
Previous studies revealed that, feedstock composition influences the elemental concentration of biochar materials (Venkatesh *et al.*, 2013a) [21]. The results pertaining to elemental composition of raw pigeon pea and cotton feed stocks and their derived biochar are presented in the Table 4. The raw pigeonpea used in the present study contains 40.21% total C, 0.82% total N and the corresponding carbon in biochar is 45.39 and nitrogen is 0.68%. Similarly, the raw cotton utilized for the biochar preparation in the study contains 32.92% total C, 0.65% total N and the corresponding carbon in biochar is 43.25% and nitrogen is 0.64%. The total N P K concentrations in biochar is graphically depicted under Fig 6. Irrespective of the feedstock type, the pyrolysis temperature had a strong influence on the total carbon and nitrogen concentrations of biochar (Bera *et al.*, 2018) [3]. The increase in carbon content of biochar after subjecting feedstock to slow pyrolysis temperature (350 to 400 ºC) attributed to loss of H and O in greater proportion than C under pyrolysis of the residue (Bera *et al.*, 2014) [2].

The results revealed that, the nutrient compositions of the feed stock and pyrolysis temperature while converting to biochar strongly influence the nutrient concentrations of the resulting biochar. Higher phosphorus content in biochar produced under low temperature pyrolysis attributed to loss of C and presence of less crystallized P associated minerals, similarly, the higher amount of K in biochar is due to the greater solubility of K associated minerals in biochar formed during slow pyrolysis (Bera *et al.*, 2014) [2].

Table 4: Elemental composition of pigeon pea and cotton residue and their derived biochar

Nutrient element	Pigeonpea feed stock	Cotton feed stock	Pigeonpea Biochar	Cotton Biochar
Carbon **(%)**	40.21 ± 0.61	32.92 ± 0.04	45.39 ± 0.03	43.25 ± 0.04
Nitrogen **(%)**	0.82 ± 0.01	0.65 ± 0.02	0.68 ± 0.02	0.64 ± 0.01
Phosphorus **(%)**	0.38 ± 0.02	0.34 ± 0.02	0.47 ± 0.02	0.43 ± 0.01
Potassium **(%)**	0.59 ± 0.01	0.55 ± 0.02	0.68 ± 0.01	0.51 ± 0.02
C N Ratio	49.24 ± 0.51	50.59 ± 0.64	67.10 ± 1.48	67.59 ± 1.00

Values are given as mean ± standard deviation for triplicate measurements



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Nutrient element	Pigeonpea feed stock	Cotton feed stock	Pigeonpea Biochar	Cotton Biochar
Carbon **(%)**	40.21 ± 0.61	32.92 ± 0.04	45.39 ± 0.03	43.25 ± 0.04
Nitrogen **(%)**	0.82 ± 0.01	0.65 ± 0.02	0.68 ± 0.02	0.64 ± 0.01
Phosphorus **(%)**	0.38 ± 0.02	0.34 ± 0.02	0.47 ± 0.02	0.43 ± 0.01
Potassium **(%)**	0.59 ± 0.01	0.55 ± 0.02	0.68 ± 0.01	0.51 ± 0.02
C N Ratio	49.24 ± 0.51	50.59 ± 0.64	67.10 ± 1.48	67.59 ± 1.00

Values are given as mean ± standard deviation for triplicate measurements

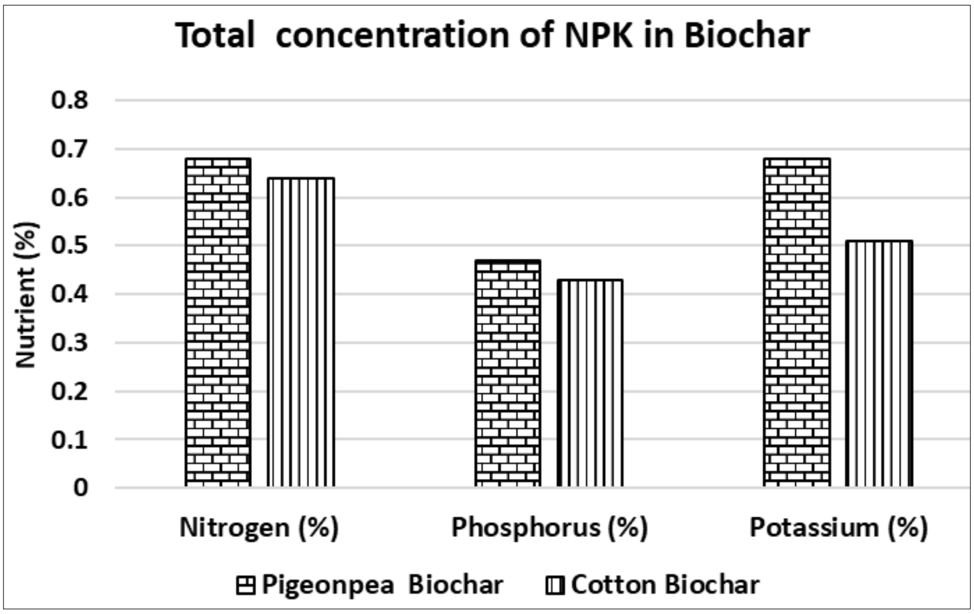

Fig 6: Total concentration of nutrients in pigeonpea and cotton derived biochar

Conclusion

Biochar production through thermo-chemical conversion process (slow pyrolysis) is gaining importance as an alternative way of managing unusable and excess crop residues. However, farmer needs easily adoptable and low cost method. The fabricated drum kiln suits the small farmer necessity to convert pigeon pea and cotton residues into biochar. The biochar produced had the potential soil amendment qualities such as amelioration of soil acidity, positive influence on physical properties such as bulk density there by improving soil aeration, pore space and water holding capacity. In addition, the carbon recovery from the residues contributing to carbon sequestration potential of soil. As a porous material, biochar refuges many beneficial microorganisms thereby influencing nutrient transformations besides supplying small amount of stalk derived nutrients. Hence, the pigeonpea and cotton stalk derived biochars are potentially valuable soil amendments in semi-arid regions. Further, location and crop specific research work is most necessary to study the long term impact of residual biochar on soil types, nutrient conservation, carbon sequestration potential, crop productivity and soil health.

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