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Biochemical characterization of cotton stalks biochar suggests its role in soil as amendment and decontamination

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Abstract. Cotton is the major fiber crop in Pakistan that accounts for 2% of total national gross domestic product (GDP). After picking of cotton, the dry stalks are major organic waste that has no fate except burning to cook food in villages. Present research focuses use of cotton stalks as feedstock for biochar production, its characterization and effects on soil characteristics. Dry cotton stalks collected from agricultural field of Bahauddin Zakariya University, Multan, Pakistan were combusted under anaerobic conditions at 450℃. The physicochemical analysis of biochar and cotton stalks show higher values of % total carbon, phosphorus and potassium concentrations in biochar as compared to cotton stalks. The concentration of nitrogen was decreased in biochar. Similarly biochar had greater values of fixed carbon that suggest its role for carbon sequestration and as a soil amendment. The fourier transformation infrared spectroscopic spectra (FTIR) of cotton stalks and biochar exposed more acidic groups in biochar as compared to cotton stalks. The newly developed functional groups in biochar have vital role in increasing surface properties, cation exchange capacity, and water holding capacity, and are responsible for heavy metal remediation in contaminated soil. In a further test, results show increase in the water holding capacity and nutrient retention by a sandy soil amended with biochar. It is concluded that cotton stalks can be effectively used to prepare biochar.

Keywords: biochar; characterization; cotton sticks; FTIR; soil amendment

1. Introduction

Waste management is the most important issue of the today’s world. Biochar production while utilizing organic wastes such as crop residues and manures has been proved to be the most efficient way of management. Therefore, at a time biochar plays duel role one as a suitable waste

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management tool and other due to its beneficial role in agricultural and environmental aspects (Cha *et al.* 2016, He *et al.* 2016, Kwapinski 2010).

Biochar is also a kind of charcoal fine grained and porous matter formed after anaerobic pyrolysis of organic wastes at 450-650°C (Krull 2010, Sohi *et al.* 2009). The use of biochar as soil amendment dates back 2500 years ago, at Amazon Basin where people used to perform slash and burn activities and incorporate the burnt materials in soils (Duku 2011, Mann 2005). The resulting soils termed as “black soils” are rich in organic matter and nutrients and have higher productivity as compared to non-fertile adjoining soils.

Biochar positively influences the biochemical, physical, and microbial activities of soils. It is a natural host in the form of organic matter for soil micro-organisms (Amonette and Joseph 2009). Moreover, biochar has an indirect role in its function as a catalyst for beneficial soil reactions (Beesley and Dickinson 2010, Cha *et al.* 2016, Gu *et al.* 2016, Verheijen *et al.* 2010). Biochar addition in soil increases the nutrients and water retention in soils (Amonette and Joseph 2009, Gu *et al.* 2016, Gundale and DeLuca 2007) therefore limits the need for fertilizer. Biochar also stabilizes soil structure and decreases soil erosion (Whalen *et al.* 2003). Due to all above discussed properties, the biochar has gained much attention as a soil amendment due to its role in carbon sequestration, soil fertility, crop production, soil decontamination (Cao *et al.* 2010, Cha *et al.* 2016, He *et al.* 2016, Placido *et al.* 2016, Zhang *et al.* 2010).

Most of the biochars have high specific surface area and high adsorption capacity but it depends on the feedstock and production conditions. Though all kinds of organic biomass can be converted into biochar but its use must be careful because there are certain phytotoxic chemicals which can affect soil micro-biota and plants. Due to high sorption capacity, biochar can be used to remediate polluted soils (Atkinson *et al.*, Namgay *et al.* 2010). As soil amendment for remediation purposes, the functional groups developed during pyrolysis play a key role. Similarly, the cotton stalks remained as waste material after harvesting and mainly used as a fuel for domestic purpose. But the manufacturing of biochar from wasted cotton stalks is very useful as they contain some essential ions required by the plant, so biochar reduce the need of fertilizer and improve plant growth and other attributes.

The purpose of this research study was to prepare biochar using harvested cotton stalks as feedstock and to characterize the cotton stalks and biochar using fourier transformation spectroscopy. The effect of biochar on soil water and nutrient retention, and heavy metal remediation was also investigated.

2. Materials and methods

2.1 Biochar production and characterization

Harvested cotton stalks were used for biochar production. The dry stalks were threshed into small sized pieces of less than 5 mm. Biochar production was done according to Kwapinski (2010). Briefly the threshed cotton stalks were filled in stainless steel furnace and pyrolysis was done at 450°C for a period of two hours.

The volatile matter, ash content and fixed carbon in biochar were determined using gravimetric analyses according to McLaughlin *et al.* (2009). The pH and EC of biochar were determined in filtered aliquots of biochar and distilled water at ratio of 1:10 (Singh *et al.* 2010). The concentration of total carbon in biochar was determined using elemental analysis. The

Table 1 The physicochemical properties of cotton sticks and biochar

Parameters	Material	
	Cotton sticks	Biochar
pH	7.86	8.51
EC (ds/m)	1.46	1.52
N (%)	1.12	0.47
P (%)	1.06	0.96
K (%)	0.9	0.82
Zn (ppm)	10.88	6.69
Cu (ppm)	2.1	0.94
Fe (ppm)	290.46	241.18
Mn (ppm)	13.6	7.54
Volatile matter (%)	41	26
Ash (%)	38	62
Fixed carbon (%)	13	23

concentration of nitrogen in biochar as well as in cotton stalks was measured after digestion of samples in concentrated H_2SO_4 followed by Kjeldahl distillation.

For phosphorous and potassium determination, cotton stalks and biochar were wet digested using HNO_3-$HClO_4$ (Chapman and Pratt 1961). After digestion analysis of phosphorous was carried out through spectrophotometer (by Ammonium Vanadate-Ammonium Molybdate yellow color method), and for potassium determination, flame photometer was used.

2.2 Fourier transform infrared spectroscopy (FTIR)

The Fourier transform infrared spectroscopy (FTIR) of dried cotton stalks and biochars was performed to observe the functional groups (Silverstein and Webster 1998). About 1.5-2.0 g sample was mixed with 200 mg of potassium bromide (KBr), and finely ground. The pellets were made under vaccum in a standard device by applying pressure of 75 kN cm^{-2} for 2-3 minutes. The pellets were used for spectral analysis. The resolution of spectra was 4 cm^{-1} with scanning range of 400-4000 cm^{-1}.

2.3 Water holding capacity of biochar

The water holding capacity of biochar was measured in a sandy soil. For this purpose three percentages of biochar i.e., 1%, 3% and 5% were used and mixed with 100 g of sandy soil. After that water holding capacity of soil was determined by following method of Piper (1966).

$$Water\ holding\ capacity, \% = \frac{weight\ of\ saturated\ soil - oven\ dry\ weight\ of\ soil}{oven\ dry\ weight\ of\ soil} \times 100$$

2.4 Biochar nutrient retention capability

The nutrient retention capacity of biochar was determined by adding 50 ml nutrients solution

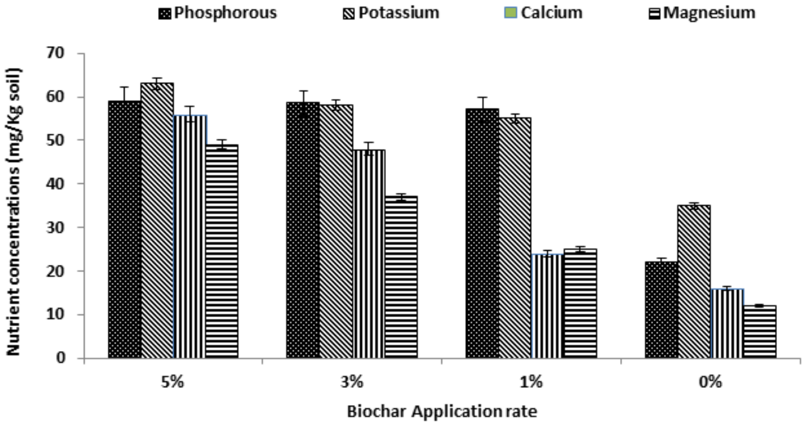

Fig. 1 Macronutrient (mg/Kg of soil) sorbed by biochar in sandy soil

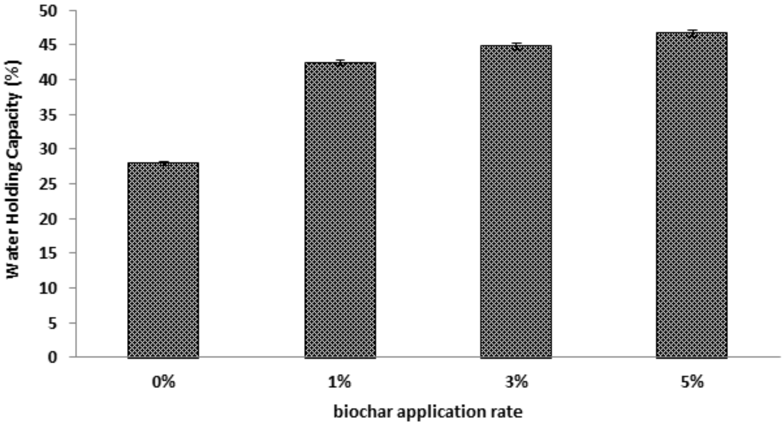

Fig. 2 Biochar addition effect on water holding capacity (saturation percentage) of sandy soil

(100 ppm) in 100 g sand amended with various biochar application levels (control, 1%, 3% and 5%). The samples were kept for two hours with nutrient solution. After that excess water was removed by simple filtration and then soil samples were analyzed for the amount of different nutrients like phosphorous, potassium, calcium and magnesium. Potassium was estimated by Flame Photometry, Ca and Mg by Atomic Absorption Spectroscopy (Rashid 1986).

3. Results

3.1 Biochemical characterization of cotton stalks and biochar

The cotton stalks contain 1.12% nitrogen that was decreased to 0.47% in biochar, whereas, percentages of phosphorus and potassium were higher in biochar as compared to nitrogen because these are not volatilized during pyrolysis (Table 1). In cotton stalks the micronutrients like Zn (10.88 ppm), Fe (290.46 ppm), Cu (2.10 ppm) and Mn (13.60 ppm) were present. Most of these micronutrients remain stable and available in biochar.

Table 2 FTIR vibrational modes assigned to various functional groups in general cotton stalks and biochar (Silverstein and Webster 1998)

Wave numbers (cm^{-1})	Functional Groups
3600 to 3200	O-H
1630 to 1650	carboxylate anion
1650 to 1780	carbonyl/carboxyl functional group
2800 to 3000	C–H stretching band appears
3000 to 2840	waxes and oils
3500 to 2800	stretching of O-H and C-H give vibrational modes
1800 to 1500	presence of functional group and carbonyl group due to interference of C-H bending
1650 to 1780	carbonyl/carboxyl functional group due to strong band around
1630 to 1650	carboxylate anion
~1600	carboxylate anion frequency shift due to H- bonding
3200-3500	multitude of O-H stretching frequencies in cellulose
Cotton	
~ 1733	COOH carbonyl/carboxyl group
~1600	COOH (H-bonding Frequency shift)
~ 3300	polymeric hydrogen bonded O-H/hydroxyl group
3147	corresponding stretching of C-H of (–CH2–) Methylene
1055	Due to C-OH stretching vibration secondary –OH
2923	aliphatic C-H stretching vibration of cellulose
Biochar	
2330-2360	Biochar show a doublet corresponding to a concentration of CO_2 greater than atmospheric
875	aromatic C-H
1585	aromatic C=C
1430	asymmetric C-O stretching in carbonates
1615	For many frequencies of H_2O bending vibrations

Data show increase of the fixed carbon from 13% in cotton stalks to 23% in biochar. The ash content was higher in biochar as compared to cotton stalks. The volatile matter was higher in cotton sticks than in the biochar. The values of pH show slightly basic nature of biochar (Table 1).

3.2 Nutrients and water retention

The macronutrients, which were applied in sand amended with various levels of biochar, were significantly sorbed by the biochar. In case of phosphorus, 5% biochar addition showed the maximum sorption following by 3% and 1%. In control treatment, where no biochar was added sand could retain approximately 1/3 of applied phosphorus. Similar pattern was observed in case of potassium, calcium and magnesium. The trend of sorption of nutrients was in the order of 5% biochar+sand>3% biochar+sand>1% biochar+sand>control (Fig. 1). The saturation percentage and water holding capacity of sand were increased gradually with addition of biochar (Fig. 2). The

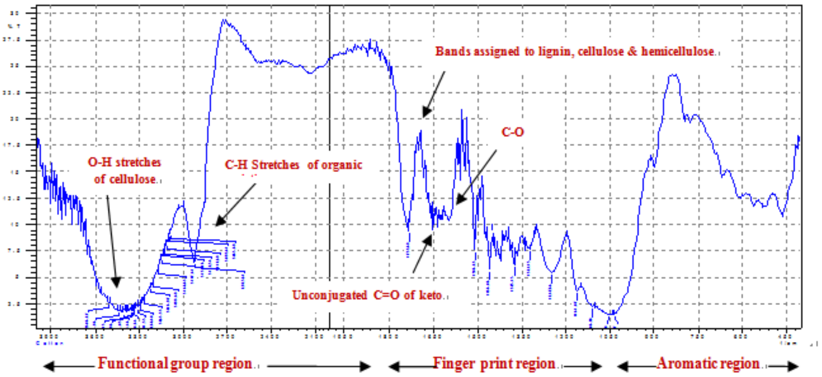

Fig. 3 FTIR spectra of cotton in functional group, finger print and aromatic region

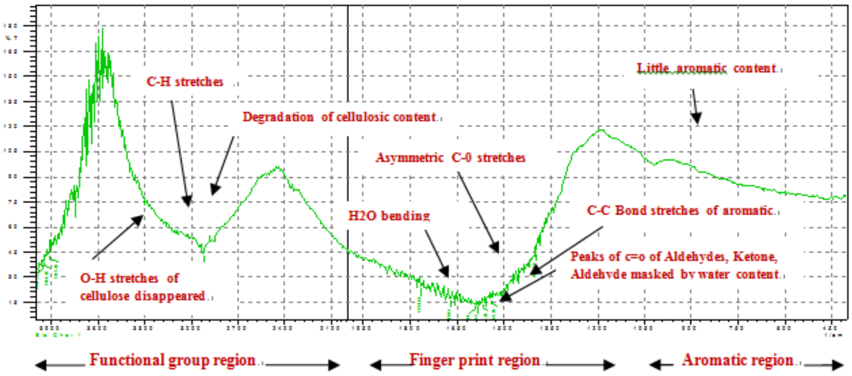

Fig. 4 FTIR spectra of biochar in functional group, finger print and aromatic region

maximum values were observed at 5% biochar application and least in control (sand without biochar).

3.3 FTIR Spectroscopy

From the FTIR spectroscopic data, peak by peak correlation was used to identify the bond vibrations i.e., stretching and bending of functional group and finger print region (Table 2). At first momentary look, a clear cut variation can be seen in the spectra of cotton stalks biochar and cotton stalks. The spectra of cotton stalks show higher cellulose that can be observed by the absorption peaks at ~3300-3600 cm^{-1}. The disappearance of these bands in biochar spectra correlates to a partial loss of cellulosic content. Biochar showed a doublet at 2330-2360 cm^{-1} corresponding to an amount of CO_2 larger than atmospheric, most probably adsorbed within micro pores. The little indication of aromatic bonds was present in biochar spectra, as it was seen by the weak response for aromatic C-H and aromatic C=C at 875 cm^{-1} and 1585 cm^{-1}, respectively. In biochar, aliphatic C-H stretch is observed at 2923 cm^{-1}, confirming that the cellulose is not entirely carbonized during pyrolysis. The aromatic C-H stretching at 3147 cm^{-1} matches the value expected from lignin aromatic residues, but a contribution from the aromatization of cellulose residues during pyrolysis would be expected.

The spectra in Figs. 3 and 4, show strong peak at 3147 cm^{-1} consequent of C-H stretching of methylene (–CH2–) groups in long alkyl chains. These peaks prove the presence of waxes. The intensities of $–CH_2–$ peaks at 3147 cm^{-1} indicate the amount of waxes present in cotton stalks but disappeared in its biochar. Other information can be obtained in the region of 1600–1800 cm^{-1}. A broad asymmetrical peak ~1733 is due to C-O stretching are present in cotton stalks are shifted to ~1650-cm^{-1} in biochar. These peaks comprise a diversity of C-O containing functional groups such as carboxylic acids esters, ketones and anhydrides. Several peaks near 1605 cm^{-1} can be assigned to C-C bond stretching resulting from aromatic rings of lignin, as well as newly carbonized and aromatized material from dehydration and cyclization of carbohydrate ring during pyrolysis. In cellulose a variety of peaks estimated from deformation bands and other single bond stretching in the region 1500 to 650 cm^{-1} were also observed. These were numerous, overlapped and difficult to assign in the averaged spectrum.

4. Discussion

4.1 Biochar role in soil amendment

Based on the results of present study we offer an excellent way for management of cotton stalks (a major agricultural residue often left for burning) in the form of biochar. The biochar prepared using cotton stalks is alkaline in nature that is due to the formation of ash during the pyrolysis process, which typically consists of Ca, Mg, K and Na carbonates (Yuan *et al.* 2011). This pH may be useful in reclamation or increasing uptake of nutrients in acidic soils (Herbert *et al.* 2012). However, owing to alkaline pH range of Pakistani soils, high pH of biochar is of major concern but in a study, no significant effect of 1 % application of cotton-stalks derived biochar on soil pH was found (Qayyum *et al.* 2014). It may be due to the fact that, the week organic acids present in biochar contribute to buffering capacity of soils and the pH is not changed to significant levels. In the cotton-stalks derived biochar most of the micronutrients such as iron (Fe) and manganese (Mn) are retained during pyrolysis (Amonette and Joseph 2009).

The fixed carbon contents in biochar indicates the portion that contain condensed aromatic rings (black carbon) and is likely to stick with soils, while, the volatile constituents are more easily degraded by soil micro-organisms, depending on its physical accessibility (Keiluweit *et al.* 2010). In our findings this increase in fixed carbon may be responsible for soil carbon sequestration and increased nutrients retention. Sohi *et al.* (2010) demonstrated the role of biochar as a soil amendment and remediation of decontaminations. They described that biochar increased the availability of nutrients in soil under stress condition than in any other soil.

4.2 Spectroscopic characterization of biochar

The fourier transform infrared spectroscopy (FTIR) was used to characterize biochar because carbon impurities, water and functional groups can be detected that may modify the sequestration capability. Various differences in the spectra were outcome of concentration of the mineral concentrations and combustion of organic matter that was distorted when heated (Cao *et al.* 2010). Our results show a number of similarities and differences between the functional groups present for the biochar and cotton stick.

The stretching of O-H and C-H gives vibrational modes in the region of 3500 to 2800 cm^{-1} (Silverstein and Webster 1998). The presence of functional groups and carbonyl groups as well as

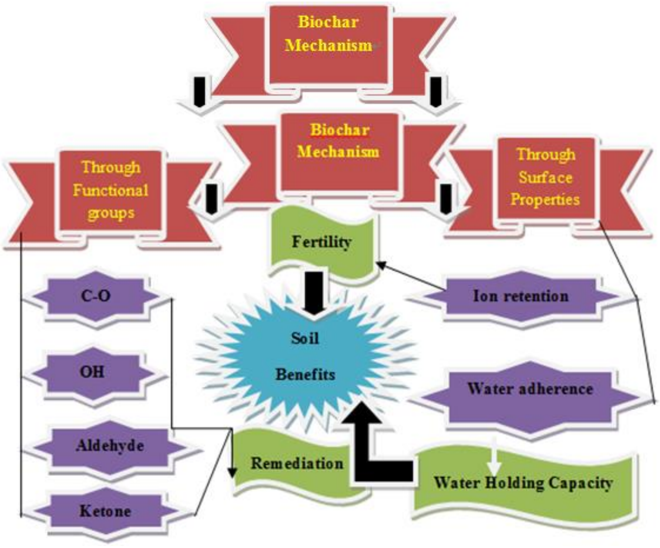

Fig. 5 Biochar functionality in soil with respect to its characteristics

due to interference of C-H bending vibrations in the region of 1800 to 1500 cm^{-1} was described in the work of Himmelsbach *et al.* (2003, 2006). Our results also indicate that the presence of a carbonyl/carboxyl functional group is due to strong band around 1650 to 1780 cm^{-1}. Cotton sticks exhibit the presence of this group at ~1733 cm^{-1}. The peak at 1630 to 1650 cm^{-1} is associated with the carboxylate anion which is mainly responsible for the chelation of heavy metals that is present in the biochar. According to Harvey *et al.* (2011) adsorption of transition elements like Cd^{+2} in biochar is resulted due to presence of functional group –C=O. Cotton sticks exhibit the presence of this group at ~1733 cm^{-1}. In biochar the peak at 1630 to 1650 cm^{-1} is associated with the carboxylate anion which is mainly responsible for the chelation of heavy metals. Berek *et al.* (2011) found that COOH, OH and ketone are the basic functional groups that provide the biochar an ability to absorb the heavy metals like Cd and As. Velazquez *et al.* (2003) stated that the frequency and shift of this peak is dependent on hydrogen bonding with water. Sample spectrum shows a strong band at ~1600 cm^{-1}, indicating a consistent frequency shift because of hydrogen bonding.

By the spectral and biochemical studies of biochar our study demonstrates the role of biochar in chelation of heavy metals which is possible only due to the presence of different function groups (Fig. 5). Soil amendment with cotton stalks biochar also enhances the uptake of nutrients due to its ability of nutrients retention. In our study phosphorous, potassium, calcium and magnesium had positive correlation with biochar addition that was useful in proving the role of biochar in soil amendment. The biochar also had more fixed carbon percentage or black carbon a role in carbon sequestration. A further soil study is suggested in order to demonstrate the beneficial role of cotton stalk biochar in field conditions.

5. Conclusions

The cotton-stalks can be successfully converted in to biochar that is a safe way of its utilization.

The biochar derived from cotton stalks has more carboxylic groups which makes its use for remediation of heavy metals. Moreover, cotton stalk-biochar is useful in case of less fertile soil to increase nutrient uptake by plants.

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