Research Article

Quality Evaluation of Biochar Prepared from Different Agricultural Residues

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Abstract | Biochar gained recognition as an efficient material for carbon sequestration and soil fertility. For its suitable application, it is important to know its chemical and physical properties which depend on the type of the initial material used. Agricultural residues of different types (e.g., *Sesbania* stems (*Sesbania punicea*, SS), Chickpea plant (*Cicer arietinum*, CP), Maize cobs (*Zea Mays*, MC), Wheat straw (*Triticum aestivum*, WS), Sugarcane (*Saccharum officinarum*) bagasse (BG) and Animal dung (AD) were pyrolyzed at temperature range of 300-500 °C in order to compare the biochar samples obtained on the base of its physical and chemical properties. Physico-chemical characteristic such as pH, EC, proximate composition, macro and micronutrients, surface functional groups identification by Fourier Transform Infrared spectroscopy (FTIR) and structure morphology by Scanning Electron Microscope (SEM) were carried out in biochar samples by using standard methods. Statistical analysis of the data revealed significant difference for proximate composition, pH, EC and macro and micro nutrients. Biochar obtained from *Sesbania* stem was found high in fixed carbon content (66.9%) and volatile matter (27%) but low in ash content (4.5%). AD biochar contained lowest amount of fixed carbon (49%) and volatile matter (17%) but highest ash content (30%) among different biochar samples under investigation. All the samples were alkaline in nature with substantial values for electrical conductivity. Macro and micronutrients such as Zn, Mn, Mg and P were high (19.6, 20.4, 37.31 and 237 mg L^{-1}, 510 mg $100g^{-1}$ respectively) in AD biochar while minimum values for Mn, Cu, P and Na (12.0, 20.4 mg L^{-1}, 150 and 38 mg $100g^{-1}$) were noticed in SS biochar samples. All the biochar samples were highly porous except AD and MC biochar samples. The most abundant functional groups found in spectra of all biochar samples were C=C aromatic and O-H except in MC biochar. It was concluded from the results that being highly porous in structure, high in carbon content and containing all the identified functional groups, SS biochar was found the best to be used as soil amendment that could have positive effect on improving the soil health. Moreover, the animal manure is recommended to be used as manure in soil rather than converted to biochar.

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Keywords | Biochar, Agricultural residues, Proximate composition, FTIR, SEM

Introduction

Biochar is a carbon (C) rich material produced by thermal decomposition of biomass under oxygen (O_2) limited conditions (Sohi, 2012). Biochar is a charcoal substance but different from charcoal as it is used as a soil amendment. The relative quality of biochar used as soil amendment is greatly influenced by the type of organic matter and conditions used in biochar production (McClellan et al., 2007; McLaughlin et al., 2009). High adsorption, cation-exchange capacities and low level of mobile matters (resins, tars and other short-lived compounds) are the important parameters to measures the quality of biochar (Liang et al., 2006; McLaughlin et al., 2009; McClellan et al., 2007). The application of heat to organic matter for biochar production results in expansion of its surface area and persistence in soil with limited biological degradation (Lehmann and Rondon, 2006). Plants and soil microorganisms use raw organic matter as nutrients source while the same organic matter in the form biochar acts as a catalyst that helps plants in water and nutrient uptake. Being highly porous and large surface area, biochar retain water and nutrients in the surface soil and also provide shelter for useful microorganisms to flourish. Compared to other soil amendments as compared to other soil amendments (Lehmann and Rondon, 2006; Warnock et al., 2007). With the passage of time decline was noticed in the adsorption capacity of biochar with an increase in its cation exchange capacity. Initial adsorption and porosity is low due to high content of mobile matter but these effects alleviate due to high susceptibility of mobile matter to biological degradation in soil. Pore size is the main physical property of the feedstock that is thermally modified in biochar and this property greatly influence the surface area, biological usage and water holding capacity of biochar. Micro pore is produced due to thermal processing, which have greater nutrient holding capacity due to higher surface areas. Microorganisms that are present in soil cannot utilize such small spaces, they get benefit from some large-pore space. Physical, biological and chemical properties of soil for plant growth are significantly improved due to the presence of biochar in soil with various pore sizes.

Biochar used as a soil amendment increases C storage (Lehmann, 2007; Woolf et al., 2010) mitigates CO_2 and emissions of N_2O (Amelooet et al., 2013; Spokas et al., 2009) and enhances inorganic N (NH^{+4} and NO^{-3}) custody in soils (Ding et al., 2010; Zhao et al., 2013). The latter feature has generated increasing attention in using biochar and activated biochar as soil amendments to adsorb NH^{+4} and to reduce N losses as N_2O emissions (Brun et al., 2011) or through NH_3 volatilization (Sarkhot et al., 2012).

Biochar is one of the simple but effective method in climate change mitigation. Greenhouse gasses such as CO_2 and CH_4 that are produced as result of organic materials decomposition are twenty one times powerful as greenhouse gases. However by charring process, most of the carbon dioxide get tied into a more stabilized form and when applied to the soil in the form of biochar effectively sequestered carbon into the soil (Liang et al., 2008). It is calculated that global carbon emission has been reduced to 10% due to charring process (Woolf et al., 2010).

Biochar application has been investigated in number of reports as plant growth enhancer, soil fertility and for removal of pollutants such as heavy metals, pesticides and hydrocarbons are the benefits investigated in various reports (Cabrera et al., 2011). Physicochemical properties of biochar governed its diversified application which in turn depends on thermal conditions used in pyrolysis process such as heating temperature, duration and the type of biomass used (Enders et al., 2012; Brewer et al., 2011; Zimmerman et al., 2011). Therefore, it is necessary to consider the different aspects during pyrolysis process for its best suitable end use. Nutrients and carbon availability has been changed variably due to different physicochemical properties of biochar.

Biochar produced by low temperature pyrolysis has high amount of volatile matter that contains readily available substrates, which is helpful in plant growth (Robertson et al., 2012; Mukherjee and Zimmerman, 2013). In contrast, large surface area and high content of aromatic carbon are attained by high temperature pyrolysis. This might help in bioremediation by increasing its adsorption power as well as its recalcitrant property which is necessary for effective carbon sequestration in soil (Lehmann, 2007). Secondly, type of biomass dictates biochar properties that may determine its effects on soil and final application. For example, biochar obtained from manure has higher cation exchange capacity than biochar derived from woody material (*Eucalyptus*) (Singh et al., 2010). Also Switch grass and Corn stover biochar produced under same temperatures had higher ash content and lower aromatic carbon than the biochar produced from woody substance (Brewer et al., 2011). Likewise, higher saturated hydraulic conductivities are produced due to application of wood chip biochar than by applying biochar of manure origin (Lei and Zhang, 2013). As it is evident from the literature that type of feedstock has key role in determining the quality of biochar and ultimately its final use. Therefore, this project has been undertaken to determine the physicochemical characteristics of biochar derived from different agricultural resides to recommend it for its best suitable use in agriculture.

Materials and Methods

Preparation of biochar from agricultural residues

The biochar was prepared from different Agricultural residues viz., Sesbania (*Sesbania punicea*) stems, chickpea (*Cicer arietinum*) plant, maize (*Zea Mays*) cobs, wheat (*Triticum aestivum*) straw, sugarcane (*Saccharum officinarum*) bagasse and animal dung. It was produced using a traditional 'on-farm' method. In Pakistan "On-farm" method is a common method used for small scale production of charcoal. All these biomasses were pyrolyzed at 300-500 ˚C for 3-4 h and pulverized to form a coarse powder (<5mm) (Arif et al., 2015).

Chemical analysis of biochar

After the pyrolysis process, all samples were ground. The ground powder was sieved through a sieve less than 0.5 mm in diameter. The analytical methods applied for biochar characterization were proximate analysis, pH, elemental analysis, EC, mineral content, biochar structure profiling and surface functional groups identification. The analyses were performed in duplicate.

Proximate composition

Moisture content was analyzed by drying the samples at 105 ˚C according to ASTM standards # D 4442-07. One gram of biochar in a crucible with lid, was burnt at 950 ^{0}C for 11 minutes (ASTM Standards # D 3175-11) for the determination of volatile matter contents (% dry weight basis). The sample in uncovered crucible (ASTM Standards # E1755-01) was burnt at 750 ^{0}C for determination of ash content (dry weight basis) for two hours. ASTM Standards # D3172-07 was used for the determination of fixed carbon contents by calculating the difference between hundred and sum of percentages of moisture, ash and volatile matter.

Fixed carbon content(%)=100-(%moisture+% Ash+% Volatile matter)

pH and electrical conductivity

Biochar and water mixture with ratio of 1:10 (w/v) was shaken for 24 hours. pH and electrical conductivity meter was used for the measurement of pH and electrical conductivity, respectively (Jindo et al., 2014).

Mineral analysis

Powdered samples of known quantity were burnt at 760 °C in muffle furnace for 6 hours for the determination of minerals contents such as Cu, P, Zn, Mn, Mg, Na and K. The resulting ash was mixed with HCl, diluted with deionized water and then used for further mineral analysis. Flame photometer was used for the determination of sodium and potassium in biochar samples. Atomic absorption spectrophotometer was used for the quantification of Mg, Mn, Zn and Cu. Phosphorus content in biochar samples was determined by spectrophotometer (Cantrell et al., 2012).

Biochar structure profiling

Scan Electron Microscope (JEOL Model JSM -5910 SEM) at 5 kV imaging at different magnification levels was used for the determination of structure and surface morphology of various biochar samples (Al-Wabel et al., 2013).

Fourier Transform Infra-Red spectroscopy (FTIR)

Biochar samples derived from different Agricultural residues were analyzed on FTIR (Model Prestige-21 Shimadzu, Japan) for obtaining transmittance spectra at 4 cm^{-1} resolution and mirror velocity of 0.48 cm S^{-1} with a wave number 400-4000 cm^{-1}. Potassium bromide (KBr) pellets were used for dilution and homogenization. Qualitative comparison of vibrating transmittance spectra of biochar was analyzed by FTIR (Rafiq et al., 2016).

Statistical analysis

The statistical analysis of the data was performed by using Statistic 8.1 for Analysis of Variance. Means were separated by Least Significant Difference Test (Steel and Torrie, 1980).

Results and Discussion

Standard methods and techniques were used for the determination of physico-chemical properties of biochar samples prepared from different agricultural residues. Following are the results of each analysis.

The date inserted in Table 1 showed significant ($p \leq 0.05$) results for moisture, ash, volatile matter and fixed carbon contents of biochar prepared from different agricultural residues. The volatile matter, fixed carbon and ash content of biochar samples were ranged between 4.5 to 30%, 17 to 25% and 49 to 66.9% respectively. Maximum ash content (30%) was observed in animal dung biochar while minimum (4.5%) was recorded in *Sesbania* stem biochar. Maximum volatile matter and fixed carbon contents were found in *Sesbania* stem biochar (25 and 66.9%) while minimum (17 and 49%) was noticed in animal dung biochar. Chen et al. (2011) recorded almost similar results for ash and fixed carbon. Higher values were obtained for the two parameters in biochar come from animal dung than biochar made from woody biomass. However, the values were higher in animal manure biochar than the present data that might by due to difference in nutrient content of feed given to the animals. Moreover, the difference in nutrient composition of biochar has been influenced by wide range of factors including difference in feedstock quality and charring conditions (Hammes et al., 2006; Chan and Xu, 2009).

Table 1: *Proximate composition of biochar derived from different Agricultural residues.*

Biochar Samples	Ash (%)	Volatile Matter (%)	Moisture (%)	Fixed Carbon (%)
Sesbania Stem	4.5±0.3d	25.0±1.7a	3.6±0.2abc	66.9±0.9a
Wheat Straw	20±0.6c	23.0±1.3b	3.0±0.5c	54.0±3.0bc
Maize Cob	21±1.0c	19.0±1.1cd	3.4±0.2bc	56.6±2.3b
Chickpea plant	23±1.0b	21.0±1.3bc	2.0±0.4d	54.0±0.6bc
Animal Dung	30±1.7a	17.0±1.0d	4.0±0.6ab	49.0±1.0d
Sugarcane Baggase	22±1.2c	22.0±1.1b	4.5±0.8a	53.5.0±2.7c

Mean ± SD; Means in each column with common letters are non-significant at 5% significance level

pH and EC of biochar

Significant differences (p≤0.05) were found among different biochar samples for mean pH values as shown in (Figure 1). Maximum mean values of pH (9.8) was noticed for chickpea plant biochar while minimum mean value (8.2) was recorded for Wheat straw biochar. Statistically significant differences (p≤0.05) were found among various biochar samples for mean EC values as shown in (Figure 2). Mean values of EC varied between 0.44 dS/m to 2 dSm^{-1} with a maximum mean value (2 dSm^{-1}) noticed for Chickpea biochar while minimum mean value (0.44 dSm^{-1}) was recorded for *Sesbania* Stem biochar. All the samples were found alkaline in nature. Chan et al. (2007) also observed alkaline nature for all the biochar samples under examination which is in agreement with the present results.

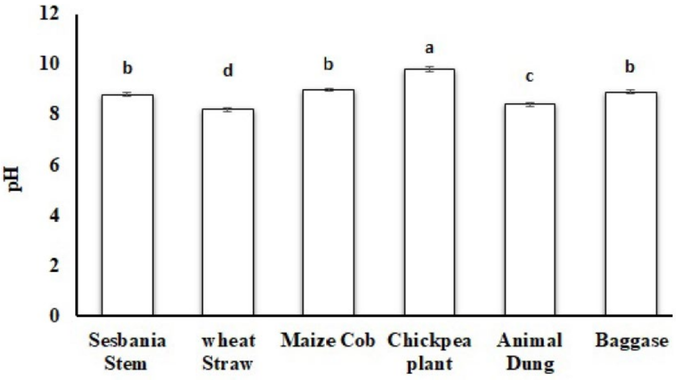

Figure 1: *pH of biochar samples obtained from different Agriculture residues.*

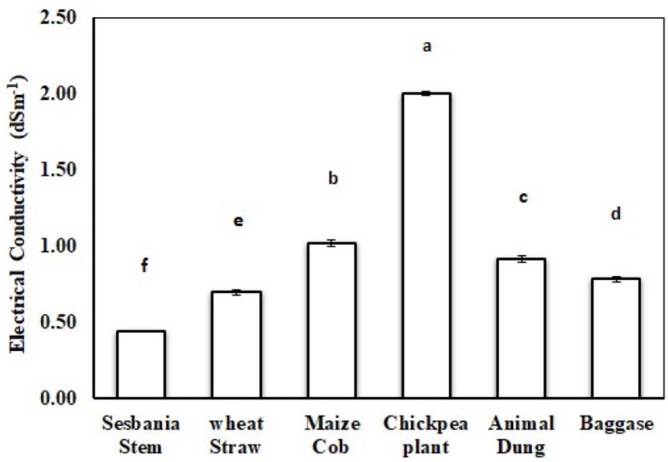

Figure 2: *EC of biochar samples obtained from different Agricultural Residues.*

Mineral composition of biochar

Statistical analysis of the mineral data of biochar obtained from different agricultural residues revealed significant (p≤0.05) results (Table 2). Maximum mean values for Zn (19.6 mg L^{-1}), Mn (20.4 mg L^{-1}), Cu (37.31 mg L^{-1}), Mg (237 mg L^{-1}) and P (510 mg $100g^{-1}$) were observed for animal dung biochar. Minimum mean values of 12.0, 20.4 mg L^{-1}, 150 and 38 mg $100g^{-1}$ for Mn, Cu, P and Na respectively were found in Sesbania stem biochar. Minimum mean values of 13.4 mg L^{-1} and 30 mg $100g^{-1}$ for Zn and K respectively were noticed in maize cob biochar samples.

Sukarato et al. (2011) documented high values of nutrient contents especially for Ca, P and Mg in biochar samples which is at par to the present result. Biochar made from woody materials have lower nutrient contents as compared to the biochar obtained from animal manure (Chan et al., 2007). Animal Dung was a rich source of high nutrients which represent the nutrition that was removed when animals were fed by fodder-biomass.

Table 2: *Mineral Composition of biochar derived from different agricultural residues.*

Biochar Samples	Minerals (Micro and macro)						
	Zn (mg L^{-1})	Mn (mg L^{-1})	Cu (mg L^{-1})	Mg (mg L^{-1})	P (mg 100g^{-1})	Na (mg 100g^{-1})	K(mg 100g^{-1})
Sesbania Stem	14.6±1.2 d	12.0±0.5 d	20.4±1.0 e	193.8±1.0 b	150±2.0 e	38±2.5 d	50±2.6 c
Wheat Straw	13.8±1.1 d	14.6±1.0 c	25.07±0.7 d	165.5±1.4 c	220±2.6 f	50±3.1 c	40±3.1 d
Maize Cob	13.4±1.7 d	13.3±1.2 cd	21.38±1.0 e	163.3±1.3 d	350±2.0 c	70±2.0 b	30±3.5 e
Chickpea plant	17.1±0.6 c	20.8±1.5 a	34.72±0.6 b	137.6±1.0 f	390±2.6 b	100±2.6 a	130±2.2 a
Animal Dung	19.6±0.7 b	20.4±1.2 a	37.31±0.9 a	237.0±1.6 a	510±3.0 a	40±3.0 d	70±1.0 b
Sugarcane Baggase	24.4±1.5 a	17.2±0.8 b	27.32±0.8 c	141.4±0.8 e	320±1.4 d	50±2.0 c	32±2.0 e

Mean ± SD; Means in each column with common letters are non- significant at 5% significance level.

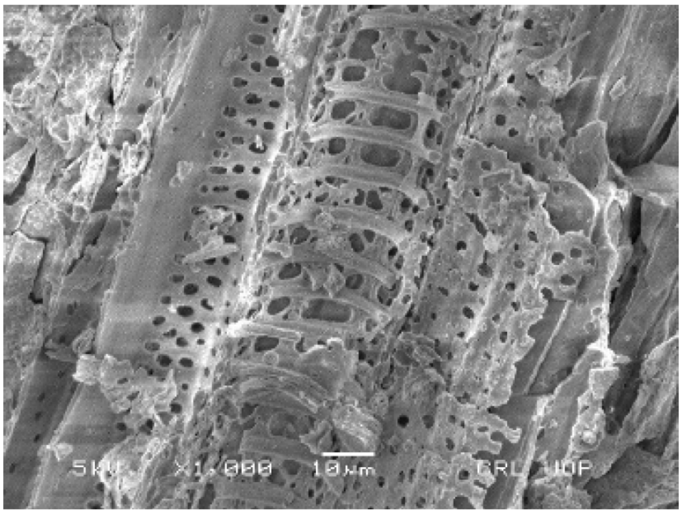

Figure 3: *SEM micrograph of biochar derived from animal dung.*

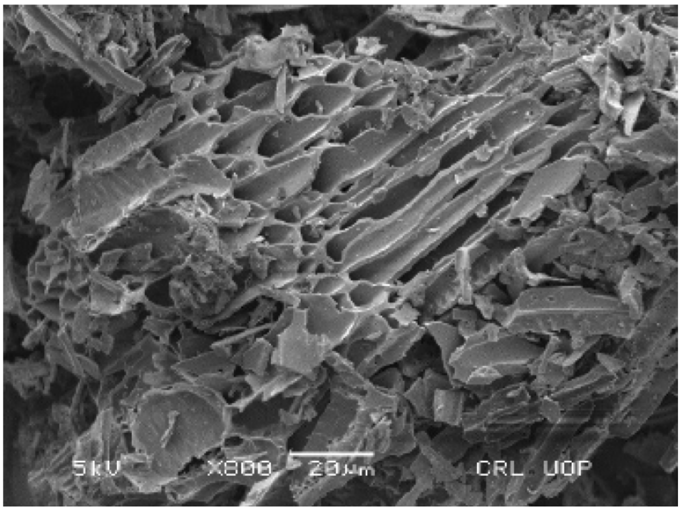

Figure 4: *SEM micrograph of biochar derived from chickpea plant.*

Structure profiling of biochar

SEM micrographs of biochar samples made from different biomasses were depicted in figures 3-8. Structural variations in terms of porosity in char particles can be studied through SEM images after carbonization process of biomass. The SEM micrographs of all the biochar samples were found highly porous except animal dung Figures 3 and maize cob biochar which were comparatively less porous in structure (Figure 5). This might be due to devolatilization during pyrolysis process supported by low values of volatile matter observed for animal dung and maize cob biochar samples (Table 1). Densities, porosities and pore structure of biochar produced are significant to the extent of devolatilization. Biochars with higher porosities, lower densities and significantly different pore structure can be produced from higher volatile matter (Haykiri et al., 2001).

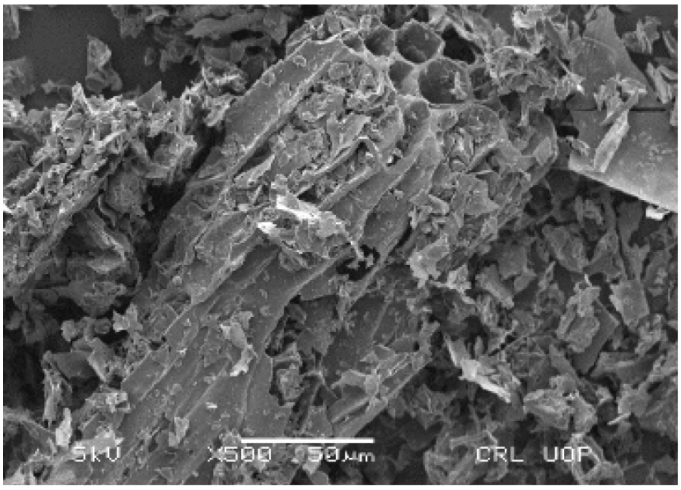

Figure 5: *SEM micrograph of biochar derived from maize cob.*

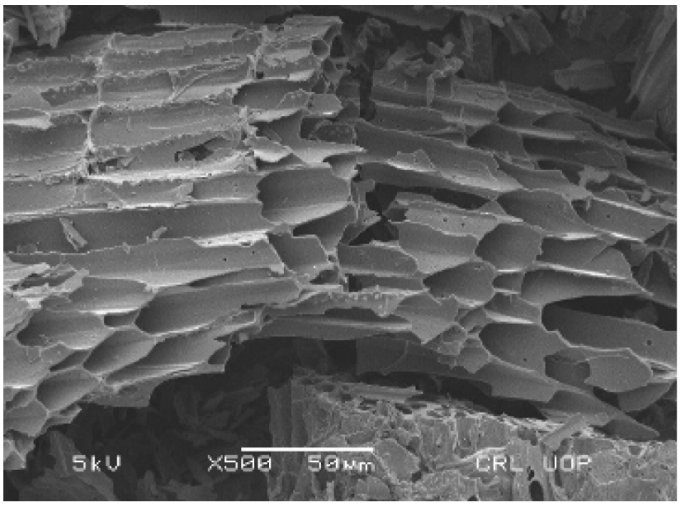

Figure 6: *SEM micrograph of biochar derived from Sesbania Stem.*

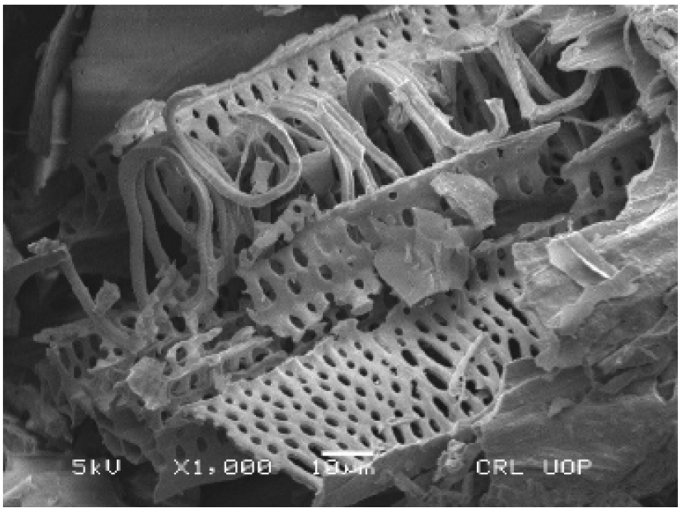

Figure 7: *SEM micrograph of biochar derived from wheat straw.*

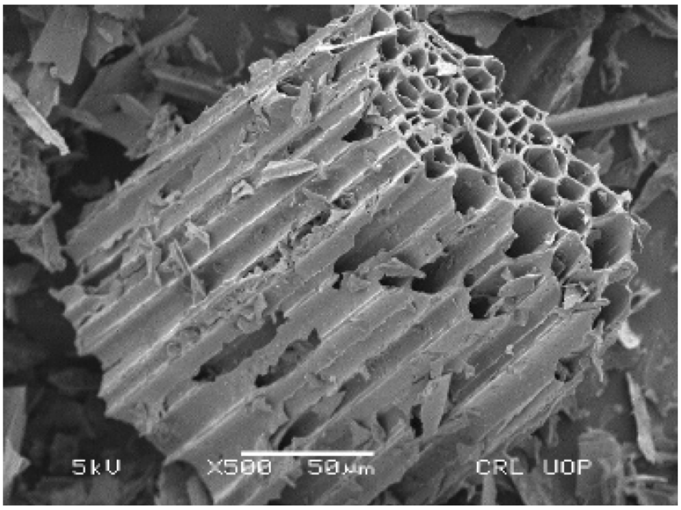

Figure 8: *SEM micrograph of biochar derived from sugarcane bagasse.*

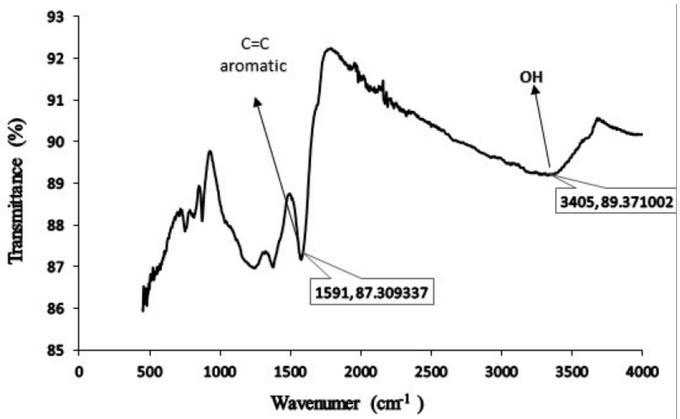

Figure 9: *FTIR Spectra of biochar produced from Sesbania Stem.*

FTIR analysis of biochar

The functional groups which were identified from the FTIR spectra of biochar samples obtained from different agricultural residues are given in Figures 9, 10, 11, 12, 13 and 14. Absorption bands between 4000 cm^{-1}- 500 cm^{-1} were used for the FTIR spectra for all biochar samples. The Infra- red spectra of all types of biochar contained different functional groups except maize cob biochar which had no functional groups in its spectrum. Aromatic (C=C) and Hydroxyl (O-H) were the most abundant functional groups. The broad O–H peak around 3600– 3200 cm^{-1} is clearly visible in all the biochar samples except maize cob produced from different agricultural biomasses demonstrating dehydration of cellulose and ligneous compounds (Keiluweit et al., 2010; Rafiq et al., 2016).

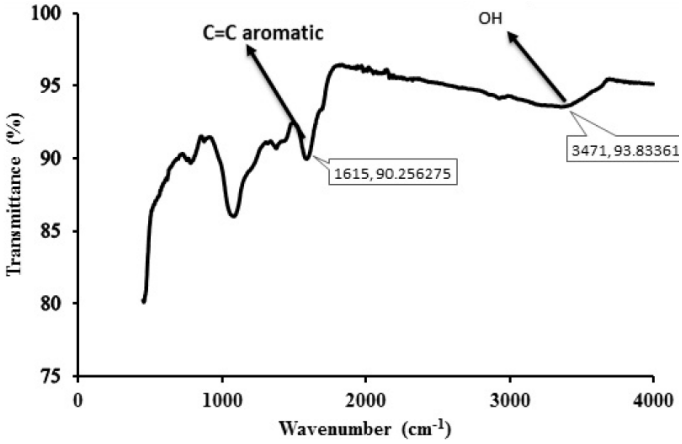

Figure 10: *FTIR Spectra of biochar produced from wheat staw.*

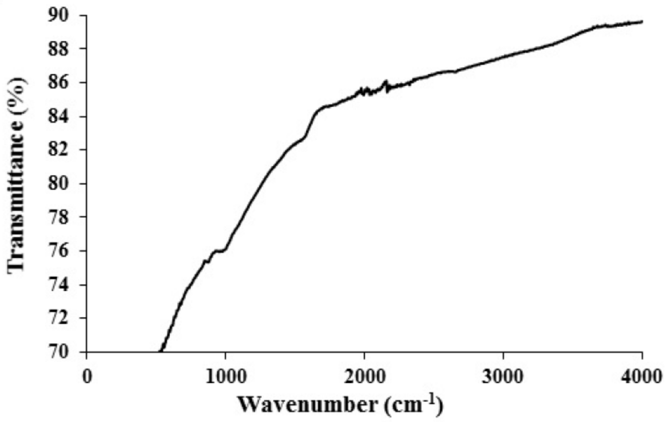

Figure 11: *FTIR spectra of biochar produced from maize cob.*

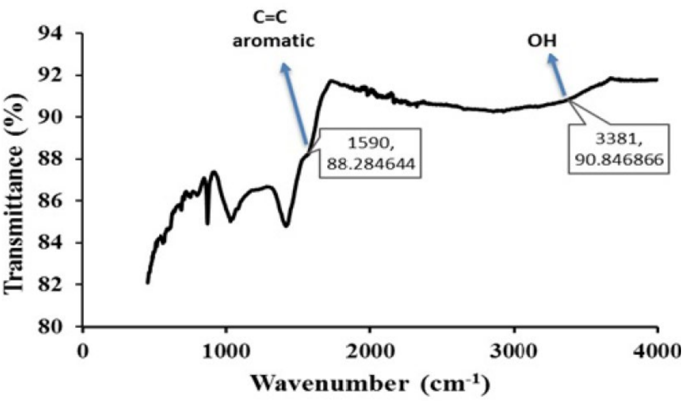

Figure 12: *FTIR Spectra of biochar produced from chickpea plant.*

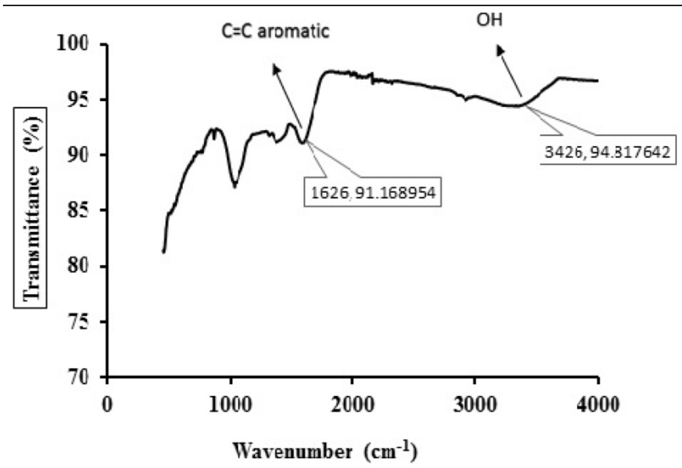

Figure 13: *FTIR Spectra of biochar produced from anmial dung.*

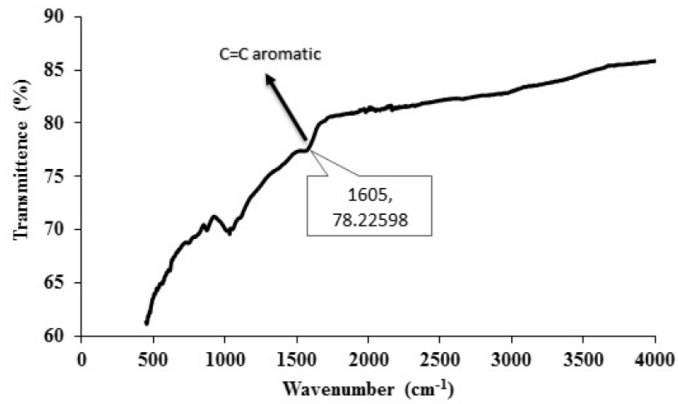

Figure 14: *FTIR Spectra of biochar produced from sugarcane bagasse.*

Conclusions and Recommendations

It was concluded from the present study that *Sesbania* stem biochar contained high content of fixed carbon and low ash and mineral content compared to other biochar samples. Maize cob and animal dung biochar samples were found comparatively less porous to other biochar samples under investigation as evident from the SEM imaging. All the biochar samples were found alkaline in reaction. Animal dung biochar had higher content of all minerals, viz; Zn, Cu, Mn, Mg and P except sodium and potassium. Chemical bonds including C=C and O-H were present in all biochar samples as shown from FTIR analysis except maize cob biochar.

Being porous in structure, containing high amount of carbon and surface functional groups, *Sesbania* stem biochar could be used for soil amendment in order to increase aeration, microbial activity, water and nutrient retention in highly weathered soils. Being alkaline in nature, all the biochar samples could be used in acidic soils for increase in soil pH. Low in fixed carbon, less porous in structure while high in mineral content; animal dung biomass is recommended to be used as manure in soil rather than converted to biochar. All biochar samples contained high carbon with aromatic structures, its application for soil amendment could have positive effect on improving stability of soil carbon.

Further studies are recommended to evaluate the effect of different temperatures on the physicochemical characteristics of these biomass sources while making biochar.

Author's Contribution

Babar Tasim: Carried out the researcha nd wrote the manuscritp.
Tariq Masood: Planned the research and helped in writing the manuscript.
Zafar Ali Shah: Helped in interpretation of FTIR and SEM data.
Muhammad Arif: Provided/collected samples.
Ata Ullah: Proof read the article.
Ghazal Miraj: Utilized her labatory for some of the analysis.
Muhammad Samiullah: Help in statistical analysis and proof reading.

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