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THE PHYSICO-CHEMICAL PARAMETERS OF BIOCHAR PRODUCED FROM THE STEMS OF DIFFERENT CROPS (RICE, MILLET AND SORGHUM) AND ITS INFLUENCE ON SELECTED HEAVY METALS IN LEAD CONTAMINATED SOIL AT A MINING AREA OF ZAMFARA STATE, NIGERIA

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ABSTRACT

The experiment involved the production of biochar from the pyrolysis of three crop stems i.e. Rice, Millet, Sorghum stems and combination of all the three and thus labeled as A,B,C and D respectively. Their physiochemical properties, namely: Cation Exchange Capacity (CEC), pH, % Total organic Carbon (TOC), Ash and Moisture contents, were determined. The biochars produced were applied to the soil and the soil analyzed for lead and cadmium level after 2 to 4 weeks. The results of physicochemical analyses of the biochars showed that millet stem biochar had the highest CEC (33.40±0.001 %), while sorghum stems biochar had the lowest (14.80±0.085 %). The pH ranged from 11.56±0.10 to 12.30±0.202, thus the biochars had % TOC of (41±0.001) for A, (37.70±0.0034) for B, (54.10±0.004) for C and (52.30±0.021) for D. Millet stem biochar had the highest % moisture content with 3.5±0.002% and rice showed the lowest moisture content of 2.5±0.001%. The results of heavy metal level in the soil after application of biochar indicated that the levels of both metals under investigation reduced drastically with time. Hence the need to monitor the level of these metals over a long period of time to ascertain the possibility of using the char from rice, millet and sorghum for remediation of the contaminated soil.

Keywords: Biochar, Heavy metals, Soil remediation, Physico-chemical, Cadmium, Lead.

INTRODUCTION

Biochar is a carbon-rich product obtained when living by-products, such as wood, manure or crop stems is heated in a closed container under limited supply of oxygen, relatively low temperature (Lehmann and Joseph, 2009) and use as soil amendment (Simon *et al*., 2011). Biochar is a charcoal produce by pyrolysis of biomass (Taylor and Mason, 2010). It is distinguished from other charcoals in its intended use as a soil amendment (Lehmann and Randon, 2006; Srinivasarao *et al*., 2013). It serves as a catalyst that enhances plant uptake of nutrient and water compare to other soil amendment, the high surface area and porosity of biochar enables it to adsorb or retain nutrients and water and also provides a habitat for beneficial microorganisms to flourish (Glaser *et al*., 2002). Biochar have been used in controlling soil pH (Rodriguez *et al.,* 2009). It acts as liming material in acidic soils (Lakaria *et al*., 2012; Srinivasarao *et al*., 2013). Figure 1 shows a model of a micro crystalline graphitic structure of biochar on the left and an aromatic structure containing oxygen and carbon free radicals on the right.

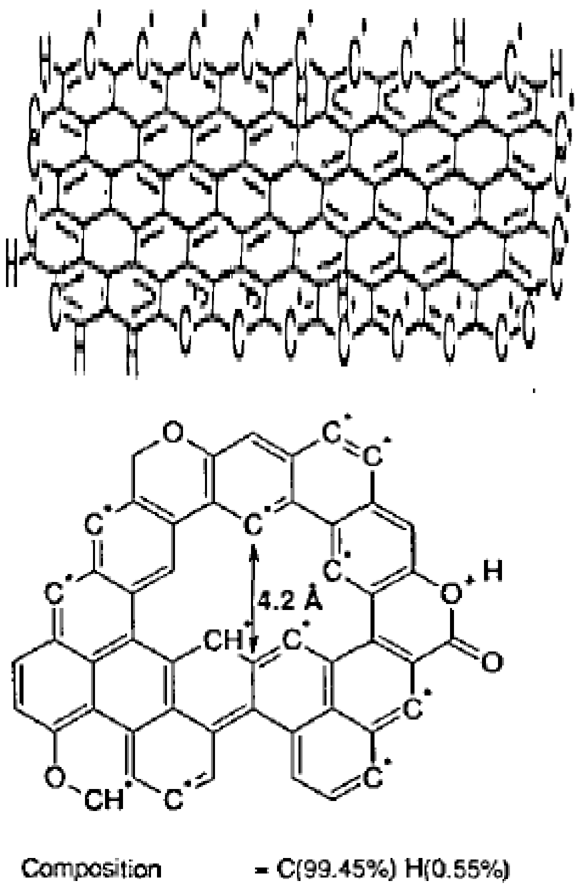

Figure 1: Putative structure of charcoal (adopted from Bourke *et al.*, 2007).

It is commonly accepted that each biochar particle comprises of two main structural fractions: stacked crystalline graphene sheets and randomly ordered amorphous aromatic structures (Bourke *et al.,* 2007). The presence of heteroatoms such as oxygen is thought to be a great contribution to the highly heterogeneous surface chemistry and reactivity. Biochar composition is highly heterogeneous, containing both stable and labile components (Sohi *et al*., 2009). Carbon, volatile matter, ash (Antal and Gronli, 2003) and moisture are generally regarded as its major constituents (DeLuca *et al*., 2006).

Biochar is most commonly incorporated into the soil by first evenly spread the desired amount onto the soil, followed by tilling in with machinery or by hand. it can be applied to the soil surface and preferably covered with other organic materials, mixed with compost or mulch, and as a liquid slurry if finely ground (Glaser *et al*., 2002). The rate of application depends on many factors including the type of biomass used, the degree of metal contamination in the biomass, the types and proportions of various nutrients (N, P, etc.) and also on edaphic, climatic and topographic factors of the land where the biochar is to be applied (Taylor and Mason, 2010)

Effect of biochar on heavy metals

Heavy metals are not biodegradable, and persist for a longtime in contaminated soils. It is expensive and time consuming to remove heavy metals from contaminated soils (Tsafe *et al*., 2014). Stabilization of heavy metals in situ by adding soil amendments such as lime and compost is commonly employed to reduce the bioavailability of metals and minimize plant uptake. It has been reported that "the use of biochar in soil amendments can stabilize heavy metals and improve the quality of the contaminated soil (Ippolito *et al.,* 2012) and has a significant reduction in crop uptake of heavy metals. Research has also shown that biochar derived from bamboo adsorbed Cu, Hg, Ni, and Pb from both soils and water, and Cd in polluted soils (Cheng *et al*., 2006). It has been observed that biochar has been use as effective tools for remediating heavy metals contaminated soils (Tang *et al*., 2013). The hardwood-derived biochar have been reported to reduce cadmium concentration in cadmium contaminated soil (Beesley *et al*., 2010).

The most important measures of biochar quality include cation exchange capacity, pH and type of organic matter feedstock used. The adsorption capacity of biochar decreases over time, whereas its cation exchange capacity increases (McLaughlin, *et. al*., 2009). Namgay *et al*., (2010) conducted a pot experiment to investigate the influence of biochar on the availability of As, Cd, Cu, Pb, and Zn to maize (*Zea mays* L.). After 10 weeks of growth, plants were harvested, shoot dry matter yield was measured, and concentration of trace elements in shoots was analyzed. The results showed that biochar application can significantly reduce the availability of trace elements to plants which indicated that biochar application can manage soils contaminated by trace elements. Purakayastha *et al.* (2013) characterized the chemical composition of biochar prepared from various crop residues at 400°C. The biochar prepared from rice residues showed highest CEC, and pH value for maize (10.7) and pearl millet (10.6) biochar was higher than that in wheat (8.8) and rice (8.6) biochar. Total carbon content was highest in pearl millet biochar (61%) followed by wheat (52%) and rice biochar (49%) whereas maize biochar had lowest carbon content (37%). However, maize biochar was richer in major (N, P, K), secondary (Ca, Mg) and micronutrient (Fe, Mn, Zn and Cu) contents.

Several studies have been carried out in order to proffer solutions to the devastating effects of lead poisoning in some mining areas of Zamfara state. But the information on the use of organic residue to remediate the affected soil in such area are either limited or completely absent. Therefore, application of biochar could potentially provide a new solution for remediation of the soils contaminated by heavy metals. Hence, attempt to bridges this gap is what inform the basis for this research

MATERIALS AND METHODS

Materials

The materials used in this work are soil samples, rice, millet and sorghum stems, pyrolysis stove, and AAS. All the reagents used in this work were of analytical grade.

Fabrication of pyrolysis stove

The pyrolysis stove consists of a cylindrical drum made up of zinc alloy sheet fabricated at Kara market. It consists of combustion chamber, ventilation cone, outer tin and lid. The diameter of combustion chamber and outer chamber were 11 cm and 13.5 cm, respectively and the height of the cylinder was 29 cm. The volume of combustion and outer chamber were 2756.32 cm^3 and 4151.56cm^3 respectively. The distance between combustion and outer chamber was 5 cm. The height of the ventilation cone was 10.5 cm.

The process of pyrolysis

The combustion chamber was filled with fuel materials (wood pellets, dry twigs, etc) which were used for lightening purpose. The biological waste raw material (rice stem, millet stem and sorghum stem) was placed in the gasifier space (space between combustion chamber and outer chamber). A rag soaked with kerosene was used as a fire starter. The lid was placed on the stove, when the fire had started burning. The fuel material burned hotter after 10 – 15 min, which showed the flame in yellow color, but the waste biomass in the outer chamber began to burn after 30 min. At that time it released gases and the flame turned blue with little smoke, implying there was complete burn of the fuel. This process was completed within 2 hr and the stove was cooled down in 1 hr. At the end of the process all the biomass had turned into char. The Biochar samples were collected from the pyrolysis stove sieved (< 0.2 mm) and their important characteristics were analyzed. The biochars obtained were coded A (rice), B (millet), C (sorghum). A fourth which was a composite of A. B and C was prepared and coded D.

Application of biochar to soil

The biochars (A, B, C, D) produced from the pyrolysis of biological wastes were made into a slurry form and incorporated into the soil on a farmland in four different respective locations in Daretta village of Anka local government, Zamfara state.

Soil sample collection and pre-treatment

Stratified random sampling method were used to collect soil samples at ten (10) different point at a depth of 5-20 cm at the four different locations labeled A, B, C and D. The samples were mixed, homogenized and representative samples was obtained using cone and quartered methods at each of the four locations for the period of four (4) weeks at two (2) weeks interval. The representative samples were then air dried, ground using pestle and mortar, sieved with a 2mm sieve, stored in a plastic container and later analyzed (Radojevic and Bashkin, 2006).

Digestion of the soil sample and AAS analysis

One gram of each samples was weighed and put separately in an empty Kjeldhal digestion flask, 10 mL of nitric acid (HNO_3) and 2 mL of 60% perchloric acid ($HClO_4$) were added to each sample in the Kjeldhal digestion flask (Popoola *et al*., 2012). The flasks were heated in digestion block in fume cupboards for 15 min, and allowed to cool at room temperature. The cool digest was and filtered into a standard 50 mL sample bottle and made up to the mark with distilled water for Atomic absorption spectrophotometer analysis (AAS) (Radojevic and Bashkin, 2006). Physico-chemical parameters (pH, Organic carbon, moisture, ash, cation exchange capacity, nitrogen, potassium and phosphorus) were determined according to Radojevic and Bashkin (2006) standard procedure.

RESULTS AND DISCUSSION

The results of analyses of some important characteristics of Biochar samples produced from the pyrolysis of Rice (A), Millet (B), Sorghum(C) stems, and the combination (D) of all the three are presented in Table 1. The physicochemical properties of the biochars (Table 1) revealed that all the char were basic in nature. The pH of biochar can vary but it is often above 9.0 and biochar can have liming value in order of several tens of percent (Van Zwuetan *et al*., 2010). The percentage cation exchange capacity (% C.E.C) was higher in all the samples with exception of sample C which was lower. These values were much greater than those reported by (Liang *et al*., 2006). The total organic carbon (TOC) content varied among the samples. The biochar from C had the largest amount followed by the combination of all three stems and it followed the order C>D>A>B. One of the important characteristics of biochar is its carbon (C) content which decides its agricultural and environmental benefits.

As it was observed, the lowest carbon content biochar was from the millet stem. Such variation had been reported for varieties of biochar produced from different feedstock (Rondon *et al.,* 2007). The results of this study falls within the ranges of 33.0 – 82.4 % reported from different feedstock (DeLuca *et al*., 2006). The results of percentage moisture content showed that millet stem biochar has the highest percentage moisture with biochar from rice having the lowest. However, sorghum biochar showed the highest percentage of ash with the rice and combination of the three having the same ash content.

Table 1: Physico-chemical Parameters of Biochars

Parameters	A	B	C	D
pH	11.95±0.139	12.30±0.202	11.56±0.10	12.20±0.017
CEC (%)	32.60±0.00	33.40±0.001	14.80±0.085	23.80±0.00
TOC (%)	41.00±0.001	37.70±0.0034	54.10±0.004	52.30±0.021
Ash (%)	14.50±0.01	16.50±0.00	23.50±0.00	14.50±0.01
Moisture (%)	2.5±0.001	3.5±0.002	3.0±0.004	3.0±0.00

Key: A = rice, B = millet, C = sorghum, D = mixture of ABC; TOC = Total Organic Carbon, CEC = Cation Exchange Capacity. Values are presented as mean± standard deviation of three analyses

Table 2: Physico-chemical parameters of the untreated soil

	Samples			
Parameters	**A**	**B**	**C**	**D**
pH	8.6±0.003	8.7±0.032	8.7±0.00	8.5±0.005
TOC (%)	2.23±0.003	1.54±0.030	1.92±0.100	1.84±0.003
CEC (%)	19.20±0.0034	10.80±0.120	14.8±0.003	15.0±0.0045
TP (mg/kg)	1.90±0.002	1.69±0.0034	1.28±0.002	1.83±0.0098
K (mg/kg)	21.79±0.101	18.46±0.0034	24.61±0.00	18.70±0.00
TN (%)	0.095±0.955	0.091±0.00	0.095±0.0012	0.084±0.045

Key: TOC = Total Organic Carbon, CEC = Cation Exchange Capacity, TN = Total Nitrogen, TP = Total Phosphorus. Values are presented as mean± standard deviation of three replicate analyses

Table 2 showed physicochemical parameters of the untreated soil with total phosphorus ranges between 1.28 – 1.90 mg/kg, and the values were below the value reported by (Tsafe *et al*., 2014) in similar work. The pH of the soil showed that it was mildly basic in all location. The TOC, CEC and total nitrogen (TN) were low. The low level of nitrogen indicated that the soil fall within the low fertility class. The potassium content was also low in all the samples. The physico-chemical parameters of the soil treated with biochar indicated that pH value did not vary much from the untreated soils (Table 3). The TOC decreased and increased after two and four weeks, respectively. The CEC increases after the application of the biochar throughout the samples apart from sample A which shows slight decrease after four weeks.

Generally, (NPK) concentration in the treated soil (Table 3 and 4) could be seen to have reduced more from the second week to the fourth week of application. The phosphorus content follows a similar pattern in all the samples before and after treating the soil with biochar, in which it increase and decrease in samples A and C, decreases and increase in sample B and decrease in sample D. In case of potassium (K) there was increase in samples A, C and D but sample B reduces from 19.23±0.00 to 8.720.0032 mg/kg. While the nitrogen content increase was only after the fourth week of application in sample C.

Table 3: Physico-chemical parameters of the soil treated with biochar after two (2) weeks of application.

	Samples			
Parameters	**Soil + A**	**Soil + B**	**Soil + C**	**Soil + D**
pH	8.3±0.0021	8.8±0.008	8.6±0.001	8.7±0.032
TOC (%)	1.67±0.0032	1.78±0.0076	1.80±0.00	1.78±0.00
CEC (%)	17.20±0.043	14.8±0.0054	14.60±0.006	16.80±0.012
TP (mg/kg)	2.03±0.005	1.44±0.0021	1.79±0.005	1.77±0.003
K (mg/kg)	5.64±0.00	19.23±0.00	4.62±0.007	6.67±0.004
TN (%)	0.098±0.00	0.088±0.00	0.074±0.003	0.095±0.054

Key: TOC = Total Organic Carbon, CEC = Cation Exchange Capacity, TN = Total Nitrogen, TP = Total Phosphorus. Values are presented as mean± standard deviation of three replicate analyses

Table 4: Physico-chemical parameters of the soil treated with biochar after four (4) weeks of application.

	Samples			
Parameters	**Soil + A**	**Soil + B**	**Soil + C**	**Soil + D**
Ph	8.6±0.003	8.9±0.0043	8.8±0.0082	8.3±0.0065
TOC (%)	2.84±0.0034	2.1±0.0043	1.98±0.0054	1.88±0.005
CEC (%)	17.0±0.0023	17.40±0.223	16.80±0.008	17.60±0.0023
TP (mg/kg)	1.55±0.2100	1.62±0.332	1.58±0.00	1.46±0.0012
K (mg/kg)	8.72±0.0032	7.18±0.004	7.18±0.0043	17.96±0.00
TN (%)	0.084±0.003	0.084±0.00	0.092±0.032	0.084±0.110

Key: TOC = Total Organic Carbon, CEC = Cation Exchange Capacity, TN = Total Nitrogen, TP = Total Phosphorus. Values are presented as mean± standard deviation of three replicate analyses

Table 5: Heavy Metals Concentrations (mg/kg) in soil before and after biochar application

	SAMPLES	Cd	Pb
Untreated Soil	A	1.190±0.014	4.350±0.005
	B	61.150±0.523	4.170±0.005
	C	68.200± 0.112	3.460±0.003
	D	47.200±0.223	2.790±0.007
Treated Soil After 2 Weeks	A1	0.912±0.026	4.110±0.003
	B1	52.000±0.124	4.130±0.003
	C1	49.200±0.221	1.900±0.018
	D1	ND	2.690±0.007
Treated Soil After 4 Weeks	A	0.753±0.332	3.910±0.008
	B	30.300±0.988	3.610±0.009
	C	ND	1.640±0.008
	D	ND	2.180±0.012

Values are presented as mean± standard deviation of three replicate analysis, ND = Not Detected

The concentration of heavy metals monitored in this work (Pb and Cd) is presented in Table 5. From the table it could be seen that both metals contents reduced sufficiently after treating the soil with the biochar. This agrees with (Cui *et al*., 2011). Biochars made from pine and oak wood and bark have been reported to remove ~100% Pb and ~50% Cd from the contaminated soil (Mohan *et al*., 2007). The decrease in the concentration was observed to be high for cadmium in D and C after fourth week of application of the char. But for lead the decrease was observed to be higher in sample C. Bioavailability of Pb in the soils had been found to decrease by 75.8 % with biochar treatment (Ahmad *et al*., 2012).

CONCLUSION

The results of this study showed that biochar has the potential to be developed as a viable tool for remediation of cadmium and lead contaminated soils. However, the biochar in this study were more effective for remediation of Cd contaminated soil than for Pb. Obviously, the biochar C & D could conceivably reduce the bioavailability and efficacy of both heavy metals in soil due to its high CEC, carbon content and high nutrient retention capacity. While arguments on the effectiveness of biochar appear reasonable, further research is needed prior to widespread application of biochar in soil remediation**.** Biochar as a potential technology for remediation of contaminated agricultural soils still have many aspects to be developed. Large scale field trials of laboratory findings are essential before operational scale remediation projects could implemented. Furthermore, incorporation of the char with other soil amendments techniques which can mitigate contamination problems could be studied to determine the advantages or otherwise.

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