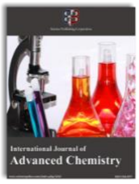
International Journal of Advanced Chemistry, 9 (2) (2021) 150-155

International Journal of Advanced Chemistry

Website: www.sciencepubco.com/index.php/IJET

Research paper



Pretreatment of biomass through torrefaction, for improved soil quality

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Abstract

Growing concerns of emissions from the burning of biomass residues have led to a demand for more efficient technologies to mitigate the effect of excess residues. The moisture content of the biomass used decreased from 8.1% to 5.5% after the torrefaction process. Results of the % volatile content indicated a significant reduction from 70.05% to 12.4%. The ash content increased from 5.25% to 10.7%, the percentage amount of fixed carbon increased significantly, from 16.6% to 71.4%. % C increased from 46.51% to 65.15%, while % O reduced significantly from 47.44% to 29.57%. There was a slight increase in % N, with a reduction in % H. Torrefied corn stalks were applied to soil samples, using three different application rates (1%, 3% and 5% w/w). The addition of torrefied corn stalks led to increase in soil pH across all 3 treated samples. Electrical conductivity values increased from the control value of 1.87 to as high as 4.05. There were also positive improvements in the cation exchange capacity and water holding capacity of all treated soil samples. Treatment of soil with torrefied biomass improved soil quality significantly, with the potential of fostering food security.

Keywords: *Biomass; Corn Stalks; Pretreatment; Soil Quality; Torrefaction.*

1. Introduction

Biomass, which can broadly be defined as an organic material, derived from plant and animal origin, occurs abundantly in nature (Contescu et al. 2018). They can exist as non-woody biomass, forest residues and agricultural biomass (Jawaid et al. 2017). Biomass is mostly associated with their usage as a source of green energy (Bridgeman et al. 2008). However, besides their energy applications, biomass can also be used as feedstock in other sectors such as the chemical, metallurgical and agricultural industries (Sobek & Werle, 2018). In our environment, soil degradation has been an age long problem and can cause considerable damage to land productivity, leading to a negative impact on food security around the world (Tully et al. 2015). In Nigeria, improving nutrient-poor soil is essential for increasing agricultural productivity. Appropriate strategies to promote improved soil management policies are therefore necessary to satisfy the need for increased food production (Mohawesh & Durner, 2017). The use of naturally-derived soil enhancers particularly torrefied biomass made from post-harvest biomass residues to increase soil water and nutrient holding capacity have become a significant tool to improve soil management, particularly in regions with low water availability and fragile soil ecosystems (Mohawesh 2016).

Raw biomass materials have several challenges that limit their utilization for industrial applications. Some of these challenges include low heating value, high moisture content, hygroscopicity and substantive smoke emission during combustion (Tumuluru et al. 2011). Also, due to rigidity, mechanical strength and poor flow, raw biomass requires high grinding energy (Eseyin et al. 2015). As a result, it is necessary for biomass to undergo pretreatment to overcome these challenges. One of the most feasible methods for improving the properties of raw biomass is the process of torrefaction (Van der Stelt et al. 2011).

Torrefaction is a thermal conversion method of biomass operating in the low temperature range of (200 – 300 °C) under atmospheric conditions in the absence of oxygen. During this process, the biomass undergoes a series of decomposition reactions that cause the liberation of volatile organic compounds (Bergman & Kiel, 2005). After complete devolatilisation of the biomass, the final product that remains is referred to as the torrefied biomass (Ciolkosz & Wallace, 2011). This process improves both the physical and chemical composition of the biomass, thereby, increasing its viability for application in various sectors (Medic et al. 2011).

This resultant torrefied biomass with improved characteristics, when added to soil as an enhancer, generally upgrades the physicochemical properties. It improves soil-water holding capacity (Karhu et al. 2011; Lal, 2013) increases cation exchange capacity (CEC) (Abdelhafez et al. 2014; Martin et al. 2015), decreases bulk density (Mukherje & Lal, 2013) and minimizes the loss of nutrients and other agricultural chemicals in soil run-off (Lehmann & Joseph, 2009). It decreases soil penetration resistance, increases aggregation and infiltration, and also improves electrical conductivity (Busscher et al. 2010). Torrefied biomass can persist in soils for years and is highly resistant to microbial decomposition, hence it can enhance sequestration of carbon for long periods (Schmidt & Novak, 2000; Downie et al. 2011). It is also known to have a liming effect on the pH of soils that are generally acidic (Dume et al. 2015).

The goal of this research is to apply torrefaction, as a viable pretreatment method for raw biomass, using corn stalks and subsequently, applying the resultant torrefied corn stalks to improve the quality of soil used for farming.

2. Methods

2.1. Sampling, preparation and torrefaction of corn stalks

The corn stalks used in this research were sampled from a farming area in Utan, Jos North Local Government Area of Plateau state, Nigeria. The corn stalks were part of the residue left behind, after crop harvest. Samples were taken from different portions of the farm and then chopped into smaller pieces and left to dry in the open for 72 hours. After this, the corn stalks were oven dried at 120 ^oC for 24 hours, to further remove moisture. The biomass sample was then ground and sieved through a 2mm mesh, ready for the bio-thermal treatment. The torrefaction process was carried out, using a GLM carbolite furnace. The dried corn stalks were placed in a covered crucible and put in a carbolite furnace at 280 ^oC for thirty minutes.

2.2. Proximate and ultimate analysis

Moisture content: Both raw and torrefied samples (2g each), were weighed into 2 separate petri dishes of known weight. Both petri dishes were then placed in an oven at 100 ^oC for 24 hours. The weights of the samples were then retaken and moisture content was calculated using the formula

$$\frac{Wo-W}{Ws} \text{ x } 100 \tag{1}$$

Where;
W_o = Initial weight of the sample + petri dish, W = Weight of sample + petri dish after drying, W_s = Weight of the sample used.
Ash content: Both raw and torrefied samples (2g each) were weighed into two different crucibles of known weight. The crucibles were then placed in a GLM Carbolite furnace at 400 ^oC for 4 hours. After this, the crucibles were removed and put in a desiccator to cool and the samples were then re-weighed. The ash content of both the raw and torrefied corn stalks were then determined, using the formula

$$\frac{Wo-W}{Ws} \text{ x } 100 \tag{2}$$

Where;
W_o = Initial weight of the sample + crucible, W = Final weight of sample + crucible after ashing, W_s = Weight of the sample used.
Volatile matter (VM): Both raw and torrefied samples (2g each) were weighed in 2 separate crucibles of known weight and covered with a lid before it was placed in a GLM Carbolite furnace at temperature of 400 ^oC for 10 minutes. The crucibles were first cooled inside a desiccator and then, the resulting samples were weighed again. Weight loss was reported as the volatile matter on a percentage basis, using the formula

$$\frac{Wo-W}{Ws} \text{ x } 100 \tag{3}$$

Where;
W_o = Initial weight of the sample + crucible, W = Final weight of sample + crucible, W_s = Weight of the sample used.
Fixed Carbon (FC): Fixed carbon of both the raw and torrefied corn stalks was calculated, using the equation of the sum of the percentages of ash content, moisture content and volatile matter subtracted from 100. Therefore

$$\% \text{ FC} = 100 – (\%\text{Ash} + H_2O\% + \%\text{VM}) \tag{4}$$

Ultimate analysis was carried out, using an Exeter Analytical Incorporation (EAC CE – 440) elemental analyzer. The elemental composition (C, H and N) of the raw and torrefied corn stalks was determined. The amount of oxygen (O) that was present in the samples was calculated as the difference between the sums of C, H and N from 100, that is;

$$(\%\text{Oxygen} = 100 – \%\text{C} + \%\text{H} + \%\text{N}) \tag{5}$$

The ultimate analysis of both the raw and torrefied biomass was carried out, using the ASTM D3176-09 (ASTM D3176 2009).

2.3. Calorific values (HHV and LHV)

Higher heating value (HHV): The HHV of both the raw and torrefied biomass was calculated theoretically, from the Dulong's equation which is a mathematical model used to calculate heating value, based on the elemental composition of the sample (Demirbas 2006). The Dulong's equation is given as:

$$\text{HHV (MJ/Kg)} = \frac{33.5 \text{ x } \%C}{100} + \frac{142.3 \text{ x } \%H}{100} + \frac{15.4 \text{ x } \%O}{100} + \frac{24.5 \text{ x } \%N}{100} \tag{6}$$

Where %C, %H, %N, %O are the percentage weights of the elements obtained from the ultimate analysis of the raw and torrefied samples.
Lower heating value (LHV): The LHV of both the raw and torrefied corn stalks were calculated from the HHV, using the IPCC formula (IPCC 2006) given as;

$$\text{LHV (MJ/Kg)} = \text{HHV} – 0.212 \text{ x } H – 0.0245 \text{ x } M – 0.008 \text{ x } Y \tag{7}$$

Where;
HHV = Higher heating value, H = Percentage hydrogen (from ultimate analysis), M = Percentage moisture, Y = Percentage oxygen (from ultimate analysis), 0.212, 0.0245 and 0.008 are constants.

2.4. Fourier transforms infrared spectroscopy (FTIR)

The FTIR analysis of both raw and torrefied corn stalks was carried out, using ASTM E1252 (ASTM E1252-98 2021). Each sample for FTIR analysis was prepared, by grinding and mixing 1 mg of the raw and torrefied corn stalks respectively with 200 mg of IR-grade KBr. The FTIR spectrum was recorded over a wave-number range of 4000 cm^{-1} – 400 cm^{-1}.

2.5. Sampling of soil and preparation of the treated soil

The soil used for this research was sampled from a farming area in Utan, Jos North Local Government of Plateau state, Nigeria. The farming area from which the soil samples were taken was the same as the one from which the corn stalks were sampled. Various samples of soil were taken from different spots at a depth of 0-20 cm and then mixed thoroughly to provide a composite sample. The soil was air dried for 24 hours and a portion of the soil was stored in a polybag without any form of treatment to serve as the control sample. Three different mixing rates of soil/torrefied corn stalks were then carried out on the remaining sample by thoroughly mixing 3 different portions of 2Kg soil samples with 20g, 60g and 100g of the torrefied corn stalk in a 1%, 3% and 5% w/w application rates respectively.

The treated samples were allowed to stay for 24 hours, transferred into a nursery polybag and returned to the farmland where the soil was originally sampled. A portion of the farmland was used as a test ground and the treated soil samples were left for one month to settle under natural conditions. The treated soil samples were then retrieved and air dried for 24 hours after which characterization of both untreated and treated soil samples were carried out to investigate changes in selected soil properties.

2.6. Determination of pH and electrical conductivity of soil samples

The pH and electrical conductivity (EC) of the soil were determined, using HI 9813-6 pH meter, Hanna Instrument USA and applying the ASTM-D 7946 (ASTM-D 7946 2017). 50g of sample was poured into a 200 ml beaker and 50 ml of deionized water added. The electrode was rinsed with distilled water and then calibrated with a buffer of 4, 7 and 9 and then placed in the solution, while waiting for 30 seconds for the electrode to reach thermal equilibrium with each of the buffer solutions respectively. The electrode was placed in the sample to measure the pH. After the measurement was stabilized, the pH reading was taken. The MODE was then changed to that of electrical conductivity to read the measurement in Siemens per meter (S/m).

2.7. Determination of cation exchange capacity

Cation exchange capacity (CEC) of the samples was determined, using ASTM D7503-10 (ASTM D7503 2010) for soil sample, by saturating the sample's exchange complex with 1N sodium acetate solution (pH 8.2). 1g of each sample was leached with sodium acetate solution, for replacement of exchangeable cations; Ca^{2+}, Mg^{2+}, K^{+} and Na^{+} ions (base cations). The excess salts were washed down by ethanol and the adsorbed respective ions were released, using 1N ammonium acetate (pH 7.0) solution. The ions released from the exchange spots were measured, using a flame photometer. The cation exchange capacity was determined by the summation of exchangeable acidity (acid cations) and the base cations in meq/100 g sample. It is important to note that if the buffer pH is 7 or above, then there is no exchangeable acidity and CEC will be the sum of base cations only.

2.8. Determination of water holding capacity

The water retention was done following the ASTM D2216 (ASTM D2216 2019), in which 20 g of each soil sample of equal grain size, was air dried, weighed and completely soaked in water in a beaker. The sample was then poured into a Whatman filter paper and placed under vacuum filtration to drain off the excess water. After it was noticed that no more water was coming out of the filter paper, the sample was reweighed to see the difference in weight. The amount of the soil weighed (20g) was then subtracted from the weight after drainage of excess water (new sample weight – old sample weight) to determine the maximum water retained.

3. Results and discussion

3.1. Characterization of raw and torrefied corn stalks

Table 1: Proximate Analysis of Raw and Torrefied Corn Stalks

Sample (corn stalks)	%Moisture content	% Ash content	% Volatile Matter	% Fixed Carbon
Raw	8.10	5.25	70.05	16.60
Torrefied	5.50	10.70	12.40	71.40

The results of the proximate and ultimate analyses of the raw and torrefied corn stalks are shown in Table 1. It was observed that the moisture content of the corn stalks decreased from 8.1% to 5.5%, after the torrefaction process, this can be attributed to the loss of hydroxyl (OH) groups caused by dehydration, during the process. Bridgeman et al., reported that other organic and inorganic products which are also released from the volatile components during torrefaction, contribute to moisture loss (Bridgeman et al 2008). Results of the %volatile content indicated a significant reduction after torrefaction, from 70.05% to 12.4%. This was due to the increase in torrefaction temperature that resulted in a considerable amount of mass loss from the release of volatiles.

The ash content was observed to have increased from 5.25% for the raw sample to 10.7% after torrefaction. The increase is due to increased carbon content of the torrefied corn stalks, relative to the raw sample. The higher the carbon content, the more ash residue left behind after the process. From the results, the percentage amount of fixed carbon increased significantly, after torrefaction from 16.6% to 71.4%, due to conversion of hemicellulose into more thermally stable compounds (Ciolkosz & Wallace, 2011).

Table 2: Ultimate Analysis and Calorific Values of Raw and Torrefied Corn Stalks

Sample (corn stalks)	% Carbon	% Hydrogen	% Nitrogen	% Oxygen (by difference)	HHV (MJ/Kg)	LHV (MJ/Kg)
Raw	46.51	5.70	0.35	47.44	31.08	29.33
Torrefied	65.15	4.66	0.62	29.57	33.16	29.75

Results from Table 2 show an increase in % C from 46.51 % to 65.15% after torrefaction, while % O reduced significantly from 47.44% to 29.57 %. Also there was a slight increase in % N after torrefaction, with a reduction in % H. The liberation of volatile groups resulted in an increase in carbon content and a decrease in hydrogen and oxygen contents, due to the formation of H_2O, CO and CO_2 (Wu et al. 2012). HHV value of the torrefied corn stalks was observed to increase from 31.08 to 33.16 MJ/Kg after torrefaction. The increase in the heating value was due to a decrease in the moisture content and an increase in the carbon content of the samples.

The bonds left behind after torrefaction were mostly C-C bonds, which generally have more energy than C-O or C-H bonds and therefore, directly results in a higher calorific value for the torrefied biomass (Stelte et al. 2012). The lower heating value (LHV) depends on the HHV and so, an increase in HHV of the torrefied corn stalks resulted in a corresponding increase in LHV when compared to the raw sample.

3.2. FTIR analysis of raw and torrefied corn stalks

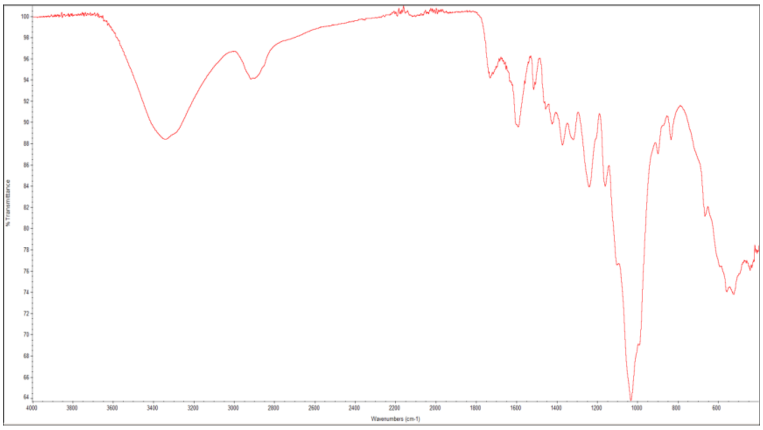

Fig. 1: FTIR Spectrum of Raw Corn Stalks.

FTIR spectrum of the raw sample from Fig. 1 shows absorption bands at 3600-3000 cm^{-1} corresponding to O-H stretching of alcohol. Absorption bands at 1750-1735 cm^{-1} indicated the presence of C=O stretching bonds which corresponded to the presence of esters in the sample. The band observed at 1620-1610 cm^{-1} was indicative of the C=C stretching of an αβ unsaturated ketone. The presence of a band at 1550-1500 cm^{-1} corresponded to the N-O stretching of a nitro group. There was also a band at 1050-1040 cm^{-1} is indicative of the C-O stretching of a carbonyl group.

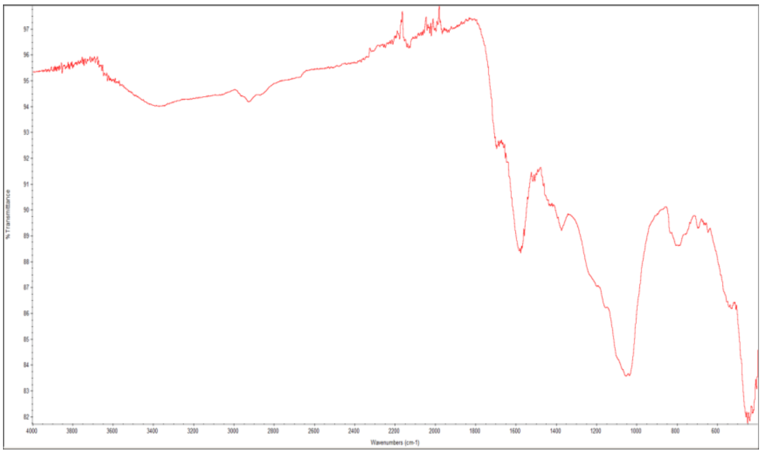

Fig. 2: FTIR Spectrum of Torrefied Corn Stalks.

The spectrum of the torrefied corn stalks from Fig. 2 shows differences from Fig. 1, due to the distortions in the structuring of the cell wall of the biomass that was caused by increase in temperature, during the torrefaction process. The absorbance of OH at 3600-3000 cm^{-1} decreased significantly which indicated the loss of OH group as a result of dehydration. A band at 2140-2100 cm^{-1} indicated C≡C alkyne stretching. Band absorbance at 1085-1050cm^{-1} was due to C=O stretching of a carbonyl group while the band at 840-790 cm^{-1} indicated a C=C bending of an alkene. The general increase of absorption in the carbonyl region was due to the degradation and depolymerization of hemicelluloses, lignin condensation, carboxylation of polysaccharides and opening of aromatic rings. This agrees with studies carried out by Gonzalez-Pena & Hale, (Gonzalez-Pena & Hale 2009).

3.3. Characterization of normal and treated soil samples

Table 3: Characterization of Normal and Treated Soil Samples

Parameter	Normal soil (control)	1 % w/w treated soil	3 % w/w treated soil	5 % w/w treated soil	FAO standard
pH	6.5	6.6	6.8	7.0	6.5 – 7.5
EC (S/m)	1.87	2.22	3.69	4.05	2 – 4

Exch. Ca^{++} (meq/100g)	3.55	4.23	5.49	6.23	
Exch. Mg^{++} (meq/100g)	0.22	0.26	0.36	0.41	
Exch. K^{+} (meq/100g)	1.25	1.43	2.29	2.35	
Exch. Na^{+} (meq/100g)	0.09	0.10	0.13	0.14	
CEC (meq/100g)	5.11	6.02	8.27	9.13	8 – 15
Water Holding Capacity (%w/w)	5.50	7.25	10.30	14.45	

CEC: Cation Exchange Capacity; EC: Electrical Conductivity; Exch: Exchangeable

Table 3 shows that relative to the control sample, it was clear that the addition of torrefied corn stalks, led to increases in soil pH across all 3 treated samples. The optimal pH range of farming soils according to the Foods and Agricultural Organization, (FAO 2020) is 6.5 – 7.5. Therefore, results show an increase in pH, within the acceptable limits. The increase in pH across the treated soil samples can be explained by the potential effect of torrefied biomass pH to modify soil pH, as torrefied biomass is a highly alkaline product, due to the existence of organic ions and inorganic carbonates. Hence, the application of torrefied biomass could be a sufficient remedy for treating acidic soils. This agrees with the work reported by Novak et al. (Novak et al. 2009).

Electrical Conductivity (EC): The results obtained for the treated soil samples in comparison with the control, showed significant increases in the electrical conductivity of the treated samples, as torrefied corn stalks application rate increased. Optimal range of EC for sandy loam soils is 2-4 S/m (FAO 2020). The results indicated an EC value below the standard for the control soil sample. However, all 3 treated soil samples showed an increase in EC which clearly indicated that the application of torrefied corn stalks had a positive effect on the EC of all treated samples in this study. The increase of soil EC with increasing application of torrefied corn stalks can be attributed to the salts contained within the torrefied samples, which was expected to increase salinity and consequently, elevate soil EC. This is in line with studies carried out by Chintala et al., (Chintala et al. 2014) and Al-Wabel et al., (Al-Wabel et al. 2017) who reported increase in soil EC on treatment with torrefied biomass.

Cation Exchange Capacity (CEC): It was inferred from the results in Table 3 that the contents of exchangeable base cations (Ca^{2+}, Mg^{2+}, K^{+} and Na^{+}) increased gradually with increasing application rate which in turn resulted in a corresponding increase in CEC. The increase in CEC in the treated soil samples could be attributed to the nature of the torrefied corn stalks, which had high surface area, with the presence of polar and non-polar surface sites, making it a good soil nutrient-retaining additive (Martin et al. 2015). Soils with low CEC are often low in fertility and more vulnerable to soil acidification. The consequence of improving CEC is that it improves soil fertility through greater retention of soil nutrients and protects against leaching.

Soil Water Holding Capacity: Results from Table 3 show a progressive increase in the water holding capacity of treated soil samples as application rate of torrefied corn stalks increased. This agrees with the studies of Ouni et al., (Ouni et al. 2013) who reported increases in soil water holding capacity when treated with torrefied biomass. Suliman et al., (Suliman et al. 2017) explained that the increase in soil water retention may be due to the high oxidation rates of torrefied biomass that could make soil surface more water friendly by increasing the amount of oxygen containing functional groups.

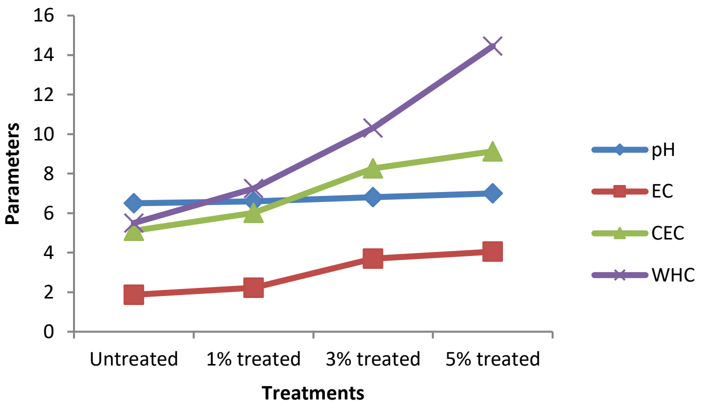

Fig. 3: Effects of the Different Application Rates of Torrefied Corn Stalks on pH, Electrical Conductivity, Cation Exchange Capacity and Water Holding Capacity of Untreated and Treated Soil Samples.

Fig. 3 shows the graphical illustration of the effects of the addition of torrefied corn stalks at different application rates, to soil samples with data obtained from Table 3. The graph shows positive increases in all of the soil parameters studied. The addition of torrefied corn stalks had the least significant effect on soil pH, while the most significant effect was observed on the soil water holding capacity.

4. Conclusion

The treatment of soil samples with torrefied corn stalks, resulted in an increase in pH from 6.5 to 7.0, the EC of the soil samples increased significantly from 1.87 S/m in the control sample to 4.05 S/m in the 5% w/w treated sample. CEC of all 3 treated samples showed varying increases in value from 6.02 meq/100g to 9.13 meq/100g while the WHC improved across all treated samples from 5.50 %w/w to 14.45 %w/w. It can be concluded therefore that the treatment of soil samples with torrefied corn stalks, had significant positive effects on the pH, electrical conductivity, cation exchange capacity and water holding capacity of the soil samples used.

Increase in the application rate of torrefied corn stalks, correlated with corresponding improvements in soil properties. The above information therefore suggests that treatment of soil with torrefied biomass can provide an environmentally friendly way of improving soil quality and also curtail the extent of environmental pollution, caused by littering of post-harvest residues. Improving the quality of soil with the use of torrefied biomass can play an essential role in increasing the fertility of farmlands, thereby advancing food security to meet the needs of our ever growing population.

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