

Pyrolysis of maize cob at different temperatures for biochar production: Proximate, ultimate and spectroscopic characterisation

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Abstract: Adopting the concept of the waste to wealth approach, agricultural waste from maize cob could be transformed into a renewable form of energy through thermo-chemical methods of treating the biomass. This method can be utilised for biochar production. The utilisation of biochar has several significant applications. These applications include the enhancement of the soil through amendment, stimulation of crop production by a variety nutrient inputs in the soil, etc. In this research work, a biochar was obtained through a slow pyrolysis process of maize cob waste. This experiment was carried out using a small-scale muffle furnace and subjecting the feedstock to heating at different temperatures (300, 400, 500 °C). The biochar was produced and characterised by a proximate analysis, scan electron microscope (SEM), Fourier transform infrared (FTIR) spectroscopy, while the surface area was determined by Saer's method. The effect of the temperature on the yield of the biochar was investigated. The results show that the biochar yield decreases with an increasing temperature for the maize cob biochar at 300, 400 and 500 °C. The results of the physiochemical properties showed that the temperature has a great impact on the physicochemical properties of the biochar. The biochar produced at 300 °C has the highest fixed carbon content of 60.5%. The largest surface area was (281.8 m²·g⁻¹) at 500 °C.

Keywords: agriculture; bioenergy; characteristics; Saer's method; temperature

Energy stands out as the most essential factor for the advancement of any nation. The overreliance on petroleum derivatives as a preeminent source of energy has caused adverse environmental changes. This has led to global climate change, environmental degradation, and several health issues (Goldemberg et al. 2004). Climate change occurs as a result of the anthropogenic emanation of toxic gases from human engagement in the environment. Carbon dioxide (CO₂) accounts for around 78% of the total greenhouse emissions from the combustion of fossil fuels and industrial processes are a major contributor to global warming. The expeditious growth in the world's economy and population has been a major factor contributing to the increase in carbon emissions (IPCC 2014). Adequate steps to address this global crisis are desperately needed.

One of the key solutions to these environmental problems is bioenergy (energy obtained from plants and animal wastes). Biomass is seen as an alternative way to solve this energy crisis.

Biomass can be defined as any organic substance derived from natural occurring matter. This includes all plant and animal matter (Loppinet et al. 2008). The United Nations Framework Convention on Climate Change (UNFCCC 2005) defines biomass as an organic material derived from plants, animals and microorganisms that are not fossilised, but rather biodegradable. These include agriculture, forestry and its associated materials, by-products, as well as municipal and industrial waste that is non-fossilised and has a biodegradable biological fraction. Biomass has been considered by numerous researchers as a significant source of alternate energy and a tool for combating

climate change (Berdes et al. 2003; Demirbas 2005; Goldemberg 2007; Lund 2007). A huge amount of biomass residue from plants can be converted into a useful form which could be beneficial and serve as a renewable source of energy. Plant-based feedstocks could be transformed into biochars, bio-oils, and pyrolysis gases (Nanda et al. 2014).

Two techniques can be used to primarily transform biomass into renewable energy sources. Through thermochemical pathways while the other one is through biochemical pathways (McKendry 2002). Bio-chemical pathways require the use of catalysts and microbes as the main source of energy from the biomass, while thermal and chemical catalysts are used in the thermochemical pathway to produce energy. The biochemical pathway of processing the biomass is aimed at the derivation of ethanol while the thermochemical pathway of processing the biomass is aimed at obtaining biochars, bio-oils and gases (Decker et al. 2017).

Pyrolysis is a thermo-chemical process which involves the disintegration of large molecules of a biomass under a limited air supply. This biomass is further broken down into relatively small fractions of biochar, bio-oil and biogas (Demirbas and Arin 2002). Pyrolysis can be divided into three main classes according to the process conditions used (Guedes et al. 2018), which could be slow, flash and fast pyrolysis. Slow pyrolysis operates at a lower temperature with a lower heating rate and a prolonged residence time which contributes to the char output. The flash pyrolysis method typically occurs over a short time usually in seconds, usually at a short residence time with a high heating intensity. Fast pyrolysis occurs at a moderate temperature and a short residence time, generally this favours bio-oil products. To produce the biochar, the slow pyrolysis process was used to obtain a biochar from maize cobs.

Biochar has several applications which could be physical and chemical. Adding biochar to soils will boost the soil fertility and alters the soil properties, leading to a boost in crop yields that allows for reduced agrochemical applications. Liquid and gas co-products derived from biomass pyrolysis can be used as a source of fuel and power generation, thereby reducing fossil fuel consumption and minimising greenhouse gas emissions.

For bioremediation and adsorption purposes, the application of biochars cannot be overemphasised due to the specific surface area and the pores that exist in the inner layer of the biochar. This enables the application of biochars to adsorb substances, both or-

ganic and inorganic pollutants. Biochars are also suitable for wastewater treatment (Ahmad et al. 2014). The textile industries are known for the discharge of a large amount of dyes and pigments into the surrounding wastewater. Biochars can be an economically viable means to adsorb these dyes. There is, therefore, a need to study the physicochemical properties of a maize cob biochar to aid its applications.

Agricultural waste from maize cobs is a high potential biomass feedstock for biochar production. In this research work, corn cobs were used as feedstock to obtain a biochar. Slow pyrolysis was adopted to produce the biochar at three different temperatures (300, 400, and 500 °C). The purpose of this research is to understand and compare the feedstock characteristics and to study the temperature effect on the percentage of the biochar produced.

MATERIAL AND METHODS

Sample collection and preparation

The feedstock from maize cob waste was obtained from Landmark University maize shelling unit located at landmark research farm in Omu-Aran, Kwara. For 24 h, the samples were dried at 106 °C with the aid of a gen laboratory oven before embarking on the experimental procedure to extract the moisture.

Carbonisation of biochar

The pyrolysis experiments were carried out in a box type muffle furnace (at a 5-kW power output, 280 V, a temperature of 1 400 °C, and a workroom size 300 × 200 × 120 cm). The feedstock was placed in a ceramic crucible having 15.5 cm internal length, 10.2 cm internal width and 5.5 cm internal height with a lid and subjected to pyrolysis at different temperatures (300, 400 and 500 °C) for 30 minutes. A pyrolysis heating rate of 10 °C·min⁻¹ was employed. After pyrolysis, the biochar sample was left inside the furnace to cool to room temperature. The biochar samples obtained were labelled as MC300, MC400 and MC500, respectively. The biochar samples were weighed after cooling. The yield of the biochar was calculated using following Equation (1):

$$\text{Yield (\%)} = \frac{\text{mass of biochar (g)}}{\text{oven dry mass of feedstock (g)}} \times 100 \quad (1)$$

Physiochemical characterisation of the biochar

Determination of pH. The pH was calculated using the Abdolali et al. (2015) method. One gram each

of the MC 300, MC 400 and MC 500 carbon samples were weighed and put in a 100 mL water-containing beaker and then boiled in a shaking mantle for 5 minutes. Each solution was diluted to 200 mL and cooled at room temperature. After this procedure, the pH was measured using a C720 pH meter (Consort bvba, Belgium).

Determination of the moisture content. In determining the samples' moisture content, the thermal drying method was used. One gram of the biochar sample was measured in triplicate and placed in a dried crucible and measured. The crucible was placed in an oven at 105 °C and the sample was dried to a constant weight for 4 h according to the method of Rengaraj et al. (2002). The difference between the initial and final mass of the carbon representing the moisture content is shown in Equation (2).

$$\text{Moisture (\%)} = \frac{(\text{initial mass} - \text{moisture mass})}{\text{initial mass}} \quad (2)$$

Determination of the volatile matter. One gram of each sample was heated at a temperature of 500 °C for 10 minutes. The volatile matter was calculated using this Equation (3):

$$\text{Volatile matter (\%)} = \frac{\text{weight of volatile (g)}}{\text{oven dry weight (g)}} \quad (3)$$

where: the volatile weight – the difference in the weight before and after heating the sample.

Determination of the ash content. In order to determine the ash content, three crucibles of MC 300, 400 and 500 was preheated to about 500 °C. This was followed by rapid cooling in a desiccator and was later weighed after cooling. One gram of MC 300, 400 and 500 was transferred into the crucible and reweighed. The crucibles containing the maize cob biochar were then placed in the furnace and the temperature was allowed to rise to 500 °C for about 1 h and 30 min, and allowed to cool in a desiccator to room temperature (30 °C) and reweighed. The ash and fixed carbon (C) was calculated using the Equation (4–5).

$$\text{Ash (\%)} = \frac{\text{weight}}{\text{oven dry weight}} \times 100 \quad (4)$$

$$\text{Fixed C (\%)} = 100 - (\text{ash \%} + \text{volatile matter \%}) \quad (5)$$

Determination of the bulk density. The bulk density was determined by using the methods of Sugumaran et al. (2012) with a slight modification. A glass cylinder (25 mL) was filled to a specified volume with a 40-mesh powder carbon sample of each of the samples and dried in an oven at 105 °C overnight. The cylinder was tapped for 1–2 min to compact the carbon and the bulk density was calculated and presented as g·mL⁻¹ in the following Equation (6):

$$\text{Bulk density} = \frac{\text{mass of dry sample (dry)}}{\text{volume of measuring cylinder}} \quad (6)$$

Determination of the surface area by Saer's method. The surface area determined by Saer's method was carried out following the procedure from the literature and a previous study (Saer 1956; Dada et al. 2013). This was undertaken by introducing 1.5 g of the biochar (MC300, MC400 and MC500) into a 250 mL conical flask containing 100 mL of 0.1 M HCl at pH 3. Thereafter, 30 g of NaCl was added and the volume was made up to 150 mL using deionised water. The resulting solution was titrated with 0.1 M NaOH on a temperature regulated magnetic stirrer at 298 K with constant stirring. The volume required to raise the pH from 4 to 9 was recorded and this was used to compute the surface area using Equation (7).

$$S \left(\text{m}^2 \cdot \text{g}^{-1} \right) = 32V - 25 \quad (7)$$

Spectroscopic characterisation (FTIR, SEM)

Fourier transform infrared spectroscopy (FTIR analysis). Characterisation using FTIR was used to obtain the infrared spectrum resolution data over a wide spectral range. FTIR provides the functional group distinction and analysis of the chemical bonding data by an analytical operational tool. FTIR was used in this project to determine the functional group of the most active elements of the material and because the rate of the carbon reaction is based on the group of chemical-active biomass components that are used as the feedstock. A FTIR-8400S spectrometer (Shimadzu, Japan) was used.

Scanning electron microscopy (SEM) analysis. SEM is an extraordinary spectrum-depth device with high-resolution imaging. It shows topographical, structural and elementary data up to 200 000 × at low magnifications (Abdolali et al. 2015).

In this research, SEM was used for the morphological surface view of the material to determine wheth-

er the material is a sufficiently carbonaceous material that would be suitable for amendment purposes and adsorption due to the microspores. A model JSM-7900F (Jeol, USA) was used for the SEM.

RESULTS AND DISCUSSION

Determination of the pH of biochar. Table 1 shows the obtained results from the determination of the pH of the maize cob biochar at three different temperature (300, 400 and 500 °C). A pH value of 7.08 was recorded at 300 °C, 7.69 at 400 °C and 7.07 at 500 °C. These results were considered to agree with existing literature references, which suggest that dry biochars through the pyrolysis process are mostly alkaline (Inyang et al. 2010; Enders et al. 2012). The decrease in the pH at 500 °C could be associated with the presence of a high ash content.

Effect of temperature on the physiochemical characteristics of the biochar

Table 2 shows the percentage composition of the ash, volatile matter, fixed carbon and bulk density of the biochar obtained through pyrolysis (at 300, 400 and 500 °C) for the maize cob biochar. The results show that pyrolysis temperature has a significant impact on the percentage yield of the fixed carbon and volatile matter in comparison to the ash and bulk density. For the maize cob, the results show that at a higher temperature, a greater fixed carbon yield could be obtained. This is in agreement with the results of other existing literature involving pyrolysis of woodchips (Mašek et al. 2013) and pomegranate seeds (Uçar et al. 2009).

The percentage of the obtained fixed carbon increased from 47% to 57%. The result of the obtained fixed carbon is dependent on the results of % volatile matter of the biochar sample. The volatile matter in the biochar sample was obtained through high heating of the feedstock in which the transformation of the biochar to a gaseous phase occurs during pyrolysis of the biomass. The more the volatile matter was forced out during pyrolysis, the greater the fixed carbon content that could be obtained as reported

Table 1. pH of maize cob biochar

Biochar samples	pH reading
MC300	7.08
MC400	7.69
MC500	7.07

Table 2. Proximate analysis of the maize cob biochar

Biochar samples	MC300	MC400	MC500
Yield (%)	34.30	24.00	22.00
Ash (%)	35.00	31.00	20.50
Volatile (%)	18.00	20.50	22.50
Fixed carbon (%)	47.00	48.50	57.00
Bulk density (kg·m ⁻³)	9.64	8.93	7.90

by Paethanom et al. (2012). The results show that the percentage of the volatile matter in the MC sample biochars increases from 18% to 22.5%. This shows that the pyrolysis temperature has a significant impact in expelling the volatile component in the biochar during pyrolysis.

Effect of temperature on the surface area (Sear's method). Figure 1 presents the effects of the temperatures on the surface area of the biochar. During the heating process, the surface area of the biochar has been observed to increase with an increase in temperature. The higher the temperature, the more the surface area expands, this is in agreement with the findings of previous studies on the surface area of biochars (Lehmann and Joseph 2009). The surface area of the biochar from the MC sample at 300, 400 and 500 °C were 199 m²·g⁻¹, 231 m²·g⁻¹ and 291.8 m²·g⁻¹, respectively. The surface area of the biochar at 500 °C was shown to have the highest surface area due to the impact of the pyrolysis temperature.

Effect of temperature on the yield of the biochar. Figure 2 shows the temperature effect at different pyrolysis temperatures on the percentage yield of the maize cob biochar. The percentage yield was determined by using of crucibles at different temperatures. During the pyrolysis process, the temperature of the muffle furnace was raised and maintained at a peak temperature for 30 min before it was

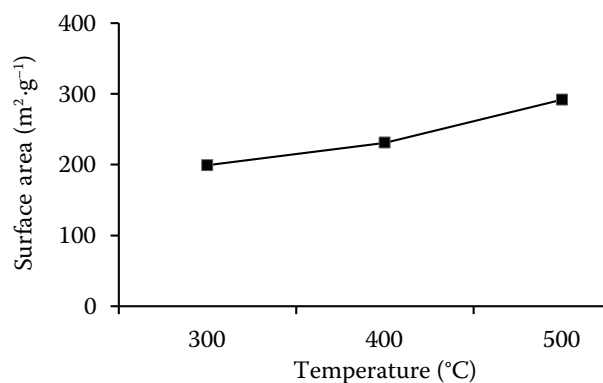


Figure 1. Surface area of the maize cob biochar

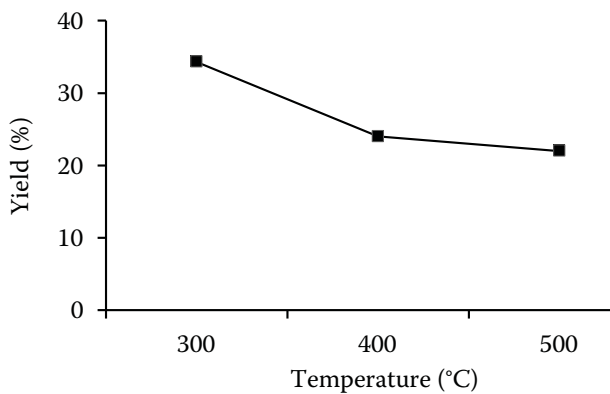


Figure 2. Biochar yield of maize cob

cooled down at room temperature. As the temperature rose from 300 °C to 400 °C, the biochar yield fell dramatically from 34.3% to 24% for the MC samples. This could be as a result of the decomposition of the lignocellulose material found in the various biochars at different temperature ranges according to (Intani et al. 2016). The temperature was then further increased from 400 °C to 500 °C, where the obtained biochar yield decreased from 24% to 22% for the MC samples. The obtained results reveal that most of the volatile matter had been removed at a low temperature. Ultimately the % yield of the biochar decreases with an increasing temperature.

Ultimate analysis. Table 3 shows the ultimate analysis of the generated biochar at different temperatures. This reveals the elemental composition of the biochar with respect to the temperature. The carbon (C) content rises with respect to the temperature from 42.39% to 54.36%, and then 59.48% for tor MC300, MC400 and MC500, respectively. The hydrogen (H) contents declined with the temperature from 6.49% to 4.32% and 2.7% for MC300, MC400 and MC500, respectively. The obtained results were considered to agree with (Wang et al. 2015). The decline in the hydrogen and oxygen content with an increase in the pyrolysis temperature is considered to be as a result of the decomposition of the oxygenated bonds and the release of low molecular weight by-products containing hydrogen and oxygen (Suliman et al. 2016). The nitrogen (N) content decreased with an increase

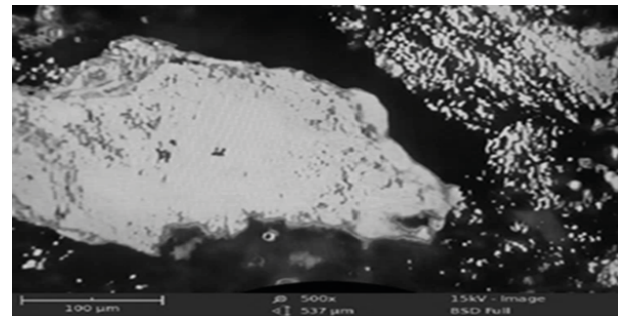
Table 3. The ultimate analysis of the maize cob biochar

Biochar samples	MC300	MC400	MC500
C	42.39	54.36	59.48
N	0.82	0.63	0.42
H	6.49	4.32	2.70
O	48.04	39.02	38.96

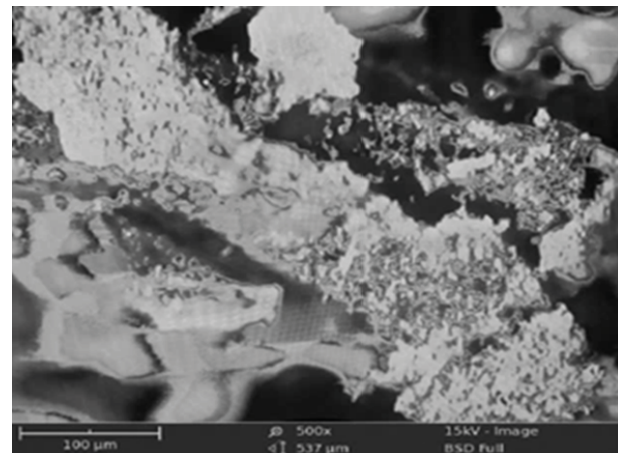
in the temperature, which is in agreement with the findings from (Zhao et al. 2018).

Scan electron microscope (maize cob biochar at 300, 400 and 500 °C). The images in Figure 3 illustrate the temperature effect on the surface morphology on the maize cob biochar. At 300 °C, the biochar has no porous structure, this could be as a result of the thermal cracking that has been initiated by the release of the volatile matter. While at 400 °C, the biochar appears to be more porous when com-

(A)



(B)



(C)

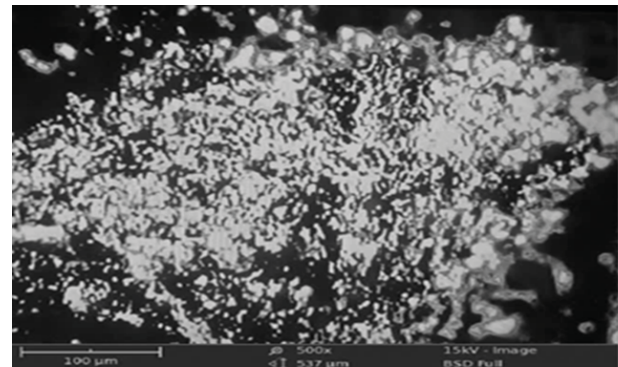


Figure 3. Scanning electron microscopy (SEM) micrographs (magnification 500 ×) of the MC biochar samples pyrolysed at: (A) 300 °C; (B) 400 °C; (C) 500 °C

pared to 300 °C. At 500 °C, as a result of the heat, a more porous structure was created.

FTIR analysis of the maize cob biochar (300, 400 and 500 °C). Figure 4 and Figure 5 show the FTIR spectra of the maize cob biochar produced at 300 °C and 400 °C, respectively. The functional groups of the maize cob biochar at 300 °C and 400 °C are present-

ed in Table 4 and Table 5. The results of the FTIR spectra of the maize cob biochar produced at 500 °C are shown in Figure 6. Table 6 reveals the functional group of the maize cob biochar at 500 °C. The results of the FTIR spectra of the maize cob biochar at 300, 400 and 500 °C reveal the effect the temperature has on the surface functional group and compound class

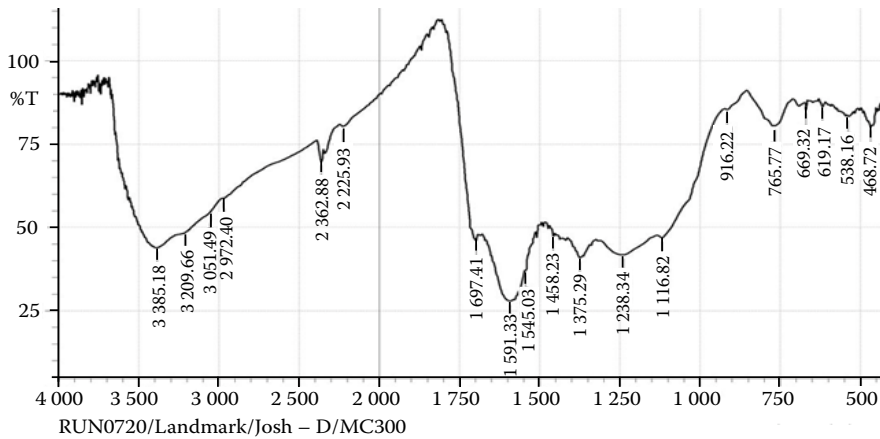


Figure 4. FTIR spectra of the maize cob biochar produced at 300 °C

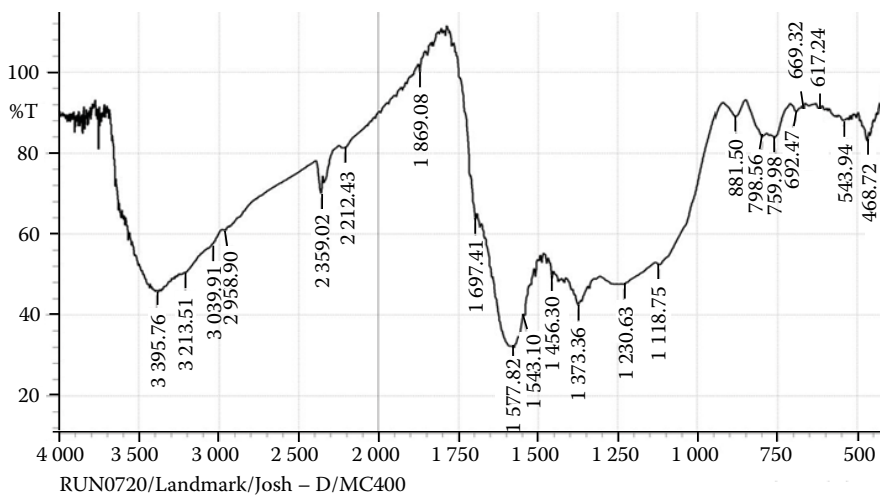


Figure 5. FTIR spectra of the maize cob biochar produced at 400 °C

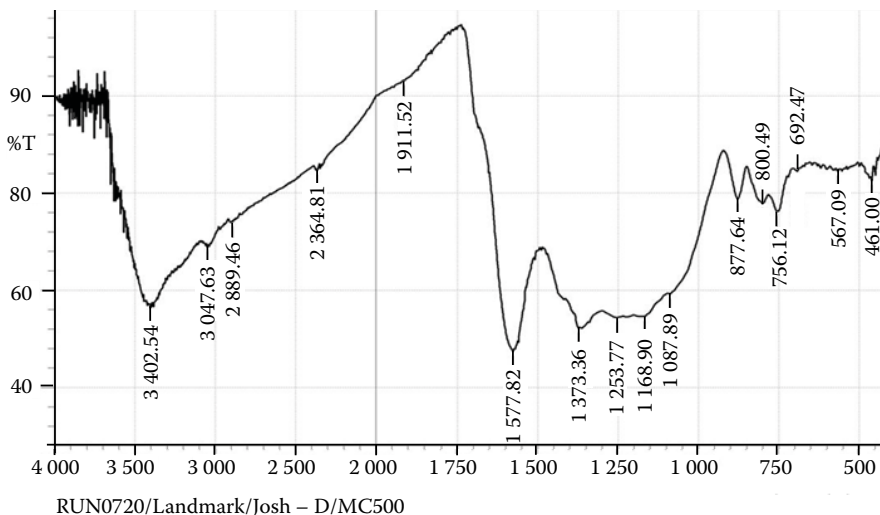


Figure 6. FTIR spectra of the maize cob biochar produced at 500 °C

Table 4. Functional group of the maize cob biochar at 300 °C

Wave numbers (cm ⁻¹)	Functional group
3 383.18	O–H stretching (alcohol)
1 591.33	N–H stretching (amine)
1 238.34	C–N stretching (amine)
1 116.82	C–O stretching (aliphatic ethers)
765.71	C=C bending (alkene)
669.32	C=C bending (alkene)

Table 5. Functional group of the maize cob biochar at 400 °C

Wave numbers (cm ⁻¹)	Functional group
3 396.76	O–H stretching (alcohol)
2 958.90	O–H stretching (carboxylic, alcohol)
1 869.08	C–H bending (aromatic compound)
1 373.36	O–H bending (phenol)
1 230.63	C–O stretching (ethers)
798.56	C=C bending (alkene)

Table 6. Functional group of maize cob biochar at 500 °C

Wave numbers (cm ⁻¹)	Functional group
3 402.54	O–H stretching (alcohol)
1 911.52	C–H bending (aromatic compound)
1 577.82	C=C stretching (cyclic alkene)
1 087.89	C–O Stretching (aliphatic ethers)
756.47	C=C bending (alkene)
798.56	C=C bending (alkene)

that exists in the biochar. It was observed that an increase in the temperature leads to an increase in the stretching of the O–H group (3 383–3 402 cm⁻¹). The O–H functional group is responsible for initiating the rate of the condensation reaction by hydroxide through the thermal cracking of the cellulose content in the material with an increase in the temperature of the furnace (Anukam et al. 2017). The presence of the C–H functional group is likely due to the alkanes present, which causes degradation of the hemicellulose (Fang et al. 2016). The C=C bonds represent the existence of alkenes which quicken the decomposition of lignin. The functional C–O group belongs to the cellulose and hemicellulose which are the (carboxylic) groups. These groups speed up the reaction rates, i.e. decarboxylation,

which leads to the disruption of the glycosidic bonding in the inner layer of the biochar during heating. This results in the formation of several compounds which contain less oxygen atoms, such as ethers, acids, aldehydes (Anukam et al. 2016).

CONCLUSION

Maize cob agricultural waste was pyrolysed using a resistance box muffle furnace in order to obtain a biochar at different temperatures (300–500 °C). The produced biochar was successfully characterised by varying the physico-chemical and structural properties of the biochar. The effects of the temperature on the physical and structural properties were investigated. The results show that the temperature had a significant effect on the biochar yield, proximate analysis, ultimate analysis, surface area, functional groups and surface morphology.

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