

## RESEARCH ARTICLE

## EFFECT OF PYROLYSIS TEMPERATURE ON THE STRUCTURE, TEXTURE AND THERMAL STABILITY OF MAIZE COB BIOCHAR

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## ARTICLE DETAILS

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## ABSTRACT

The aim of this study was to investigate the effect of pyrolysis temperature on the structural, textural, and thermal stability characteristics of maize cob biochar. Biochar was produced by pyrolysis of maize cobs at different temperature (250°C, 300°C, and 350°C), and the resulting biochars were characterized using scanning electron microscopy (SEM), Brunauer–Emmett–Teller (BET) surface analysis, X-ray fluorescence spectroscopy (XRS-FP), thermogravimetric analysis (TGA), and Fourier-transform infrared spectroscopy (FTIR). Results showed that increasing pyrolysis temperature led to the development of more pronounced pore structures and increased BET surface area (251.0–325.4 m<sup>2</sup>/g), with the highest micropore surface area observed at 300°C. Elemental analysis revealed a decrease in nutrient and mineral content with increasing temperature. TGA indicated improved thermal stability at higher temperatures, particularly for the 350°C biochar. FTIR spectra demonstrated a decline in labile functional groups and increased aromaticity with temperature. The findings suggest that maize cob biochars produced at lower temperatures retain more functional groups and mineral elements beneficial for soil amendment, while those produced at higher temperatures exhibit greater thermal stability and aromaticity, making them suitable for long-term carbon sequestration and soil remediation applications.

## KEYWORDS

## 1. INTRODUCTION

Biochar is produced by the pyrolysis of organic material under oxygen-limited conditions and at relatively low temperatures (< 700°C). Pyrolysis of biomass consists of a series of complex reactions that can be divided into three stages according to the degradation temperature for hemicellulose (220–315°C), cellulose (315–400°C) and lignin (> 400°C) (Yang et al., 2006). Several factors and parameters influence the properties of biochar, of which pyrolysis temperature and nature of biomass feedstock are considered more significant (Antal and Gronli, 2003). Pyrolysis temperature has direct influence on the elements in the feedstock that are potentially lost to the atmosphere, fixed into the more stable carbon fraction or released as soluble forms during pyrolysis (Song and Guo, 2012). Therefore, knowledge of the effect of pyrolysis temperature on composition and surface chemistry of biochar is a necessary requirement for their application for agronomic and environmental benefits. Converting low-cost agricultural residues into biochar has potential for reducing waste disposal problem. Maize (*Zea mays*) cob is low-cost agricultural by-product that is poorly disposed in Nigeria and constitutes environmental and public health concerns. Reports on the production of biochar from maize cobs for ameliorating environmental contaminants are relatively few (Cao et al., 2004; Wang et al., 2015). This report examined the effect of pyrolysis temperature on the textural, structural and thermal stability characteristics of maize cob biochar that are relevant to their beneficial soil application.

## 2. MATERIALS AND METHODS

Maize cobs were obtained from Uselu market in Benin City, cut into small pieces, washed thoroughly to remove extraneous materials, air-dried and then dried at 60°C for 8 hrs. The pre-treated maize cobs were pyrolysed at 250, 300 and 350°C and the biochars obtained were milled, sieved through a 300µm mesh and stored for analyses.

## 2.1 Characterization of biochar

## 2.1.1 Surface morphology

Scanning electron microscope (FEL Quantaim 450 FC type 20331 A) analysis of biochar samples was carried out to determine changes that occurred on the surface structures of the biochar during pyrolysis.

## 2.1.2 Surface area

Surface area and pore volumes of the biochars were determined by the applications of the Brunauer-Emmett-Teller (BET) and t-plot analysis available with Micromertic ASAP 202 and a Micromertics Tristar II 3020 (version 2.0).

## 2.1.3 Elemental composition

The relative elemental content of the biochar samples was determined by XRS-FP.

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### 2.1.4 Thermogravimetric analysis

Thermogravimetric analysis (TGA) of the biochar was performed on a Parkin Elmer TGA 400 coupled with a differential thermal gravimetry (DTG) to understand their pyrolytic behavior and thermal resistance. For the TGA experiment about 10mg of each sample was processed and heated at a heating rate of  $10^{\circ}\text{C}\cdot\text{min}^{-1}$  under nitrogen up to  $950^{\circ}\text{C}$ .

### 2.1.5 Functional group

Chemical functional groups were determined by Fourier transfer infrared spectroscopy (Bruker IFS 3V).

## 3. RESULT AND DISCUSSION

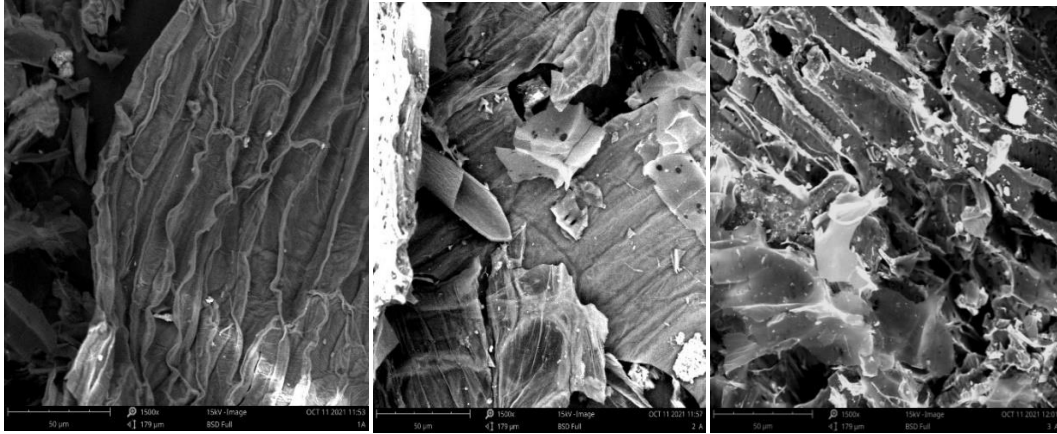


Figure 1: SEM images of maize cob biochars obtained at pyrolysis temperatures of  $250^{\circ}\text{C}$  (a);  $300^{\circ}\text{C}$  (b) and  $350^{\circ}\text{C}$  (c)

### 3.2 Surface area

The effect of pyrolysis temperature on the BET surface areas and microporosity of the biochars are given in Table 1.

Table 1: Effect of pyrolysis temperature on surface area and porosity of maize cob biochar			
Surface area and pore structure characteristics	Pyrolysis temperature ( $^{\circ}\text{C}$ )		
	250	300	350
BET surface area ( $\text{m}^2\cdot\text{g}^{-1}$ )	251.0	305.11	325.37
Pore volume ( $\text{cm}^3\cdot\text{g}^{-1}$ )	0.109	0.136	0.109
Micropore surface area ( $\text{m}^2\cdot\text{g}^{-1}$ )	306.10	383.22	306.29
Average pore width (nm)	6.50	5.75	6.50

### 3.1 Surface morphology

Figure 1 shows the scanning electron microscope (SEM) images of the biochar samples obtained at  $250$ ,  $300$  and  $350^{\circ}\text{C}$ .

Structures of the biochar samples revealed porous surface caused by volatilization of organic materials during pyrolysis. It can be seen that appearance of deep channels and pores in the biochar samples became more prominent with increasing pyrolysis temperature. Increase in pyrolysis temperature is generally associated with the random release of volatile matter, such as  $\text{H}_2\text{O}$ ,  $\text{CO}_2$ ,  $\text{CO}$  etc from biomass with implication for the textural features of biochar (Kelluweit, et al., 2010).

The devolatilization of biomass materials developed porosity in the biochars resulting in particles with essentially micropore structure. BET surface areas were generally moderate for maize cob biochars produced at all the pyrolysis temperatures but tended to increase with increase in temperature within the range of this study. The range of values of BET surface obtained in this study are higher than reported for similar biomass materials (Fu et al., 2011; Kim et al., 2012). Micropore volume and area of biochars were not markedly affected by pyrolysis temperature, reaching a value of  $0.136\text{cm}^3\cdot\text{g}^{-1}$ ,  $383.22\text{m}^2\cdot\text{g}^{-1}$  respectively at  $300^{\circ}\text{C}$ . This trend was observed by other workers (Angin and Sensoz, 2014) and has been explained in terms of shrinkage of biochar structure which leads to a decrease of the mean-size and pore volume of biochar particles.

### 3.3 Elemental composition

The results of the relative elemental content of the biochars are given in Table 2.

Table 2: XRS-FP analysis of biochars produced at different pyrolysis temperatures			
Element	Weight %		
	Temperature of pyrolysis ( $^{\circ}\text{C}$ )		
	250	200	350
Mg	-	-	6.571
Al	4.713	2.869	2.617
Si	7.170	9.466	3.623
P	1.171	1.551	0.705
S	1.250	0.870	0.444
Cl	4.907	6.379	3.314
K	38.675	38.942	20.152
Ca	7.107	7.671	1.345
Cr	0.033	0.013	-
Mn	0.264	0.177	-
Fe	3.359	1.594	-
Co	0.050	0.009	-
Ni	0.023	0.007	-
Cu	0.300	0.119	0.086
Zn	0.340	0.126	0.109
Sn	0.911	0.893	0.765

The elements represented in the results in Table 2 are those present in the biochar samples at levels  $\geq 0.01\%$ . It can be seen that the biochar samples are generally relatively rich in nutritional and mineral elements. With increase in pyrolysis temperature, the nutritional and mineral elements decreased markedly. These results suggest that biochar produced at low pyrolysis temperature may have greater agronomic benefits when applied to soil than biochar produced at high pyrolysis temperatures.

### 3.4 Thermogravimetric analysis

Figure 2 shows TG curves and differential analysis of the weight loss (DTA) of the biochars. The TG and DTG curves showed two stages of weight loss. The mass loss occurring slowly at 200°C accounted for less than 3% and is commonly associated with loss of moisture content of the samples (Santos et al., 2015). The main mass loss started around 350°C for all the biochar samples. A sharp weight decrease took place at around 380°C for biochar

sample produced at 250°C and 400°C for samples prepared at 300 and 350°C. This was due to the fact that the generated biochars had undergone a previous heat treatment before the thermal analysis, then the tested biochar samples were thermally stable below the temperature at which they were produced (Wu et al., 2016). It is known that secondary pyrolysis reactions could be easily detected and observed at the temperature exceeding the biochar's primary decomposition temperatures (Santos et al., 2015). Thus the weight loss over a wider temperature range could be attributed to degradation and decomposition of organic materials (Sun et al., 2014). The temperature at which maximum weight loss occurred varied somewhat, with the biochar produced at 350°C showing better thermal stability, consistent with the results of previous studies (Sun et al., 2014). Above 600-700°C, decomposition for all the biochars finished and the curves became stable with total weight loss from thermal degradation of about the same order of magnitude of about 96%.

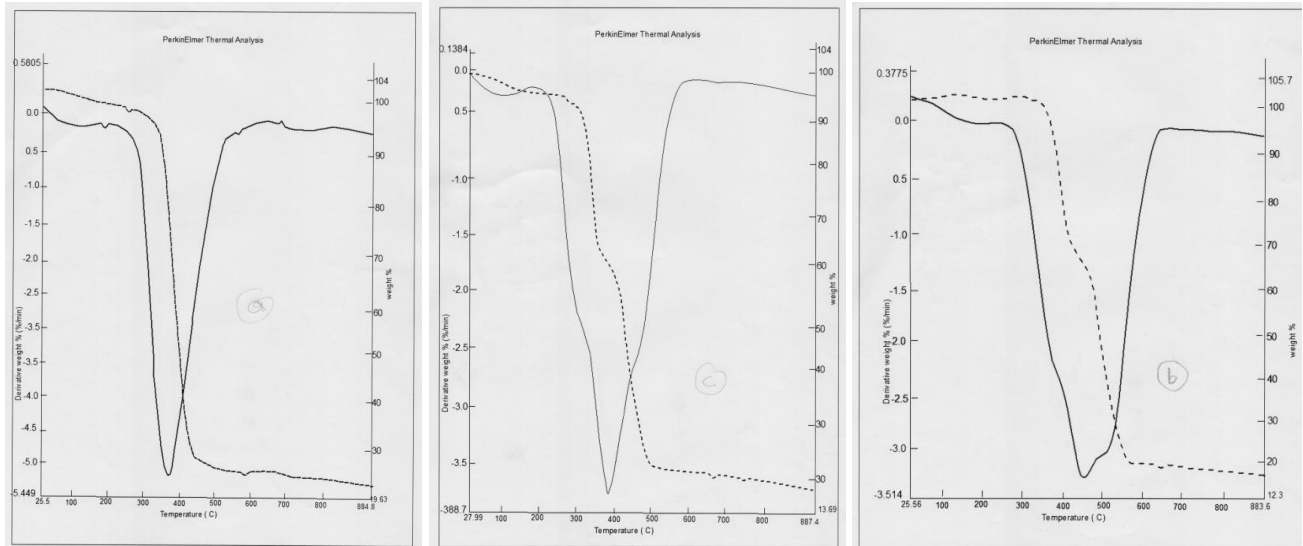


Figure 2: TGA and DTA curves of biochar samples prepared at various temperatures of 250°C (a); 300°C (b) and 350°C (c).

### 3.5 Functional group

FTIR spectra of maize cob biochars prepared at different temperatures are

shown in Figure 3 and the functional groups observed are given in Table 3.

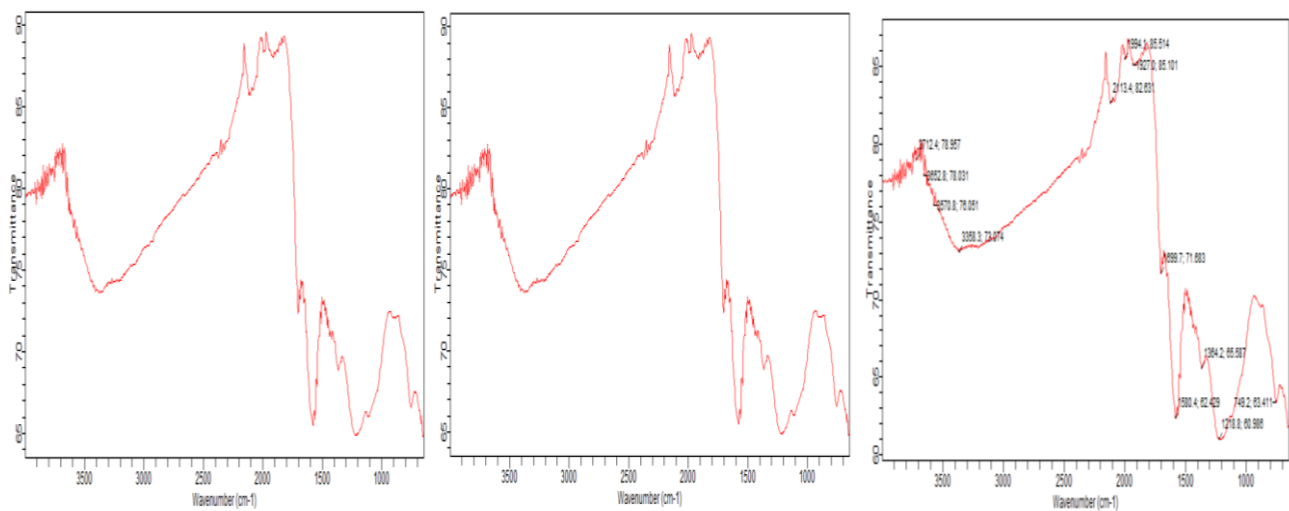


Figure 3: FTIR spectra of maize biochars produced at different temperatures 250°C (a); 300°C (b) and 350°C (c)

Table 3: Functional groups observed in the Fourier-transform infrared (FTIR) spectra of biochars

Wave numbers (cm <sup>-1</sup> )	Characteristic vibrations (functionality)
3200-3500	OH- stretching (water, hydrogen-bonded hydroxyl)
2935	C-H stretching
1600	Aromatic C=C and C=O stretching of conjugated ketones and quinines
1440	C=C stretching
1325	O-H bonding (phenols, phenolics)
1030-1100	Symmetric C-O stretching

As pyrolysis temperature increased, FTIR spectra of biochars revealed a decrease in the stretching of O-H ( $3200\text{-}3500\text{ cm}^{-1}$ ) and C-H ( $2935\text{ cm}^{-1}$ ) which was attributed to increase in dehydration reaction (Chen *et al.*, 2012). The absorption peak at  $2560\text{-}3000\text{ cm}^{-1}$  which represents aliphatic C-H stretch vibration, appeared in the biochar spectra. The assignment of the peaks of the biochars including the aromatic C-H stretch around  $800\text{ cm}^{-1}$  and aromatic C=C stretch at  $1600\text{ cm}^{-1}$  indicate an increasing degree of condensation and aromaticity for the biochars which became more pronounced as the pyrolysis temperature increased. The stretch band at  $1712\text{ cm}^{-1}$  attributed to the carboxyl and carbonyl groups originating from carbohydrate appeared with seemingly more intensity for the biochar sample prepared at  $300^\circ\text{C}$ .

Biochar produced from carbonization of organic matter have been known to sequester C in soils (Lehmann, 2007). However, long-term C sequestration efficiency depends largely on the biochar's stability dependent on production conditions including pyrolysis temperature (Al-Wabei, *et al.*, 2013). It has been suggested that biochars produced at relatively high temperatures ( $> 400^\circ\text{C}$ ) could be more resistant to mineralization than those pyrolysed at lower temperatures ( $\leq 400^\circ\text{C}$ ), thus representing an efficient technique for mitigating greenhouse gas emissions into the environment (Ippolito *et al.*, 2012). Additionally, high temperature biochars on account of alkalinity and alkaline forms have ameliorating effects on acidic soils (Wan *et al.*, 2014). Maize cob biochars produced at the lower pyrolysis temperatures ( $250\text{-}350^\circ\text{C}$ ) have organic functional groups on their surface, high cation exchange capacity, low pH values as well as less aromatic content may be useful in enhancing soil nutrient exchange sites as well as soil cation exchange capacity when applied to soils.

#### 4. CONCLUSION

The results from this study showed that biochars produced at different pyrolysis temperatures differ markedly in their textural and thermal stability characteristics that could influence agronomic and environmental benefits following soil application.

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