



Biochar from the Thermochemical Conversion of Orange (*Citrus sinensis*) Peel and Albedo: Product Quality and Potential Applications

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Abstract

Orange (*Citrus sinensis*) is a popular fruit in west Africa that generates residues such as peels (OP) and albedo (OA) from its consumption. In this study, the biochar obtained from the char-optimised thermochemical conversion of orange peels and albedo were evaluated. The products obtained was characterised using Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM–EDS) and Branueur–Emmett–Teller (BET) Analyses and potential applications were discussed. FTIR analysis revealed similar spectra for both samples and possessing polar groups such as alcohol, esters, ketones, aldehydes, carboxylic, ether and phenols which are characteristic of low-temperature biochar. The EDS analysis showed that OP biochar possess higher carbon content than OA biochar whilst the latter contains more inorganic elements. SEM analysis revealed that OP biochar possess a smooth surface as compared to the highly convoluted surface of the OA biochar. BET analysis revealed that the surface area was $352.5 \text{ m}^2 \text{ g}^{-1}$ and $356.3 \text{ m}^2 \text{ g}^{-1}$ for OP and OA biochar, respectively. Several key conclusions on the potential applications were proposed based on the analytical findings and these include soil amendment, adsorbents and as catalysts.

Keywords Orange peels · Orange albedo · Bio-char · Characterisation · Thermochemical conversion

1 Introduction

In light of the global research drive towards energy and environmental sustainability, numerous investigations on thermochemical processing of biomass has been conducted. Plant derived feedstock tends to give good yield of biochar due to their high carbon content and these includes forest residues, grasses, agricultural residues and algae [1]. Plant derived feedstock considered for biochar production over the years includes cocoa pod husk [2], cassava rhizome [3], sugar cane bagasse [4, 5], wood chips [6–11], coconut fibers [12], rubber wood sawdust [13], rice hulls/husks [7, 8, 14], eucalyptus leaves [12], hazelnut shells [10, 15], rice straw [8, 14], switch grass [16], elephant grass [17], bamboo [5,

18], hickory wood [5], furniture wood [11], rapeseed cake [19], apricot stone [15], pine sawdust [20], grape seed [15], chestnut shell [15] amongst others.

Orange (*Citrus sinensis*) is also a popular fruit in west Africa that generates residues such as peels and albedo from its consumption. Orange peels have been known to contain essential oils possessing antioxidant, anti-carcinogen and germicidal properties [21, 22]. Orange peels can be used as animal feeds, fertilizers, feedstock for the growth of single cell protein [23] and others [24, 25]. Alternatively, it has been shown that citrus peels can be used as feedstock for thermochemical processes too [26]. Biochar is solid residue of biomass thermochemical processing that is rich in carbon (65–90%) and possesses a porous structure [27]. The physico-chemical properties of the biochar-determines its potential application [8]. These properties are affected by the type of feedstock and the conditions of pyrolysis or gasification [5].

Biochar can be used as an adsorbent in separating some chemical species from aqueous solutions [4, 28], as a bio-composite [20], as a solid fuel [9, 12], and for agricultural purposes as a sequestrate and soil conditioner [29, 30]. Biochar from orange peels have been utilised in a variety of

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environmental applications in recent times. It has been used in the adsorption of ammonium [31], lead [32] and other ionic contaminants [33], as catalyst in syn-gas production [34] and for the conditioning of loess soil [35]. The thermochemical conversion biochar of orange residues in a top-lit updraft biomass conversion reactor have not been evaluated in open literature (within the scope of the authors' search). Besides, a comparative study of this nature considering the potential applications of both residues is not reported. The interest in biochar from orange peels and albedo is its relatively relative abundance in this part of the world and lack of a major competitive use. The goals are in solid waste management and biomass valorisation.

The aim of this study is to evaluate the nature, properties and characteristics of biochar obtained from the low-temperature thermochemical conversion of orange peels and albedo in a top-lit updraft biomass conversion reactor with a view of determining the suitability of the product in a variety of potential applications. The potential applications of the char were discussed based on the observations from the analytical findings. The chars were characterised using Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS) and Branueur-Emmett-Teller (BET) analyses.

2 Methodology

2.1 Product Development

The orange (*Citrus sinensis*) peels and albedo were sourced from the market in Ilorin, Nigeria. The orange peels are referred to as OP and orange albedo as OA, respectively. Both feedstock was sundried to remove all moisture until crisp. The biochar was produced using an updraft biomass conversion reactor with retort heating. Details of reactor design, configuration and operation are discussed elsewhere [17]. The peak temperature of the process was 300 °C and it lasted for 2 h for both samples. Low temperatures for

thermochemical processes lead to more solid phase products. Since the reactor operates by retort heating, the thermal energy for the process is the recycled heat from the outer combustion region hence the achieved temperature [36].

2.2 Product Characterisation

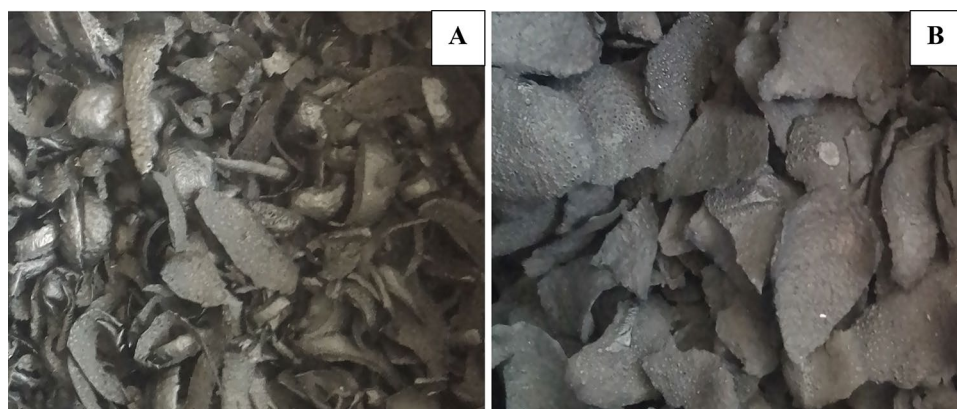
The products obtained were characterised using Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS) and Branueur-Emmett-Teller (BET) analyses. FTIR (Shimadzu, FTIR-8400S, Japan) was used to determine the functional groups and complexes present in both biochar samples. The spectra were recorded using transmittance method in the 4000–650 cm^{-1} region with 30 sample scans. Scanning Electron Microscopy (SEM, Phenom proX, Phenom-World BV, The Netherlands) was used to study the surface morphology of the particles of the biochar. The acceleration voltage of the microscope was set to 15 kV and magnification of 500–1000 times. Branueur-Emmett-Teller (BET) analysis was used to study the particle and pore dimensional characteristics of the bio-char. The micropore volume, surface area and pore width of the biochar were measured using a BET analyser (Quantachrome NovaWin ©1994–2013, Quantachrome Instruments v11.03). The surface area and micropore volume of the biochar samples were calculated based on the nitrogen adsorption-desorption isotherm obtained using the multipoint BET and the Dubinin-Radushkevich (DR) methods, respectively whereas the total pore volume and pore diameter were estimated using the Barette, Jovner & Halenda (BJH) method.

3 Results and Discussion

3.1 Visual Inspection

Figure 1a, b show the visual images of the biochar obtained after the low temperature thermochemical conversion of

Fig. 1 Camera images of biochar from OP (a) and OA (b)



orange peels and albedo in a top-lit updraft biomass reactor. The char from the peels had a noticeable and distinctive glossy appearance which was not observed for the albedo chars. To gain more insight into the nature of the biochar obtained and their potential applications will be elucidated in further sub-sections.

3.2 Functional Group Analysis

The chemical and structural characteristics of agricultural waste products wastes such as surface area, porous structure and functional groups are highly significant in assessing their suitability in removing water pollutants [37]. The

functional groups present in the biochar obtained from orange peels and orange albedo (pith) is presented Table 1 based on the spectra obtained by FTIR analysis (Fig. 2). The tiny peaks ranging from 3942 to 3217 cm^{-1} correspond to the OH bond stretch of alcohols, carboxylic and phenolic groups [17, 38]. It may also correspond to the amino group NH associated with proteins present in the biomass cell wall [39, 40]. The peaks at approximately 2900 cm^{-1} correspond to the aliphatic C–H groups [41]. The peak 1681 cm^{-1} correspond to the C=O (carbonyl) stretch of ketones aldehydes probably with a small quantity of amides [42–44]. The presence of a peak at 1527 cm^{-1} strongly suggests the presence of a nitro-compound characterised by the N–O asymmetric

Table 1 FTIR assignments for biochar samples OP and OA

Observed peaks (intensity)		Possible functional groups
OP	OA	
3896 cm^{-1} (very weak)	3865 cm^{-1} (weak)	O–H (alcohols, phenols, carboxylic acid)
3541 cm^{-1} (very weak)	3595 cm^{-1} (weak)	
2900		C–H (methyl)
1681 cm^{-1} (medium)	1681 cm^{-1} (medium)	C=O (ketone, aldehydes, amides)
1527 cm^{-1} (medium)	1527 cm^{-1} (medium)	N–O (nitro)
1411 cm^{-1} (medium)	1411 cm^{-1} (medium)	COO– (carboxylate)
1026 cm^{-1} (weak)	1026 cm^{-1} (medium)	C–O (carboxylic acid, esters)
779 cm^{-1} (weak)	779 cm^{-1} (weak)	Aromatic C–H

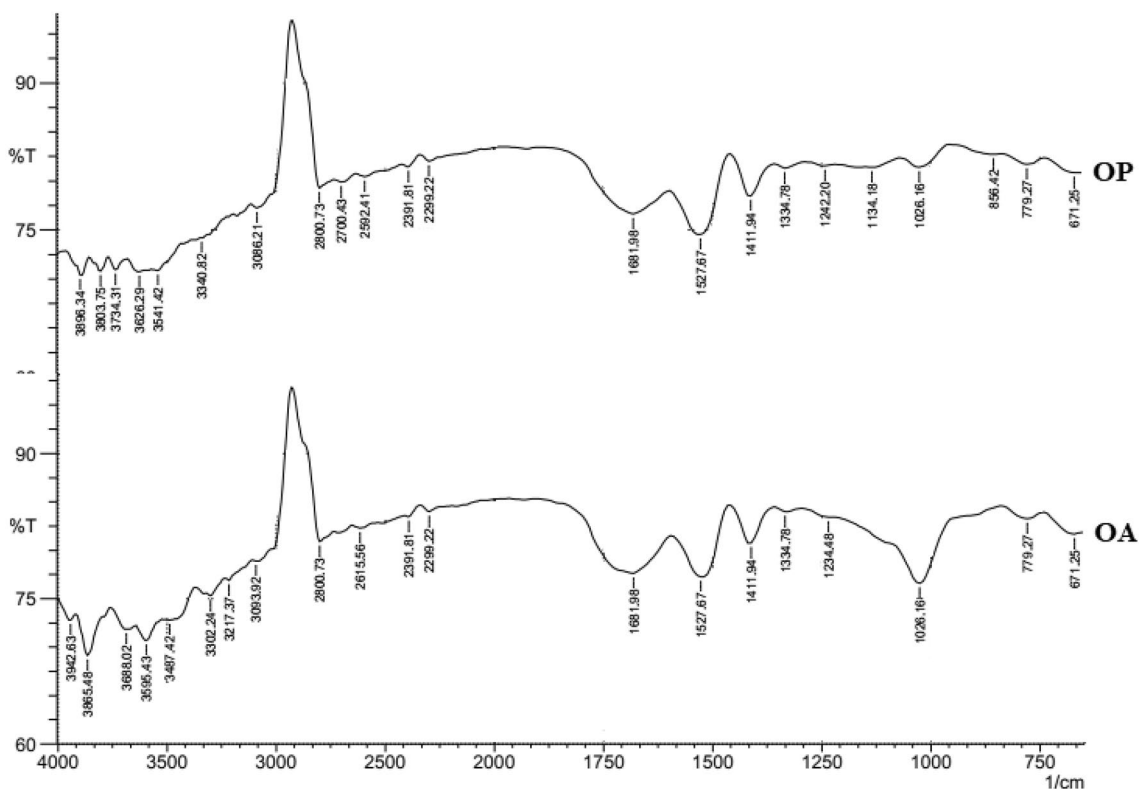


Fig. 2 FTIR spectra of biochar samples OP and OA

Table 2 Inorganic composition of the biochar samples

Element symbol	Element name	Weight concentration (%)	
		OP	OA
C	Carbon	80.72	55.33
O	Oxygen	16.16	33.15
N	Nitrogen	2.55	3.47
Fe	Iron	–	2
Fe	Fluorine	0.37	–
Si	Silicon	–	1.56
Ca	Calcium	–	1.07
Cl	Chlorine	0.13	0.44
Al	Aluminium	–	0.76
K	Potassium	–	0.66
Na	Sodium	–	0.57
S	Sulfur	0.08	0.36
Mg	Magnesium	–	0.32
P	Phosphorus	–	0.31
Total	100.01	100	–

stretch [45, 46]. The peak at 1411 cm^{-1} may also correspond to the asymmetric and symmetric stretching of the carboxylate (COO^-) group [47–49]. The peaks at 1026 cm^{-1} can be attributed to the C–O stretch of carboxylic acids and esters of carbohydrates [50, 51] while the peak at 779 cm^{-1} correspond to the vibrational stretch of the C–H methyl group [30, 52].

The spectra obtained for both samples were relatively similar with little differences in peak intensity especially the peak observed at 1026 cm^{-1} . The presence of polar groups such as alcohol (OH), esters, ketones, aldehydes, carboxylic, ether and phenols suggests that the biochar samples has the potential to be used as adsorbent for aqueous pollutants [53] and as soil amendment for improving cation exchange [54]. These important functional groups are present because the thermochemical conversion was a low temperature ($300\text{ }^\circ\text{C}$) and char-optimised one. Biomass degradation beyond $400\text{ }^\circ\text{C}$ would however cause a loss of these groups via dehydration thereby forming larger number of aromatic C–C structure.

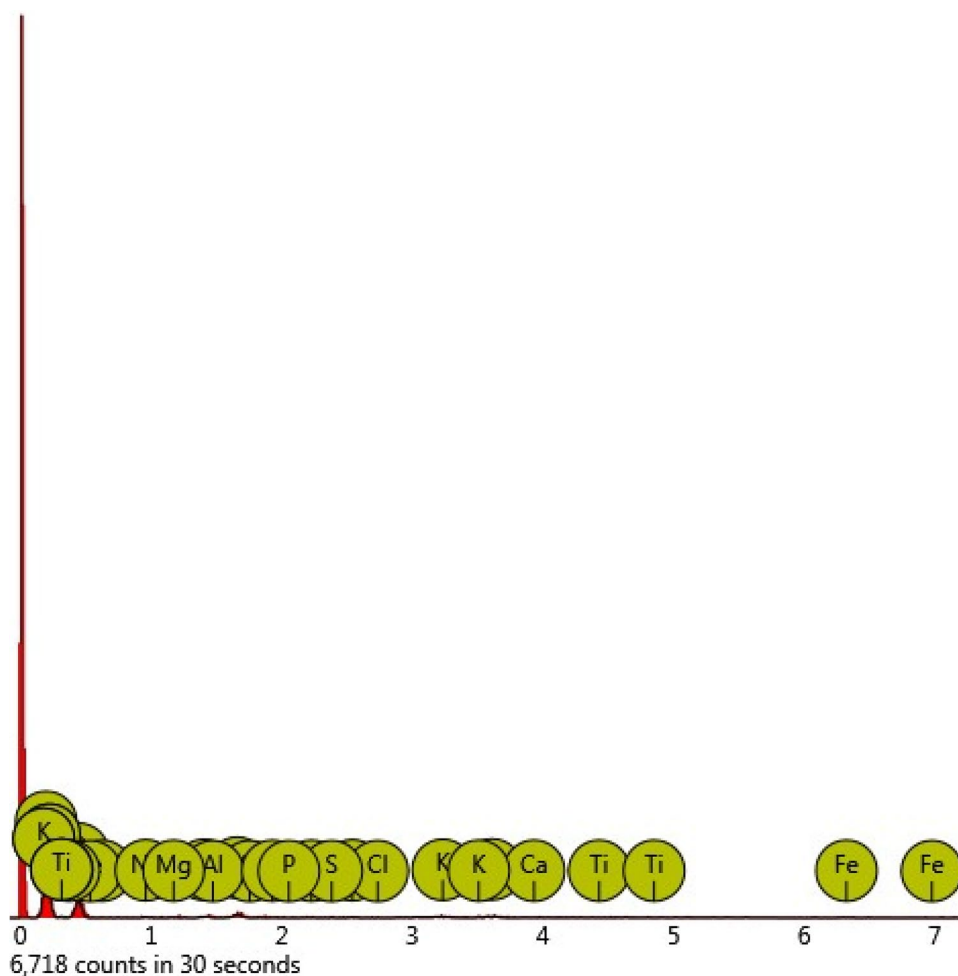
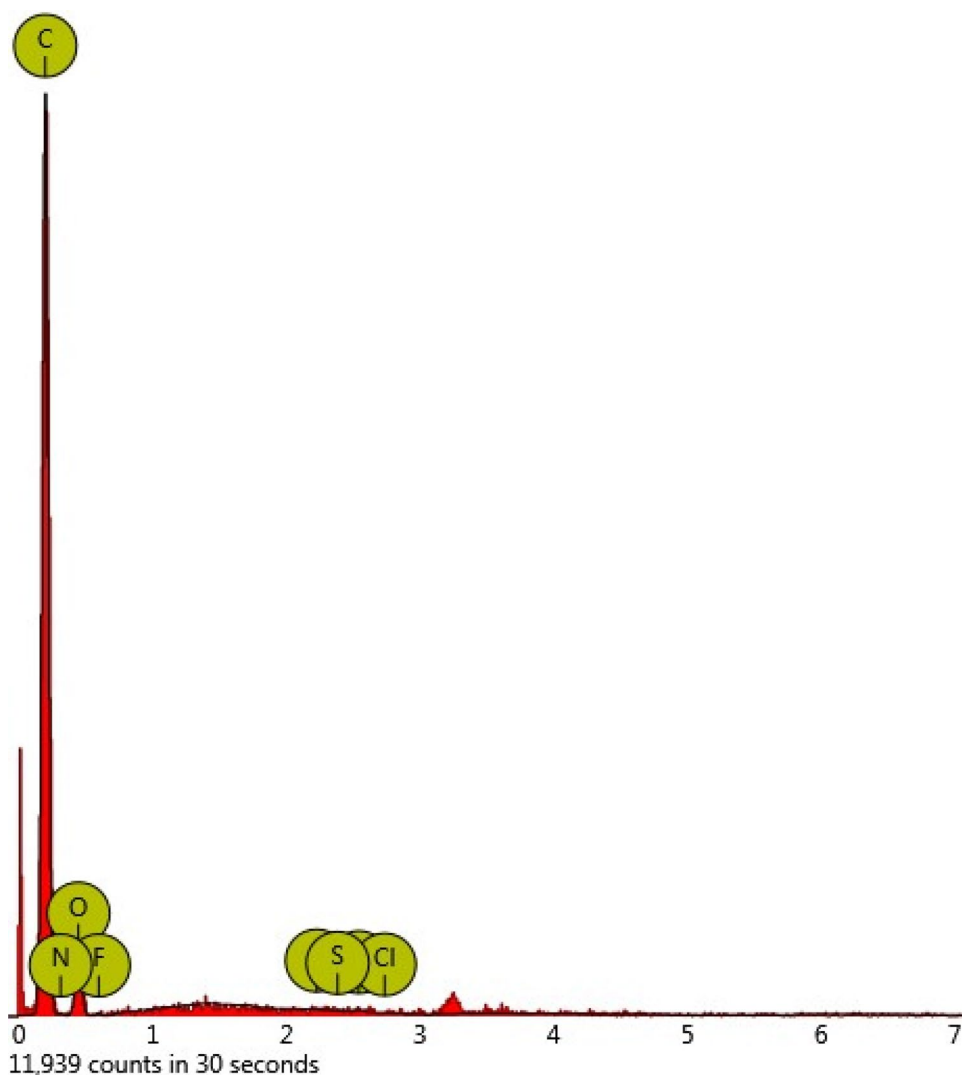
Fig. 3 EDS spectrum for OA biochar

Fig. 4 EDS spectrum for OP biochar



3.3 Biochar Composition

The inorganic elements present in the biochar samples with their respective weight concentrations as analysed by EDS are presented in Table 2. The corresponding EDS spectrums for OA and OP biochar are shown in Figs. 3 and 4. The data presented in the table showed that OP biochar possess higher carbon content than OA biochar whilst the latter contains more inorganic elements than OP. Since the thermochemical conversion of both biomasses was done under the same condition, it can be inferred that orange peel would give quality carbon rich biochar than orange albedo making the OA biochar more effective in pollutant adsorption both from water and soil [41]. However, the presence of other elements such as Calcium, Nitrogen, Phosphorus and Potassium in OA biochar enhances its soil nutritional value and application in the production of fertilizer [17, 55]. Apart from its capacity to remediate pollutants in water [53], carbon rich bio-chars also has applications in greenhouse gas (GHG)

emissions [1] and carbon sequestration [56] in soils. Both biochar had higher carbon content than some of the agricultural residues studied in literature [55–58] confirming their use a proper feedstock for biochar production.

3.4 Biochar Morphology

SEM analysis was used to further understand the porous structure of the biochar. Images showing the morphological structure of OP and OA biochar are shown in Figs. 5 and 6, respectively. The surface morphology of both samples was different in despite being from the same fruit source. OP biochar possess a smooth surface as compared to the highly convoluted surface of the OA biochar. Biochar from OA has a spongy-like surface, with lots of crevices and interstices with relatively higher porosity which could provide adequate surface area for catalytic adsorption, nutrient retention (soluble organic and inorganic) and water holding capacity when

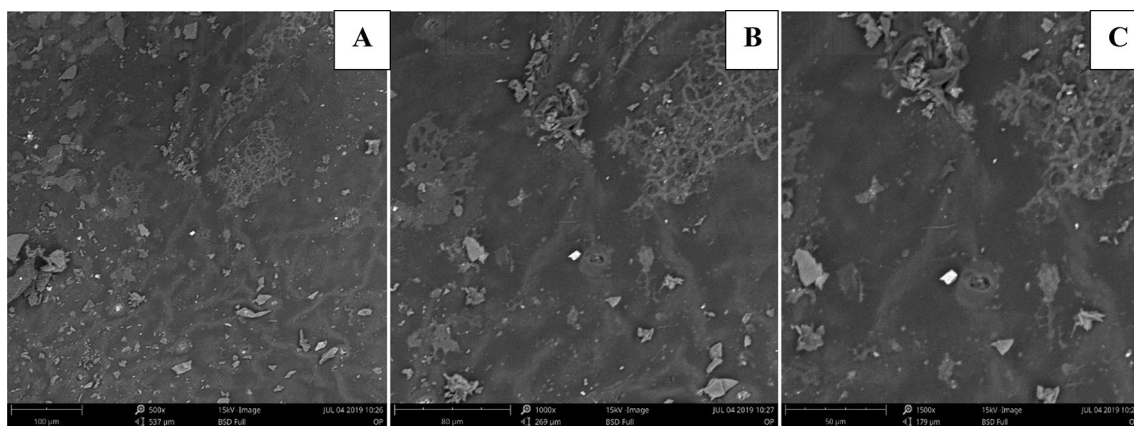


Fig. 5 SEM images for orange peel biochar with magnification **a** $\times 500$; **b** $\times 1000$; and **c** $\times 1500$

incorporated in soil [55–57, 59]. The OA biochar can be said to have a relatively lower pore volume compared to the OP biochar due to the nature of the surface as can also be seen from the BET analysis (Table 3). The smooth surfaces reduce porous characteristics and hence lower the adsorptive potential of the biochar [60].

3.5 Porous Characteristics

The surface area and micropore volume of the biochar samples were calculated based on the nitrogen adsorption–desorption isotherm obtained using the multipoint BET and the Dubinn–Radushkevich (DR) methods respectively whereas the total pore volume and pore diameter were estimated using the Barette, Jovner & Halenda (BJH) method. The data obtained from these methods are presented in Table 3. Although the surface area of OP biochar was slightly lower

than that of the OA biochar, it was found to be more porous. However, the pores in the OA biochar was found to be larger than that of OP biochar as it can also be seen in the structural images (Figs. 5, 6). The surface characteristics of the biochar samples would enable them function as adsorbents as well as in the enhancement of soil quality. Biochar porosity is highly advantageous in gasification and combustion applications as well as soil quality enhancement [17]. The surface functionality coupled with the porosity of biochars has a large effect on its capability to retain water in soil [61].

The BET analysis was carried out using Nitrogen physisorption at 77 K. More details on the results are shown in the supplementary material. The pore size distribution for biochar samples of OA and OP are shown in Fig. 7a, b, respectively. It can be observed that they are quite similar. Both biochar revealed occurrence of pores around 2.8 nm with a broad size distribution of pores up to 6 nm. The biochar can be considered to be mesoporous as the pore

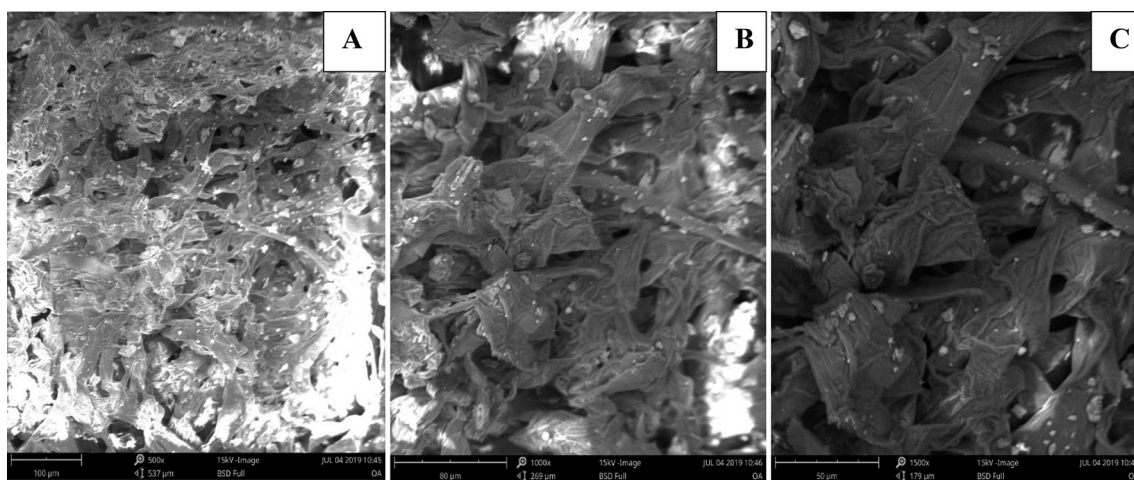


Fig. 6 SEM images for orange albedo biochar with magnification **a** $\times 500$; **b** $\times 1000$; and **c** $\times 1500$

Table 3 Porous characteristics of biochar from orange residues

Properties	OP	OA
BET surface area ($\text{m}^2 \text{g}^{-1}$) ^a	352.5	356.3
Micropore volume ($\text{m}^3 \text{g}^{-1}$) ^b	0.148	0.145
Total pore volume ($\text{m}^3 \text{g}^{-1}$) ^c	0.217	0.215
Pore diameter (nm) ^c	2.132	2.138

^aMultipoint Brunauer, Emmett and Teller (BET) method

^bDubinini–Radushkevici (DR) method

^cBarrett, Jovner & Halenda (BJH) adsorption method

diameter is $> 2 \text{ nm}$ but $< 50 \text{ nm}$. Examination of the pore size distributions and adsorption isotherms help to not only provide useful knowledge on the porous characteristics of the char but also evaluate their potential performance in adsorptive systems [62, 63].

3.6 Practical Implications of the Study

In this approach involving the co-carbonisation of orange (*Citrus sinensis*) peels and albedo, the first significant implication is in solid waste management. Haven previously examined perennial grasses [17] and an agricultural residue [36], a biomass waste generated from fruit consumption was successfully converted in this study. Besides the Size reduction of the waste stream, the development of quality biochar for a variety of applications was achieved. Waste-to-wealth is achieved in a relatively simple process that is easy to understand and use. Furthermore, the study utilises energy from the controlled combustion of biomass for the co-carbonisation process in a retort heating system. A key advantage in such a design is the lack of dependence on electricity. In some developing countries in Africa with issues with electricity supply (Nigeria as a case study), such a technology will gain ready acceptability. No electrical power requirement also signifies less cost and usability in remote locations. These considerations make this

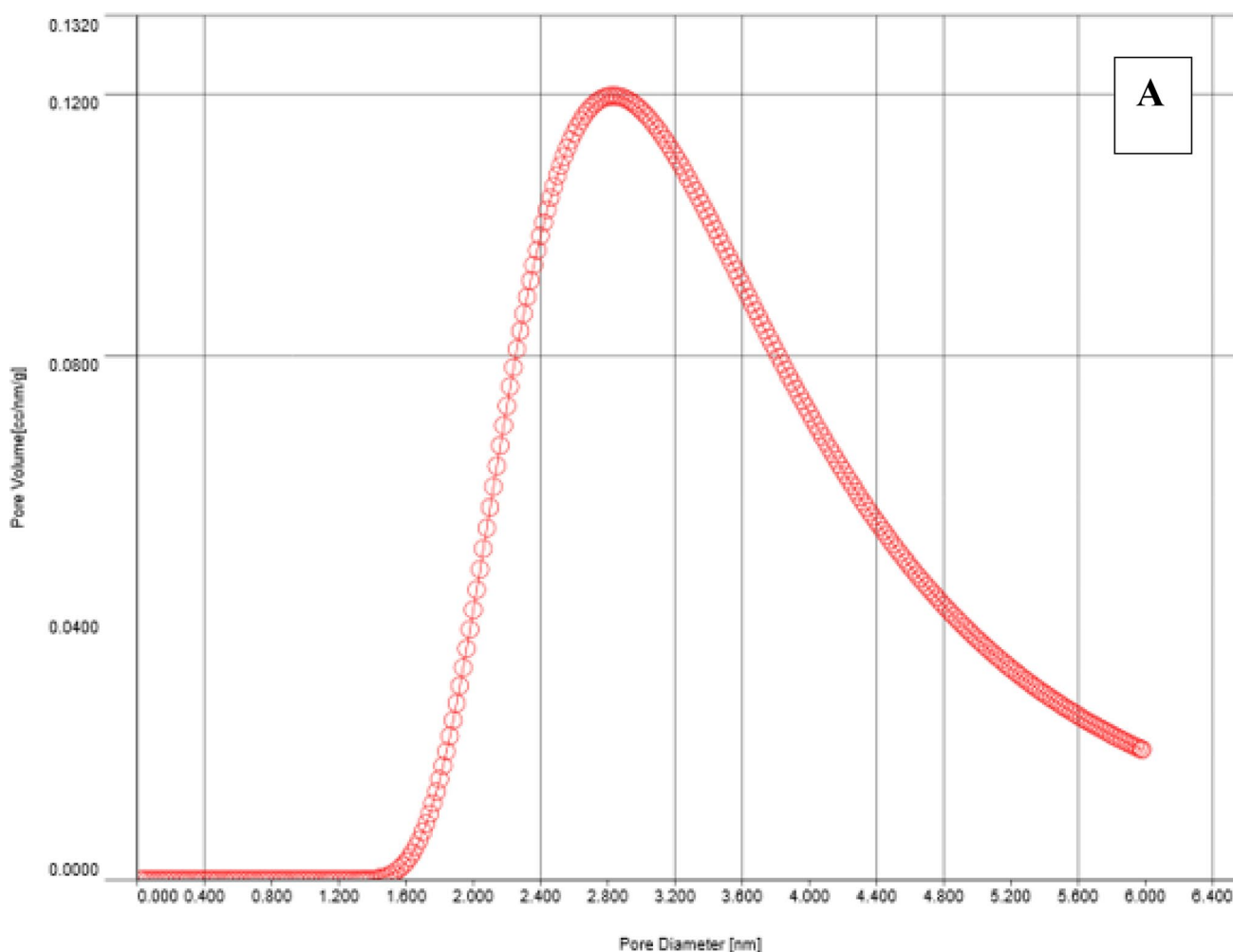


Fig. 7 Pore size distribution curve for biochars OA (a) and OP (b)

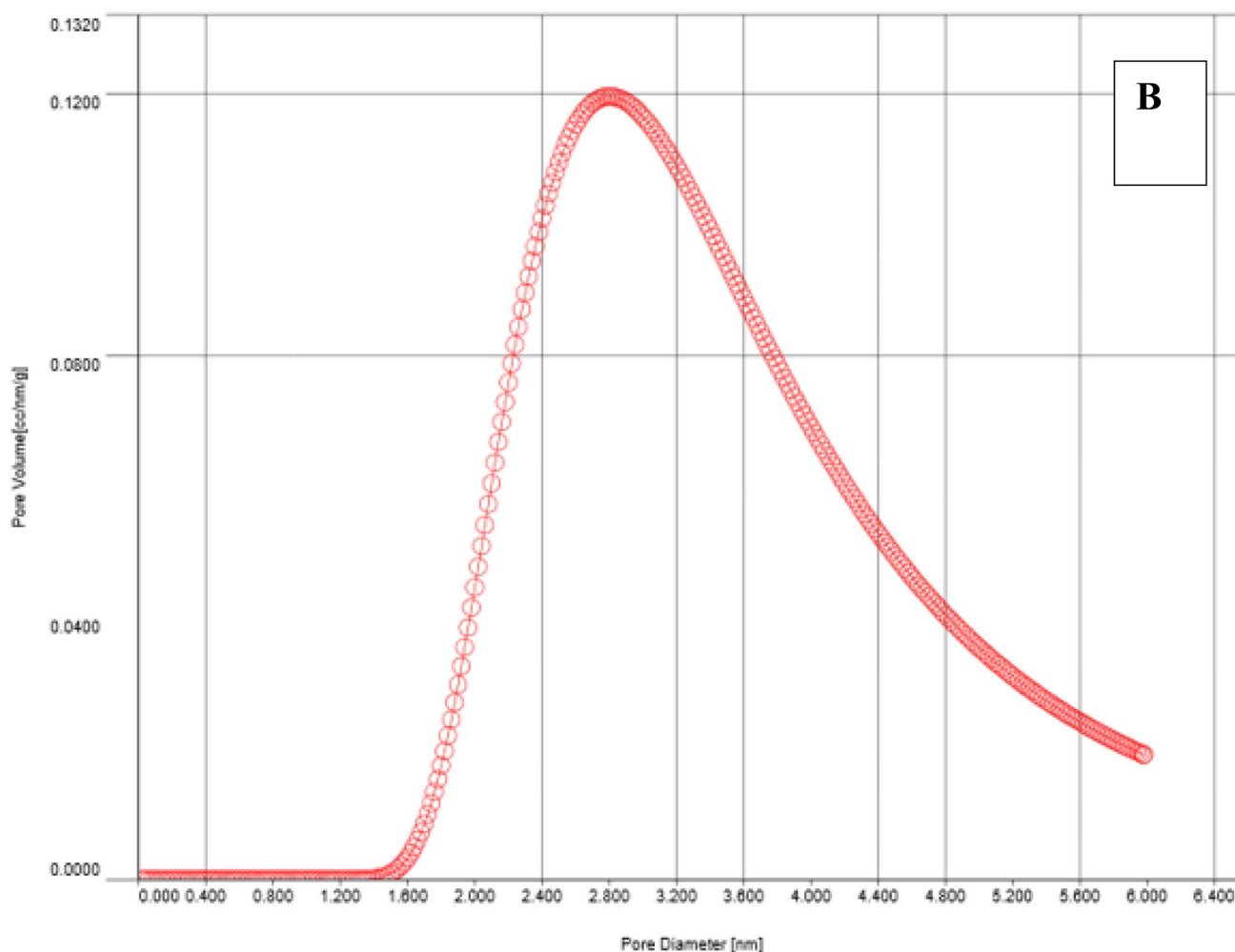


Fig. 7 (continued)

study an important one in the race for energy and environmental sustainability in developing African countries.

4 Conclusion

The albedo (OA) and peels (OP) of orange were converted in a top-lit updraft biomass reactor with retort heating at a peak temperature of the process was 300 °C and process time of 2 h. FTIR analysis showed that the spectra obtained for both samples were relatively similar with little differences in peak intensity. The presence of polar groups such as alcohol (OH), esters, ketones, aldehydes, carboxylic, ether and phenols suggests that the biochar samples has the potential to be used as adsorbent for aquatic pollutants and as soil amendment for improving cation exchange. The EDS analysis showed that OP biochar possess higher carbon content than OA biochar whilst the latter contains more inorganic

elements. This suggest that OA bio-chars would be more effective in pollutant adsorption both from water and soil while OA bio-chars would be more effective in GHG emissions and carbon sequestration applications. SEM analysis revealed that OP biochar possess a smooth surface as compared to the highly convoluted surface of the OA biochar. The OP biochar would possess a better catalytic activity, water retention capacity and adsorption capacity due to it having a larger surface area and possibly more active sites. BET analysis revealed that though the surface area of OP biochar was slightly lower than that of the OA biochar, it was more porous. The paper has been able to evaluate the characteristics of bio-chars obtained from the thermochemical conversion of orange residues. It can be clearly seen that the product if of high quality and can be used in a variety of important application.

Compliance with Ethical Standards:

Conflict of interest The authors declare that there are no conflicts of interest.

Ethical standards This article does not contain any studies involving human or animal subjects.

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