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# Evaluating the nature of captured exhaust soot from a retort heating carbonization system

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Abstract. A deeper understanding of all aspects of biomass thermochemical conversion is necessary as researchers pursue multiple avenues for energy and environmental sustainability. In this paper, the agglomerative accumulation of soot observed, captured on the inner surface of the exhaust pipe, during the operation of a top-lit updraft biomass carbonisation system was evaluated to understand its nature and qualities. The soot was evaluated using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy with Energy Dispersion Spectroscopy (SEM-EDS), and Brunauer-Emmett-Teller (BET) analyses. The combustion fuels were bamboo (Bambusa vulgaris) stalk and stem of African balsam (Daniellia olivieri). FTIR analysis revealed the presence of hydroxyl, aromatic double bond, aldehyde, thyiol, and carbonyl functional groups. Energy Dispersion Spectroscopy (EDS) analysis revealed that the elemental carbon content of the soot was found to be 75.05% carbon with 15.13% oxygen. Scanning Electron Microscopy (SEM) analysis showed that the soot had a hollow morphology and a lustrous appearance with white tiny grit grains of carbon nano-spheres. The BET analysis revealed that the specific surface area of the soot was 500  $m^2/g$ , while the pore volume and pore diameter were measured to be 0.218 cc/g and 2.113 nm, respectively. The material can find use in water purification purposes and also, as an additive to lubricating oils.

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## 1. Introduction

Thermochemical processing is an established technique of biomass valorization [1]. Such thermochemical processes include pyrolysis, gasification, combustion, hydro-thermal carbonisation, and torrefaction [2,3]. Gasification is one of the most popular techniques and it has been investigated for numerous feedstocks over the years [4,5]. The types of thermochemical conversion reactors include fixed bed, fluidized bed, ablative reactors, rotating cones, kilns, and furnaces [6,7]. Operation mode can be batch, semi-batch, and continuous [6].

For gasification reactors, a stream of inlet air is essential to the process. Fluidized bed gasifiers are fluidized by the inlet air stream by either circulation or bobbling [8], while fixed bed reactors have lower air velocity and are non-suspended in the inlet fluid. The inlet air-flow for the fixed bed takes three basic configurations: updraft [9], downdraft [10–13], and cross-draft [8]. Reactors can also be top-lit or bottom-

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lit based on the point of ignition of the combustion fuel [9]. Recent studies have utilized a low-temperature biochar-optimized carbonizer design in which an outer combustion space generates heating for the inner carbonization chamber. It has been evaluated for biochar production from elephant grass [14,15], plantain fibres [16], orange peel and albedo [17], almond leaves [18], sugarcane bagasse [19], and oil palm fibre [20]. In such hybrid systems where combustion is involved, soot is also produced and trapped.

Several studies have attempted to gain an understanding of combustion soot, albeit for vehicular combustion engines [21–23], domestic stoves [24], and various oxidation-gasification systems [25–27]. Some thermochemical conversion systems do not produce soot as their reaction does not involve combustion and flame. However, the current design under consideration [14,16] generates thermal energy for carbonization from an outer combustion region of which exhaust fumes are released. A significant agglomerative accumulation of fine soot char was observed to be trapped in the inner sections of the exhaust.

In this paper, the soot noticed from the experimentations in Adeniyi et al. [14,16] was evaluated to understand its nature and qualities with suggested possible application. The captured soot was evaluated using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy with Energy Dispersion Spectroscopy (SEM-EDS), and Branueur-Emmett-Teller (BET) analyses.

## 2. Methodology

## 2.1. Experimental

The sizing and design, operational considerations, and yield of products for the top-lit updraft biomass carbonizer with retort heating have been discussed elsewhere [14,16]. However, the soot is formed by the agglomerative accumulation of the fumes of the combustion fuels trapped and captured onto the inner walls of the exhaust pipe. The combustion fuels were bamboo (Bambusa vulgaris) stalk and stem of African balsam (Daniellia olivieri). The soot was retrieved from the exhaust pipe using a spatula and placed within sample bottles for analysis. The samples were employed in two carbonization runs individually for biochar production from elephant grass by Adeniyi et al. [14] and from plantain fibres by Adeniyi et al. [16]. Since the same combustion fuel (bamboo stalk and stem of African balsam) was used for retort heating, the soot in both studies was analyzed in this current investigation as it has been derived from the same parent material. The mechanisms of soot formation and methodologies for the characterization include FTIR, SEM-EDS, and BET analyses, to be discussed in the subsequent subsections.

## 2.2. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR was conducted to determine the functional groups and complexes present in the soot char. The spectrophotometer model used for the FTIR analysis was SHIMADZU FTIR-8400S. The sample holder was cleaned using ethanol on wet wipes and care was exercised to prevent the splash or spill of the reagent on the equipment. On the user interphase, the scan range was set to 650-4500 cm<sup>-1</sup>. The sample was placed on the holder and then, scanned to obtain the spectrum.

## 2.3. Scanning Electron Microscopy with Energy Dispersion Spectroscopy (SEM-EDS)

SEM was employed to examine the surface morphology of the soot char particles. A double adhesive was placed on the sample stub. The soot char was sprinkled on the stub and subsequently taken to a sputter coater (Quorum-Q150R Plus E) with 5nm of The stub was placed on a charge reduction gold. sample holder and introduced into the column of the SEM machine (Phenom ProX by Phenom world Eindhoven Netherlands). It was initially viewed from a navigation camera (NavCam) before being sent to SEM mode. The spectrum was recorded using transmittance method in the 4000-650  $\text{cm}^{-1}$  region with 30 scans. The acceleration voltage of the microscope was set to 15 kV and magnification of 500 to 1500 times. Different magnifications were stored after adjustment of brightness and contrast.

## 2.4. BET analysis

BET analysis was used to study the particle and pore dimensional characteristics of the soot char. The surface area, pore volume, and sizes of the bio-char were measured using a BET analyzer (NOVA Station A). The surface properties of the soot were considered using multipoint BET surface area and DR (Dubinin-Radushkevic) method for the pore volume and width (diameter), respectively. The bio-char was characterized by N<sub>2</sub> adsorption test at 77 K. A small amount of dry nitrogen gas was introduced to the sample tube to prevent contamination of the clean surface. The tube was then removed and the sample weighed. The tube was fixed to the volumetric apparatus; then, the sample was evacuated down to 2 Pa pressure. Adsorbate was introduced to give the lowest desired relative pressure; then, the adsorbed volume was measured.

## 3. Results and discussion

## 3.1. The mechanism of the soot formation

The general scheme of the formation of soot is illustrated in Figure 1. Controlled combustion occurred in the heating space (i.e., the combustion chamber) and the fumes produced are evacuated from the system via



Figure 1. The general scheme of the process.

the exhaust pipe. The fumes go through the exhaust pipe; soot is captured on the inner rough surfaces. This zone in the exhaust pipe is of reduced geometry and is centrally located on top of the reactor. The ratio of exhaust pipe to that of the combustion chamber is 1:10. The key reaction of the coke formation is shown in Eq. (1):

$$2CO \leftrightarrow C + CO_2. \tag{1}$$

The coke deposition is induced by the conversion of carbon monoxide into carbon dioxide, especially in a system with limited oxygen (like that used in the study). This coke is the substance that agglomerates on precursors to form soot.

The transport of the produced smoke out of the combustion zone occurred between the space above the carbonisation chamber and the exhaust pipe through a reduced geometry. The formation of soot along the exhaust pipe diameter is enhanced by the reduced geometry. The interacting and consecutive steps involved include formation of soot precursors on the exhaust pipe, soot particle inception, and aggregation of soot particle, as shown in Figure 2. Once the precursors are formed, larger numbers of particles are produced along



**Figure 2.** Formation mechanism of soot via soot precursors formation (a); soot particle inception (b); and the aggregation of soot particle (c).

the internal surface of the exhaust pipe; they collide to produce larger particles that eventually agglomerate to produce aggregates of soot.

## 3.2. Fourier Transform Infrared Spectroscopy (FTIR)

Spectra of soot samples were obtained from FTIR analysis. Figure 3 shows the peaks observed in the region between  $4000 \text{ cm}^{-1}$  and  $600 \text{ cm}^{-1}$ . The peaks and their corresponding assignments are given in Table 1. The strong peaks at  $3857 \text{ cm}^{-1}$  and  $3711 \text{ cm}^{-1}$  correspond to the residual O-H surface groups present in the soot char [28], while the presence of the N-H stretching band is shown by the peak at  $3402 \text{ cm}^{-1}$  [29]. The peak at 2978  $\rm cm^{-1}$  is assigned to the CH stretching vibrations of the  $CH_3$  group [30,31]. The small peaks at  $2754~\mathrm{cm^{-1}}$  and  $2492~\mathrm{cm^{-1}}$  correspond to the aldehyde (-CHO) and thiol (S-H), respectively [32,33]. The possible functional group of the peak detected at 2283  $\rm cm^{-1}$  has not been previously reported, as noted by Matthews [34]. The C=O stretching of carboxylic acids can be observed at 1720 cm<sup>-1</sup> [35], while the peak at  $1512 \text{ cm}^{-1}$  corresponds to the C=C stretching of aromatic or alkene groups [22]. A broadening is observed in the region of 1000–1300  $cm^{-1}$  in the spectrum. This region is a complex section of the IR spectrum, where aromatic C-C and C-H plane deformation structures are observed; however, the most important structure corresponds to ether C-O-C stretching groups [22,36]. The peak at  $1273 \text{ cm}^{-1}$  corresponds to the C-O stretch vibrations of carboxylic acids and ethers [37]. The small peak at  $1010 \text{ cm}^{-1}$  corresponds to the unsaturated hydroxyl (C-C-O) stretch [35], while the



Figure 3. IR spectra of biomass soot.

Peaks $(cm^{-1})$	Assignment	Reference (s)
3857 (sharp)	O-H (hydroxyl)	[28]
$3711 \; (sharp)$	O-H	[67]
3402	N-H	[29, 68]
2978	C-H	[30, 31]
2754	-CHO (aldehyde)	[33]
2492	S-H (thiol)	[32]
2283	Possible functional group could not be identified	
1720	C=O (carbonyl)	$[35,\!69]$
1512	C=C (aromatic)	[22]
1273 (broad)	C-O	[37]
941	P-OH	[38]
694 (broad)	C-H (out of plane deformations)	[39]

Table 1. Peak assignments.

origin of the peak at 941  $\rm cm^{-1}$  corresponds to the phosphonic group [38]. The peak observed at 694  $\rm cm^{-1}$  corresponds to the CH out of plane deformations [39]. The FTIR analysis confirms the presence of carbon, oxygen, nitrogen, sulphur, hydrogen, and phosphorus.

## 3.3. Scanning Electron Microscopy with Energy Dispersion Spectroscopy (SEM-EDS)

Tables 2 and 3 give the elemental analysis of the soot char with and without inclusion of carbon/oxygen/nitrogen, respectively. The percentage of carbon is not included in Table 2 to best illustrate hetero-element concentrations. The tables quantitatively confirm the presence of some elements earlier observed from the FTIR analysis. The data from Tables 2 and 3 are plotted in Figures 2 and 3, respectively.

Table 2.	Elemental	analysis	(carbon	included	)
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Element name	Weight	
Element name	concentration $(\%)$	
Carbon	75.05	
Oxygen	15.13	
Iron	2.92	
Nitrogen	2.25	
Copper	0.76	
Aluminium	0.51	
Calcium	0.48	
Sulphur	0.48	
Potassium	0.47	
Magnesium	0.47	
Silicon	0.42	
Phosphorus	0.41	
Sodium	0.38	
Manganese	0.28	

Element name	Weight	
Element name	concentration	
Iron	32.65	
Aluminium	8.54	
Copper	8.48	
Magnesium	7.98	
Silicon	7.25	
Sulphur	7.22	
Phosphorus	6.55	
Sodium	6.19	
Potassium	6.00	
Calcium	5.99	
Manganese	3.15	

Table 3. Elemental analysis (carbon excluded).

From Table 2, it can be seen that the elemental carbon content of the soot was found to be 75.05% carbon with 15.13% oxygen with the carbon content greater than biochar obtained from other biomass like rice, bamboo [40], and date palm [41].

The exclusion of carbon in the elemental analysis enhanced the presence of the elements earlier observed in the carbon inclusive analysis. The carbon rich soot can serve as a favorable adsorbent for nonbiodegradable or inorganic pollutants [42]. The oxygen content and oxygen-containing functional groups enhance the adsorption of organic pollutants by interacting with the pollutants via hydrogen bonding or complexation [43]. The addition of the carbon soot to soils combined with bioenergy can be used as a means to mitigate climate change [44]. The soot can also be explored as a binder-free electrode material for high performance energy storage devices such as super capacitors [45-47]. Iron, which is the most abundant element in addition to carbon and oxygen, increases the adsorption capacity in pollutant removal and toxicity reduction [48, 49].

As previously applied with diesel soot [50,51], the obtained soot char can also be considered for use as an additive to lubricating oils to reduce the friction and wear rate reduction. In this respect, it could serve as an alternative to the use of diesel soot and carbon black as lubricant additives. Dispersion of soot nanoparticles to lubricating oils in small concentrations strengthens the anti-friction effects of the lubricants [50,52]; however, this depends on the oil formulation [51]. Soot can also be used as a solid lubricant [53,54].

The SEM analysis of the soot materials is shown in Figure 4 with a magnification ranging from 500 to 1500 times. The images show that the soot has a hollow morphology and a lustrous appearance with large surface particles. Moreover, there is no cluster formation because of the inherent stability of the



Figure 4. SEM micrographs of the biomass soot.

 Table 4. Surface properties of the soot.

Parameter	Value
Multipoint BET surface area	$500.8 \mathrm{m}^2/\mathrm{g}$
Pore volume	$0.218 \mathrm{~cm^3/g}$
Pore diameter	2.113 nm

soot [21]; there are white tiny grit grains/spheres (red arrow) commonly referred to as carbon nano-spheres [22] on the surface, affirming the large surface area as confirmed by the BET analysis results.

## 3.4. BET analysis

The surface area, pore volume, and pore diameter of the soot char were analyzed using a BET analyzer, and the results obtained are presented in Table 4 The BET specific surface area of the soot below. was measured to be 500  $m^2/g$ , while the BJH pore volume and pore diameter were measured to be 0.218 $cm^3/g$  and 2.113 nm, respectively. The surface area and pore of the soot were found relatively higher than bio-chars previously obtained from the carbonization unit [14,16], which is the main product. The specific surface area was also found to be higher than candle soot [55], diesel soot [56], and functionalized carbon soot [57], which possessed surface areas of 78.6  $m^2/g$ ,  $55 \text{ m}^2/\text{g}$ , and  $137.93 \text{ m}^2/\text{g}$ , respectively. High surface area increases the potential of the soot as -an adsorbent for heavy metals [58,59] and other wastewater pollutants [60–62]. The pore size distribution is shown in Figure 5. It is observed that a significant volume of the soot has a pore size/diameter of 2-6 nm. It is in the mesoporous range according to the International Union of Pure and Applied Chemistry (IUPAC) classification. This reveals that the soot obtained from the process is mesoporous in nature. The soot properties also make it a potential alternative to carbon black as a filler/additive to plastic composites [63,64], polymers, thermoplastic, and rubber blends [65,66].

## 4. Conclusion

A significant agglomerative accumulation of fine soot char was observed to be trapped in the inner sections



Figure 5. Pore size distribution of the soot obtained.

of the exhaust. In this paper, the soot derived from the experimentations in Adeniyi et al. [14,16] was evaluated to understand its nature and qualities. FTIR analysis revealed the presence of hydroxyl, aromatic double bond, aldehyde, thiol, and carbonyl functional groups. EDS analysis revealed that the elemental carbon content of the soot was found to be 75.05% carbon with 15.13% oxygen. SEM analysis revealed that the soot had a hollow morphology and a lustrous appearance with white tiny grit grains of carbon nano-spheres. The Branueur-Emmett-Teller (BET) analysis revealed that the specific surface area of the soot was  $500 \text{ m}^2/\text{g}$ , while the pore volume and pore diameter were measured to be 0.218 cc/g and 2.113 nm, respectively. The very high carbon content and surface area imply that it can serve as an adsorbent for water purification purpose. It can be considered for use as an additive to lubricating oils to reduce the friction and wear rate reduction, serving as an alternative to the use of diesel soot and carbon black as a lubricant additive.

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