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Thermogravimetric Study of Maize Cob Carbonization for Bioenergy Recovery: Product Yield Estimation and Bio-Energy Potentials

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Abstract

The study investigated the bulk fuel, microstructure, morphological, mineral, and functional group characteristics of maize cobs (MC) along with carbonization through thermogravimetric analysis (TGA) for potential energy recovery. To the best of the authors' knowledge, this is the first study on the micro-scale analysis of the fuel properties and bioenergy recovery potential of MC in the scientific literature. The results showed that MC contains high carbon, hydrogen, oxygen, volatile matter and fixed carbon but low moisture, and ash contents. The functional group (FTIR) analysis revealed MC contains alcohol, ester, and carbonyl functional groups in its chemical structure, which could be attributed to the lignocellulose components of biomass. The analysis of MC microstructure and morphology showed a rough yet compact surface comprising fibres. The TGA carbonisation process revealed MC experienced significant mass loss (ML) ranging from 73–76%, whereas the residual mass or mass yield (RM or MY) was from 23.6–27.2% with increasing carbonisation temperatures from 450–650°C. Furthermore, the findings indicated that the optimal temperature for carbonization of MC is 550°C, based on the conditions examined in this study. The HHV of the solid MY ranged from 26.66 – 26.99 MJ/kg, whereas the energy yield (DE) was 95.42 – 95.63%, and energy density (DE) 3.52 – 4.04. The findings indicate that while the HHV and EY increased, the MY and EY decreased with increasing carbonisation temperatures. In general, the study demonstrated that MC is a potentially suitable raw material or biomass feedstock for the sustainable recovery of bioenergy through carbonization.

Keywords: *Maize cobs, Biomass, Carbonization, Energy recovery, Thermogravimetry.*

1. Introduction

Over the years, anthropogenic activities have continued to exacerbate the effects of climate change and global warming on the planet [1-2]. It is estimated that human activities such as agriculture, land use, food wastes, transportation, and energy use among others have resulted in ~ 0.8°C to 1.2°C global warming, which is higher than during the pre-industrial era [3-4]. Based on the current data, the IPCC report projects that the warming of the planet could soar to 1.5°C by the year 2052 [5], which could spell disastrous consequences for humanity and future generations [6-7]. Therefore, there is a growing need to curb and or mitigate the soaring emissions of greenhouse gases (GHG), particularly from energy use as the sector accounts for

~73% or about 49.4 billion tonnes of CO_2 eq. annually [8]. Furthermore, energy analysts posit that the burning of coal accounts for over 40% of carbon dioxide (CO_2) emissions on the planet. Since energy production and consumption play crucial roles in the warming of the planet and changes in climatic conditions; the transition from fossil fuels to greener alternatives will help to alleviate or reverse the environmental damages already caused by GHG [9]. To achieve these goals, Paris Accord of 2015 posits that humanity needs to reduce GHG from 50 billion tonnes annually to zero over the next 50 years [10-11]. Therefore, it has become imperative to seek, adopt and implement alternative sources of energy such as solar, wind, geothermal, tidal, or biomass.

Biomass is a widely accessible, socially acceptable, and carbon-neutral source of energy with the potential to address the world's growing need for renewable and sustainable alternatives [12-13]. Therefore, it is envisaged that humanity's switch from fossil fuels to renewable energy technologies (RET) will enhance environmental sustainability by lowering GHG emissions and diversifying the global energy mix [14-15]. From an economic perspective, the large scale adoption and implementation of RETs will also create long-term jobs, improve living standards, and address the energy crises, particularly in developing countries [16-18]. Ultimately, the transition to RETs will also greatly enhance human health, safety, and the environment, which are critical tenets of the sustainable development goals [18-19]. However, the recovery and utilisation of energy from biomass is prone to numerous challenges owing largely to its intrinsic fuel properties. For example, biomass feedstocks are bulky (or heterogeneous), which results in pre-treatment, processing, handling, storage, and transportation problems [20-21]. Likewise, raw or harvested biomass is characterised by high moisture, ash, mineral matter and low heating value, volumetric and gravimetric densities, which hamper energy recovery potential [22-23]. The wide variety, age, sources, location, and soil conditions result in a wide range of biomass feedstocks with various properties. However, the lack of comprehensive data on the fuel and energy properties of a wide range of biomass properties has also hampered utilisation for energy recovery.

Maize (*Zea mays L.*) is a staple food and cash crop in many developing nations such as Nigeria. It is estimated that 49.7% of households in Nigeria cultivate maize [24], which in addition to commercial agricultural production accounts for ~11 million tonnes annually [25]. Conversely, the cultivation and processing of Maize generate large quantities of solid wastes or stover comprising leaves, stems and the cobs, which account for over half the mass of the plant. Current strategies for the waste valorisation include the production of animal feeds, organic manure, mulching, cooking fuel [26-27], whereas other inefficient, unsustainable, and costly approaches such as landfilling, dumping or open-air burning, have been proposed in the past to address the challenges of maize wastes [28]. Since the maize cobs account for a significant part of the wastes, it has become imperative to explore sustainable approaches for their valorisation into valuable products. One approach is the conversion of maize cobs (MC) into energy through biomass conversion technologies such as pyrolysis or carbonisation.

Therefore, this study aims to critically examine the bulk fuel, microstructure, morphological, mineral, and functional group characteristics of MC and carbonization potential through thermogravimetric analysis (TGA). To the best of the authors' knowledge, this is the first study on the micro-scale analysis of the fuel properties and bioenergy recovery potential of MC in the scientific literature.

2. Materials and methods

The experimental methods, standards, and equipment employed to examine and estimate the product yield, distribution, and bio-energy potentials of Maize Cob (MC) through carbonization are presented in this section of the paper. In addition, the physicochemical, microstructure, mineral, and functional group characteristics of MC are also presented in detail. The outlined properties are critical to understanding the fuel properties, potential applications, and waste profiles after thermochemical conversion [29-30].

2.1. Bulk fuel properties

The bulk fuel properties of MC comprising the ultimate, proximate, and calorific characteristics were examined in this study. The ultimate analysis was carried out using the CHNS analyser (Vario Macro Cube, Germany) to determine the elemental composition of carbon, hydrogen, nitrogen, and sulphur content of MC, respectively. The oxygen content was calculated from the sum of the CHNS composition in weight percentage (wt.%). The proximate analysis of MC was carried out on the thermogravimetric analyser (Shimadzu TG-50, Japan) to determine the moisture content (MC, Temperature 110°C and hold time 6 minutes), volatile matter (VM, Temperature 900°C and hold time 5 minutes), and ash content (AC 750 °C and hold time 5), and the fixed carbon (FC by difference). The proximate analysis was conducted based on the heating program described in the literature [31]. Lastly, the calorific analysis was carried out on the bomb calorimeter (IKA C200, USA) under isoperibolic conditions to determine the higher heating value (HHV) of MC.

2.2. Fourier Transform Infra-Red (FTIR)

The functional group analysis of MC was conducted through Fourier Transform Infra-Red (FTIR) spectroscopy using the FTIR analyser (Shimadzu Prestige 21, Japan). For each test, the powdered MC was mixed with potassium bromide (KBr) in the ratio of 1:100 before pelletization using pellet and die apparatus. Next, the sample was analysed using the FTIR analyser based on 20 runs and 16 cm resolution to obtain the functional group compositions of MC. The FTIR spectra as recorded from 4000 cm^{-1} to 400 cm^{-1} as presented in Figure 1.

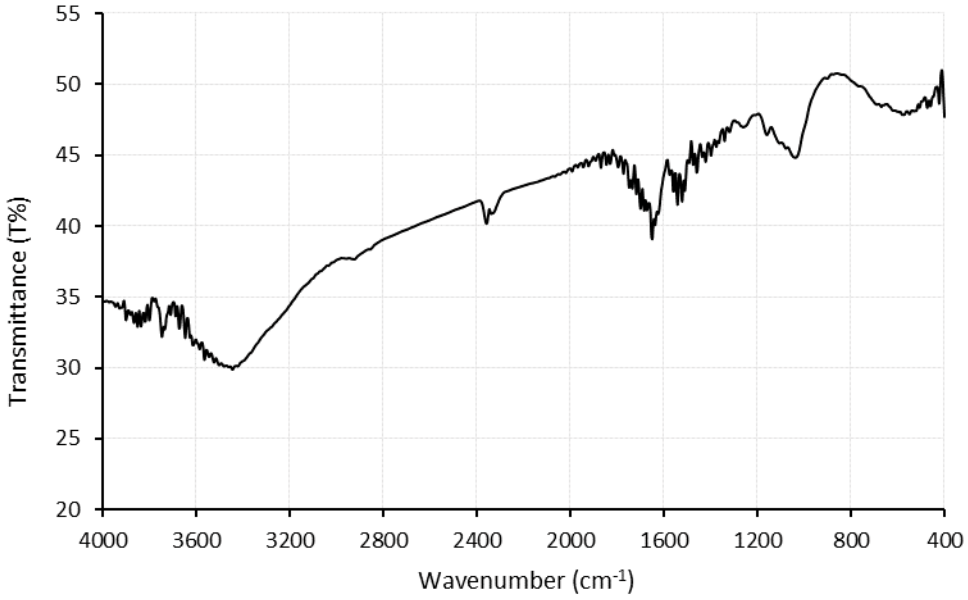


Figure 1. FTIR spectra of maize cobs

2.3. Scanning electron microscopy (SEM)

The morphology and microstructure of MC were analysed by scanning electron microscopy (SEM) using the microscope (JEOL JSM IT-300 LV, Japan). The powdered MC sample was deposited on carbon epoxy tapes and sputter-coated with gold to enhance image quality and prevent charging before the SEM analysis. Next, the sample surface was scanned in vacuum to examine the morphology and microstructure at a magnification of ×1000 through the point ID technique. The SEM micrograph depicting the morphology and microstructure of MC is presented in Figure 2.

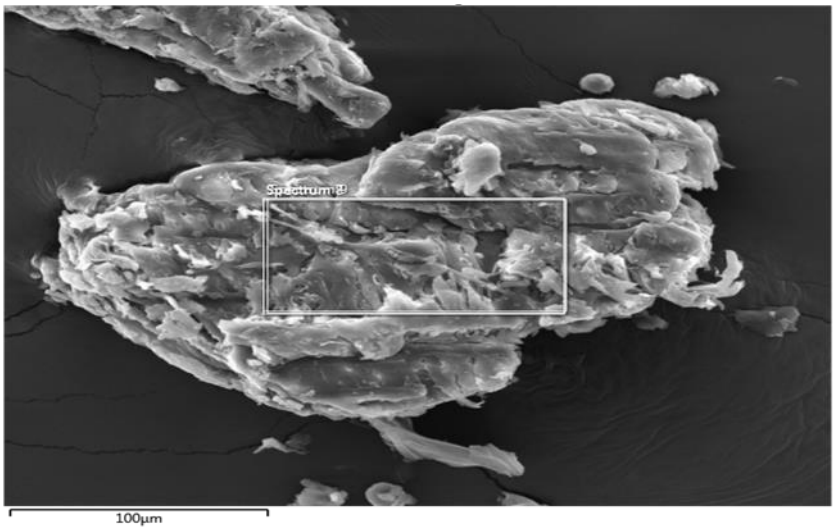


Figure 2. SEM micrograph of maize cobs

2.4. Energy dispersive X-ray (EDX)

The metal and metal compositions of the MC were examined by electron dispersive X-ray (EDX) using the EDX analyser (JEOL JSM IT-300 LV, Japan) based on the capture zone and point analysis technique. Next, the compositions of metal and metals in the MC sample calculated based on charge balance and the EDX spectra are presented in Figure 3.

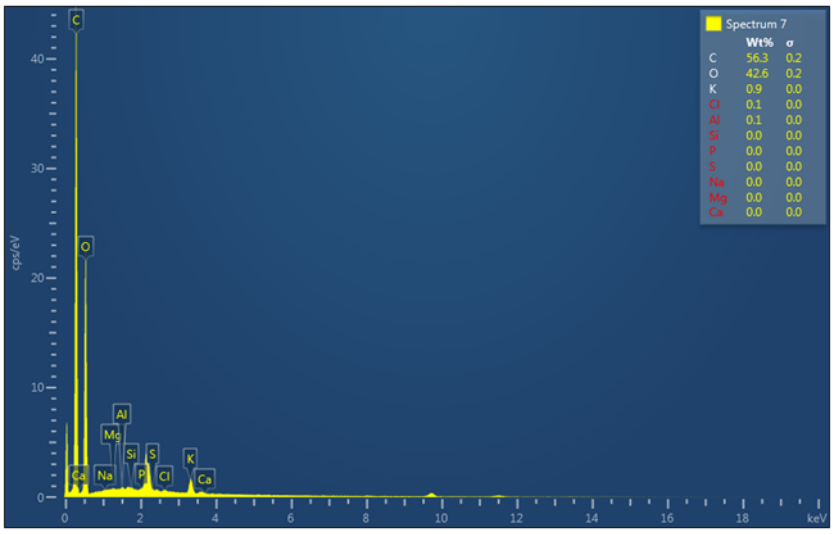


Figure 3. Elemental composition of maize cobs by EDX analysis

2.5. Thermogravimetric analysis (TGA)-based carbonisation of MC

The carbonization of MC was examined through thermogravimetric analysis (TGA). The analysis was performed at the selected non-isothermal/isothermal heating programme of the TGA. For each test, 7 ± 0.20 mg of MC was weighed in an alumina crucible and heated from 30°C to the reaction temperatures (450°C, 550°C, and 650°C) at a heating rate of 30°C/min and hold time of 30 minutes. The evolved species were purged with nitrogen gas at a flow rate of 50 mL/min. On completion, the TGA furnace was cooled and the thermogram analysed to obtain the mass loss (TG, %) and derivative mass loss (DTG, %/min) which were plotted against time (minutes).

2.6. Product yield, distribution, and energy analyses

The product yield, distribution, and energy analysis of the carbonization process were computed from the mass loss (ML) and residual mass (RM) data based on the following equations. Next, the carbonization parameters, namely; higher heating value (HHV), mass yield (MY), and energy yield (EY) were calculated from the mass loss (ML) using equations 1-4 [32], while the energy density (ED) was calculated from the MY [33];

$$HHV\left(\frac{MJ}{kg}\right) = 19.85 + 9.35ML(\%) \quad (1)$$

$$MY(\%) = 100 - ML(\%) \quad (2)$$

$$EY(\%) = 1 - 0.06ML(\%) \quad (3)$$

$$ED = \left(\frac{EY}{MY}\right) \quad (4)$$

3. Results and discussion

3.1. Bulk fuel properties

The bulk fuel properties of MC comprising the ultimate, proximate, and calorific characteristics were examined in this study and presented on an as-received basis (ar) in Table 1. The aim was to reveal the elemental constituents, proximate properties, and higher heating value (HHV) of MC for potential energy recovery. The bulk fuel properties provide insights into the fuel quality, potential waste profiles, or the environmental friendliness of potential biomass [34-36].

Table 1. Bulk fuel properties of maize cobs

Fuel analysis	Elements/Property	Symbol (Unit)	Maize cobs (ar)
Ultimate	Carbon	C (wt.%)	41.73
	Hydrogen	H (wt.%)	6.29
	Nitrogen	N (wt.%)	0.82
	Sulphur	S (wt.%)	0.11
	Oxygen	O (wt.%)	51.04
Proximate	Moisture	M (wt.%)	9.88
	Volatile Matter	VM (wt.%)	68.02
	Fixed Carbon	FC (wt.%)	15.70
	Ash	A (wt.%)	6.41
Calorific	Higher Heating Value	HHV (MJ/kg)	15.85

As observed in Table 1, MC contains high carbon, hydrogen, oxygen, volatile matter, and fixed carbon but low ash, moisture, nitrogen, and sulphur. The HHV of 15.58 MJ/kg deduced for MC is below the average value of 18 MJ/kg required for thermochemical biomass conversion [37-38]. The low HHV could be ascribed to the high oxygen content of MC presented in Table 1. Furthermore, the high O content could result in over-oxidation of MC fuel particles along with the conversion of nitrogen and sulphur into NO_x, SO_x, and H_xS during thermochemical conversion. Hence, the findings indicate that the N and S content of MC could potentially result in flue gas emissions during the energy recovery and conversion of MC. Overall, the results indicate that MC is fairly good quality and environmentally friendly fuel with a low waste potential profile.

3.2. Fourier Transform Infra-Red (FTIR)

Figure 1 presents the FTIR spectra for MC from 4000 cm^{-1} to 400 cm^{-1}. The spectrum contains several broad, sharp, or medium peaks and bands of varying intensities ranging from 30 T% to 53 T%. The objective of the analysis is to determine the functional group compositions of MC.

The sharp peaks observed from 4000 cm^{-1} to 3600 cm^{-1} could be assigned to the rota-vibrational bands of water vapour, which confirms the existence of inherent moisture in the

MC chemical structure. Likewise, the broad peak observed between 3600 cm^{-1} and 3200 cm^{-1} could be assigned to the stretching vibrations of the O-H functional groups in alcohols, phenols or lignin. However, the medium-sharp peak observed at 2300 cm^{-1} could be assigned to the stretching vibrations of the functional groups −C≡N− or −C≡C− typically found in unsaturated hydrocarbons (alkynes), nitrile, and heterocyclic compounds. Furthermore, the small- and medium-sharp peaks between 1800 cm^{-1} to 1600 cm^{-1} and from 1600 cm^{-1} to 1200 cm^{-1} could be assigned to the −C=C, C−H or C=O stretching vibrations found in esters, aldehydes, ketones, and carboxylic acids. The peaks in these regions are typically linked with lignin found in plants. Lastly, the peak observed between 1200 cm^{-1} and 1100 cm^{-1} could be assigned to the C-O-H, C-O-C, or OCH_3 vibrations related with the guaiacyl monomers and pyranose rings of hemicellulose and cellulose. Overall, the FTIR analysis showed that the chemical structure of MC is comprised of the lignocellulosic components, namely; hemicellulose, cellulose, and lignin along with alcohol, ester, ketone, carbonyl, and carboxylic acid functional groups.

3.3. Scanning electron microscopy (SEM)

Figure 2 presents the micrograph of MC determined by scanning electron microscopy (SEM). Based on the SEM micrograph, it can be surmised that the morphology and microstructure of MC consist of a rough surface characterised by layers of asymmetrical shaped fibres and particles. The fibres could be due to lignin and hemicellulose, whereas the irregular shaped particles could be ascribed to oil, waxes and extractives typically found on the surface of biomass. Further analysis indicates the MC surface is characterised by a surface structure devoid of pores, crevices or fractures. Hence, MC contains a compact surface structure with low porosity and voidage. Hence, it can be surmised that MC is characterised by compact microstructure and low porosity, which could result in the uniform thermal degradation of the particles. Furthermore, the asymmetrical shaped fibres and particles and lack of pores, crevices or fractures could result in uniform thermal degradation of the MC particles during thermal conversion.

3.4. Energy dispersive X-ray (EDX)

Figure 3 presents the EDX spectra of MC determined by energy-dispersive X-ray (EDX). The findings indicate that MC consists of carbon, oxygen, potassium, chlorine, and aluminium in major and trace compositions. As observed in Figure 3, the major elements detected during EDX are carbon (56.3 wt.%) and oxygen (42.6 wt.%), while potassium, chlorine, and aluminium were detected in trace amounts. The presence of carbon and oxygen is due to their roles as structural elements of hemicellulose, cellulose, and lignin in MC [39-40]. Furthermore, the occurrence of potassium and chlorine may be due to the alkali metals based salts in the soils or environment during the growth and development of MC [41-42]. Lastly, the aluminium may be due to oxides of the metal also found in groundwater and soils [43-44].

3.5. Thermogravimetric analysis (TGA)-based carbonization of MC

The carbonisation of MC was examined through TGA by heating a known mass of the MC sample from 450°C to 650°C (Δ100°C) at 30°C/min heating rate and hold time of 30 minutes based on the selected non-isothermal/isothermal heating programme of the TGA. Figures 4 and 5 present the TG and DTG plots for the carbonization of MC, respectively, whereas Figure 6 presents the temperature profiles for the TGA process.

The TG plots in Figure 4 indicate that the increase in holding temperature and time significantly influenced the carbonisation of MC. As observed, the slope of the TG plots decreased from right to left resulting in a higher mass loss as the temperature increased from 450°C to 650°C during the carbonisation process. However, the DTG plots revealed two sets of endothermic peaks that occurred from room temperature to 105°C and between 105°C and the holding temperatures (450°C, 550°C, and 650°C). The findings indicate that the carbonisation process occurred in three stages, namely; drying (due to loss of surface moisture from 30°C to 105°C), carbonisation (due to loss of volatile and lignocellulosic matter from 105°C to holding temperature), and lastly char degradation [45].

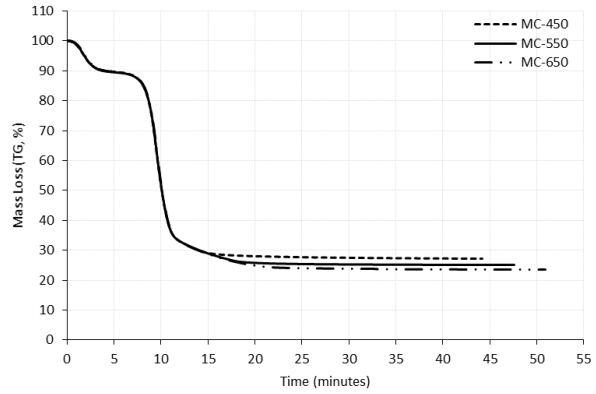


Figure 4. TG plots for the carbonisation of MC

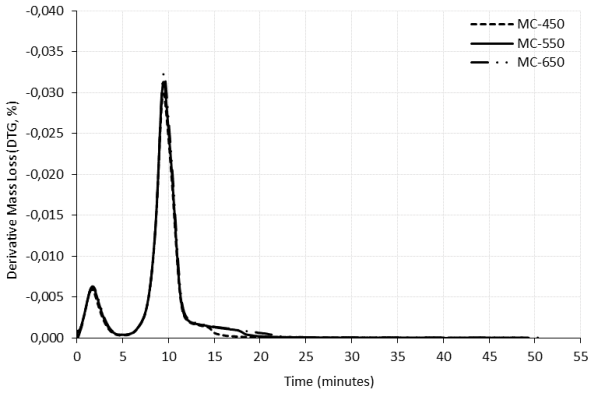


Figure 5. DTG plots for the carbonisation of MC

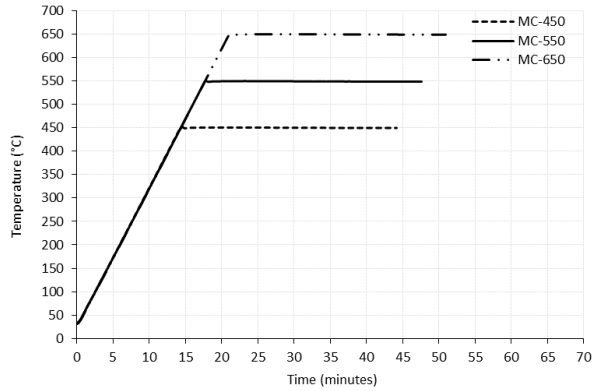


Figure 6. Temperature profiles for the carbonisation of MC

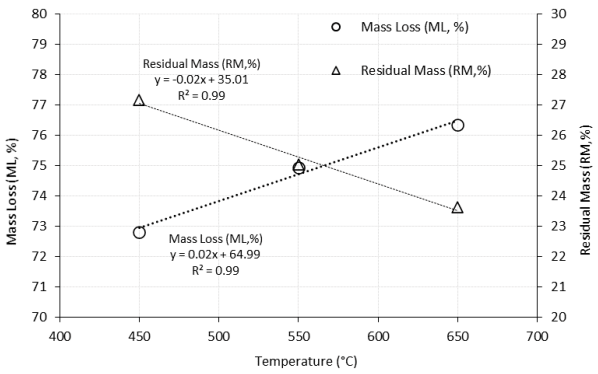
Based on the size, symmetry, and mass loss of the peaks, it can be reasonably surmised that the second stage is the rate most reactive stage. To further examine the extent of the thermal reactions, the temperature profile characteristics (TPC) of the MC during the entire TGA-carbonisation process was examined as presented in Table 2. As observed, the onset values decreased from 262.48°C to 261.75°C, whereas the midpoint and endpoint values increased from 304.58°C to 306.59°C and 347.17°C to 350.42°C, respectively, during the TGA-carbonisation process. The findings indicate that the overall thermochemical reaction of devolatilization that governs the carbonisation process occurs in the range of 261.75°C and 350.42°C. The devolatilization of MC results in various potential products, as highlighted in section 3.6.

Table 1. Temperature profile characteristics of MC during TGA-carbonisation

Carbonization temperature (°C)	Onset temperature (°C)	Midpoint temperature (°C)	End point temperature (°C)	Reaction time (mins)
450	262.48	304.58	347.17	44.18
550	261.93	305.38	348.50	47.57
650	261.75	306.59	350.42	50.93

3.6. Product yield, distribution, and energy analyses

Figure 7 shows the mass loss (ML) and residual mass (RM) of the TGA carbonisation of MC. The yield, distribution, and energy of the potential products were analysed through ML and RM. As observed, the mass loss (ML) decreased from 72.8% to 76.4%. The decrease in mass of MC during the carbonisation process can be ascribed to devolatilization (or removal of volatile matter). Furthermore, it could be due to the degradation of the biomass components, namely; hemicellulose, cellulose, and lignin, resulting in the formation of pyrolytic carbon. Hence, the increase in temperatures from 450°C to 650°C observably enhanced the devolatilization, decarboxylation, and depolymerisation of the MC biomass components. Furthermore, the findings indicate that the optimal temperature for carbonization of MC is 550°C, based on the conditions examined in this study. However, the residual mass (RM) or mass yield (MY, %) decreased from 27.2% to 23.6% with increasing carbonisation temperatures. The findings



indicate that the potential biochar components for MC carbonisation could range from 24% to 27% with potential HHV ranging from 26.66 to 26.99 MJ/kg as presented in Table 3. Furthermore, the HHV and DE increased, whereas the MY and EY decreased with increasing carbonisation temperatures.

Figure 7. Product yield, distribution, and energy analyses

Table 2. Product yield, distribution and energy analyses

Carbonization temperature (°C)	Higher Heating Value (MJ/kg)	Mass yield (MY, %)	Energy yield (EY, %)	Energy density (DE)
450	26.66	27.18	95.63	3.52
550	26.86	25.05	95.50	3.81
650	26.99	23.64	95.42	4.04

4. Conclusions

The study examined the product yield, distribution, and energy potential of the carbonisation of maize cobs (MC) through thermogravimetric analysis (TGA). The bulk chemical fuel and functional group compositions of MC along with the microstructure, morphology, and mineralogy were also examined in the study. The findings revealed a high content of carbon, hydrogen, oxygen, volatile matter and fixed carbon but low moisture, ash. FTIR analysis showed that the chemical structure of MC is comprised of alcohol, ester, and carbonyl functional groups typically ascribed to the lignocellulosic components of biomass. The microstructure and morphology analysis revealed MC is characterised by a rough yet compact surface. The product yield analyses indicated that the mass loss (ML) ranged from 73% to 76%, whereas the residual mass (RM or MY) was from 23.6% to 27.2% with increasing carbonisation temperatures from 450°C to 650°C. In general, the study demonstrated that MC is a potentially suitable raw material or biomass feedstock for the sustainable recovery of bioenergy through carbonization.

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