Effects of Densification and Torrefaction on the Thermal Properties of Pelletized Corncob

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Abstract

Biomass torrefaction into fuel pellets is deemed a sustainable energy solution, which could mitigate over-dependency on fossil fuels. This study examined the effects of pelletization and torrefaction on selected physical and thermal properties of corncob waste (CW). Corncob samples were collected, sorted and pulverized, before torrefaction pre-treatment. Raw corncob (RC) and torrefied corncob waste (TCW) were screened to 0.3, 0.5 and 1.0 mm. Using starch (5 wt.%) as binder, RC and TCW pellets were produced, at compaction pressures of 50, 75 and 100 MPa. They were characterized by proximate, ultimate, physical, thermogravimetric and thermal analyses. All samples exhibited good quality and thermal properties, but those from TCW were better. Relaxed density of RC and TCW pellets varied from 700 to 876 and 616 to 800 kg/m³, respectively. Maximum relaxation values for RC and TCW pellets were 1.43 and 1.07, respectively. TCW sample (1.0 mm) had higher heating value of 25.8 MJ/kg, which was 29% greater than those from lignite or brown coal. Thus, pellets produced from TCW are suitable for energy applications.

Keywords: compaction pressures; pelletized particles; particle size; TCW; TGA.

Introduction•

Until recent years, fossil fuels have played an important role in driving industrialization, and they are the leading source of global energy consumption [1-3]. Fossil fuel consumption is associated with negative environmental impacts such as global warming and climate change. This has required a quest for alternative sources of sustainable energy generation [4-6]. Biomass, a naturally abundant energy source is considered a promising alternative to fossil fuel, due to its renewability, global availability and carbon-neutral balance [7-9]. Biomass

[•]The abbreviations list is in page 74.

includes woody and agricultural waste from forestry activities, products and spinoff materials like cobs, stalks, pods and shells, among others [4]. Traditionally, woody waste has been the most favourable energy source. Agricultural wastes are increasingly becoming important energy sources, due to their short planting seasons and lack of impact on deforestation and environment [1]. Corncob wastes (CW) are abundant in tropic regions such as Nigeria [10]. In corn mills, corncobs are used as substitute fuel in convectional boilers with low thermal efficiency, because of their poor fuel properties and low energy density [3]. Corncobs have a higher moisture content (MC), less fixed carbon content (FCC), more volatile matter content (VMC), more fibrous and tenacious nature, and less energy than those from low-to-medium-grade fuels [11-13]. The problems associated with transportation, grinding, storage and handling become massive issues at a cogeneration plant [3]. One of the crucial steps to circumvent these limitations is through pre-treatment methods such as torrefaction and densification processes like pelletization [7, 14].

Torrefaction is a thermochemical conversion process in which biomass is heated to temperatures (T) ranging from 200 to 300 °C, while preserving atmospheric pressure in an unreactive atmosphere [15]. Torrefied solid fuels have a comparatively high mass yield, energy content and less VMC, which is regarded as an improvement on raw solids.

Pelletization is a densification process that can improve handling characteristics of raw materials and enhance calorific value (CV) of biomass. This process addresses low-density and transportation problems. Combined torrefaction and pelletization produce high energy density fuels [16, 17]. Research efforts have elaborated on the implication of bio-residue densification and pretreatment processes on fuels' physical and thermal properties for energy applications. For instance, physicochemical characteristics of tar coal and TCW with five distinct torrefaction temperature and four different holding times (HT) have been examined [17]. Proximal (PA) and ultimate (UA) analyses and physical tests have been carried out to investigate properties of TCW samples, at five distinct temperatures and four HT. Reported results have revealed that, at maximum T of 300 °C and HT of 60 min, properties of TCW have favourably compared with those from Thar coal. Also, it has been reported that torrefaction improves thermal and combustion characteristics of biomass waste. However, CV has varied from 17.26 to 18.89 MJ/kg.

Upgrading of corncobs via torrefaction has been examined by [18], to improve their combustion properties for solid fuel development and applications. Corncob samples have been characterized using ASTM standards, and their mass and energy yield (EY) has been calculated using a standard procedure. Also, a thermogravimetric analyser has been employed to investigate thermal degradation of the samples. The authors have reported that thermal decomposition of corncob has begun around 388.6 °C. The most significant weight loss has been found at 600 °C. TCW obtained at 260 °C, for 60 min, as exhibited best combustion characteristics.

In another study, [10] have carried out a quintessential characterisation of CW to investigate their potential for energy applications. Some reported properties were MC, VMC, FCC, ash content (AC), UA and TGA. Based on results, it has been reported that properties of corncob make it a favourable biomass source that could be used for energy applications. However, it has been highlighted that high percentage of AC may create some technical challenges that may lower thermal efficiency. The study has concluded that using CW seems possible and commercially viable to convert abundantly available domestic agriculture biomass into what could be considered as alternative fuels. A review of relevant studies in literature [10, 18, 19] has revealed that there are few works tailored towards pelletization of agro-waste, and in particular, there is no known published or near absent research on combined torrefaction and pelletization and torrefaction affect thermal characteristics and quality of pelletized CW.

Methodology

RC for this study has been provided by the University of Ilorin farm, while starch has been supplied by a cassava processing plant in Ilorin, Kwara State, Nigeria. The samples were held in the University of Ilorin's woodworking laboratory.

Material preparation and torrefaction

Handpicking technique was used to sift corncob samples that had been preserved. This involved removing any superfluous components and plant remnants. Corncobs were then oven-dried, based on ASTM D2444-16 [20, 21], at a temperature of 105 °C, for 2 h, to eliminate surface water interference. Materials were crushed and sieved to minimize particle size (PS), while increasing workability and compactness. Samples were screened into three different sizes of 0.3, 0.5 and 1.0 mm, for better workability. Sieved materials were torrefied in a muffle furnace for 30 min, at 280 °C. N gas was first released into the torrefying chamber and then passed through at a rate of 100 mL per min, to create and maintain an inert environment. Zip-lock polythene bags were used to keep RC and TCW samples apart during torrefaction process. Afterward, 20.5 g cassava starch were dissolved in 60 cm³ cold water, and gelatinized on a gas cooker, by continuously stirring the solution, until a slurry of smooth paste was formed.

Material characterization

AC, FCC and VMC present in the samples were estimated performing PA of RC and TCW samples. Based on ASTM D-3175-18 [22], VMC was evaluated by holding the dried samples collected in a closed crucible, at 925 \pm 20 °C, for seven min, in OMEGALUX LMF-3550 muffle furnace (after MC analysis of RC samples). According to ASTM D-3174-12 [23], residual samples acquired after VMC assessment were gradually heated to 700 \pm 50 °C, for 30 min, in the muffle

furnace. FCC was calculated by subtracting values for MC, VM and AC from 100, as shown in Eq. (1).

$$FCC = 100 - MC - VM - AC$$
(1)

UA of the samples was completed according to ASTM D-3176-15 [24], to ascertain their C, H, O, N and S contents, which were determined using Eqs. (2-4), respectively.

$$C = 0.37 FCC + 0.455 VM$$
(2)

$$H = 0.052 FCC + 0.455 VM$$
(3)

$$O = 0.304 FCC + 0.476 VM$$
 (4)

All reported values in both analyses were determined on a dry basis.

Pelletization procedure

Weighing process for samples of RC and TCW (Fig. 1) was done using a digital scale. A binder equal to 5% of each sample's total mass was applied [4]. A stirrer was used to manually mix aggregates. Then, feedstock was poured into the mold. Ingredients for manufacturing of pellets were compacted using EL31-072 hydraulic compression machine. 50, 75 and 100 MPa and compaction pressure (CP) were taken into consideration. Produced pellets were taken out of the molds, weighed and measured, before being air dried.



Figure 1: Samples of produced TCW pellets.

Physical properties

Density

The pellets' density was calculated using ASTM D2395-17 [24], by dividing their masses by corresponding volumes. As soon as produced pellet samples were removed from the mould, green density (GD) of RC and TCW pellets was assessed. Then, 30 days after pelletization procedure, relaxed density (RD) was determined. The test was carried out in triplicate, and the average of each value was determined. After pellets were expelled, their mass and geometry were determined by employing a digital

weighing balance and Vernier calliper, respectively. Compressed density (ρ) of pellets was estimated using Eq. (5).

$$\rho = \frac{m}{v} \tag{5}$$

where m denotes mass of the pellet and v is volume. Mass was measured using a digital scale. Volume was calculated by measuring diameter, height and central whole diameter of pellets at various intervals, using Vernier calliper. Volume of the pellets was computed using Eq. (6).

$$v = \pi h r^2 \tag{6}$$

where h and r denotes pellet's height and radius, respectively.

Relaxation ratio

Relaxation ratio, often defined as quotient of compressed density (ρ_c) to RD (ρ_r) of the pellet, was employed to compute pellet's relaxation ratio, using Eq. (7) [4]:

$$R = \frac{\rho_c}{\rho_r} \tag{7}$$

Drop-to-fracture

Drop-to-fracture (D) test of the pellets was carried out based on ASTM D440-86 [25], to determine their toughness during handling, storage and transportation. The procedure comprised weighing the pellet, recording its initial mass, and then dropping the sample four times onto a flat steel plate from a height of 1.85 m. Percentage, expressed as quotient of the pellet's final weight (W_2) retained after four drops to its original weight (W_1), was computed using Eq. (8) [26]:

$$D = \frac{W_1}{W_2} \times 100\% \tag{8}$$

Moisture absorption

MA test was determined using established procedure adopted by [19], in which pellet samples were dried in BST/HAO-1122 hot air oven, at a temperature of 105 °C, for 24 h. Thereafter, they were kept in DM-115 humidity chamber, for five h, at 30 °C, with 90% humidified air, to investigate their hydrophobic properties. Pellet samples' mass was then weighed using the digital balance, and recorded.

Thermal properties

Thermal properties of RC and TCW samples, such as CV, SY and EY, were determined using standard methods.

Calorific value

Parr 6400 O bomb calorimeter was used to determine the pellet's energy content or CV of both RC and TCW samples, based on ASTM D5865-13 [27]. 1 g of the

pelletized sample was placed in the crucible (i.e., sample holder), then transferred from the bomb calorimeter to a steel capsule.

Solid yield

SY was determined using established procedure adopted by [28], in which an initial mass of CW was weighed and fed into the torrefaction chamber. Afterwards, total mass of the product left was weighed and documented. SY of the TCW sample was determined using Eq. (9).

$$Y_{solid} = \frac{m_p}{m_f} \times 100 \tag{9}$$

where Y_{solid} is SY, and m_p and m_f are mass of products and feed, respectively.

Energy density

Energy density (E_d), a quotient of HHV of TCW to HHV of RC, was computed using Eq. (10) [28]:

$$E_d = \frac{HHV_{torrefied}}{HHV_{raw}} \tag{10}$$

Energy yield

EY was determined from the product of SY and energy density, as shown in Eq. (11).

$$Y_{eneroy} = Ysolid \times E_d \tag{11}$$

where Y_{energy} is EY and Y_{solid} is SY.

Results and discussion

Thermogravimetric analysis

Fig. 2 (a and b) depicts TGA/DTA of RC and TCW samples. The entire process engenders four distinct evolutional temperature regimes, ranging from below 170 to above 700 °C.



Figure 2: (a) TG and (b) DTG for RC and TCW samples.

Removal of moisture that takes place at a temperature below 170 °C corresponds to first stage. Fig. 1 depicted that only 5% and 3% mass losses were observed for RC and TCW samples, respectively, under this temperature [2]. Slight reduction in percentage mass loss that was exhibited by TCW samples could be due to loss of surface water (i.e. molecules of water held by weak forces) and elimination of light VMC that had already taken place during torrefaction processes [10]. Second stage falls within temperature range from 250 to 700 °C, which is main decomposition of RC and TCW samples, due to devolatilization and decomposition. During this stage, which can be subdivided into two parts, namely, 250 to 460 °C and above 500 °C, cellulose, hemicellulose contents of RC and TCW samples decomposed, and at 500 °C and above, lignin contents were thermally degraded. At temperatures above 700 °C, final products of the two samples were AC. This explain the constant trend until the end, which corresponds to the third stage.

Proximate analysis

Table 1 shows PA results of RC and TCW. When compared to TCW, RC exhibited a higher MC. This was caused by the surface and binding water removal from biomass due to torrefaction. In the early stage of heating, water produced by chemical reactions was also eliminated using a procedure known as thermo-condensation. Moreover, recorded MC value for TCW was 1.3%, which compares favourably with 1.33% reported for coal by [8]. Furthermore, VMC and FCC values of TCW samples met *Deutsches Institut für Normung* 51731 standards [29], and also fell within an acceptable range for values from hybrid fuel of 97% lean-grade coal [14]. As compared to coal, TCW had almost the same percentage of AC. The fraction of AC could be attributed to the large proportion of inorganic solid waste in torrefied biomass. Also, studies have revealed a higher amount of ash in lignocellulose torrefaction. This suggests that if equivalent amounts of their pellets are burnt, it may be possible to remove more ash from TCW than from RC. High AC feedstock may also make users feel unease while operating, according to [30].

Properties		RC			TCW	
PS (mm)	0.3	0.5	1.0	0.3	0.5	1.0
MC	7.30	8.00	9.30	1.00	1.30	1.35
VMC (%)	78.00	77.00	71.00	47.00	45.00	42.00
AC (%)	1.70	1.40	1.00	4.00	3.70	3.05
FCC (%)	13.00	13.60	18.70	48.00	50.00	53.60

Table 1: PA of RC and TCW.

Table 2 presents elemental composition of RC and TCW samples. It can be seen that TCW samples exhibited an increase in CC across the board for all PS. TCW had a maximum C percentage of 54% for 1.0 mm PS, which compared favourably with CC found in coal [17]. When compared to the value recorded for RC within the same series, it was discovered that O content of TCW was much lower for all PS.

Sample	PS (mm)	С	Н	0
	0.3	47.87	5.93	43.82
RC	0.5	48.23	5.91	43.67
	1.0	49.11	5.9	43.2
	0.3	52.33	5.46	37.35
TCW	0.5	53.03	5.42	37.29
	1.0	54.00	5.40	36.85

Table 2: Elemental composition of RC and TCW.

Impact of torrefaction is visible, as there was a remarkable difference in percentage C and O content of RC and TCW samples. This is because torrefaction leads to the release of water vapour and CO₂ [17]. Additionally, a modest reduction in H content was seen, on both RC and TCW pellets, for all PS. H content was found to decrease as PS increased from 0.3 to 1.0 mm. This may be due to hydrocarbons generated during torrefaction processes, such as butane and methane [28]. Torrefaction is also responsible for hydrocarbons release, which ultimately reduces net H content [17].

Compressed density

Fig. 3 depicts effects of variations in CP and PS on compressed density of produced fuel pellets.



Figure 3: Effect of variation in PS and CP on the compressed density of RC and TCW pellets.

For both pellets, GD ranged from 760 to 1,250 and 637 to 920 kg/m³, respectively. RC pellets, with CP of 100 MPa and 0.3 mm PS, had the highest GD, while TCW, with 1.0 mm PS and 50 MPa CP, had the lowest one. However, it was observed that the density of some TCW pellets showed a similar trend to what was reported

previously by [16], with the ratio for the blend of 97:3 of lean-grade coal to torrefied biomass. TCW pellets were found to have a lower density than RC pellets produced under the same conditions. One possible result of pre-treatment for torrefaction is that treated TCW has a lower density than that from untreated RC. Another reason for TCW pellets' low density could be because water that was chemically bonded and VMC were lost during densification, which would have strengthened binding activity. Additionally, disintegration of hemicelluloses during torrefaction makes it difficult to plasticize, and has a significant frictional resistance, requiring more energy to compact compared to leftovers of RC [31]. Compressed density increased with more CP but decreased with higher PS. According to [32], when pelletization is performed under the same conditions, finer grinds result in a higher compressive density than large particles. This is because finer grinds require a higher level of conditioning, since they have a larger surface area and can more easily absorb moisture than large particles.

Relaxed density

Fig. 4 depicts the variation in PS and CP effects on RD of produced pellets. It shows how RD of fuel pellets was affected by PS and CP.



Figure 4: Effects of variation in PS and CP on the RD of RC and TCW pellets.

The generated pellets' relaxed densities ranged from 616 to 800 and 700 to 876 kg/m³, respectively. For both RC and TCW pellets of CW, the pellet with 0.3 mm PS and 100 MPa had the highest relaxed densities. However, it was found that RD of RC pellets was approaching densities of TCW at 0.5 mm PS, which may be because RC pellets' weight was reduced due to moisture and certain light VMC being lost during curing [33]. However, relaxed densities of RC and TCW pellets decreased with larger PS and increased with CP. These findings compared favourably with those of similar research on RC pellets reported by [34]. The possibility of elastic-spring-back and expansion of pellets experienced by RC samples had been eliminated through torrefaction, making TCW pellets more stable [25].

Relaxation ratio

Fig. 5 illustrates how changes in CP and PS affect the resulting pellets' relaxation ratio.



Figure 5: Effects of variation in PS and CP on relaxation ratio of RC and TCW pellets.

For RC and TCW pellets, relaxation ratio ranged from 1.08 to 1.43 and 1.03 to 1.17, respectively. Torrefied pellet with PS of 0.3 mm and CP of 100 MPa had highest relaxation ratio, and torrefied pellet with PS of 1.0 mm and CP of 50 MPa had the lowest. It was found that, for both pellets, relaxation ratio varied directly with CP, and inversely with the PS. According to analysis of results from this study's relaxation ratio, pellets generated at low CP are more stable than pellets produced at higher CP, which implies those produced at low CP could have stable density [19].

Drop-to-fracture test

Fig. 6 depicts effects of variation in PS and CP on drop-to-fracture test of produced pellets, which translates to the degree of durability. For pellets made from RC and TCW materials, drop-to-fracture ranged from 60.01 to 97.80 and 34.0 to 80.0, respectively. RC pellets showed higher durability values than those from TCW. RC pellet with 0.3 mm PS and CP of 100 MPa recorded highest drop-to-fracture value. The lower value was recorded by TCW pellet, with 1.0 mm PS and CP of 50 MPa. With increasing CP and decreasing PS, pellets' durability improves. This may be because smaller PS have stronger intermolecular bonds, since there is less pore space between them, which increases adhesive forces between particles, improving their interlocking and bonding, and increasing durability. The degree of particle interlocking affects how much CP increases. TCW were less durable than RC pellets, under the same conditions. Linkages between individual particles in TCW pellets may become weaker due to the modification of hydroxyl groups in MC, and hemicellulose content during the process [34-36].



Figure 6: Effects of variation in PS and CP on percentage drop-to-fracture of RC and TCW pellets.

Moisture absorption

Effects of variation in PS and CP on MA of generated fuel pellets are depicted in Fig. 7.



Figure 7: Effects of variation in PS and CP on the percentage MA of RC and TCW pellets.

MA is an index by which the degree of pellets' shelf life can be predicted, since the ones that absorb moisture during storage are more prone to damage than hydrophobic

ones. For RC and TCW pellets, MA ranged from 17.2 to 19.4 and 2.2 to 3.7 wt.%, respectively. TCW pellets had a water absorption capacity that was equivalent to 80% reduction in MA. This may be due to TCW's somewhat different structure and composition [35].

According to [19], emission of hydrophilic hydroxyl groups from raw biomass, and increase in fraction of hydrophobic CC in torrefied biomass were due to changes in their moisture uptake. Additionally, [25] have speculated that breakdown of hydroxyl groups and lignin coating on torrefied biomass particles were the main causes of their observed lower water uptake rate. Percentage MA of RC and TCW pellets increased with larger PS and decreased with stronger CP. The findings of this study compared favourably with those of similar works [19].

Calorific value

Fig. 8 depicts profiles of CV for produced RC and TCW pellets, of which HHV ranged from 16 to 18.8 and 24 to 25.8 MJ/kg, respectively. It can be shown that RC and TCW heating value improved as their PS increased. TCW sample with the highest value (i.e., 25.8 MJ/kg) was 1.0 mm PS, which was almost identical to CV reported for coal (i.e., 24.6 MJ/kg) [17]. Reduction in MC due to torrefaction processes may be related to a rise in heating value [19]. Average improvement after torrefaction pre-treatment was 37.2%. As a result, these findings demonstrate that torrefaction affects the increase in CV of TCW. Enhanced energy density seen in TCW samples was also explained by a high proportion of lignin, and a lower fraction of hemicellulose and cellulose, with low heating values. Results of this investigation agree with findings from [18], which have revealed that RC and TCW have energy values of 18 and 26.8 MJ/kg, respectively. Similarly, [17] has reported energy density of 24.79 MJ/kg for TCW.



Figure 8: CV of samples.

Solid yield

As shown in Table 3, SY of 56.1 g (58.4%) was obtained after 96 g initial mass from CW was fed into the torrefaction chamber. According to [37], the decrease in mass yield upon torrefaction can be associated with loss of moisture and organic VMC by TCW. It was further explained that loss of water predominantly occurred during torrefaction process, when organic matter of biomass got dehydrated. Also, a reduction in mass yield could be associated with decomposition of hemicellulose and cellulose content from CW. SY of 45.4% wt for TCW, at 573 K, after 30 min, has been reported by [28]. Disparity in findings reported by [28] and the value obtained in this study can be due to the difference in torrefaction temperature and HT that the samples were subjected to. Thus, higher operating torrefaction temperature and HT results in relatively low SY [37].

Energy yield

EY of TCW is shown in Table 3 for various PS. It can be observed that an increase in PS led to a slightly higher EY. EY of 79.4% and 79% were seen at PS of 0.5 and 0.3 mm, respectively, while 80% EY was seen at 1.0 mm PS. PS only slightly affects EY, with larger ones having a higher energy output [38], due to their consistent heat transfer properties. However, if torrefaction processes are compromised, desired attributes may change. Biomass with a higher lignin content will possibly produce higher energy values. According to [37], cellulose and hemicellulose contents of biomass were almost eliminated during torrefaction, which resulted in observed differences in energy output of the samples.

Sieve size (mm)	HHV of RC (MJ/Kg)	HHV of TWC (MJ/Kg)	М _Ү (%)	EY (%)	E _d
0.3	16.9	23.0	58.4	79.0	1.36
0.5	17.6	24.1	58.4	79.4	1.37
1.0	18.8	25.6	58.4	80.0	1.37

 Table 3: Energy values of samples.

Conclusions

The study evaluated the effect of process parameters on CW processed by pelletization. Some selected physical and thermal properties of RC and TCW pellets, such as PA, GD, RD, relaxation ratio, durability, MA capacity, CV, SY and EY were characterized using standard test methods. Torrefaction had a significant effect on all other properties of the pellets. Observations of results on PA revealed differences between TCW and RC samples. For TCW samples, FCC increased, while MC decreased for all considered samples. Higher FCC in TCW samples also showed that they will emit greater CV when charred. VMC was modified and disparities in obtained values within sieve sizes were low. PS, CP and their combined effect affected properties of pellets produced from RC and TCW. HHV from RC and TCW samples improved from 16.9 to 18.8 MJ/kg and 23 to 25.6 MJ/kg, as PS increased

from 0.3 to 1.0 mm, respectively. Maximum HHV was seen for a TCW sample of 1.0 mm PS. Thus, both RC and TCW pellets, regardless of size, are suitable for use in energy systems, since their HHV are comparable to those of lignin coal, which is within the range from 10 to 20 MJ/kg. HHV of 1.0 mm TCW was 29% higher than that from lignin coal.

Author's contributions

N. S. Saidu, M. A. Alododo: conceptualization; investigation; reviewing and editing. H. A. Ajimotokan, A. A. Adeleke: investigation; methodology; writing of original draft. T. A. Orhadahwe, P. P. Ikubanni: research design; data analysis; reviewing and editing. J. K. Odusote, M. S. Lawal: conceptualization; data curation; reviewing and editing; project administration. All authors have read and agreed to the published version of the manuscript.

Abbreviations

AC: ash content **ASTM:** American Society for Testing and Materials **CC**: carbon content CO₂: carbon dioxide **CP**: compaction pressure CV: calorific value CW: corncob waste EY: energy yield FCC: fixed carbon content **GD**: green density HHV: higher heating value HT: holding time MA: moisture absorption MC: moisture content **PA**: proximate analysis **PS**: particle size RC: raw corncob **RD**: relaxed density **SY**: solid yield TCW: torrefied corncob waste **TGA**: thermogravimetric analysis UA: ultimate analysis VMC: volatile mater content

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