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Physical and mechanical characteristics of composite briquette from coal and pretreated wood fines

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Abstract Melina wood torrefied at 260 °C for 60 min was agglomerated with lean grade coal fines into composite briquettes using pitch as binder. Torrefied biomass (3%–20%) and coal fines (80%–97%) were blended together to produce the composite briquettes under a hydraulic press (28 MPa). The briquettes were cured at 300 °C. Density, water resistance, drop to fracture, impact resistance, and cold crushing strength were evaluated for the composite briquettes. The proximate, ultimate, and calorific value analyses were carried out according to different ASTM standards. Microstructural studies were carried out using scanning electron microscope and electron probe microanalyzer equipped with energy dispersive x-ray. Fourier Transform Infrared Spectrophotometer (FTIR) was used to obtain the functional groups in the raw materials and briquettes. The density of the composite briquettes ranged from 0.92 to 1.31 g/cm^3 after curing. Briquettes with $< 10\%$ torrefied biomass has good water resistance index ($> 95\%$). The highest cold crushing strength of 4 MPa was obtained for briquettes produced from 97% coal fines and 3% torrefied biomass. The highest drop to fracture (54 times/2 m) and impact resistance index (1350) were obtained for the sample produced from 97% coal and 3% torrefied biomass. The fixed and elemental carbons of the briquettes showed a mild improvement compared to the raw coal. The peaks from FTIR spectra for the briquettes shows the presence of aromatic C=C bonds and phenolic OH group. The composite briquettes with up to 20% torrefied biomass can all be useful as fuel for various applications.

Keywords Composite briquettes · Lean grade coal · Torrefied biomass · Physico-mechanical properties · Combustion properties

1 Introduction

Coal fines are by-products that are inevitably produced when lump lean grade coal is being processed, transported or handled (Adeleke et al. 2019a). Coal fines are small in particle size (often less than 3 mm) making it not to meet the requirements of numerous industrial production processes (Adeleke et al. 2019b). On the other hand, biomass has been considered as waste and discarded in an unhealthy way, which in turn causes environmental pollution (Adeleke et al. 2019c). Whereas, these materials can be effectively used for several purposes. Previous research works on the use of coal fines and biomass wastes have shown tremendous potentials in using these materials for the production of industrial and domestic fuels (Odusote et al. 2019; Zhong et al. 2016). However, there are several

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limitations to the use of these energy materials. For instance, the use of coal has attracted huge criticism in respect to the various release of greenhouse gases into the atmosphere; thereby causing environmental pollutions (Mousa et al. 2016; Lázaro et al. 2007; Basu et al. 2011; Agbor et al. 2014). Biomass has low density and high moisture than coal (Adeleke et al. 2020a) and this makes its combustion without any pretreatment not favorable (Lasode et al. 2014). Several pretreatment techniques such as torrefaction (Bach et al. 2017), carbonization (Basu 2013) and pyrolysis (Basu 2010) have been employed to upgrade biomass. These methods have been proven useful in improving the energy content of biomass (Adeleke et al. 2020b). Adeleke et al. (2019a) reported that fuel in form of briquettes could be developed from coal fines and upgraded biomass. It has been established that with appropriate briquetting parameters and conditions such as appropriate curing conditions (Mollah et al. 2015), binder contents (Adeleke et al. 2019b) and so on, briquettes with good physico-mechanical properties can be produced (Mollah et al. 2016a, b).

Zhong et al. (2017) formed coal briquette from high volatile coal fines using molasses and coal tar pitch as binder. Briquettes with good mechanical integrity in terms of compressive strength (13.06 MPa) and drop to fracture (56.6 times/2 m) were developed and reported useful as feedstock for COREX iron making process. Zhong et al. (2016) used xylene activated coal tar pitch as binding agent to produce briquettes with high mechanical integrity. Marganingrim and Estiasty (2020) also produced bio-coal briquettes from rejected coal and biomass. It was reported that an increase in the biomass content within the briquettes lowered the emission of greenhouse gases such as NO_x, CO, and H_2S. However, the mechanical properties of the briquettes were poor. Handayani et al. (2019) studied the effect of carbonization on the characteristics of bio-coal briquettes. It was similarly reported that the briquettes were with minimal CO (395 mg/Nm^3) and NO (5.6 mg/Nm^3) gases emission. Yuliansyah et al. (2019) produced and characterized bio-coal briquettes from pyrolyzed bio-coal blends with biomass up to 87.5% of the briquette composition. It was reported that more biomass content within the briquettes led to short ignition time. It also caused a significant reduction in CO and CO_2 emissions. Mursito and Widodo (2020) also characterized bio-coal briquettes blended produced from low quality coal and biomass waste treated with Garant® bio-activator. The addition of activated biomass was observed to lower the sulfur content in the low quality coal used. There is a revived interest in developing the energy generation sector of most developing nations while maximizing the grade of coal available for use. Thus, the need for the present study. Similarly, there is limited study on the physicomechanical characteristic of briquettes produced from lean grade coal fines and torrefied woody biomass (melina). Thus, the present study focuses on the use of up to 20% torrefied biomass aggregated with lean grade coal fines in developing composite briquettes with good physical and mechanical properties.

2 Materials and methods

The production procedure for the hybrid fuel briquettes is illustrated in Fig. 1, mixing of coal fines with torrefied biomass and then blended with pitch. It was then briquetted and cured. The key twin properties of fuel briquettes (physico-mechanical and combustion properties) were then measured.

2.1 Materials

2.1.1 Lean grade coal fines

Lean grade coal fines obtained from the Okaba mines (Nigeria: 7° 23′ 0″ N, 7° 44′ 0″ E) were used for the production of composite briquettes in the present study. The coal fines (< 3 mm) were sun-dried, pulverized, and screened to particle size below 0.70 mm (Adeleke et al. 2019d). It was further oven-dried at 105 °C for 30 min to remove moisture before blending it with torrefied biomass. The properties of the lean grade coal are given in Table 1.

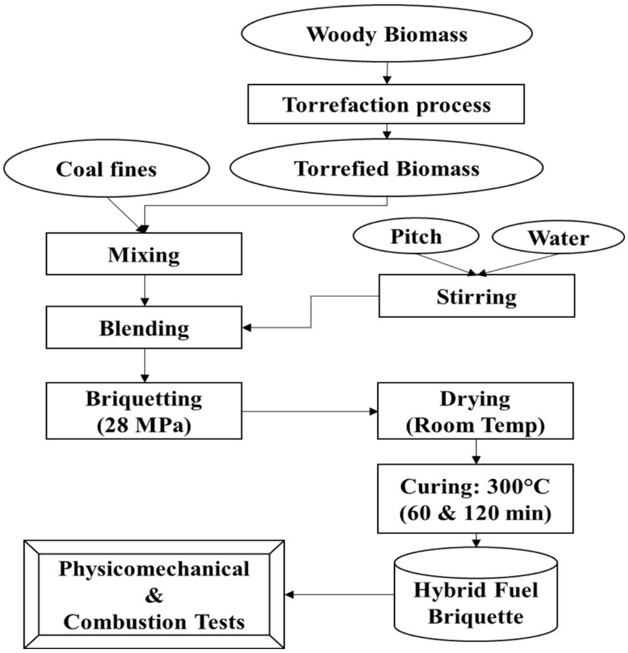


Fig. 1 Schematic for the hybrid fuel briquette production layout

Table 1 Characteristics of the lean grade coal

Sample	Proximate (wt% dry basis)				Ultimate (wt% dry basis)					HHV (MJ/kg)
	MC	VM	AC	FC	C	H	N	S	O	
LGC	1.37	13.71	18.00	64.92	71.47	2.88	0.90	0.71	24.04	24.20
Pitch	0.18	73.99	0.95	24.88	89.17	7.43	0.30	0.50	2.60	39.73
Melina	7.52	81.42	2.15	8.92	47.09	6.65	0.38	0.19	43.54	18.54
TM	2.63	54.07	2.17	41.08	66.08	5.18	0.30	0.20	26.30	23.45

TM, torrefied biomass; LGC, lean grade coal; MC, moisture content; VM, volatile matter; AC, ash content; FC, fixed carbon content; C, carbon; H, hydrogen; N, nitrogen; S, sulphur; O, oxygen; HHV, higher heating value

2.1.2 *Torrefaction of melina*

Melina (*Gmelina arborea*) lump obtained from Benin city, Nigeria (Nigeria: 6° 20′ 17.34″ N, 5° 37′ 32.70″ E) was used in this study. It was converted into chips and fines below 6.35 mm using a Saw wood cutting machine (Model No: CS 33 EB) and further pulverized into particle size less than 2 mm using a Laboratory Mill (Thomas Wiley Model 4). The pulverized sample was sun-dried for five days (5 h/d) and then subjected to torrefaction process. Pulverized sample of biomass (< 2 mm) was placed in a Tubular furnace set-up at 260 °C with an inert environment, which was achieved by a continuous flow of 2 L/min of nitrogen into the furnace at a resident time of 60 min (Adeleke et al. 2019a; Odusote et al. 2019). The torrefied biomass obtained after the process was then grounded and sieved to particle size below 0.70 mm. The properties of the raw and torrefied biomass are shown in Table 1 (Adeleke et al. 2019b).

2.1.3 *Pitch*

Pitch is a by-product of the destructive distillation of coke or char. It contains some complex mixture of poly-aromatic (Holuszko et al. 2017). Pitch was used directly at a particle size less than 0.70 mm. The proximate, ultimate and higher heating values analyses of the pitch which was previously reported by Adeleke et al. (2019b) are shown in Table 1.

2.2 Briquette formulation and blending

The entire process was carried out based on the schematic in Fig. 1. The raw materials used were in accordance with the formulation in Table 2. Coal fines were varied between 80% and 97% of the 25 g while torrefied biomass was varied between 3% and 20%. However, pitch and water were used at 10% each of the total weight of the briquette. Water was added to the pitch binder and then stirred mechanically to obtain homogeneity. The coal and torrefied biomass fines were also mixed separately. The samples were then blended together to obtain homogeneity. The mixture was then poured into a 25 mm internal diameter cylindrical steel die and compressed under a hydraulic press with a load of 28 MPa. The pressure was reduced gradually so that the hybrid fuel briquettes could be ejected from the mold.

Table 2 Composition of the hybrid fuel briquette

Hybrid ratio	Coal (%)	Torrefied biomass (%)
97:3	97	3
95:5	95	5
90:10	90	10
85:15	85	15
80:20	80	20

2.3 Curing of the hybrid fuel briquette

The green briquettes were initially dried at room temperature for 24–36 h for initial moisture removal. The sample was then cured in an inert environment orchestrated by a flow of nitrogen (0.5 L/min) into a Tubular furnace set to 300 °C for resident time of 60 and 120 min. After the protective curing, the sample was removed from the furnace, cooled to room temperature in a desiccator and then tested.

2.4 Thermogravimetric and combustion analyses

The thermal degradation behaviors of the lean grade coal fines and biomass were observed with a Thermobalance system (Model No: STA7300). Approximately 6.5 mg of test sample was loaded into the crucible for analysis in the equipment. The experiment was carried out in an inert environment obtained by continuous flow of 100 mL/min of nitrogen. Biomass samples were heated from 30 to 800 °C at different heating rates of 5 and 10 °C/min, while coal was heated up to 1200 °C at 5 and 10 °C/min. Proximate (moisture, ash, volatile matter, and fixed carbon contents), ultimate (carbon, hydrogen, nitrogen, sulfur, and oxygen), and calorific value analyses of the coal fines, raw and torrefied biomass, and crushed briquettes were carried out based on IS: 1350-1 (1984), ASTM D5373 (2016) and ASTM D5865-04 (2004) standards, respectively. The calorific value was measured using an Oxygen Bomb Calorimeter (Model No: A1290DDEE).

2.5 Physico-mechanical properties of briquettes

The physico-mechanical properties of the composite briquettes evaluated were density, water resistance, drop to fracture, impact resistance, and cold crushing strength (compressive strength). The densities of the composite briquettes were calculated using the ratio of mass (M) to volume (V) for each briquette, as shown in Eq. (1). The volume of the briquette was obtained from the measured height and diameter. Richard's (1990) method was modified in this study to evaluate the resistance of the composite briquettes to absorption of water and disintegration (WRI). Briquette with weight (W_1) was immersed in a cylindrical glass containing 220 mL distilled water at (30 ± 2) °C for 30 min. It was then removed and cleaned to eliminate water on its surface and reweighed (W_2). The relative change in weight of the composite briquettes were measured and percentage water absorbed was calculated using Eq. (2) and WRI (%) was then estimated using Eq. (3).

Cold crushing strength (CCS) (compressive strength) was carried out using a universal mechanical testing machine (10 kN Hounsfield apparatus) located at National Metallurgical Laboratory, Jamshedpur, India. The machine was operated in a compression mode as applicable for coke and briquettes (Zhong et al. 2016; Wang et al. 2013; Lumadue et al. 2012). The maximum crushing load (M_f) that the briquettes can withstand before cracking or breaking was recorded and repeated in duplicates for each sample. The average was then used to calculate cold crushing strength in accordance with Eq. (4). "D" represents the bottom circular diameter of the composite briquettes. Drop to fracture test entailed dropping three different samples of the composite briquettes separately from stationary elevation of 2 m until it breaks. The number of times per 2 m (times/2 m) taken for it to breaks was used to appraise the drop resistance. Impact resistance index (IRI) was then calculated from the drop to fracture test using Eq. (5).

$$\rho = \frac{M}{V} \tag{1}$$

$$\text{Water absorbed } (\%) = \frac{W_2 - W_1}{W_1} \times 100 \tag{2}$$

$$\text{WRI } (\%) = 100 - \%\text{ water gained} \tag{3}$$

$$\text{CCS} = \frac{4M_\text{f}}{\pi D^2} \tag{4}$$

$$\text{IRI} = \frac{100 \times \text{Average number of drops/2 meters}}{\text{Average No. of pieces}} \tag{5}$$

2.6 Microstructural studies, elemental mapping and FTIR spectrometry

The microstructural and elemental studies of the best composite briquettes based on the physico-mechanical properties analyses were carried out using scanning electron microscope (Nova Nano SEM 430) and electron probe microanalyzer equipped with EDX (energy dispersive x-ray spectroscopy-JEOL 8230 model). Fourier Transform Infrared spectrometry (FTIR) was used to obtain the functional groups present in the lean grade coal, pitch, torrefied biomass and some briquette samples (samples that cured at 300 °C for 60 min). An approximately 2 mg of samples with dry KBr (potassium bromide) powder, approximately 300 mg, were weighed into an agate mortar for pulverization and mixed until the samples dispersed into each other. The mixture was shaped by pressing into a transparent disc for 10 min using a tablet machine. The discs were analyzed by FTIR and the spectra were recorded in the range of 4000–400 cm^{-1} at a resolution of 4 cm^{-1}.

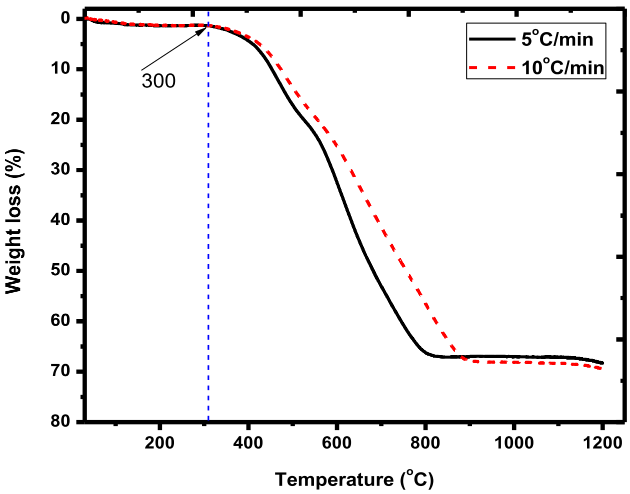


Fig. 2 Thermogravimetric curves of the lean grade coal at 5 and 10 °C/min heating rates

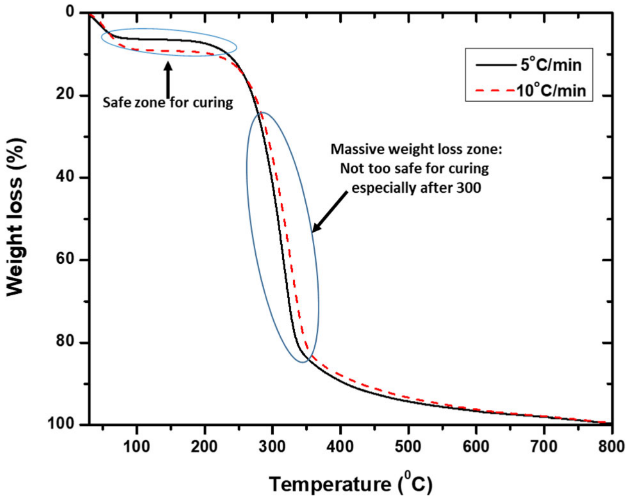


Fig. 3 Thermogravimetric curves of melina wood at 5 and 10 °C/min heating rates

3 Results and discussion

3.1 Thermogravimetric analyses

The thermogravimetric curves of the lean grade coal, which show the weight loss pattern in nitrogen environment at different heating rates, are presented in Fig. 2. The weight loss pattern of the lean grade coal is similar to that of melina (Fig. 3). However, massive weight loss terminated at around 800–900 °C, which reflected a typical characteristic of a lean grade non-coking coal (Speight 2012). Figure 2 shows that coal begins to have massive weight loss around 318 °C. This implied that exposure of coal to a temperature higher than 318 °C will bring about massive volatile loss. Thus, to produce briquettes from the lean grade coal, lower temperature range where the weight loss will be limited to bounded moisture and light volatile losses must be employed during its curing. Figures 2 and 3 serve as the basis for which curing conditions for the composite briquettes were set. To have a briquette with good mechanical integrity and a stable or improved combustion properties (ultimate, proximate and calorific value) from lean grade coal, curing in the neighborhood of 300 °C under a protected environment that is deficient of oxygen was needed (Adeleke et al. 2019a). Figure 3 also shows the thermogravimetric curves of the melina wood at 5 and 10 °C/min heating rates. After the initial low mass loss, which predominantly have been affirmed to be moisture loss, there was a slow weight loss zone which was nearly linear (Ren et al. 2013). The zone was identify as safe zone in this study for curing because the weight loss was less than 10%, which indicated that only light volatile matter (energy-lean content of hemicellulose) along with moisture have been released from the melina wood (Adeleke et al. 2019d). Beyond this zone, a further loss and degradation of energy-rich contents of cellulose and lignin compositions of the melina wood begins massively (Odusote et al. 2019; Nhuchhen et al. 2014). Therefore, curing of the blend of lean grade coal and torrefied biomass was carried out at 300 °C to avoid massive devolatilization, which in turns may weaken the physico-mechanical attributes of the briquettes.

3.2 Physico-mechanical properties

3.2.1 Density

The apparent green or initial densities (ID) and the densities after curing for the briquette samples are shown in Fig. 4. The green densities of the briquettes were in the range of 1.18 to 1.31 g/cm^3. It could be observed from Fig. 4 that as the percentage of torrefied biomass increases, the green density of the briquettes reduces. The density of the cured briquettes also varied from 0.93 to 1.31 g/cm^3 for the 300 °C–60 min samples and 0.92 to 1.26 g/cm^3 for the 300 °C–120 min samples. It is evident in the results that the curing process lowered the density of the briquettes. This may be due to weight loss during curing at 300 °C, which may not affect the dimensional variations of the briquettes. This is even supported by the fact that the more severe the curing, the higher the loss of unbounded moisture, light volatiles, and some degree of bounded moisture (Mollah et al. 2016a, b). The density of the sample produced in this studies showed a similar trend to what was reported previously by Adeleke et al. (2019d) with the highest value obtained for the blend of 97:3 of lean grade coal to torrefied biomass. Samples with 20% torrefied biomass have the lowest density. The density of a briquette has been reported to affect transportation, handling, and its combustion (Richard 1990; Mitchual et al. 2013; Ikubanni et al. 2019). Making briquette more dense impairs its combustion properties (Mitchual et al. 2013). Based on the report of Richard (1990) which has been widely referenced since there is no standard value for various properties of briquettes, most of the briquettes after curing surpassed the 1.25–1.30 g/cm^3 recommended for briquettes of strong quality. This is acceptable for both domestic and industrial applications especially as feedstock for energy generation and direct reduced iron making process.

3.2.2 Water resistance

Water resistance index (WRI), shown in Fig. 5, can be used to infer the capacity of the briquettes against absorption of water and disintegrations during handling, storage, and transportation. The higher the WRI, the better the resistance against water absorption and disintegrations

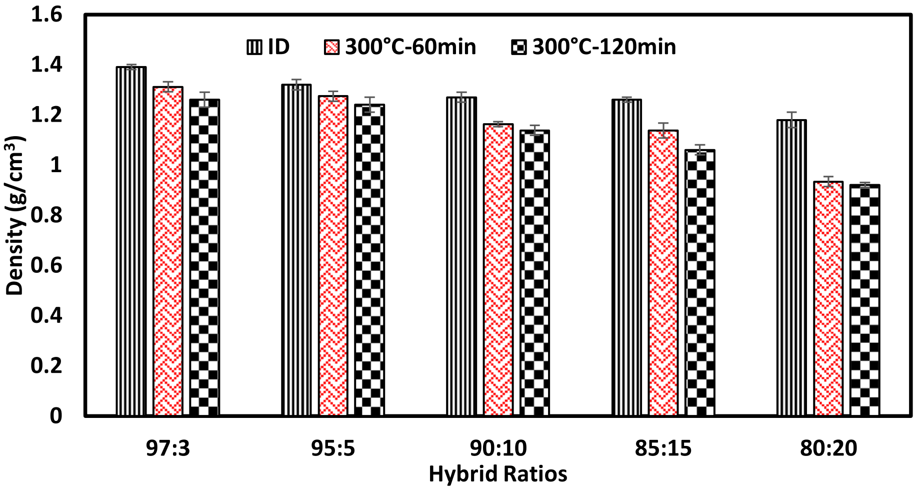


Fig. 4 The density of the briquette based on different hybrid ratios (green and cured)

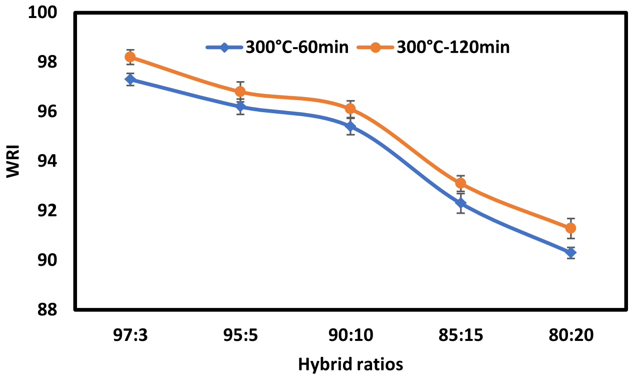


Fig. 5 Water resistance indices for the briquettes of various hybrid ratios

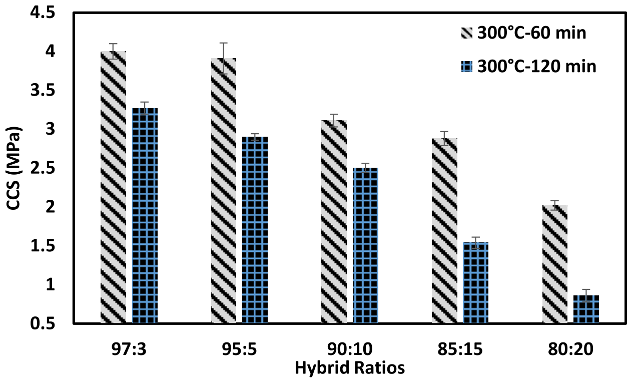


Fig. 6 The cold crushing strength (CCS) of the briquettes of various hybrid ratios

(Adeleke et al. 2019b; Richard 1990). This is important because the briquettes were produced with pitch binder and torrefied biomass, which to a certain degree still have the tendencies to attract moisture and water. The WRI decreased with increase in torrefied biomass content within the briquettes. However, a further exposure to curing beyond 60 min led to a mild improvement in the WRI, as shown in Fig. 5. This may be due to the more hydrophobic properties that was impacted on the briquettes (Zhong et al. 2017; Benk and Coban 2011). The WRI varied from 90.3% to 97.3% for samples cured at 300 °C for 60 min and 91.28% to 98.2% for those cured at 300 °C for 120 min. Meanwhile, samples with $\geq$ 15% torrefied biomass contents were below the 95%, which has been widely accepted as minimum requirement for briquettes with water resistant capacity especially those produced from water-sensitive materials. By implication, briquettes with $\geq$ 15% torrefied biomass contents absorbed more water and are highly susceptible to disintegration during handling, storage, and transportation. Torrefied biomass (up to 10%) can be partially used to replace lean grade coal fines in producing composite briquettes with good water resistance indices (95.4%–98.2%). Thus, briquettes fit for domestic and industrial applications based on WRI have been produced from the hybrid of coal and torrefied biomass in this study.

3.2.3 *Cold crushing strength*

Figure 6 represents the cold crushing strength (CCS) for the briquettes of various hybrid ratios and curing conditions. Cold crushing strength is often refer to as compressive strength (Adeleke et al. 2019b) and it represents the maximum crushing load a briquette can withstand before cracking or breaking. The CCS ranged from 2.02 to 4.00 MPa for those cured at 300 °C for 60 min and 0.86 to 3.27 MPa for briquettes cured at 300 °C for 120 min. It is obvious that the CCS reduced with increase in torrefied biomass and the trend is similar to what was obtained for the WRI of the briquettes (Fig. 6). Continued devolatilization of torrefied biomass, which was far higher than the lean grade coal, may be responsible for such a notable drop in CCS (Mollah et al. 2016a, b). The devolatilization process can imminently create cracks, which are potential sites for the initiation of breaking failure or crushing under compressive loading in trucks, bins or conveyor belt during transfer (Nieto-Delgado et al. 2014). Compared to the previous study of Adeleke et al. (2019b) where the influence of variation in binder types and percentage constituent on physico-mechanical properties was carried out, the produced composite briquettes from varying torrefied biomass contents have lower CCS. However, the whole set of briquettes produced surpassed the 350 kPa suggested by Richard (1990) as benchmark for briquettes of industrial and domestic applications. Noteworthy is the integration of larger percentage of torrefied biomass into lean grade coal fines for the production of the briquettes. Thus, this implied lower environmental pollutions and better biomass waste management.

3.2.4 Drop to fracture and IRI

Two of the key parameters that serve as general diagnostic for briquette strength are drop to fracture and impact resistance. Impact resistance is measured using the impact resistance index (IRI) and the IRI of the briquettes produced in this study are shown in Fig. 7. A high IRI indicates a high impact resistance (Richard 1990). The drop to fracture of the samples decreased with an increase in torrefied biomass content (a similar trend obtained for CCS and WRI). The number of drop from a stationary point to breakage varies from 6 to 54 times/2 m. The highest value was obtained from the sample exposed to 300 °C for 60 min curing condition. Against what was reported for the CCS and IRI by Adeleke et al. (2019b) for briquettes produced with blended binder of pitch and molasses, the CCS, WRI, drop to fracture, and IRI followed a similar trend for all the samples. The IRI of the briquettes varied from 250 to 1350 for samples cured at 300 °C for 60 min and 150 to 1175 for those cured at 300 °C for 120 min. The present results surpassed the IRI of 50 that was benchmarked for briquettes of industrial application (Richard 1990). The IRI values of the briquettes produced in the present study are higher than what was obtained for starch-bonded briquettes (25) produced from 97% coal and 3% torrefied biomass reported by Adeleke et al. (2019b). Blesa et al. (2003) reported a distinct IRI of 1000 for smokeless briquettes produced from low ranked coal whereas better results in terms of 1175 and 1350 were obtained in this study (Fig. 7). Generally, with torrefied biomass increased to 20%, the IRI of all the briquettes were extremely higher than required (50). Thus, the briquettes are very good feedstock useful domestically and industrially for thermal generation or metallurgical processes.

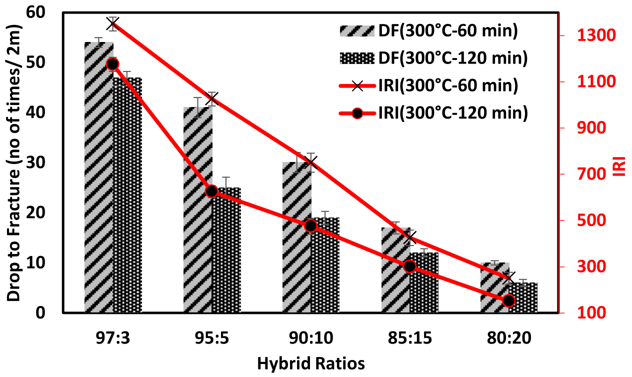


Fig. 7 The drop to fracture and impact resistance index for the briquettes

3.2.5 Proximate, ultimate and calorific value analyses

For effective utilization of the composite briquette, the proximate, ultimate, and calorific value results are essentially important as the physico-mechanical properties. Thus, the physico-mechanical properties and combustion characteristics are considered as Siamese twins. If a briquette have compacted strength and integrity but poor in combustion essence, then it is considered as poor fuel (Adeleke et al. 2019b; Chou et al. 2009). The moisture content (MC), volatile matter (VM), ash content and fixed carbon (FC) for the composite briquettes at different curing conditions are shown in Table 3. The MC varies from 2.16% to 2.53%. A mild reduction was observed as the curing time increased. The VM increased with about 1% compared to the raw coal (Table 1). A little increment in ash content was obtained in the briquettes compared to the raw coal. The increase in VM and ash are due to the presence of torrefied biomass and pitch in the composite fuel. A major parameter that indicates how efficient coal or

Table 3 Proximate contents of the briquettes

Curing conditions	Hybrid ratios	MC (%)	VM (%)	Ash (%)	FC (%)
300 °C–60 min	97:3	2.45	14.21	18.21	65.13
	95:5	2.48	14.23	18.20	65.09
	90:10	2.50	14.26	18.19	65.05
	85:15	2.51	14.27	18.18	65.04
	80:20	2.53	14.30	18.16	65.01
300 °C–120 min	97:3	2.00	14.00	18.21	65.79
	95:5	2.08	14.23	18.20	65.49
	90:10	2.10	14.25	18.19	65.46
	85:15	2.12	14.32	18.18	65.38
	80:20	2.16	14.43	18.16	65.25

Table 4 Ultimate analyses and caloric values of the briquettes

Curing conditions	Hybrid ratios	C (%)	H (%)	N (%)	S (%)	O (%)	CV (MJ/kg)
300 °C for 60 min	97:3	74.65	2.48	0.93	0.73	21.21	25.96
	95:5	74.56	2.50	0.92	0.73	21.29	26.01
	90:10	74.58	2.52	0.92	0.72	21.26	26.18
	85:15	75.54	2.53	0.92	0.72	20.29	26.22
	80:20	75.54	2.57	0.91	0.72	20.26	26.30
300 °C for 120 min	97:3	74.94	2.42	0.91	0.73	21.00	26.01
	95:5	75.00	2.45	0.92	0.73	20.90	26.11
	90:10	75.10	2.47	0.91	0.72	20.80	26.22
	85:15	75.37	2.50	0.92	0.72	20.49	26.26
	80:20	75.54	2.52	0.91	0.72	20.31	26.85

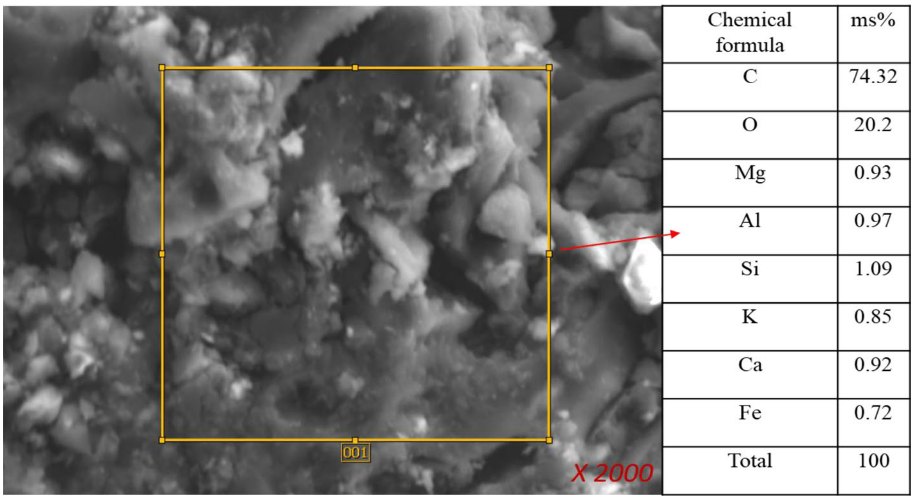
any other solid fuel is for energy generation or metallurgical application is the FC (Abnisa et al. 2013). The FC increased by about 1% compared to the raw lean grade coal. There are briquetting processes especially those with inorganic binders that impairs the proximate content of briquette fuel (Altun et al. 2003). However, the use of pitch and torrefied biomass with lean grade coal fines led to a better fuel in terms of FC. This, in turn, influenced the calorific value of the composite briquettes as shown in Table 4. Though an improvement with more curing time was observed, yet it was still abysmal. In similar trend, the variation in the ultimate contents (C, H, N, S, and O) of the composite briquettes were minimal as shown in Table 4. The carbon content ranges from 74.56% to 75.94% which is a little higher than the 71.47% of the lean grade coal fines (Table 1). The N and S contents were nearly constant for all the composite briquettes. The O and H contents were reduced with curing time and this is predominantly traceable to loss of unbounded and bounded moisture. Thermal cracking which may lead to liberation of low molecular weight compounds in coal, torrefied biomass and pitch may also be responsible for the pattern of results obtained for the ultimate contents (Adeleke et al. 2020a, b; Yip et al. 2007). However, the briquetting process does not impair these characteristics but rather mildly improve them to be useful fuel for energy and metallurgical applications.

3.3 Microstructure and elemental mapping

The morphology of the 97:3 hybrid ratio that was cured at 300 °C for 60 min is presented in Fig. 8. There are structures of granular surface, irregular arrangements and charging effects, which were previously observed for a briquettes of high volatile coal (Zhong et al. 2017). This homogenous and closely packed granular surface may be oxygen bridges, which were generated in the curing process by coal, torrefied biomass and pitch (Wang and Bai 2014; Adeleke et al. 2019b; Zhong et al. 2017). The bulk element analysis of the area map (001) shown in Fig. 8 also confirmed the ultimate analysis showing that carbon in the entire area is 74.32%. It also confirmed that C and O dominated the composite briquette fuel. Thus, a need to do the C and O mapping of the briquette, which are shown in Fig. 9. The spatial distribution of carbon and oxygen showed that both elements are enriched and distribute evenly on the entire surface. This implied that the C and O are not entangled at a corner or a segment of the briquette. This will improve the combustion of the briquettes when in use.

3.4 Analyses of briquette samples using FTIR

The FTIR spectra of the lean grade coal, pitch and torrefied melina (Mw) used for the production of the briquettes are presented in Fig. 10a while Fig. 10b represents the FTIR spectra of briquette samples cured at 300 °C for 60 min. The peaks in spectra range of 3300–3700 cm^{-1} usually connote the stretch vibration of phenolic hydroxyl group as identified by Li and Zhu (2014) and Speight (1994). Torrefied biomass possess this peak at 3342 cm^{-1}. Meanwhile, this peak appear insignificantly in the lean grade coal and is absolutely missing in the pitch. This implied that the lean grade coal contains lower proportion of phenolic hydroxyl group. This spectrum is obvious in the briquettes as the torrefied biomass increased within the samples. There is a huge tendency for weaker bond formation due to the phenolic hydroxyl group, which revealed why the physicomechanical integrity of the briquettes slightly diminishes as the torrefied biomass increased within the briquettes. The presence of deformation vibration of C–H in aliphatic carbon ($-CH_3$) and $-C(CH_3)_2$ appears in the range of 2800–2975 cm^{-1} and 1375 cm^{-1} in pitch and torrefied biomass (Fig. 10a). The relative intensity of these peaks decreased or nearly disappeared in the briquette samples as shown in Fig. 10b. This may be because of their liberation during the curing process. The spectra around 1400–1600 cm^{-1} observed in torrefied biomass and pitch is not prominent in the lean grade coal. These spectra range are attributed to the presence of aromatic (C=C) in biomass and pitch by Speight (1994). The spectra range appears in the briquettes though not vehement in 97:3 mixing ratio. However, the more condensed aromatic ring (C=C) at 1000–1300 cm^{-1} was dominant in the 97:3 mixing ratio compared to other briquettes (Zhong et al. 2017). The briquette samples revealed the presence of aromatic C=C bonds and phenolic OH groups which were established along with oxygen bridges as strengtheners for briquettes



Chemical formula	ms%
C	74.32
O	20.2
Mg	0.93
Al	0.97
Si	1.09
K	0.85
Ca	0.92
Fe	0.72
Total	100

Fig. 8 SEM of the 97:3 hybrid ratio (cured at 300 °C for 60 min)

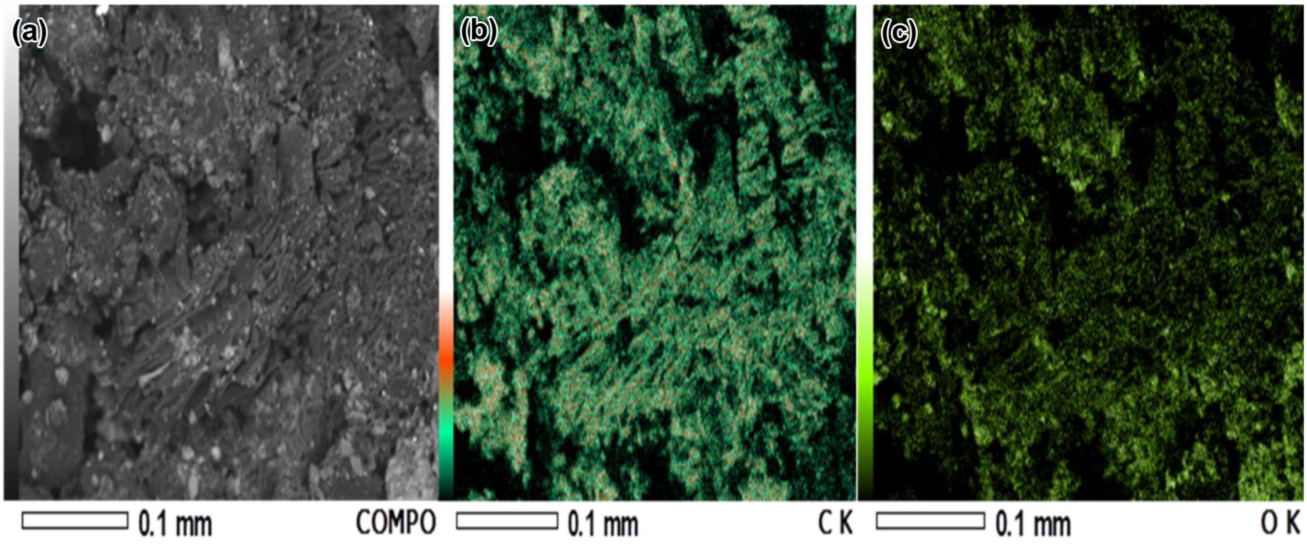


Fig. 9 Major elemental mapping (carbon and oxygen) for the major constituent of 97:3 hybrid ratio (cured at cured at 300 °C for 60 min)

when exposed to curing in inert environment (Zhong et al. 2017).

4 Conclusions

The physicomechanical properties and combustion characteristics of produced composite briquettes from coal and pretreated melina wood fines has been studied. Curing at 300 °C is good for composite briquette. Temperature in the range of 100–300 °C are as well safe zone for curing especially when scaling up considering better energy consumption. The composite briquettes with $\leq$ 10% torrefied biomass have water resistance index greater than 95% which met the suggested benchmark. The increment in torrefied biomass within the briquette lowers its cold crushing strength. The highest drop to fracture (54 times/2 m) and impact resistance index (1350) were obtained for the best briquettes. The calorific values of the composite briquettes have minimal variations (approx. 26 MJ/kg). The fixed and elemental carbons were improved by briquetting process by about 3%. Carbon and oxygen are the predominant elements in good spatial distribution within the composite briquettes. The composite briquettes are very good feedstock for energy and metallurgical applications.

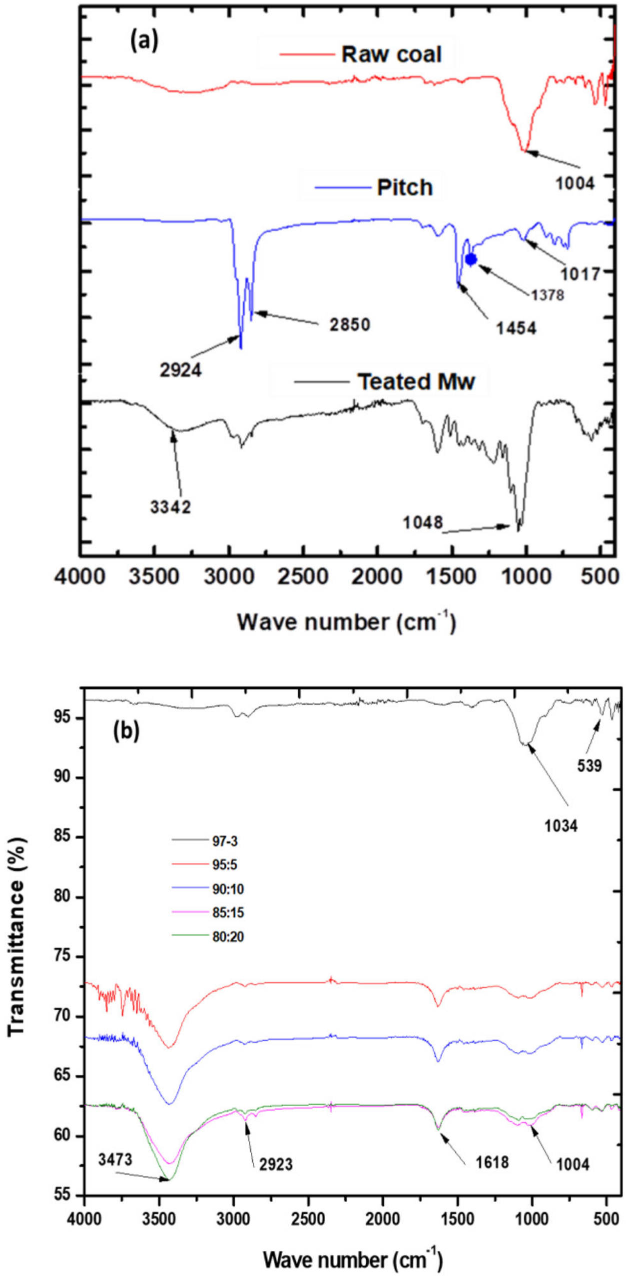


Fig. 10 FTIR spectra **a** raw materials **b** briquettes from different mixing ratio carbonized at 300°C for 60 min

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Declarations

Conflict of interest The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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