Effects of operating parameters on maize COB briquette quality

Article *in* Biomass and Bioenergy · April 2018

DOI: 10.1016/j.biombioe.2018.02.015

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EFFECTS OF OPERATING PARAMETERS ON MAIZE COB BRIQUETTE QUALITY

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Abstract

Briquetting is considered as one of the pre-treatment methods available for producing uniform sized and moisture content feedstock which is easy to handle, transport and store. The quality of briquettes in terms of density and durability depends on the physical and chemical properties of the feedstock and briquetting conditions. In this study, the effect of compacting pressure, temperature, moisture content, and particle size on the properties of briquettes for thermochemical applications were investigated. It was found that density, impact resistance, and compressive strength significantly increased with increasing compacting temperature (20-80 ^oC) and compacting pressure (150-250 MPa). However, increasing moisture content and particle size had a negative impact on briquette quality. The results showed that there was a strong interaction between briquetting parameters with the interaction between moisture and temperature significantly affecting both briquette density and mechanical strength. Briquettes with high density and durability/mechanical strength required to meet quality certification standards could be obtained with course ground material ($<$4mm) from relatively low moisture content feedstock (7-8%) with pressure of 200-250MPa and a compacting temperature of 80^{o}C.

Keywords: Briquette quality, density, impact resistance, compressive strength, maize cob, agricultural residues

1 Introduction

Biomass for energy generation has attracted much attention because it is an abundant resource [1] and CO_2 neutral [2, 3]. According to the World Energy Council [4], biomass contributes 14% out of the 18% of global energy supply from renewables and contributes 10% of total global energy consumption. It is the predominant source of energy in developing countries e.g. over 80% in sub-Saharan Africa, which is mainly used for cooking [5]. Biomass is heterogeneous in terms of size, shape and composition and has low bulk density (e.g. about 4 times lower than the bulk density of diesel) [6], leading to difficulties in handling, storage and transport. Densification of biomass into briquettes/pellets increases bulk density from 40-200 kgm^{-3} to 450-800 kgm^{-3} [5, 7] and produces a high energy feedstock with uniform moisture, shape and size which makes it suitable for storage and transportation with potential uses in combustion, pyrolysis and gasification [8]. Densification minimises particulate emissions per unit solid fuel transported and improves biomass combustion efficiency as well as conveyance efficiencies (less dust and wastage and lower labour cost) in commercial energy generation facilities [9, 10]. The classification of briquettes and pellets is commonly based on their sizes e.g. 4.0-10.0 mm diameter and 20-50mm length according to the respective Austrian (ONORM M 7135) and German (DIN 51731) quality standards for pellets [11, 12] with 10 - 200 mm diameter and 16 - 400 mm length commonly used for briquettes [13-16].

Due to the increase in the share of renewable energy required to achieve national government targets, the demand for densified products increased from 7 to 19 million tonnes for the period 2006–2012 [17]. However, shortage of feedstock and sustainability of supply for wood pellet production provides a major challenge especially in the rapidly growing EU pellet market with an urgent need to broaden the feedstock range by using agricultural residues and other sources of biomass e.g. municipal solid waste. Briquetting can be preferred over pelleting for agricultural residues because it can accommodate feedstock with large particle sizes and high moisture content [18], which in turn reduces the energy input in pre-processing of feedstock (grinding and drying). It was reported [19] that the energy required for grinding corn stover decreased 3 fold when increasing particle size from 0.8 mm to 3.2 mm at a moisture content of 6-12%.

In transport, handling and storage briquettes with high density and mechanical strength are desirable [20]. High density is desired to reduce transport and storage costs [21-23], with high compressive strength, i.e. $\geq$2.56 MPa [24] preferred to prevent breakages [25]. Durability of over 80 % [26] is reported to ensure briquettes/pellets remain intact during transport/storage and reduce the amount of fine particles/dust produced [7]. Ensuring moisture content of feedstocks between 5-22 % has been reported to facilitate stable compaction of several feedstocks such as wood, alfalfa, lignite, wheat straw and waste paper [20, 22, 27, 28].

Particle size for producing briquettes can be varied from 0.1 to 6 mm depending on type of feedstock [29-34]. However, Ahmed et al [35] also reported that particle size of 6-8 mm together with 13-15% in powder form was recommended to enhance briquette durability by increasing interlockages and minimising spaces between particles [7]. Although pellets have been studied intensively with certified quality standards available (e.g. Austrian ONORM M 7135, Swedish SS 187120, German DIN 51731 and DIN EN 15270 and European Standard Committee CEN/TE 335), very little work has been done on briquetting of agricultural residues and the only standards available for briquettes are for wood. Pellet standards therefore have often been used to determine agricultural residue briquette quality. Previous studies [21,24,30,36] showed that briquette properties were strongly dependent upon moisture content, particle size, temperature, compacting pressure and type of feedstock. However, the findings are case-specific and the results are variable. Increasing compacting pressure for mango and eucalyptus leaf [21] from 30 to 100 MPa increased the density from 600 to 1100kgm^{-3}. Similarly increasing pressure from 3 to 11MPa increased the density of palm oil mill residues from 950 to 1010 kgm^{-3} [24]. Density of tropical hard wood briquettes decreased when particle size was increased from $<$1mm to 2-3.35mm, however, there was a weak positive correlation between compressive strength and particle size [36]. The effect of moisture content varies depending on feedstock such that impact resistance (as measured by shatter index) of paper mill briquette increased from 36227 to 168875 when moisture content was increased from 5 % to 15 % and maximum compressive strength of 1299 kgcm^{-2} was reported at a moisture content of 9 % [30].

To date, interactions between different briquetting parameters (compacting pressure, moisture content, particle size and compacting temperature) on properties of briquettes have not been studied. Therefore, fully understanding how chemical composition and physical properties impact upon briquette product quality is essential. The literature shows that low pressures (5-31 MPa) [9, 34], used in the compaction of maize residues resulted in the production of low density ($<$ 1000 kgm^{-3}) briquettes which did not meet the German Standard DIN 51731 (1-1.4 gcm^{-3}). Kaliyan and Morey [33] produced maize cob briquettes at a pressure of 150 MPa and reported that density and durability were significantly affected by, moisture content (10 and 20 %), pre-heating temperature (25 and 85 ^{o}C) and particle size (mean particle diameter 0.85 and 2.81 mm), however, the impact of pressure and the interactions between briquetting parameters were not analysed. In this study, the effect of briquetting conditions (pressure, moisture, particle size and temperature) and their interactions on the properties of maize cob briquettes was investigated. The findings from this study have clear potential globally as maize is one of the major crops grown globally but particularly in sub-Saharan Africa regions where a large amount (~7 million tonnes) produced annually [37] are either burnt in open air (without heat recovery) or are dumped to decompose in uncontrollable ways. Converting residue cobs into energy would not only contribute to reducing greenhouse gas emissions but also to a more sustainable waste management strategy.

2 Materials and Methods

2.1 Material

Maize cobs were kindly provided by Barfoots of Botley Ltd, UK. Maize (supersweet varieties) was harvested at Stage R3 (milk stage) of maturity in Senegal, Morocco, United State of America, South Africa, Greece, Germany, United Kingdom, France and Spain and stored at 0-5ºC for 1-25 days. Waste cobs were sent to Newcastle University and stored in a cold room at 6ºC prior to briquetting. As a result, this work was performed on substrates of unknown provenance, for which the chain of custody is not known. Hence, the maize cultivars cannot be specified. While the authors believe that this work exemplifies the briquetting performance of cobs, there are reasonable concerns that there may be growth/substrate/chain of custody factors that have influenced the results obtained, and given the early stage of harvest (R3), the results may not be directly extrapolated to corn cobs that are harvested after the plant reaches full maturity (R6) and the bract leaves have turned brown. Residue maize cobs were cut into pieces < 5 mm and oven dried at 105ºC for 2-8 hours to obtain a range of moisture contents. All moisture contents presented in this paper are on a % wet basis. Dried maize cobs were crushed using a HGBTWTS3 laboratory blender 8010ES and separated using 2.36 and 4.00 mm sieves to study the effects of particle size.

2.2 Briquette preparation

A machine fabricated with a hollow cylindrical mould, internal diameter of 2 cm and length 12.5 cm was adapted from the work of Zafari and Kianmehr [28]. The mould was fitted inside two 150W band heaters connected to a temperature controller and was insulated with Fortaglas for operator safety and to reduce heat loss.

About 7g of ground maize cob was fed inside the mould and then manually compressed using a 10 tonne Hydraulic Bench Press (Clarke CSA10BB). A dwell time (i.e. duration for which particles under compression remain under maximum compacting pressure during briquetting) of 20s was chosen for all experiments to minimise briquette relaxation [21, 29] which could have negative impacts on briquette properties (density, impact resistance and compressive strength). The effects of temperature (20-80^{o}C), moisture content, (7-17%) particle size ($<$ 2.36 mm and $<$4.00 mm) and pressure (150, 200, 250MPa i.e. within the range of pressures used for briquetting several biomass materials [23, 38, 39]) and their interactions were studied using a 2-level factorial design of experiment. Briquettes were stored in an air tight container at room temperature (approximately 20^{o}C) for 7 days to allow stabilisation [40] prior to analysis of their properties (density, impact resistance and compressive strength).

2.3 Briquette characterisation

Moisture, ash, volatile matter and fixed carbon content of maize cobs and briquettes were determined according to ASTM D3173, ASTM D3174, ASTM D3175 and ASTM D3172 standards respectively. Ultimate analysis was carried out using a Carlo Erba 1108 Elemental Analyser to determine percentage of carbon, hydrogen and nitrogen. High heating value (HHV) was determined using a CAL2K ECO bomb calorimeter. Scanning electron microscopy (SEM) analysis was carried out using a TM3030Hitachi Microscope. Differential Scanning Calorimetry (DSC) analysis was carried out using a DSC Q20 model to identify the range of compacting temperatures to be used in the briquetting experiments. Analysis of neutral detergent fibre (NDF) was carried out by enzymatic gravimetry, while acid detergent lignin (ADL) and acid detergent fibre (ADF) were analysed using an Ankom 220 analyser. The composition of cellulose, hemicellulose and lignin were subsequently determined [41]:

$$\text{Cellulose} = \text{Neutral detergent fibre (NDF)} - \text{Acid detergent lignin (ADL)} \quad (1)$$

$$\text{Hemicellulose} = \text{Neutral detergent fibre (NDF)} - \text{Acid detergent fibre (ADF)} \quad (2)$$

$$\text{Lignin} = \text{Acid detergent lignin (ADL)} \quad (3)$$

Density was determined using the stereometric method which allows briquettes to be used for thermo-chemical applications to remain dry [42]. Height and diameter of a briquette was measured using a digital vernier calliper (error: $\pm$ 0.005 mm) to determine volume. For impact resistance, a briquette was released 4 times from a height of 1.85 m to fall freely under gravity onto a metallic plate to determine impact resistance [43]. Percentage residual weight of briquettes was determined after each drop. The remaining piece with the highest weight was taken as the residue and used for the next drop. Impact resistance was defined as the percentage residual weight after the 4th drop. Compressive strength was determined via both the cleft and simple pressure tests using a Tinius Olsen H50KS compressing machine. Briquettes were placed between two flat parallel surfaces with surface area greater than the briquette. Briquettes were placed horizontally for the cleft test and vertically for the simple pressure test. An increasing load was then applied to compress briquettes at a rate of 1 mm/min until the briquette failed/cracked. The ultimate load at the point where the briquette cracks, F was used to calculate the compressive strength using Equations (4) and (5). An average of 3 measurements for each test were carried out.

Compressive strength $\sigma = F/A$ (4)

Compressive strength, $\sigma = F / l$ (5)

Where A and l are the cross-sectional area (m^2) and length (m) of briquettes.

The physical and mechanical properties of briquettes such as density, impact resistance and compressive strength are presented as mean values of at least 6 samples/briquettes. Minitab 17 statistical software was used to analyse the impact of the variables and their interactions on density, impact resistance and compressive strength of briquettes. Statistical analysis was carried out at a significance level of $\alpha = 0.05$.

3 Results and Discussion

3.1 Characteristics of maize cobs

Fresh maize cobs used in this study had high moisture content (73.9 $\pm$ 0.74%), which is much higher than in other work e.g. 30.3 % [44]. The high moisture content is likely due to the use of fresh maize cobs which were harvested at early stage of maturity (R3 i.e. milk stage) and also stored at 0-5^{o}C prior to analysis. They cannot be used directly for briquetting according to European Standard Committee CEN/TC 335 for solid fuels as the moisture content in briquettes is required to be 5-15%. In addition, high moisture feedstock/products are prone to fungal decomposition during transportation and storage [27] and poor combustion properties such as low heat output, low combustion temperature, and long fuel residence time in the combustion chamber [17]. Therefore, these fresh maize cobs must be dried/partly dried prior to being briquetted. Maize cob (Table 1) had high volatiles (~76%) and low ash content (3.2%), which agreed well with other work [45, 46]. Fresh maize cobs had a similar high heating value to that of woody materials and anthracite.

Differential scanning calorimetry analysis was carried out to identify the range of compacting temperatures to be used in the briquetting experiments. An endothermic peak was observed at 100.9 ^{o}C associated with a loss of moisture, but no transition steps were observed (Fig 1). The non-visibility of the glass transition temperature could be due to interference from the moisture endothermic peak [47], as the glass transition step is likely to overlap with the moisture endothermic peak area.

Table 1: Properties of fresh maize cobs (dry basis)

Property	Mazie cob
Proximate analysis	
Ash (%wt)	3.2 (± 0.03)
Volatiles (%wt)	76.1 (± 0.70)
Fixed carbon (%wt)	20.7 (± 0.70)
Ultimate analysis	
C (%)	46.9 (± 0.01)
H (%)	8.1 (± 0.39)
N (%)	2.8 (± 0.06)
O (%) by difference	42.2 (± 0.33)
High heating value (HHV) (MJ/kg)	18.9 (± 0.07)

A maximum compacting temperature of 80^{o}C was therefore chosen for this study based on the glass transition temperature of 79.2^{o}C identified for corn stover [48]. Furthermore, compacting at high temperatures i.e. ≥100^{o}C is undesirable because it not only requires high energy input which in turn reduces energy efficiency but also reduces compressive strength of briquettes due to the evaporation of water which makes them brittle [49]. A certain amount of moisture is required to reduce friction between particles and the mould during compaction and to enhance the force of attraction between particles [27].

Two exothermic peaks at 283.78^{o}C and 337.73^{o}C observed in the DSC thermo-gram (Fig.1) could be due to the decomposition of hemi-cellulose, cellulose, and lignin [50]. The lignin, cellulose and hemicellulose composition identified in this study were 1.5%, 47.1 % and 29.4% respectively with the remaining 22.0% likely to be extractives (e.g. protein, starch, oil and sugar). A low lignin content in this study compared to much higher levels (3-15 %) observed by other researchers, [33, 41, 51, 52] could be due to the analysis method used in this study of which the acid detergent lignin (ADL) only gives a partial value of total lignin content [33].

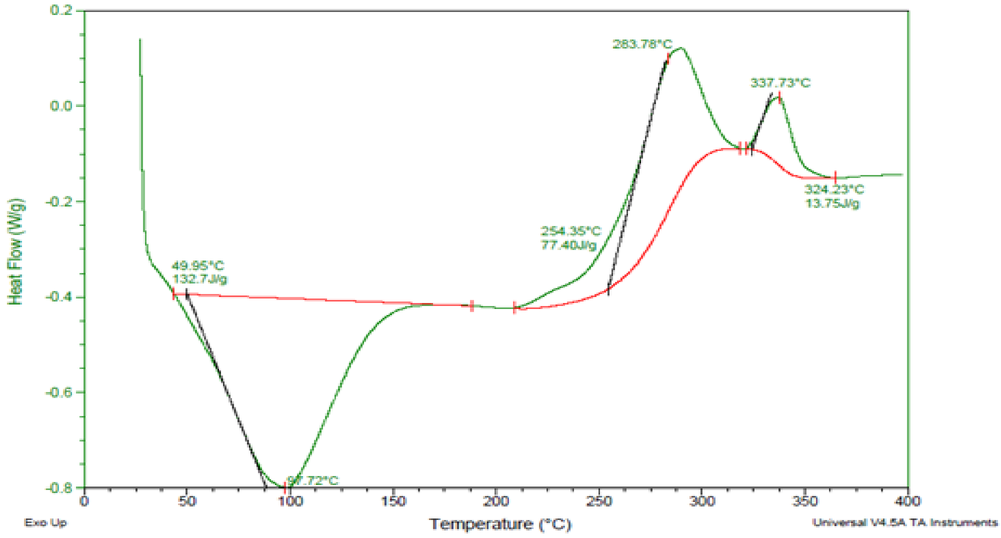


Fig 1: Differential scanning calorimetry (DSC) thermo-gram of maize cobs at 7.14 % moisture content

3.2 Density

Briquette density ranged between 516 kgm^{-3} and 1058.2 kgm^{-3} from variations in briquetting parameters used in this study. The lowest density of 516 kgm^{-3} was obtained with a low temperature (20 ^{o}C), a high moisture content (16.94 %) and a particle size <4.0 mm, the density of all other treatment combinations being >700 kgm^{-3}. With the exception of where a high compacting pressure (250 MPa), small particle size (<2.36 mm) and a high temperature (80 ^{o}C) were used, all briquettes produced from the high moisture content of 16.94 % had a density less than 1000 kgm^{-3} (Fig 5) which falls below the range of 1-1.4 gcm^{-3} required to meet the German Standard DIN 51731. Highest density briquettes (1054.4-1058.2 kgm^{-3}) were produced from particle size of <2.36 mm, moisture content of 7.14 % and pressure of 200-250 MPa Under these conditions density remained relatively constant likely due to a reduction in original void spaces between particles and an increase in inter-particle bonding at high pressures i.e. > 200 MPa. This trend is consistent with results reported for briquettes from palm oil mill residues [24] and pine [32]. Density increased with increasing compacting pressure and temperature but decreased with increasing particle size and moisture content (Fig 2a). Moisture content and pressure were the predominant factors affecting briquette density. However, Zhang and Guo [38] found that particle size (0.16-5 mm) and moisture content of 5-17 % were the predominant factors that affected density of caragana korshinskii kom briquettes within a range of compacting temperatures of 70-150 ^{o}C and compacting pressure of 10-170 MPa. Rhén et al [53] reported that density of spruce pellet was predominantly affected by moisture content (6.3-14.7 %) and compacting tempearture (26-144 ^{o}C) for particle size of <3.15 mm and compacting pressure of 46-114 MPa. A similar observation was reported [54] on density of olive tree pruning residue pellets produced from various particle size ranges <1 mm to <4 mm, moisture content of 5-20 %, compacting temperature of 60-150 ^{o}C and pressure of 71-176 MPa. Variable results for factors affecting briquette density are likely due to variation in feedstock properties in addition to which many of the comparative studies have mainly focused on the effects of single factors rather than looking at the interaction among them.

(a)

(b)

(c)

(d)

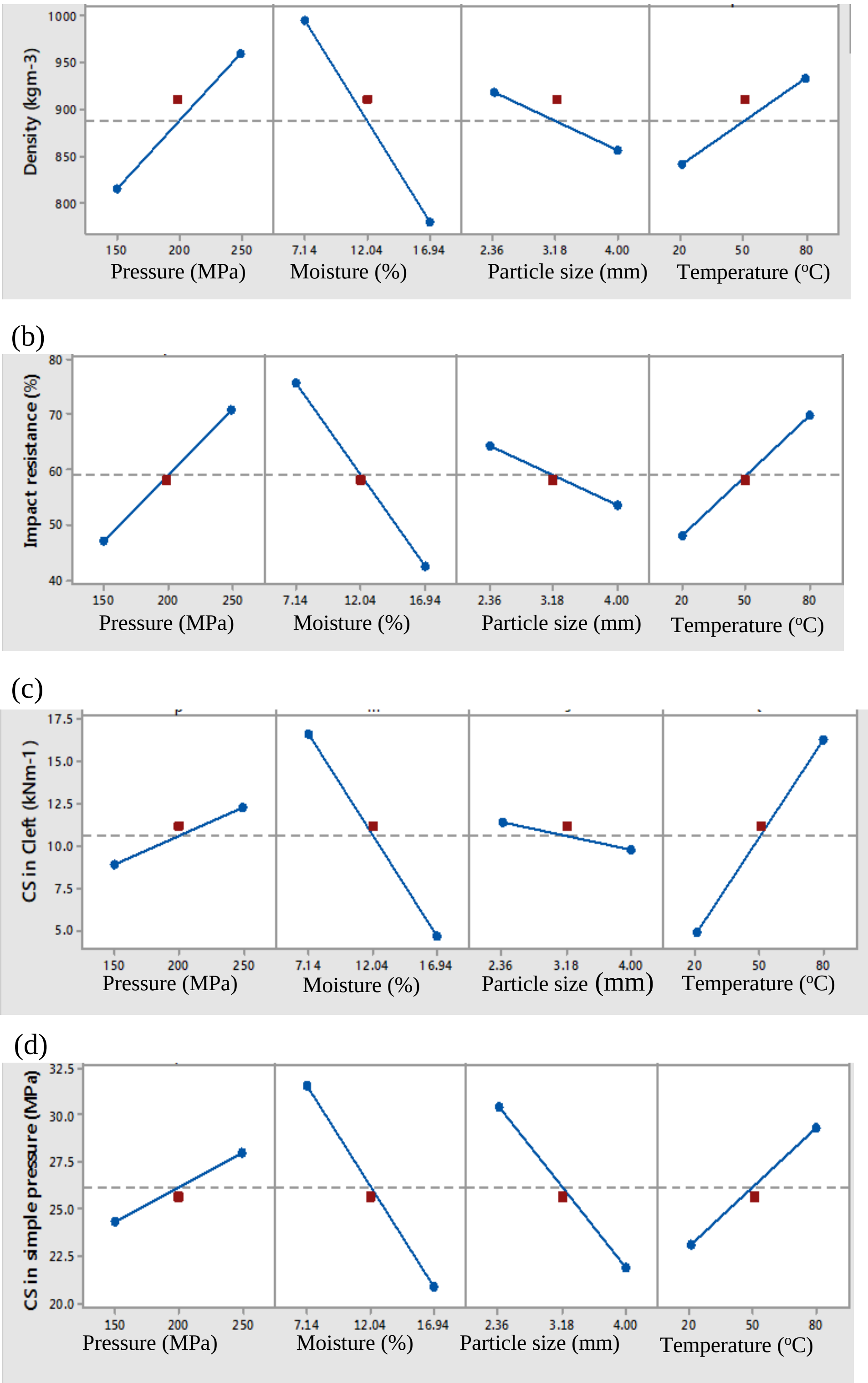


Fig 2: Effects of briquetting parameters: pressure, moisture content, particle size and temperature on (a) density, (b) impact resistance, (c) compressive strength (CS) in cleft and (d) compressive strength in simple pressure. Red square represents the mid-point.

All interactions (Table 2) had significant impact on density ($P < 0.05$) except the interaction between moisture and particle size. Briquettes produced at around 17 % moisture content and pressure <250 MPa (Fig 5) had a density below the German Standard (DIN 51731) for pellets (1-1.4 gcm^{-3}) regardless of particle size and compacting temperature. This is likely due to the incompressibility of water that prevents particles from being completely flattened at high moisture content. Furthermore, the low briquette density could have been attributed to a reduction in briquette weight or an increase in briquette volume upon drying and stabilising. It was also observed that a high proportion of large cracks (Fig. 4) were formed in briquettes produced at high moisture content i.e. 16.94 %. Matúš et al. [27] also reported appearance of cracks on spruce briquettes produced at a moisture content above 16.5 % with 2.56, 12.69, 35.92, 26.06 and 27.77 % of particles <0.50, 0.5-<1.00, 1.00-<2.00, 2.00-<4.00 and >4.00 mm in sizes. Increasing compacting pressure to 250 MPa and reducing particle size (<2.36 mm) could increase the density into the standard range ~ 1,000 kgm^{-3} but this will increase the energy requirement for producing briquettes.

Table 2: Analysis of variance: Response variable: Briquette density

	Degree of freedom	Sum of square	Mean sum of square	F-value	P-value
Pressure (*p*)	1	253650	253650	6274.78	0.000
Moisture content (*m*)	1	559678	559678	13845.27	0.000
Particle size (*s*)	1	45418	45418	1123.54	0.000
Temperature (*t*)	1	101393	101393	2508.26	0.000
$p \times m$	1	28145	28145	696.24	0.000
$p \times s$	1	37772	37772	934.40	0.000
$p \times t$	1	2997	2997	74.15	0.000
$m \times s$	1	87	87	2.14	0.153
$m \times t$	1	23069	23069	570.69	0.000
$s \times t$	1	14971	14971	370.35	0.000
$p \times m \times s$	1	414	414	10.23	0.003
$p \times m \times t$	1	1552	1552	38.38	0.000
$p \times s \times t$	1	9377	9377	231.97	0.000
$m \times s \times t$	1	3200	3200	79.15	0.000
$p \times m \times s \times t$	1	385	385	9.52	0.004
Error	32	1294	40		
Total	48	1083917			

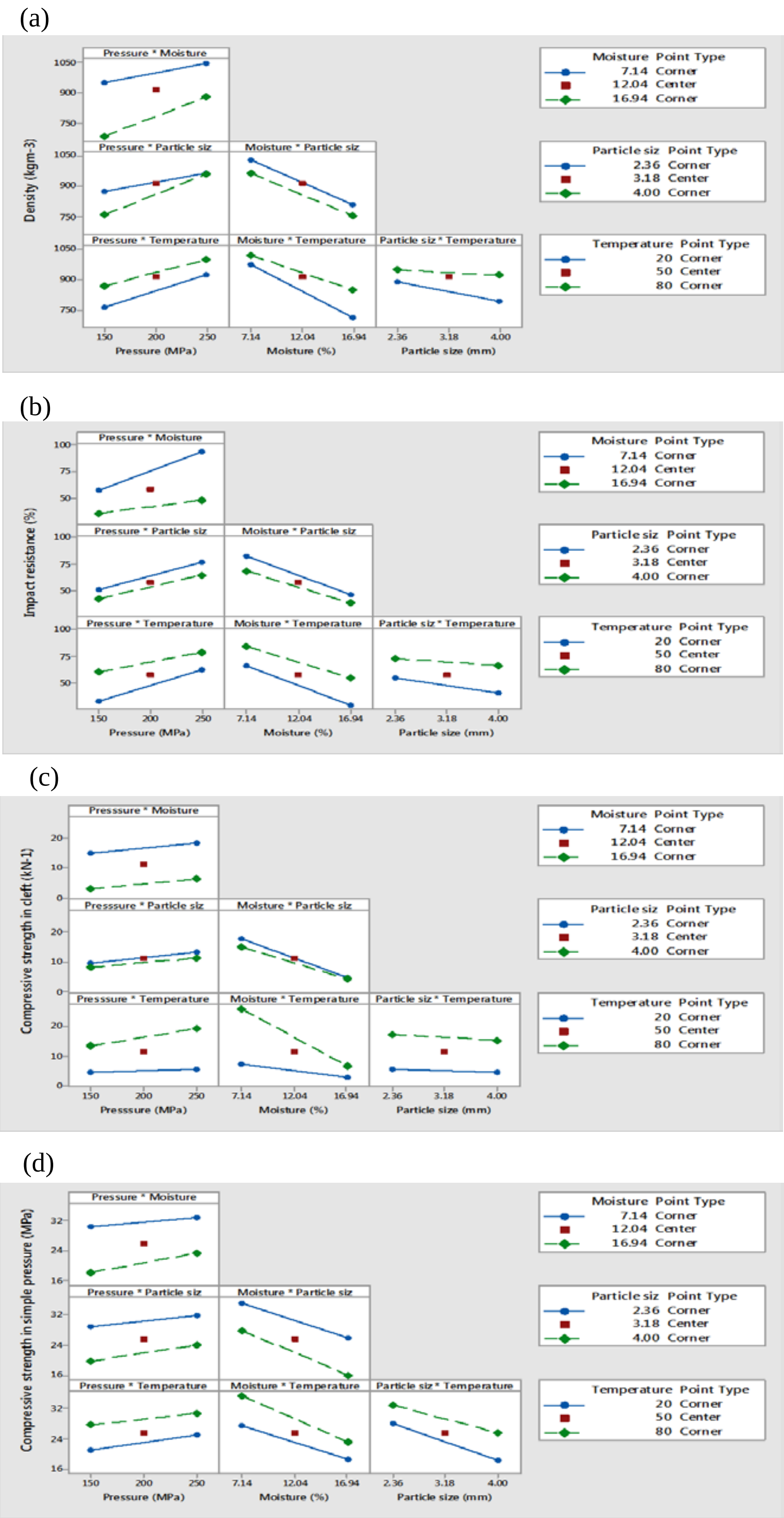


Fig 3: Interaction effects of briquetting parameters: pressure, moisture content, particle size and temperature on (a) density, (b) impact resistance, (c) compressive strength (CS) in cleft and (d) compressive strength in simple pressure. Red square represents the mid-point.

At low moisture content (7.14 %), for small particle size < 2.36 mm, compacting pressure and temperature had little effect on density. Density only increased by less than 4 % when pressure was increased from 150 MPa to 200 MPa and remained almost constant with a further increase to 250 MPa. However, at a moisture content of 7.14 % for a particle size < 4 mm, a significant increase in density (~20 %) was observed when increasing pressure from 150 MPa to 200 MPa; but with only a slight further increase of ~5 % as pressure was increased to 250 MPa. In addition, compacting temperature had a great effect at 150 MPa (~14 % increase). In contrast, at high moisture content (17 %), increasing pressure and temperature significantly increased density for both particle sizes which was probably due to the combined effect of high pressure and heat softening the particles and evaporating moisture. Therefore, with maize cob feedstock at moisture content 7.14-10%, high density briquettes could be produced at either 150 MPa/80 °C or 200 MPa/20 °C for particle size < 2.36 mm but for particle size < 4 mm a pressure > 200 MPa was required. At high moisture content (16.94 %), only a particle size < 2.36 mm could provide briquettes with a density ≥ 1000 kgm^{-3} and this was under conditions of high pressure and temperature i.e. 250 MPa and 80 °C.

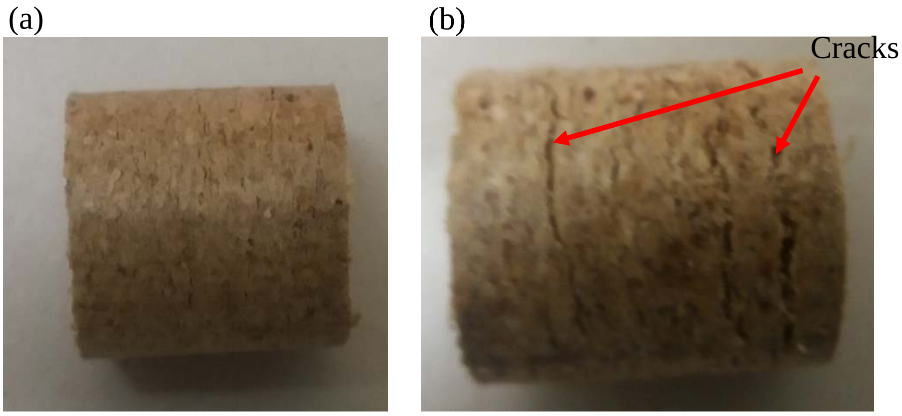


Fig. 4: Briquette produced from pressure of 200 MPa, compacting temperature of 80 °C and particle size of < 2.36 mm at moisture content of (a) 7.14% and (b) 16.94 %.

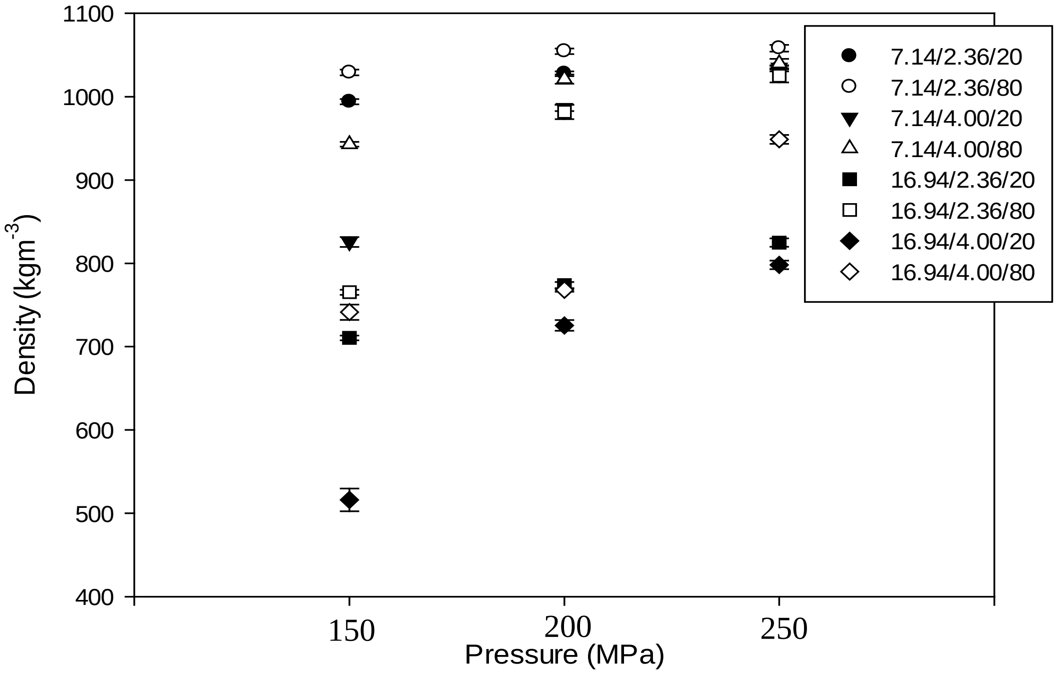


Fig 5: Effect of pressure on briquette density (legend: particle size (mm)/ moisture content (%)/ compacting temperature (^{o}C))

3.4 Impact resistance

Impact resistance is a measure of durability of briquettes which defines their tendency to produce dust or break when subjected to a destructive force. It is an indicator of the mechanical strength of briquettes [55], therefore its value should be as high as possible. In this study, impact resistances ranged from 17.7 % to 99.8 % with variations in the briquetting parameters used. Within all ranges of briquetting parameters studied, impact resistance was increased in response to increased pressure and temperature, but was reduced with an increase in moisture content and particle size (Fig 2b). The optimal moisture content and pressure identified in this study compares well with the optimal moisture content (7.5 %) and pressure (200 MPa) required to produce olive waste briquettes with high impact resistance [30]. At high temperature and pressure, moisture evaporates and increases the rate of heat transfer within biomass particles. However, very high moisture prohibits complete flattening of particles which lowers inter-particle bonds [7], causing less stable and weak briquettes. Application of temperature and pressure causes diffusion of molecules thus reducing void space and forming solid bridges which increases bonding between particles and hence the strength of briquettes. The results agreed well with previous studies for paper mill waste briquettes (prepared in a pressure range of 150-250 MPa and moisture content of 9 % [30] and mango and eucalyptus leaf briquettes (pressure of 30-100 MPa and moisture content of 8.6 % and 7.9 % respectively [21]). However, they disagreed with the findings for pulping residue and spruce sawdust briquettes [23] where impact resistance increased as moisture content was increased from 7 to

15 %. The variations are likely due to variation in the range of optimal moisture contents used for the different feedstocks.

At a fixed compacting temperature of 20 ^oC, impact resistances of briquettes prepared at high moisture content (16.94 %) and particle size <4.0 mm were not influenced by compacting pressure (likely due to the incompressibility of water) and remained around 20 %. Decreasing particle size to <2.36mm had little effect on impact resistance at low compacting pressures but led to a significant increase at 250 MPa. This could be due to the heat generated at high compacting pressure enhancing the release of water within small particles, helping the binding process. Impact resistance was almost 3 fold higher at 150 MPa when temperature was increased to 80 ^oC most likely due to solid bridge formation, however, particle size had no impact. There were significant interactions ($p<0.05$) between briquetting parameters on impact resistance (Table 3; Fig 3b) except for the: pressure x particle size, moisture content x particle size x temperature and pressure x moisture content x particle size x temperature interactions. Under high pressure and temperature, low molecular weight components become binding elements of particles whereas at high temperature and pressure, moisture evaporates and increases the rate of heat transfer within biomass particles [56].

Table 3: Analysis of variance: Response variable: Impact resistance

	Degree of freedom	Sum of square	Mean sum of square	F-value	P-value
Pressure (*p*)	1	6821.1	6821.1	360.74	0.000
Moisture content (*m*)	1	13293.4	13293.4	703.03	0.000
Particle size (*s*)	1	1342.0	1342.0	88.12	0.000
Temperature (*t*)	1	5794.8	5794.8	306.47	0.000
$p \times m$	1	1666.2	1666.2	88.12	0.000
$p \times s$	1	21.6	21.6	1.14	0.293
$p \times t$	1	357.5	357.5	18.91	0.000
$m \times s$	1	109.2	109.2	5.78	0.022
$m \times t$	1	195.2	195.2	10.32	0.003
$s \times t$	1	233.2	233.2	12.33	0.001
$p \times m \times s$	1	181.0	181.0	9.57	0.004
$p \times m \times t$	1	125.5	125.5	6.63	0.015
$p \times s \times t$	1	144.9	144.9	7.66	0.009
$m \times s \times t$	1	21.9	21.9	1.16	0.290
$p \times m \times s \times t$	1	66.3	66.3	3.50	0.070
Error	32	605.1	18.9		
Total	48	30979.3			

At low moisture content (7.14 %) and particle size ($<$2.36 mm) increasing compacting temperature from 20 ^{o}C to 80 ^{o}C significantly increased impact resistance i.e. from 50 % to 80 % at 150 MPa. However, there was no effect of temperature on impact resistance at higher compacting pressures $>$200 MPa (Fig 6). For larger particle size ($<$4 mm), compacting temperature had a significant effect resulting in high impact resistance ($>$80 %) but only at high pressure (200 MPa-250 MPa) when a compacting temperature of 20 ^{o}C was used. Impact resistance increased significantly ($P < 0.05$) with an increase in pressure from 150 to 200MPa, but was unchanged above 200 MPa.

Briquettes with high impact resistance/durability are desirable to minimise breakage and dust formation during transporting and conveying. Up to now, there are no certified standards for biomass briquettes, however, other researchers [55, 57] have reported that impact resistance of

80 - 90 % or over 90 % is required for better handling and transportation. However, very high-quality briquettes (with impact resistance above 95%) were obtained at (i) small particle size (< 2.36 mm), low moisture content (7.14 %) and high pressure (>200 MPa) and (ii) high particle size (< 4.00 mm), low moisture content (7.14 %), high temperature (80 °C) and high pressure > 200 MPa. These briquettes lost only < 3.5 % of their weight after shattering and are therefore durable thus satisfying the European Standard Committee CEN/TC 335 (durability > 95 %) and are also suitable for transportation, storage and handling with minimal breakage and dust generation.

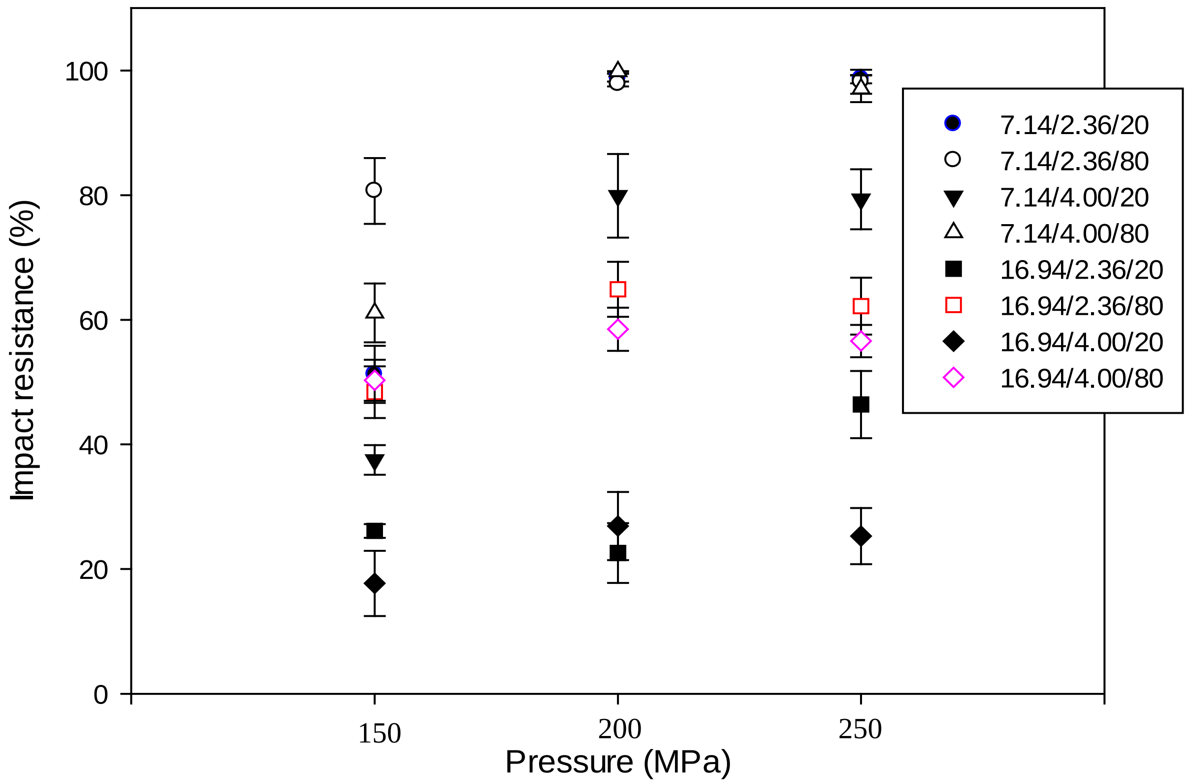


Fig 6: Effect of compacting conditions (temperature, pressure) and feedstock properties (moisture content, particle size) (legend: particle size (mm)/ moisture content (%)/ compacting temperature (°C))

3.5 Compressive strength (CS)

Compressive strength is the maximum load that a briquette can withstand before it breaks [58]. It is used to estimate the compressive stress resulting from the weight of the top briquettes on the lower briquettes during storage, transport and handling. Compressive strength (CS) tests were performed via both cleft and simple pressure tests. These two tests have been used independently [9, 13, 53, 59] to determine compressive strength of briquettes and it was found from this study (data not presented) that there was a strong positive correlation between the two tests.

Moisture content and compacting temperature were the dominant factors affecting compressive strength in cleft whilst simple pressure was mainly affected by moisture content and particle size i.e. simple pressure decreased with increasing moisture content and particle size (Fig 2c and 2d). The compressive strength (between 75 and 120 MPa) of pine briquettes increased with an increase in compacting pressure in the range of 31 - 318 MPa but was reduced with an increase in particle size i.e. 0.5 - 4.0 mm [32]. Compressive strength of hazelnut shell briquettes produced from particle size of 2-4 mm, moisture content of 8.7 % with pyrolysis oil from hazelnut shell and some wood as binder (6.5-18.0 %) increased (from around 11 to 38 MPa) when compacting pressure was increased from 300 to 800 MPa [13]. However, the effect of moisture content found in this study contradicts with others. For example, for lupin seed with an average particle size of 0.5 mm, compressive strength of briquettes increased with moisture content from 9.5 % to 15.0 % [60]. A 30% increase in compressive strength of olive refuse briquette was observed when moisture content was increased from 5 % to 15 % [30] using a compacting pressure of 200 MPa and particle size of <0.250mm. An increase in compressive strength of pulping reject briquettes from 13.0 to 37.2 MPa was reported when moisture and compacting pressure were increased from 7 % to 18 % and 300 MPa to 800 MPa respectively [23].

Both compressive strength in cleft and simple pressure increased significantly ($P<0.05$) when pressure was increased from 150 MPa to 200 MPa but with no further increase at higher pressures. One can argue that an increase in compacting pressure is associated with an increase in interparticle bonds resulting from an increase in cohesion force [36]. However, above the optimal compacting pressure, in this case 200 MPa, the phenomenon of dilation occurs, producing cracks in briquettes and consequently weakens them [61]. Interaction plots (Fig 3c and d) shows that there were significant interactions (Table 4 and 5) on compressive strength in cleft (Table 4) for all variables with the exception of: pressure x moisture, pressure x particle size, pressure x particle size x temperature, moisture x particle size x temperature and, pressure x moisture x particle size x temperature. For compressive strength in simple pressure all variables showed significant interactions with the exception of particle size x temperature and, pressure x moisture x particle size x temperature (Table 5).

Table 4: Analysis of variance: Response variable: Compressive strength in cleft.

	Degree of freedom	Sum of square	Mean sum of square	F-value	P-value
Pressure (*p*)	1	138.38	138.38	427.15	0.000
Moisture content (*m*)	1	1722.01	1722.01	5315.51	0.000
Particle size (*s*)	1	32.18	32.18	99.32	0.000
Temperature (*t*)	1	1549.28	1549.28	4782.33	0.000
$p \times m$	1	0.00	0.00	0.01	0.940
$p \times s$	1	1.05	1.05	3.24	0.081
$p \times t$	1	73.26	73.26	226.14	0.000
$m \times s$	1	17.64	17.64	54.46	0.000
$m \times t$	1	654.90	654.90	2021.56	0.000
$s \times t$	1	1.44	1.44	4.43	0.043
$p \times m \times s$	1	11.70	11.70	36.12	0.000
$p \times m \times t$	1	29.93	29.93	92.37	0.000
$p \times s \times t$	1	0.01	0.01	0.02	0.900
$m \times s \times t$	1	1.11	1.11	3.43	0.073
$p \times m \times s \times t$	1	0.11	0.11	0.34	0.564
Error	32	10.37	0.32		
Total	48	4243.70			

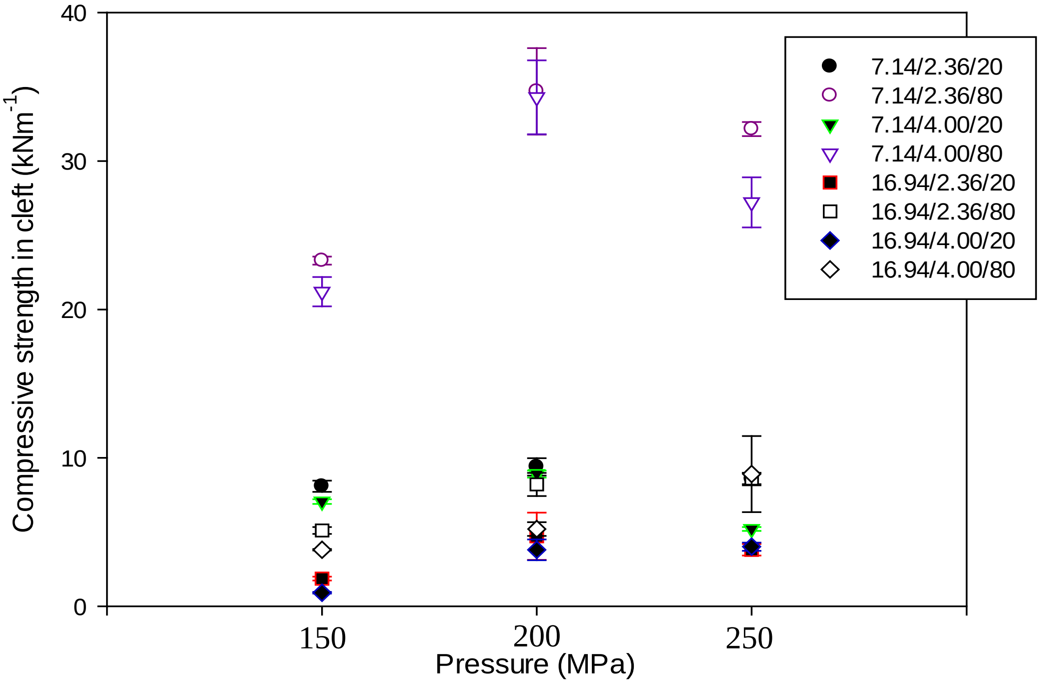
Table 5: Analysis of variance: Response variable: Compressive strength in simple pressure.

	Degree of freedom	Sum of square	Mean sum of square	F-value	P-value
Pressure (*p*)	1	161.33	161.33	125.76	0.000
Moisture content (*m*)	1	1376.02	1376.02	1072.57	0.000
Particle size (*s*)	1	884.08	884.08	689.12	0.000
Temperature (*t*)	1	466.25	466.25	363.43	0.000
$p \times m$	1	23.80	23.80	18.55	0.000
$p \times s$	1	7.36	7.36	5.74	0.023
$p \times t$	1	2.08	2.08	1.62	0.212
$m \times s$	1	21.60	21.60	16.84	0.000
$m \times t$	1	32.34	32.34	25.21	0.000
$s \times t$	1	16.80	16.80	13.10	0.001
$p \times m \times s$	1	31.04	31.04	24.20	0.000
$p \times m \times t$	1	13.87	13.87	10.81	0.002
$p \times s \times t$	1	19.25	19.25	15.01	0.000
$m \times s \times t$	1	7.21	7.21	5.62	0.024
$p \times m \times s \times t$	1	0.70	0.70	0.55	0.465
Error	32	41.05	1.28		
Total	48	3105.07			

It is recommended [24] that the minimum compressive strength in simple pressure for briquettes is 2.56 MPa to enable storage, transportation and handling with minimum breakage. Compressive strength in simple pressure of all briquettes in this study was above the recommended value (Fig. 7b). The smallest value of 10 MPa was obtained at large particle size (<4 mm), with low compacting pressure and temperature (150 MPa and 20 ^{o}C) and high moisture content (16.94 %).

At a compacting temperature of 20 ^{o}C, compressive strength in cleft was below 10 kNm^{-1} for all moisture content and particles size variations studied (Fig.7a). Increasing compacting pressure from 150 to 200 MPa resulted in more than 100% increase in compressive strength in cleft for particle size <4 mm and high moisture content but had little impact where small particle size <2.36 mm and low moisture content were used. Increasing pressure increased compressive strength because particles undergo plastic and elastic deformation, thereby increasing contact areas of particles which in turn filling void spaces and increasing inter-particle bonds [38, 54]. High compacting pressure could also crush large size particles, leading to increased densification [62]. During briquetting, pressure causes particles to rearrange to form closely packed mass and then to elastically and plastically deform when pressure increases. During the plastic and elastic deformation, particles move and fill void spaces which increases contact area, consequently increasing both density and strength [18, 54]. According to Kers [31] and Antwi-Boasiako and Acheampong [57], too much moisture in the feedstock leaves cracks/void space in briquettes due to the escape of moisture within the briquette. The formation of cracks/void spaces makes briquettes more porous thereby reducing their strength and density. Therefore, a minimum amount of moisture in a feedstock is required to act as a binding/catalyst to release low molecular mass products which binds particles together thereby improving briquette strength. However, low moisture content is associated with low rate of heat transfer between particles and therefore the requirement for high compacting pressure [56]. In addition, moisture is responsible for bringing interfacial forces and capillary pressure into play to increase forces of attraction between particles [27].

(a)



(b)

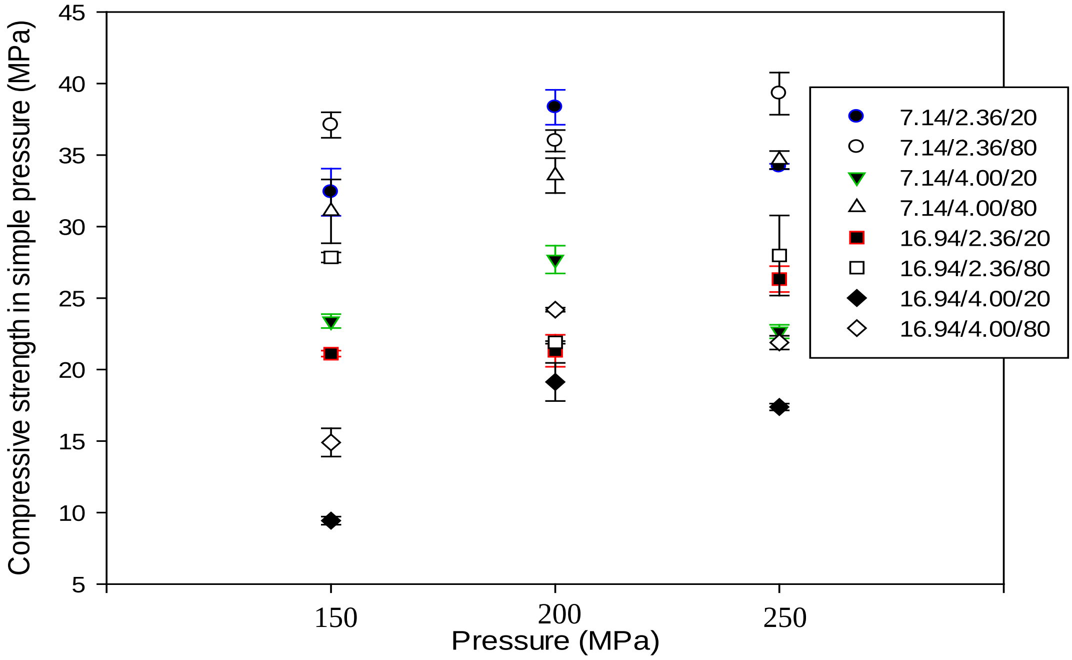


Fig 7: Effect of briquetting conditions (temperature, pressure) and feedstock properties (particles size, moisture content) on compressive strength in (a) cleft, (b) simple pressure (legend: particle size (mm)/ moisture content (%)/ compacting temperature (^{o}C))

At a compacting temperature of 80 ^{o}C, the effect of compacting pressure was highly significant both with high and low moisture content feedstocks. An increasing temperature releases natural binders such as lignin, cellulose and hemicellulose which form solid bridges upon cooling [49, 62, 63] thereby increasing strength and density. A scanning electron microscopy (SEM) image (Fig 8) of a briquette which was broken from the middle in a direction perpendicular to the axis of the cylindrical briquettes showed a relatively smooth surface and particles which were flattened to form a layer. The layer observed in the SEM image could have resulted from solid bridge formation as no evidence of mechanical interlock was observed. Application of high pressure and/or temperature during densification results in diffusion of molecules at the point of contact from one particle to another, thus forming solid bridges [7]. Particles of corn stover and switchgrass briquettes/pellets are bonded mainly by solid brigdes resulting from natural binders i.e. mainly lignin and protein [14]. Natural binders can be squeezed out of particles at temperatures near the glass transistion temperature (80 °C for maize cob) and improve particle bonding through formation of solid bridges on cooling [7]. An increase in temperature also results in evaporation of water from the particles of biomass under compression and since water is uncompressible, the density of the briquette is increased.

(a) (b)

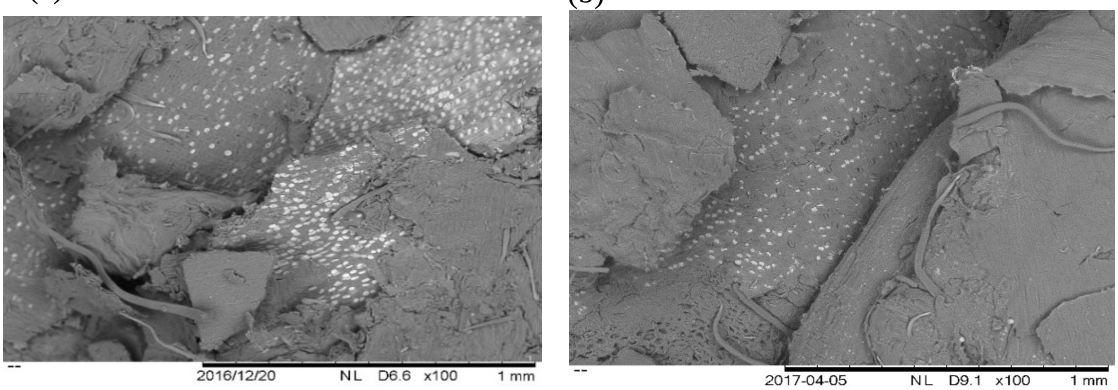


Fig 8: SEM image of broken briquette perndicular to the axis of the cylindrical briquette from crused maiez cob of 2.36 mm seive size, (a) compacting pressure of 250 MPa, compacting tempearture of 80 °C, moisture content of 7.14 % (b) compacting pressure of 150 MPa, compacting tempearture of 20 °C, moisture content of 16.94 %.

At a fixed pressure, small particles are more densely packed than large particles [43]. In addition, they have large surface area of contact which helps to create strong inter-particle bonding, while large particles cause cracks which reduces density and strength [28]. The larger surface area of small particles also facilitates better heat transfer (necessary for strong bond formation) between particles thereby improving density and strength [54]. High porosity would lower both density and strength. Valence and Van der Waals' forces can contribute to bonding when seperation between particles are about 10 Å and 0.1μm respectively [14]. Therfore, the forces contributing to bonding become less effective for large pore sizes, thereby weakening the briquettes.

4 Conclusions

Briquettes properties are an important character to meet the increasing demand for biomass feedstocks, enabling long-term handling, storage and transport. In this study, an increase in compacting pressure and temperature and a decrease in moisture content and particle size increased density, impact resistance and compressive strength of corn cob briquettes. The results showed that compacting pressure of 150MPa led to low quality and is not suitable for briquette production regardless of the other parameters used in briquetting process. Pressure ≥ 200MPa and temperature had no effect on properties of briquettes made from low moisture content (< 10 %), or small particle size (< 2.36mm) maize cob. However, by increasing compacting temperature up to 80°C, the particle size could be increased without trading off any durability properties. This is because temperature releases components such as lignin, cellulose and hemicellulose which act as binders. Compressive strength in simple pressure was in the recommended range (≥ 2.56MPa) for all tested conditions. There was a strong interaction between briquetting parameters and the interaction between moisture and temperature significantly affected all the briquette properties studied most likely because moisture accelerates heat transfer between maize cob particles which ease elastic and plastic deformation during compression and also facilitates the release of natural binders.

Acknowledgements

The authors would like to express their sincere gratitude to the Commonwealth Scholarship Commission for financial support; to technicians at the School of Chemical Engineering and Advanced Materials for their help in the fabrication of the briquetting machine and to Barfoots of Botley Ltd for providing maize cobs for this study.

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