

EFFECTS OF OPERATING VARIABLES ON DURABILITY OF FUEL BRIQUETTES FROM RICE HUSKS AND CORN COBS

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ABSTRACT

Biomass densification processes increase fuel energy density for more efficient transport. This study presents new data to show that blending different types of biomass improves the properties of densified biomass briquettes. The specific objectives were to investigate the effects of sample batch (biomass source), material ratio (rice husks to corn cobs), addition of binder (starch and water mixture) and compaction pressure, on briquette properties, using a factorial experiment.

Briquettes had a unit density of up to 1.9 times the loose biomass bulk density, and were stronger than briquettes from the individual materials. Considering average values from two biomass sources, an unconfined compressive strength of 176 kPa was achieved at a compaction pressure of 31 MPa for a 3:7 blend of rice husks to corn cobs with 10% binder. These briquettes were durable, with only 4% mass loss during abrasion and 10% mass loss during shattering tests. They absorbed 36% less water than loose corn cobs. Statistical analysis of the results showed that starch and water addition was required for adequate briquette strength, but significantly reduced green and relaxed densities. The source of the biomass had a significant effect on densification, which emphasises the need to understand factors underlying biomass variability.

Key words: fuel; briquettes; blends; rice husks; corn cobs; density; durability

1 INTRODUCTION

1.1 Energy from agricultural biomass

Biomass has received tremendous attention both in developed and developing countries as a renewable energy source [eg. 1,2]. A major drawback of biomass energy is the competition between energy and food crops for cultivable land [3,4]. This issue is resolved by use of agricultural residues, which would otherwise be wasted, for energy generation. However, although agricultural residues form one of the biggest potential sources of biomass energy in most developing countries, their efficient exploitation for energy is presently uncommon [1,5]. At present, agricultural residues are combusted directly without optimisation of energy efficiency or control of air emissions, or they are left on farm land/processing sites to decay, potentially releasing greenhouse gases and/or polluting surface waters.

1.2 Biomass densification

Since direct use of unprocessed biomass feedstock can lead to problems during storage, transportation, handling and processing [6], numerous strategies have been developed to convert various types of biomass into secondary fuels that have better characteristics compared to the parent material(s). These strategies include biomass densification.

Biomass densification involves its compaction into a pellet or briquette of up to ten times higher density than the parent material(s) [7,8]. Such processing increases biomass bulk and energy density per unit volume, leading to lower storage requirements, more efficient transportation, reduced particulate emissions per unit volume of material transported or combusted, and uniform feeding into industrial equipment such as boilers, gasifiers and domestic stoves for rural applications [1,9,10].

Research by other workers has demonstrated that agricultural residues such as rice husks, corn cobs, olive husks etc. can be densified into briquettes [e.g.,1,10,11]. Due to variations in properties of different biomass materials, some feedstocks are more easily densified than others. Biomass materials with a higher lignin, starch or protein content exhibit better compaction than those with higher cellulosic content [8]. This has prompted addition of biomass containing higher amounts of these components to other biomass. For example, blending sawdust from Scots pine with wheat straw resulted in more durable pellets compared to wheat straw alone [12], rice bran was used as a binder in briquetting rice straw [13] and olive refuse blended with fibrous paper mill waste [55], for reportedly improved briquette durability.

1.3 Rice husks and corn cobs

Rice and corn are examples of major crops that result in generation of huge amounts of waste from their cultivation and processing, including rice husks and corn cobs. Around 134 Mt of rice husks were produced globally from 671 Mt of rice production in the year 2008 [14]; in the same year, approximately 135 Mt of corn cobs were produced from 797 Mt of corn production [15].

Table 1 compares energy, ash, moisture contents, bulk density and porosity of rice husks and corn cobs, as gathered from sources in the literature [1,14,16,17,18,19,20-29]. It can be estimated that the total annual generation of rice husks and corn cobs has an energy content of 4 EJ, which represents about 1% of the world total primary energy consumption [30].

Problems have been encountered with the use of briquettes produced from these individual materials. Briquettes produced from rice husks have been reported to cause clogging of industrial boilers and domestic stoves due to their high ash content [31], which is also abrasive and wears equipment quickly due to the high silica content of the rice husk ash [14]. Briquettes produced from corn cobs have a tendency for water absorption due to the high porosity of corn cob particles. High moisture contents are undesirable in thermochemical processes such as pyrolysis and gasification due to the energy requirement for drying of biomass and the reduced heating value of the product gas [32,33].

1.3.1 Material ratio

Blending of rice husks and corn cobs will result in fuel briquettes with a lower ash content compared to briquettes produced from rice husks alone. On the other hand, briquettes containing rice husks are expected to absorb less water than briquettes from corn cobs alone, due to the lower water absorbency of the thick outer walls of rice husks [20]. Material ratio is the proportion of individual rice husks or corn cobs residue in the blend of both residues.

1.3.2 Sample batch

The variability of biomass materials have resulted in inconsistency in the characteristics of fuel briquettes produced from different types of residues [8,57], this may even apply to the same type of residues grown at different season or different locations. It becomes necessary to understand the influence of the variability on biomass densification and fuel briquette's quality.

1.3.3 Binder

Despite the additional cost, additional binders are often added in densification of biomass residues, as they may not naturally contain adequate proportions of binders.

Starch has been used as a binder in some densification processes, such as in compaction of sorghum residue, and corn cobs individually [34,35], and has been reported to improve briquette characteristics. Starch is a polysaccharide, which is widely available. It has a high energy content and is a good binding agent due to its chemical and structural properties [36]. Addition of water and heat to starch granules causes swelling, which results in the formation of intermolecular hydrogen bonds between the amylose and amylopectin components of starch, followed by loss of the individual crystalline structure of the two components [37]. This leads to formation of a viscous solution that undergoes retrogradation, i.e., gelling, during cooling or storage. The viscosity of hydrated starch increases its shear and tensile strengths. The fluidity and viscoelasticity of the produced solution [37] gives it the ability to occupy the void spaces present within and between biomass particles, forming solid bridges that become stronger upon air-drying.

1.3.4 Pressure

During the densification process, an increase in pressure results in plastic and elastic deformation, molecule diffusion and closing up of void spaces between particles to form a compacted solid. Briquettes manufactured at lower pressures of 30 to 60 MPa crumble easily, while those produced at higher pressures of 150 to 250 MPa remain compacted and durable [57], for example, increasing the compaction pressure from 1 to 10 MPa increased the shear strength of briquettes from 2.8×10^{-2} kPa to 9.6×10^{-2} kPa [43]. Currently, efforts are directed towards improving the quality of fuel briquettes produced at lower pressures.

1.3.5 Objectives

This study investigated the effects of sample batch (biomass source), material ratio, addition of binder, and compaction pressure, on properties related to the durability of fuel briquettes made from blends of rice husks and corn cobs, including their strength, resistance to impact and abrasion, and water absorption.

2 MATERIALS AND METHODS

2.1 Sourcing, preparation and characterisation of raw materials

Two bulk samples of air dried rice husks, and corn cobs, as well as a sample of starch were sourced and collected from local farms and milling sites in Niger state, Nigeria. Rice husks were used as received from the milling site, since they have a particle size of <2 mm, which can readily undergo densification. The mass median diameter (“D50”) of the rice husks was 0.7 mm. Corn cobs were used with a particle size of <1.6 mm, based on preliminary experiments which found that larger particles (2-10 mm) were less easily compacted. Corn cob particles obtained using a hammer mill fitted with 1 mm screen were blended with larger particles (1-1.6 mm) that had been manually crushed. The mass median diameter of the resulting blend was 0.8 mm. Characterisation of rice husks and corn cobs included determination of bulk density by BS EN 15103 [38], moisture content by BS EN 14774-2 [39], particle size by sieve analysis according to DD CENT/TS 15149-2 [40], water absorption by adaptation of BS EN 772-21 [41] and specific gravity using a Micromeritics helium pycnometer (ACCU Pyc 1330). The porosity of materials was determined using equation 1.

$$\text{Porosity} = \left(1 - \frac{\rho}{SG}\right) \times 100 \dots \dots \dots (1)$$

where

ρ = density of material (kg/m³ dry basis)

SG = specific gravity of material (kg/m³)

The particle morphology of three replicates of each sample of rice husks and milled corn cobs was examined using a Jeol JSM-6480LV high-performance, variable pressure analytical scanning electron microscope (SEM). Energy-dispersive x-ray spectroscopy (Oxford Instrument INCAx-sight EDS-system) was used for microanalysis of the solid phases viewed by SEM.

Pure unrefined cassava starch was prepared as a binder by mixing into a paste with water at a mass ratio of 2:3, for 5 minutes prior to its addition to the rice husk and corn cob blends. The binder contents for the experiments reported here were chosen based on preliminary experiments with 5 to 35% starch mixture.

2.2 *Experimental design and analysis*

A factorial experimental design method involving 24 runs was employed for production of briquettes. The variables investigated in this study were chosen based on their expected influence on briquette quality: sample batch (S), i.e. two different samples, A and B, of rice husks and corn cobs obtained for two different seasons and farms sites, material ratio (M), i.e., percentage mass of rice husks in the blend of rice husks and corn cobs, and binder addition (B), i.e. mass of starch and water added as a percentage of the rice husk and corn cob blend, and compaction pressure (P). The response variables measured were green (immediately after extrusion from the mold) and relaxed (after 24 hours curing) unit density, unconfined compressive strength, mass lost in abrasion and shattering tests (i.e., “durability rating”) [59], and water absorption. The methods used to measure these response variables are summarised in Table 2. Unless otherwise specified, each test was repeated for three briquettes.

Two levels (low and high) were selected for each of the independent variables; the 2^4 factorial design that was used for briquette production is shown in columns 2 to 5 of Table 4, which also shows the measured responses.

Since the effect of water in the binder was confounded with that of the starch in this experiment, additional experimental runs were conducted to assess the effect of water on the response variables, with and without starch. The responses for briquettes containing water only (without starch) are presented in rows 21** to 24** of Table 4, for comparison with the results for otherwise similar briquettes produced with both water and starch mixture in rows 9 to 12. In the statistical analysis, the effect of dry starch on briquette responses was assumed to be the same as when only rice husks and corn cobs residues were used (i.e., with no water or starch in rows 17** to 20** of Table 4).

Statistical effects of variables and their interactions on the responses were calculated based on the individual replicate results shown in columns 7, 9 and 11 of Table 4 [42]. Effects of the variables and interactions between the variables on a response are estimated as the differences between the averages for the high and low levels of a variable or interaction, and the total mean response. The highest order interactions of variables were assumed to be largely due to random noise [42]. Normal probability plots of the effects can be used to visualize the significance of the effects of individual variables on the responses [42]. The estimated effects can be read from the abscissa, against the standard deviation of the normal

distribution on the ordinate. The scale of the ordinate has been adjusted such that a normal distribution appears as a straight line, i.e., points that lie on the straight line may be a result of normal random variability, whereas those that deviate from the straight line indicate significant effects of these variables or interactions on the response. Analysis of variance was also used to determine the statistical significance of the observed effects [42].

The fitted model for the predicted responses is shown as Equation 2 [42], and Equation 3 was used to calculate the residuals (ϵ) of the responses.

$$\hat{Y} = \bar{Y} + \left(\frac{j^1}{2}\right) * x_1 + \left(\frac{j^2}{2}\right) * x_2 + \dots + \left(\frac{j^n}{2}\right) * x_n \dots \dots \dots (2)$$

$$\epsilon = y - \hat{Y} \dots \dots \dots (3)$$

Where;

\bar{Y} is the grand mean for each set of response data (eg. green density)

$j_1, j_2 \dots j_n$ is the observed main or interaction effect of the variables

$x_1, x_2 \dots x_n$ is the respective sign of the observed effects for each response value

A normal probability plot of the residuals was used to visualize the normality and check that all effects other than those included in the model are explained by random noise.

2.3 Briquette production

Biomass and binder blends were weighed out in the proportions indicated in columns 3, 4 and 5 of Table 4 and densified using a simple hand-held laboratory steel mold with an inner diameter of 32 mm, an outer diameter of 51 mm, and length of 100 mm (Figure 1).

A hydraulic compression testing machine (Controls-04600/FR) was used to compress the blended biomass in the mold to the desired load (column 6 of Table 4), at a rate of 200 N/s as shown in Figure 2. Based on previous findings by the author and other researchers [43,44] that a hold time under compression in the mold in excess of 40 seconds has little effect on briquette characteristics, the compacted briquettes were held at the desired compaction pressure of 19 and 31 MPa, for 60 seconds, and then extruded from the mold through the hole shown on the right side of the mold base plate in Figure 1.

2.4 Briquette curing

All briquettes produced in the factorial design experiment were cured for 24 hours at $23 \pm 2^\circ\text{C}$ and relative humidity of $50 \pm 5\%$ before testing.

New batches of the briquette formulations with the highest relaxed density and those with the highest unconfined compressive strength were made for further testing after curing as follows:

- 24 hours + 6 days at $23 \pm 2^\circ\text{C}$
- 24 hours at 35°C + 6 days at $23 \pm 2^\circ\text{C}$.

The briquettes were cured at 35°C to assess the effect of warm weather conditions, e.g., in the source country of the raw materials, Nigeria.

Figure 3 shows some of the briquettes produced.

3 RESULTS AND DISCUSSION

3.1 Properties of loose rice husks and corn cobs

Table 3 shows the properties of the rice husks and corn cobs measured in this study.

The ash content of the rice husks in Table 3 appears to be consistent with the literature value in Table 1, while that of the corn cobs, though almost twice the literature value, is much lower than that of the rice husks. The moisture contents determined for both the rice husks and corn cobs in this study were similar, although moisture content of corn cobs is commonly found to be higher than that of rice husks, and both our measurements are slightly lower than the ranges reported by others [1,17] (Table 1). This may be due to handling and storage conditions of the materials before the moisture content test, whereby our materials were not fresh, and air drying and hammer milling of the corn cobs is associated with significant moisture loss [17].

Furthermore, the measured solids specific gravities were consistent between samples of each material, but those of the rice husk solids were slightly higher than those of the corn cob solids. This observation may be attributable to the higher ash content of the rice husks, or other differences in composition between rice husks and corn cobs as shown in Table 1 and Table 3. The loose bulk densities of both the rice husks and corn cobs in this study fall within the ranges determined by other workers (Table 1), except for corn cob Sample A which is

about 40% higher than expected. The porosities of the rice husks and corn cobs were similar, but also higher than results by others reported in Table 1, with a notable difference between the porosities measured for the two samples of corn cobs, reflecting the difference in bulk density.

These results indicate a greater variability in the properties of lignocellulosic biomass than has hitherto been reported in the literature, potentially caused by factors including growth conditions, cultivation methods, and post-harvesting handling of the crop.

Based on visual observation, rice husk samples A and B appeared similar. On the other hand, corn cob sample B appeared to have flat, flakey and porous particles while sample A had round particles with a thick outer layer that appeared less porous. The SEM investigation of the biomass particle morphology confirmed a difference in the texture between the rice husks (Figure 4) and corn cobs (Figure 5), and between the two samples of corn cobs. Corn cobs consist of softer, porous particles (Figure 5b), which can aid water penetration, while rice husks have thicker cell walls and fewer openings on particle surfaces, which resist rapid water penetration. The microstructure of corn cob particles implies existence of a capillary network and an associated tendency to absorb water [23].

Consistent with the literature [20,23] (Table 1), the water absorption of the corn cobs was found to be higher than that of the rice husks. For all four biomass samples, the available pore spaces within the biomass residue were oversaturated. The oversaturation of the porosity is observed as swelling (% volume change in Table 1Table 3), which is typical of most lignocellulosic materials when immersed in fluids such as oil [52] and water.

3.2 Briquette density and compressive strength

Columns 7, 9 and 11 of Table 4 show the mean unit densities and compressive strengths of briquettes obtained for each run of the factorial design experiment; the standard deviations for the three replicates of each test are shown in columns 8, 10 and 12.

Results showed that blending rice husks and corn cobs produced briquettes with a unit density of up to 1.9 times the average bulk density of the loose biomass, and of better strength than briquettes made from the individual materials. The bulk densities of the fuel briquettes in this study were estimated to range from 366 to 570 kg/m³; the higher value compares well with the ≥ 500 recommended value for standard solid biofuels under the UK code of good practice [54].

For various blends of rice husks and corn cobs, the compressive strengths and relaxed densities obtained were in the range of 25 to 237 kPa and 490 to 712 kg/m³ respectively. Considering average values from both biomass sources investigated, an unconfined compressive strength of 176 kPa was achieved at a compaction pressure of 31 MPa for a 3:7 blend of rice husks to corn cobs with 10% binder (starch/water = 2:3). These briquettes were found to be durable with only 4% mass loss during abrasion and 10% mass loss during shattering tests. They absorbed 36% less water than the loose corn cobs. The durability properties of briquettes obtained in this study compare well with those obtained by other researchers [e.g., 17,35,43] for individual agricultural biomass and [e.g., 12,57], for mixed woody biomass. The durability properties fall within the specification of $\leq 12\%$ moisture content and $< 10\%$ mass loss during shattering and abrasion tests for CEN/TS 14961, the European standard for solid fuel quality [54].

3.3 Effects of briquetting variables on response variables

The main (individual) and interaction (two-factor and three-factor) effects of the sample batch, material ratio, binder content and compaction pressure, on the green densities, relaxed densities and compressive strengths of the briquettes in the 2⁴ factorial design experiment (Runs 1 to 16), are presented in normal probability plots in Figure 6a to c. The effects that deviate from the straight line in the probability plot are the most significant. The probabilities that the shown effects are attributable to random error, p , were determined based on the F-statistics calculated in the analysis of variance (ANOVA). An effect is generally considered as statistically significant when $p < 0.05$ [42]. A normal plot of the residuals from equation 2 showed a straight line, indicating good model fit.

The use of corn cob sample A rather than B had a highly significant positive effect on both green and relaxed densities ($p = 0.0001$ and $p = 0.0001$, respectively), but only a small positive effect on compressive strength ($p = 0.562$). Since densification is the primary motivation for briquetting, this effect emphasises the importance of understanding the variability of biomass properties, and possibly the need for consistent pre-treatment of residues before their application as biofuels. The lower rice husk content had a highly significant positive effect on briquette relaxed density and compressive strength ($p = 0.0001$ and $p = 0.0001$, respectively). The smaller particle size of the corn cobs and their porous nature may have resulted in better compaction. This observation also compares well with findings by other authors [17,35], where briquettes produced from smaller particles sizes

exhibited less relaxation. There is an increased energy cost associated with biomass grinding, but energy is saved in compaction, as smaller particles are more easily densifiable due to their greater surface area [8] which increases the effect of short range electrostatic and magnetic forces, and causes particles to adhere to each other [56].

The use of the starch/water binder decreased briquette density ($p = 0.003$), which may be attributable to the low density of the starch/water gel, in comparison with the residues that it replaced, and the possible expansion of briquettes due to heat development during densification. However, the briquettes produced without binder (experimental runs 17** to 20**), were crumbly and with negligible compressive strength, and the use of the binder was thus critical to achieve a useful compressive strength, as expected (1.3). It was postulated that the effect of the binder on the briquette responses may be due to the natural presence of binders in most biomass materials, which are activated using moisture or temperature [17]. Therefore, to assess the effect of water separately from that of starch, an ANOVA was conducted for experimental runs 9 to 12 (including starch and water) and 21** to 24** (including water only), in **Error! Reference source not found.** The results compared well with those for the 2⁴ factorial design experiment, and showed that starch had an effect of 16 kPa ($p = 0.0001$) on strength, on top of the effect of water alone. There was also an interaction between sample batch and binder content, which significantly reduced the green density of the briquettes made with corn cob sample B containing the starch binder ($p = 0.0040$). This interaction effect was not apparent for the relaxed density ($p = 0.135$), but had a strong negative effect on the compressive strength ($p = 0.0001$). The use of the higher pressure of 31 MPa yielded a significant positive effect on briquette relaxed density ($p = 0.001$), which is consistent with rational expectations and the literature [43].

Table 5 summarises the durability properties of the briquettes with the highest density and compressive strength from Table 4.

Despite the addition of the binder containing water to the blends of rice husks and corn cobs, briquette moisture contents in Table 5 appear within range for good quality briquettes ($\leq 12\%$) recommended by the European standards for solid fuels CEN/TS 14961 [54]. The briquette moisture contents in this study can also be compared with the range of 9 to 14 % achieved for binderless corn cob briquettes by Kaliyan & Morey [17]. Curing at 35°C significantly reduced the moisture content of briquettes, as a result of increase moisture loss due to elevated temperature. Briquette expansion was mainly due to longitudinal with an

average of 15 % longitudinal expansion compared with 4 % diametrical. An average reduction in density of briquettes sample A and B was found to be 22 % (Table 5Table 5).

Water absorption of briquettes produced at 3:7 rice husks to corn cobs (repeats of runs 4+,12) is almost twice that of 1:1 rice husks to corn cobs (repeats of runs 3+,15). This may be due to the high ratio of porous corn cobs (Figure 5) in the 3:7 blend of rice husks to corn cobs. The over-saturation observed in briquettes can also be related to the swelling nature of lignocellulosic residues that was observed in the raw feed samples (section 3.1).

Shattering and abrasion resistance of briquettes produced from 3:7 blend of rice husks to corn cobs conform with the <10% mass loss required by standards for quality assurance of solid biofuels CEN/TS 14961[54]. This compares well with 8 to 12% mass loss for corn cob briquettes produced at 150 MPa and 85°C [17]. The difference in porosity of the briquettes and their individual parent materials was relatively small at 8 to 22%. Whereas relatively low compaction pressures were investigated in this work to reduce energy and equipment costs, preliminary results for the 3:7 blend of the B samples of rice husks and corn cobs, without use of a binder, suggest that a relaxed density of 774 kg/m³ could be achieved at an increased compaction pressure of 80 MPa. The blend ratio may also affect the briquetting process energy consumption; for example, there was a decrease in energy consumption when the blend ratio changed from 30/70 to 50/50 (manuscript in preparation).

4 CONCLUSIONS

This study has demonstrated that briquettes of good and consistent quality, which conform to CEN/TS 14961[54], can be produced by blending rice husks and corn cobs.

Statistical analysis of the results showed that the proportion of corn cobs and higher compaction pressure had positive effects on briquette green and relaxed densities, as well as compressive strength. Starch and water binder addition were necessary to achieve measurable unconfined compressive strengths, but significantly reduced the green and relaxed densities of the briquettes. Although the source of the biomass did not affect briquette strength, it had a significant effect on biomass densification.

Further study of the causes and effects of biomass variability is recommended. The negative effect of starch binder on briquette density also indicates the need to explore other sources of binder that do not result in swelling during biomass densification. Further research into the impact of blend ratio on the briquetting process energy consumption is also required. Since

the maximum storage period for briquettes used in this paper was 7 days, it is important to look into the effect of longer storage periods on briquette quality.

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List of abbreviation

M = Material ratio

B = Binder

P = Pressure

S = Sample batch

ST = Starch

p = Probability

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LIST OF FIGURES

Figure 1: Blend of rice husks and corn cobs loaded into the 32 mm diameter mold

Figure 2: Mold with sample subjected to load in compression machine

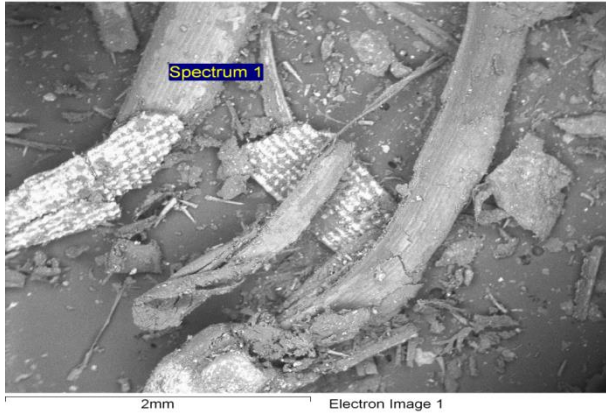
Figure 3: Briquettes produced from blends of rice husks and corn cobs

Figure 4: Morphology of rice husks under SEM (Sample B)

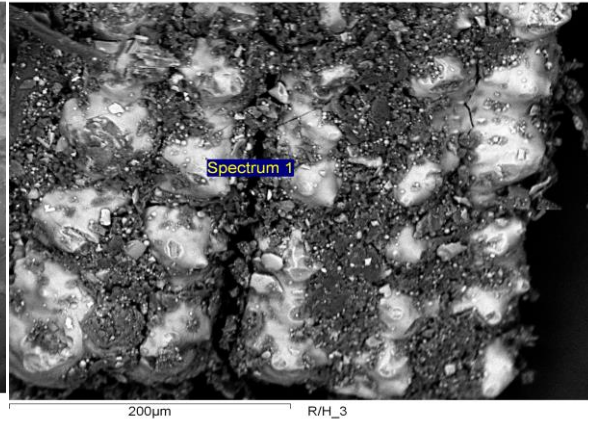
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Figure 6: Normal probability plots of the effects of sample batch (S), material ratio (M), binder addition (B), compaction pressure (P), and their interactions (MS, BS, PS, MB, MP, PB, MBS, MPS, PBS, MPB, on briquette a) green density, b) relaxed density and c) compressive strength

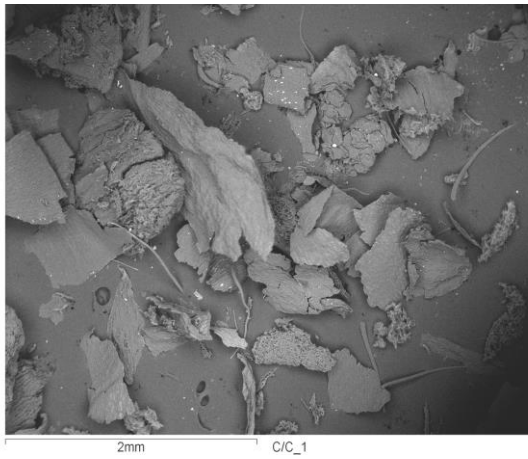




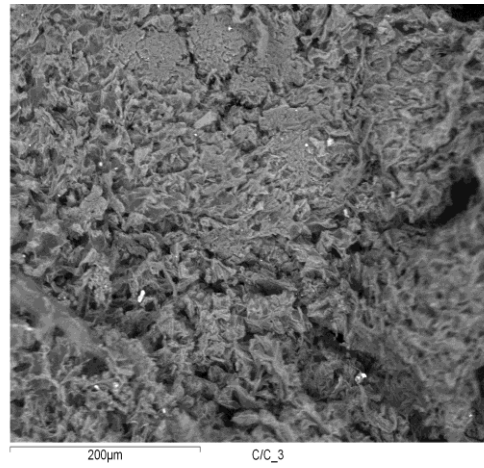
(a)



(b)



(a)



(b)

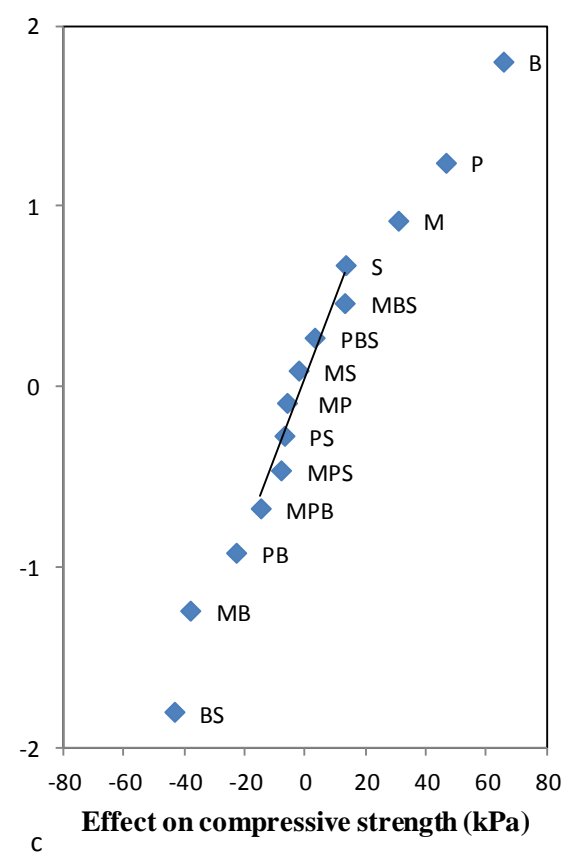
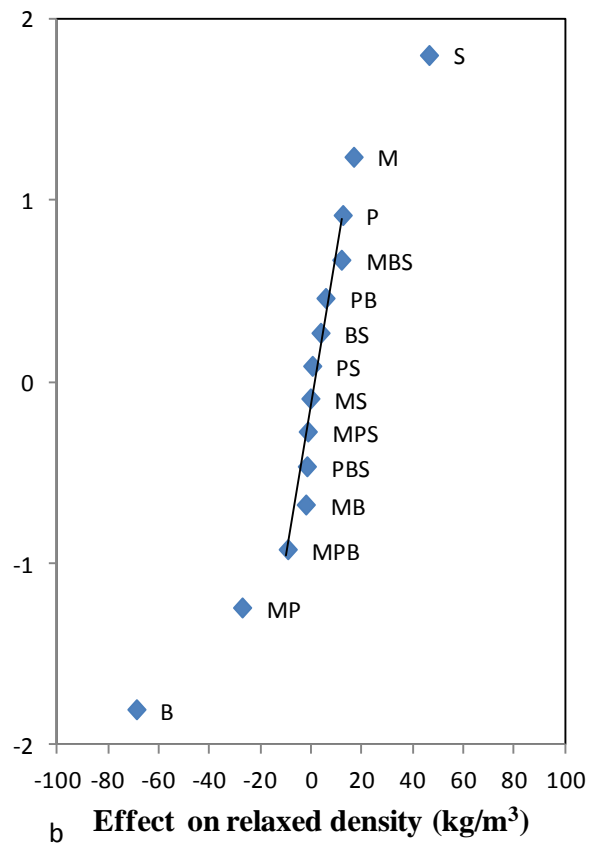
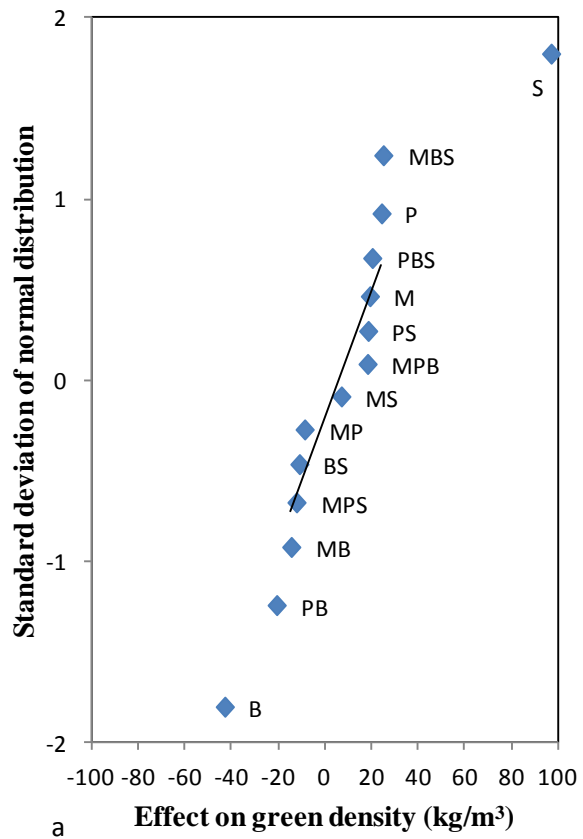


Table 1: Comparison of basic properties of rice husks and corn cobs

Properties	Rice husks	Corn cobs	Reference
Calorific value (kJ/kg dry mass)	16000	18000	[14,16,18,19]
Ash content (% dry mass)	20	<2	[1,14,18,21,22]
Moisture content (% undried mass)	8-12	20-55	[14,17,19,21,22]
Bulk density (unprocessed) (kg/m ³ dry mass)	100-150	160-210	[14,17,21,22,23]
Bulk density (ground to <0.85 mm) (kg/m ³ dry mass)	331-380	282	[21,22]
Porosity (% dry volume)	63-73*	68	[21,22]
Water absorption (% dried mass)	105	327**	[20,23]
Lignin (% dry mass)	19.2	15.3	[25,29]
Protein (% dry mass)	1.8	2.7	[24,26,27]
Starch (% wt dry mass)	<1	1.61	[24,28]]

*range of 4 different types including long and short grain rice

**average water absorption on whole small cobs

Table 2: Briquette characterisation methods

Briquette Property	Method Summary	Standard Test Method	Reference
Unit density	Ratio of cylinder mass to volume	DD CEN/TS 15405:2010	[45]
Moisture content	Mass lost in drying at 105°C ±2	BS EN 14774-2	[38]
Unconfined compressive strength	Failure loading of axially loaded cylinder	ASTM C39-96 (adapted)	[46]
Abrasion resistance	Mass lost in tumbling for 24 h	DD CEN/TS 15639	[47]
Shattering resistance	Mass lost in drop from 1 m		adapted from [48, 49,50,51]
Water absorption	Mass gained after soaking in water at room temperature	BS EN 772-21	[41]

Table 3: Feed material properties (averages of three measurements)

Raw feed sample	Rice husks		Corn cobs	
	Sample A	Sample B	Sample A	Sample B
Ash content (% dry mass)	19.6	ND	4.1	ND
Moisture content (% undried mass)	7.0	7.0	6.9	6.8
Specific gravity	1.50	1.50	1.47	1.46
Bulk density (dried mass, kg/m ³)	363	354	395	278
Porosity (% of uncompact volume)	75	76	73	81
Water absorption (% dried mass)	112	160	168	289
(% saturation of porosity)	48	109	130	251
(% volume change)	29.4	ND	40	ND
Particle size (mm)	<2	<2	<1.6	<1.6

ND = Not determined

Table 4: Briquette density and strength measured in a factorial design with sample batch, material ratio, binder content and compaction pressure

Run	VARIABLES					RESPONSES*					
	Sample batch (S)	Material ratio (M) (% mass of rice husks in rice husk/corn cob blend)	Binder (B)		Pressure (P) (MPa)	Unit Green Density (kg/m ³)		Unit Relaxed Density (kg/m ³)		Compressive Strength (kPa)	
			(% mass of starch in rice husk/corn cob blend)	(% mass of added water in rice husk/corn cob blend)		mean	SD	mean	SD	mean	SD
1	A	50	4	6	19	815	15	616	18	70	4
2	A	30	4	6	19	867	14	671	41	152	7
3	A	50	4	6	31	896	17	673	19	158	1
3+	A	50	4	6	31	830	19	664	8	148	1
4	A	30	4	6	31	874	17	631	11	183	7
4+	A	30	4	6	31	870	11	660	10	179	3
5	A	50	6	11	19	767	40	556	13	151	19
6	A	30	6	11	19	808	26	592	9	155	4
7	A	50	6	11	31	815	36	583	16	171	9
8	A	30	6	11	31	846	25	596	22	175	14
9	B	50	4	6	19	698	23	593	22	25	7
10	B	30	4	6	19	766	10	612	33	64	9
11	B	50	4	6	31	761	21	586	39	59	8
12	B	30	4	6	31	795	9	629	15	189	14
13	B	50	6	11	19	767	4	490	24	168	11
14	B	30	6	11	19	715	20	556	29	191	6
15	B	50	6	11	31	707	27	572	10	237	21
16	B	30	6	11	31	703	15	512	5	177	16
17**	B	50	0	0	19	316	0	316	0	0	0
18**	B	30	0	0	19	607	28	301	0	0	0

Table 4: Briquette density and strength measured in a factorial design with sample batch, material ratio, binder content and compaction pressure

Run	VARIABLES					RESPONSES*					
	Sample batch (S)	Material ratio (M) (% mass of rice husks in rice husk/corn cob blend)	Binder (B)		Pressure (P) (MPa)	Unit Green Density (kg/m ³)		Unit Relaxed Density (kg/m ³)		Compressive Strength (kPa)	
			(% mass of starch in rice husk/corn cob blend)	(% mass of added water in rice husk/corn cob blend)		mean	SD	mean	SD	mean	SD
19**	B	50	0	0	31	659	46	316	0	0	0
20**	B	30	0	0	31	615	37	549	13	0	0
21**	B	50	0	6	19	752	10	623	26	59	4
22**	B	30	0	6	19	791	36	695	14	98	4
23**	B	50	0	6	31	777	16	642	11	48	4
24**	B	30	0	6	31	812	27	712	11	70	4

* Average of three responses; SD is standard deviation

** Experimental runs for effect of water only on briquette responses

Table 5: Durability properties of briquettes at different curing conditions

Briquette properties	Run from Table 4	Curing temperature (°C±2° C)	Curing time (d)	Response (sample A)	Response (sample B)
Unit relaxed density (kg/m ³)	3+	23	1	664	ND
	15	23	1	ND	572
	3 (repeat)	23	7	645	ND
	15 (repeat)	23	7	ND	616
	15 (repeat)	35	1	ND	586
	3 (repeat)	35	1	586	ND
	3 (repeat)	35	7	531	ND
	4+, 12	23	1	660	629
	4, 12 (repeat)	23	7	644	669
	4 (repeat)	35	1	660	ND
4 (repeat)	35	7	600	ND	
Reduction in unit density (after storage) (% of green density)	3+	23	1	20	ND
	15	23	1	ND	19
	3 (repeat)	23	7	28	ND
	15 (repeat)	23	7	ND	13
	15 (repeat)	35	1	ND	17
	3 (repeat)	35	1	35	ND
	3 (repeat)	35	7	40	ND
	4+, 12	23	1	24	21
	4, 12 (repeat)	23	7	26	16
	4 (repeat)	35	1	24	ND
4 (repeat)	35	7	30	ND	
Densification (proportion of average loose biomass density of 348 kg/m ³)	3+	23	1	1.9	ND
	15	23	1	ND	1.6
	3 (repeat)	23	7	1.9	ND
	15 (repeat)	23	7	ND	1.8
	15 (repeat)	35	1	ND	1.7
	3 (repeat)	35	1	1.7	ND
	3 (repeat)	35	7	1.5	ND
	4+, 12	23	1	1.9	1.8
	4, 12 (repeat)	23	7	1.9	1.9
	4 (repeat)	35	1	1.9	ND
4 (repeat)	35	7	1.7	ND	
Moisture content (% undried mass)	3+	23	1	9	ND
	15	23	1	ND	12
	15	35	1	ND	6
	4+, 12	23	1	10	10
Porosity (% volume)	3+	23	1	59	ND
	15	23	1	ND	65
	4+,12	23	1	60	60
Water absorption (% dried mass)	3+	23	1	70	ND
	15	23	1	ND	66
	4+,12	23	1	142	151
(% saturation of porosity)	3+	23	1	118	ND
	15	23	1	ND	100
	4+,12	23	1	237	245
Compressive strength (kPa)	3+	23	1	148	ND
	15	23	1	ND	237
	3 (repeat)	23	7	98	ND

Briquette properties	Run from Table 4	Curing temperature (°C±2° C)	Curing time (d)	Response (sample A)	Response (sample B)
	15 (repeat)	23	7	ND	180
	3 (repeat)	35	1	73	ND
	3 (repeat)	35	7	60	ND
	4+, 12	23	1	179	189
	4, 12 (repeat)	23	7	167	167
	4 (repeat)	35	1	135	ND
	4 (repeat)	35	7	130	ND
Shattering resistance	3+	23	1	14	ND
(% undried mass loss)	15	23	1	ND	4
	4+,12	23	1	9	11
Abrasion resistance	3+	23	1	20	ND
(% undried mass loss)	15	23	1	ND	3
	4+,12	23	1	3	4