

Utilization of Lignin from Waste Degumming Liquor as Fuel Additive and Binder in Sugarcane Bagasse Briquettes

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Keywords: briquette quality, briquetting, lignin, binder, sugarcane bagasse.

Abstract. As the world shifts from a fossil-based to bio-based economy, energy production via the valorization of biomass residues is promising. However, raw biomass utilization for energy production proves to be challenging owing to their low bulk densities and hygroscopic nature. These problems are addressed through briquetting. This work focuses on the utilization of sugarcane bagasse (SCB) – an abundant agricultural residue in the Philippines – with lignin as a binder for solid fuel briquettes. The effects of SCB-to-lignin ratio and compaction pressure on the fuel characteristics and quality of the briquette were investigated. Briquetting experiments were carried out in a Carver press at a constant pressing temperature of 150°C and varying compaction pressures (CP = 5, 8 and 11 MPa) and binder content (BC = 4, 8 and 12%). Briquetting of SCB resulted in an increase in bulk density by 7 to 8 times (0.817 ± 0.006 - 0.974 ± 0.029 g/cm³) compared to loose SCB (0.110 ± 0.000 g/cm³). From ANOVA, it was found out that CP significantly affects bulk density ($p < 0.05$). The higher heating value (HHV) and compressive strength (CS) rose to as high as 19.7 ± 0.1 MJ/kg_{db}, and 113.14 ± 2.81 MPa, respectively. It was found out that BC significantly affects the HHV ($p < 0.05$) but has an insignificant effect on the CS ($p > 0.05$) while CP has insignificant effects on both HHV and CS ($p > 0.05$). Overall, SCB-lignin briquettes are able to overcome storage and handling challenges through improved bulk density, stacking capability of up to ~550,000 briquettes at the lowest achieved CS, retarded moisture uptake rate (0.0611 ± 0.0017 wt% per hr) and an equilibrium moisture content of 8.02 ± 0.15 wt%, making this fuel viable for domestic usage as determined by current briquetting standards.

Introduction

At present, the world is strongly dependent on the use of fossil fuels for energy production and continued dependence will ultimately lead to depletion of fossil reserves, as well as aggravate the effects of global warming. A statistical review of world energy by British Petroleum Global showed that the fossil fuel demand in 2023 constitutes 81% of the world's energy consumption. Fossil fuels include oil, gas and coal contributing 32%, 23%, and 26%, respectively in the global energy mix[1]. Around the world, initiatives have been done to move from today's fossil-based economy to a more sustainable economy that is based on renewable resources such as biofuels from biomass residues as alternatives for fossil fuels and coal. Utilization of biomass residues not only effectively closes the carbon cycle [2] but also eliminates the problem biofuel production poses on the food industry where the production of biofuels competes with the food industry for raw materials [3].

The Philippines, being an agricultural country, generates a considerable amount of biomass residues. According to a 2020 report by the Philippine Statistics Authority (PSA), 53.7% of the total

agricultural output is attributed to crop production. Sugarcane has the highest production percentage out of all the agricultural crops, amounting 17.408 million metric tons for the 1st two quarters of 2020 [4]. From this thriving agricultural crop, 15.1 kg of dry trash per 100 kg stalks or harvested cane could be recovered for utilization as a bioenergy source [5]. A type of residue that is produced from the processing of sugarcane is sugarcane bagasse (SCB). It is the fibrous residue of sugarcane after the crushing and the extraction of juice. SCB is mainly composed of cell-soluble matter, cellulose, hemicellulose, lignin, ash, crude protein, and glucose [6].

Over the last two decades, the country has endeavored to improve the management and operation of the biomass residues through the implementation of several national laws such as the “Renewable Energy Act of 2008” (RA 9513). In the aspect of utilizing biomass residues to generate energy, SCB is directly burned to generate heat. Although direct combustion practically consumes the entirety of the biomass fuel and gives off energy, it is possible that the fuel is not utilized to its fullest potential due to certain factors such as high moisture content, irregular shape and sizes, and low bulk density. These factors pose difficulty for transportation, storage, handling and energy content [7]. Thus, a more efficient method would be to densify the biomass. Biomass densification is a means of increasing the energy density of biomass by mechanical or thermochemical means before feeding it for combustion [8]. Mechanical densification involves the application of pressure to densify the material. Briquetting and pelletization are examples of mechanical densification techniques. This is a relatively simple and cost-effective densification technique compared to thermochemical densification such as pyrolysis which is described as a relatively complex process that entails additional energy and cost [9]. Bales, pellets, and briquettes are products of mechanical densification whereas bio-oil and bio-char are products of thermochemical densification [9].

Briquetting is a mechanical treatment to compress loose biomass into a product with higher density, energy content and with less moisture [10]. Briquettes and their characteristics mainly depend on the feedstock, process conditions and equipment used. For instance, the HHV of the fuel is mainly dictated by the inherent calorific value of its biomass components. The compacting pressure affects mainly the briquette strength, density and consequently loss of moisture of the biomass fuel [13]. The moisture uptake and equilibrium moisture content of the fuel is highly dictated by the storage conditions (e.g. relative humidity, temperature) and feedstock characteristics. The particle size of the components also affects the compressive strength and density of briquettes [13].

Briquettes may also be enhanced with binders. Binders aid in the agglomeration of the biomass particles through adhesion and cohesion forces, attractive forces between solid particles, mechanical interlocking and solid bridge type mechanisms [14]. This results in improved mechanical strength of the briquette while also reducing the energy required for compression of biomass [15]. A promising binder to be used in solid fuel applications is lignin. Lignin is a biopolymer which comprises ~20% of lignocellulosic material. It is a waste product of industries that are based on cellulose, hemicelluloses, and their derivatives [16]. Such industries include pulp and paper, textile, and bioethanol production. Utilization of this by-product as a binder and fuel additive is attractive since it is capable of forming solid bridges between particles which increases the mechanical integrity of the densified biomass [17] while positively contributing to the fuel calorific value with increasing binder concentrations [18,19]. A study by Hu et al. [20] showed that bio-char pellets added with 10% lignin as binder have increased compressive strength, decreased compression energy, and lowered ignition temperature. However, moisture uptake increased with increasing binder concentrations but still within acceptable limits. The improvement in these properties showed that lignin binders show good potential for utilization in biofuels.

The goal of this work was to improve biomass briquette quality with the addition of lignin from industrial black liquor. Despite the potential of lignin as a fuel additive and binder, studies in literature focusing on lignin-binder biofuels have been scarce. In order to address this gap, as well as the viability of using lignin-binder SCB briquettes for fuel applications, this study aimed to investigate effects of SCB:lignin ratio and compression pressure on the briquette quality (bulk density, higher heating value and compressive strength) and fuel characteristics (briquette proximate composition, combustion indices, burning rate and equilibrium moisture content). A 32 factorial design was

employed to evaluate the effect of SCB:lignin ratio (4%, 8%, 12%) and compaction pressure (5 MPa, 8 MPa, 11 MPa) at constant briquetting conditions of 150°C at 30-minutes pressing time.

Materials and Methods

Sample Acquisition and Preparation.

SCB was sourced from BOMEDCO, a sugar-milling company in Bogo, Cebu. The collected samples were sun-dried then milled using a Wiley Mill (Thomas-Wiley Model 4, New Jersey, USA) equipped with a 2-mm mesh aperture screen. The milled SCB were stored in polypropylene boxes for subsequent characterization and briquetting.

Lignin (LGN) was sourced from the waste degumming liquor of the fiber-processing plant of the Department of Science and Technology - Philippine Textile Research Institute (DOST-PTRI) in Taguig City. The precipitation procedure followed was based on the study by Alriols et al. [21]. One (1) liter of liquor was transferred to a 2-L glass beaker. The liquor was then acidified to pH 2 by slowly adding concentrated sulfuric acid. Brown precipitates of lignin are expected to appear during the pH adjustment. The resulting mixture was placed in a water bath (25°C) until the precipitate settled at the bottom of the vessel and formed a big clump. The supernatant liquid was removed and the precipitate was filtered using a cheesecloth. The filter cake was then washed with warm water and the pH of the spent wash was measured using a pH meter after each washing. The washing step was stopped after the pH of the spent wash became constant (around pH 5.5). The washed solids were then dried in a vacuum tray dryer at 60°C and 300 mmHg until a constant weight reading was achieved in the dryer balance. The dried solids were then reduced in size with a 1000W food processor at 20,000 rpm (BN1000W Gorenje) and stored in a polypropylene container for subsequent characterization and briquetting.

SCB and LGN Characterization.

The dried SCB was characterized in terms of the mean particle size, loose and tapped bulk density, proximate composition namely, moisture content (MC), volatile matter (VM), fixed carbon (FC) and ash, higher heating value (HHV), whereas LGN was characterized in terms of the proximate composition and HHV only.

SCB and LGN Characterization: Particle Size Distribution.

Pre-weighed standard Tyler sieves with sieve apertures of 2mm, 850µm, 450µm, 250µm, and 180µm were stacked in descending order of mesh size. About 50 g of milled SCB was placed on top of the sieve having 2mm aperture size and covered. The stack of sieve trays was then mounted to a sieve shaker (Mod. A5911, Interest Benelux, Netherlands), and was shaken for 15 minutes. After shaking, the mass of SCB retained on each of the sieve was determined [13]. The mean particle size was then calculated using Equation 1.

$$d_m = \sum \frac{m_i}{m_T} d_{ave} \quad (1)$$

where m_i (g) is the mass of sampled retained on a particular sieve tray, d_{ave} (mm) is the average size of the mesh through which the SCB samples have passed through and on which it was retained, and m_T (g) as the total mass of the sample.

SCB and LGN Characterization: Loose and Tapped Density Determination.

The loose and tapped densities of the raw SCB were determined based on the standard method in ASTM D7481-09 [22]. A pre-weighed and pre-dried 100-mL graduated cylinder was placed on an analytical balance and tared, SCB was poured into the cylinder until a set mass was obtained. The loaded cylinder was weighed to the nearest 0.1 mg. The loose density ρ_{loose} was determined using Equation 2.

$$\rho_{loose} \left[\frac{g}{mL} \right] = \frac{m_{loaded} - m_{empty}}{v_{loose}} \quad (2)$$

where m_{loaded} is the mass of the loaded graduated cylinder, and m_{empty} (g) is the mass of the empty cylinder, and v_{loose} (mL) is the volume occupied by the loose biomass. For tapped bulk density ρ_{tapped} , the cylinder was tapped for 500, and 1250 times from a height of 3 mm onto a flat and hard surface to settle the loose biomass and their corresponding volume measurements were recorded. If the difference between the volume of the biomass tapped for 500 and 1250 times was less than 1 mL, the volume reading tapped at 1250 times will then serve as the tapped volume of the biomass. If the difference was greater than 1 mL, additional tapping (increments of 1250 taps) will be done until the difference of less than 1 mL was achieved. The value was then used as the tapped volume v_{tapped} and was then used in Equation 3.

$$\rho_{tapped} \left[\frac{g}{mL} \right] = \frac{m_{loaded} - m_{empty}}{v_{tapped}} \quad (3)$$

SCB and LGN Characterization: Proximate Analysis.

The standard test method for chemical analysis of wood charcoal by the American Society for Testing and Materials (ASTM) procedure D1762-84 [23] was used for proximate analysis. SCB, lignin, and SCB briquettes were characterized in terms of their proximate components, namely moisture, volatile matter, ash, and fixed carbon. The proximate analysis was carried out twice for each sample following the method described in NREL/TP-510-42622 [24] and ASTM D1762-84 [23]. To ensure that any residual materials adhering to the crucibles and their covers were volatilized, the crucibles and covers were pre-fired by heating them at 950°C for 1 hour, and cooling to room temperature, prior to the analysis. About 1 gram of sample (SCB, Lignin, or SCB briquettes) was weighed (to the nearest 0.0001 g) into the pre-fired and pre-weighed crucibles. Moisture content was determined by placing the sample-containing crucibles in a convection oven (Memmert Oven, Model UN50) at 105°C for 2 hours then cooled in a desiccator for 30 minutes prior to weighing using an analytical balance (Kern ABJ-NM/ABS-N, Model ABS 220-4N). The drying procedure was repeated until a constant weight was obtained, that is, the change in the calculated moisture content is less than 0.05 %. The percent moisture content, MC (wt.%) was then calculated using Equation 4.

$$MC (\%) = \frac{m_s - [(m_{sc})_{MC} - (m_c)_{empty}]}{m_s} \times 100 \% \quad (4)$$

where m_s is the mass (g) of the sample before drying, m_c is the mass (g) of the dry crucible, and m_{sc} is the mass (g) of the crucible containing the sample, $(m_{sc})_{MC}$ is the mass (g) of the crucible containing the sample after MC determination. Volatile matter content was then determined by covering the crucibles containing the samples obtained from moisture analysis with pre-fired crucible lids before placing in a furnace (PF3/SPEC, Vecstar, UK) set at 950°C. With the furnace door open, the crucibles were placed on the outer ledge of the furnace for 2 minutes, then moved to the edge of the furnace for 3 minutes. Afterwards, the crucibles were then transferred to the rear of the furnace with the furnace door closed. The crucibles were then taken out from the furnace after 6 minutes, allowed to cool for 15 minutes in a stainless-steel tray, then placed into the desiccator for 30 minutes before weighing. The percentage of volatile matter, VM (wt.%), was calculated using Equation 5. The lid was not included in the weighing.

$$VM (\%) = \frac{(m_{sc})_{MC} - [(m_{sc})_{VM} - (m_c)_{empty}]}{m_s} \times 100\% \quad (5)$$

where $(m_{sc})_{VM}$ is the mass (g) of crucible containing the sample after heating at 950°C and MC is the fractional moisture content determined previously. Afterwards, the uncovered crucibles containing the sample was heated in a muffle furnace (PF3/SPEC, Vecstar, UK) at 575°C for 4 hours. After heating, the crucibles containing the residue were taken out of the furnace, allowed to cool for

15 minutes in a stainless-steel tray, then placed into the desiccator for 30 minutes before weighing. The percentage of ash, Ash (wt.%) was calculated using Equation 6.

$$Ash (w/w) = \frac{(m_{sc})_{Ash} - (m_c)_{empty}}{m_s(1 - M)} \times 100\% \quad (6)$$

where $(m_{sc})_{Ash}$ is the mass (g) of the crucible containing the sample after heating at 575 °C. The percentage of fixed carbon of the sample, FC (wt.% of fixed carbon in sample), was calculated by difference using Equation 7(7).

$$FC (w/w) = 100\% - VM - Ash \quad (7)$$

SCB and LGN Characterization: Higher Heating Value Determination.

The higher heating value of the pre-treated samples were determined using an adiabatic bomb calorimeter (Parr 1108 Oxygen Combustion Bomb) and procedures prescribed by the Parr Instruments operating manual [25, 26] which are compliant with ASTM D2015-96 [27]. About 0.7-1.1 g of sample, accurately measured to the nearest 0.1 mg, was used for every analysis. Nickel-chromium fuse wire (Parr 45C10) was used to ignite the samples. About 10 cm of the wire was used for each run and the length of the wire after ignition was recorded for correction of the actual heat released by the consumed wire. Temperature readings were recorded prior to firing, at 30-second intervals for 5 minutes after firing, then at 1-minute intervals for the next 7 minutes. The correction for nitric acid formation was adjusted using standard sodium carbonate (0.0709 N) titration while there was no correction for sulfur [28]. The HHV was determined using Equation 8.

$$HHV \left[\frac{MJ}{kg} \right] = \frac{C\Delta T - (\Delta \hat{H}_{HNO_3}^f + (l_{wire,i} - l_{wire,f})\Delta H_{wire}^c)}{m_s} \quad (8)$$

where C is the calorimeter constant (6.03 kJ/°C), ΔT is the temperature change from before ignition to the temperature reading at the 12th minute after ignition (°C), $\Delta \hat{H}_{HNO_3}^f$ is the heat of formation of nitric acid (kJ) which is determined by the volume of titrant (4.18×10^{-3} kJ/mL of titrant), $l_{wire,i} - l_{wire,f}$ is the change in wire length measured before and after ignition (cm), ΔH_{wire}^c is the heat of combustion of the fuse wire (9.6232×10^{-3} kJ/cm for Parr 45C10), and m_s is the mass of the sample or standard benzoic acid (grams) used in the ignition. Equation 9 was used to express HHV in dry-basis.

$$HHV_{ab} \left[\frac{MJ}{kg} \right] = \frac{HHV}{1 - \frac{\% MC}{100}} \quad (9)$$

Briquetting.

The mold used is an assembly of 3 parts namely the mold piston Fig. 1a, the mold cylinder Fig. 1b, and the mold base Fig. 1c. The mold was designed to produced briquettes with dimensions compliant to the Austrian ONORM M7135 standard for briquettes.

Sugarcane bagasse and lignin binder mixtures of 25 grams with different ratios (0 wt.% lignin, 4 wt.% lignin, 8 wt.% lignin, and 12 wt.% lignin) were prepared. Exact amounts of SCB and LGN for a specific ratio are illustrated in Table 1. A beaker was placed on a top loading balance and tared. SCB was poured first into the beaker until a specified mass was obtained. LGN was then added to the beaker until the total mass of the mixture reached 25 g. It was then mixed thoroughly using a spatula. The prepared mixtures were carefully poured into two of the six cylinders (42.5 mm inner diameter) using a funnel. The remaining cylinders were loaded with pure SCB (~25g) to act as fillers to avoid mold damage. After each compartment was loaded with the mixtures, the piston was gently placed on the cylinders. The mold was placed on the base of the hydraulic press (Carver Hydraulic Press, Model C) preheated at 150°C (Figure 1e). The mold was centered well on the preheated base. Once the briquette mold was in place, the hydraulic jack was pumped to obtain the desired compaction pressure (5 MPa, 8 MPa, 11 MPa). After the pressure was reached, the pressed biomass was held at

the desired pressure for 30 minutes. After 30 minutes, the release knob on the hydraulic press was turned counter clockwise to release the pressure. After releasing the pressure, the briquette mold was removed from the press and the briquettes were removed from the mold. The weight and the dimensions of the briquettes were measured directly after compaction and then stored in re-sealable polyethylene bags for subsequent characterization and testing.

Table 1. Formulations of 25-g briquettes and number of briquettes produced per mixture proportion.

Mixture proportions	Mass of lignin (g)	Mass of SCB (g)	No. of briquettes produced
0% LGN	0	25	6
4% LGN	1	24	6
8% LGN	2	23	6
12% LGN	3	22	8

In order to assess the briquette quality, European standards for the domestic use of biomass briquettes were employed. The Austrian ONORM M7135 and the EN 14961-3 were considered in this study. Although these standards are based on compressed wood and compressed bark, which may not be necessarily applicable for herbaceous biomass like SCB, they provide a means to classify the quality of the briquette [13]. Details of each standard are listed in Table 2.

Table 2. European standards for the domestic use of biomass briquettes [29].

Standard	Diameter/Length (mm/mm)	Bulk Density (g/cm ³)	Moisture content (wt.%)	Heating Potential (MJ/kg)	Ash content (wt.%)	Sulfur content (wt.%)
Ö-NORM M7135 (Austria)	40-100/up to 1000	≥1	≤10	≥18	≤0.7	≤0.08
EN 14961-3 (Europe)	>25-125/ up to 400	≥0.9	≤15	≥15.3	≤0.7	≤0.04

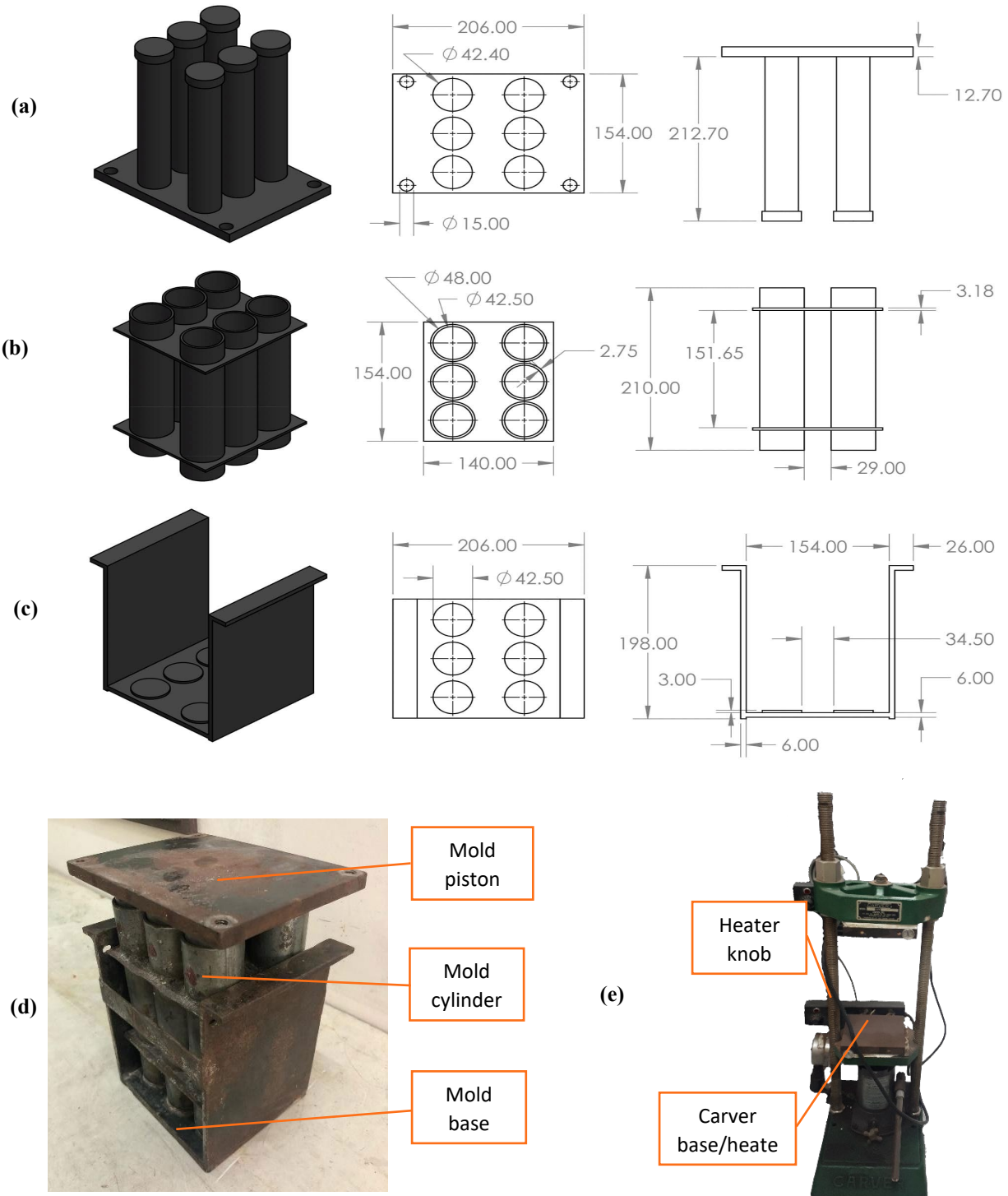


Fig. 1. Redrawn schematic diagram based on measurements used for fabrication.: (a) Mold piston isometric, top & front view; (b) 6-cylinder mold isometric, top, & front view; (c) mold base isometric, top, & front view; (d) briquette mold assembly isometric view; (e) Carver press.

Briquetting: Briquette Density.

After the briquetting process, the briquettes were weighed and corresponding dimensions were measured. The diameter d_{briq} and height h_{briq} of the briquettes were measured using a Vernier caliper. In measuring the diameter and the height of the briquette, 2 sides of the briquette were measured. The average of the two sides were then calculated and the average value was then used as the d_{briq} and the h_{briq} . The briquette density was calculated using Equation 10.

$$\rho_{briq} \left[\frac{g}{cm^3} \right] = \frac{4m_{briq}}{\pi(d_{briq}^2)(h_{briq})} \quad (10)$$

where m_{briq} is the mass of briquette immediately after briquetting. The density ratio and degree of compaction are then determined to evaluate the briquetting performance.

$$Density\ Ratio = \frac{\rho_{briq}}{\rho_{loose}} \quad (11)$$

$$Degree\ of\ Compaction = 1 - \frac{V_{briq}}{\frac{m_{biomass}}{\rho_{loose}}} \quad (12)$$

Briquetting: Moisture Reabsorption.

The moisture reabsorption was determined using procedures prescribed by Conag et al. [28]. Briquette samples were oven dried at 105°C for 3 hours. The dried briquette sample was weighed and thus taken as the initial mass of the dried briquette. The briquette sample was exposed to atmospheric conditions for a test period of 15 days. Mass of the briquettes were recorded at fixed intervals (1-hour intervals for the first 12 hours, then 12-hour intervals in the succeeding days). Weight (in grams) acquired by the briquette was taken as reabsorbed moisture. Wet bulb temperature, dry bulb temperature and relative humidity (RH) readings were also determined using a sling psychrometer at the same intervals as weight readings.

Briquetting: Normalized Burning Rate.

An iron stand with a wire mesh was mounted on a receiving tray on top of a top loading balance. An alcohol lamp was positioned such that it does not contribute to the total system mass. Briquette samples of known weight were loaded on the wire mesh and the lamp ignited. The rate of mass loss of the briquette was monitored at 10-second intervals using a stopwatch until no change in mass was observed. Ash was not removed for the entirety of the burning period. Readings were recorded as soon as the flame contacted the briquette.

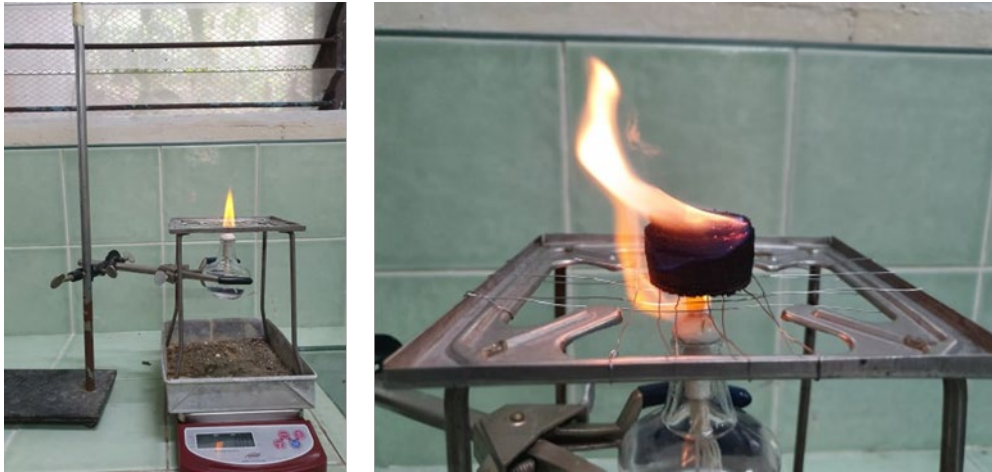


Fig. 2. Normalized burning rate setup.

The determination of the burning time was based on the work of Chaney et al. [30]. There are three phases of the burning process: Phase 1 is the ignition phase where no drastic change in mass occurs; phase 2 is the steady state flaming combustion phase where the fuel is enveloped in a luminous flame (refer to Fig. 2) and it starts with a sharp decrease in mass which continues to decrease over time and; phase 3 is when the flame dies and the briquette decomposes further by char combustion mechanism. Only phase 2 of the burning process was considered. Equation 13 was used to determine the normalized burning rate.

$$NBR \left[\frac{\% \text{ loss}}{\text{min}} \right] = \frac{(m_{briq,i} - m_{briq,f})/m_{briq,i}}{t_{burn}} \times 100\% \quad (13)$$

where $m_{briq,i}$ and $m_{briq,f}$ initial and final mass of briquette, respectively, and t_{burn} is the time elapsed during the steady state flaming combustion phase.

Briquetting: Compressive Strength.

The compressive strength of the briquette was measured using the Universal Testing Machine (UTM) WEW-600, Serial no. 195, following the procedures by Emerhi [31]. The briquette samples were mounted to the UTM and the compressive load was applied to the sample. The load was increased until the briquette started to fracture and the compressive load was recorded immediately. Equation 14 was used to calculate the compressive strength, CS.

$$CS [MPa] = \frac{F_c}{A} \quad (14)$$

where F_c , the compressive load exerted the moment the briquette started to fracture in Newtons, which was specifically determined by using the stress-strain diagram, and A is the surface area of the briquette sample (mm^2). From the compressive load data F_c , the number of stackable briquettes was calculated.

$$F_{\text{weight of briquette}} [N] = m_{\text{briquette}} \times g \quad (15)$$

$$N_{\text{stackable}} = \frac{F_c}{F_{\text{weight of briquette}}} \quad (16)$$

where $m_{\text{briquette}}$ (kg) is the mass of briquette, g (m/s) is the gravitational acceleration constant and $N_{\text{stackable}}$ is the number of stackable briquettes.

Briquetting: Briquette Combustion Indices.

The proximate analysis and higher heating value of the briquetted samples are determined based on the procedures described in the previous subsections. The potential of the briquettes to be co-fired with conventional coal was assessed by obtaining the fuel ratio FR [32], combustibility index CI [33], and the volatile ignitability VI [34]. These were computed using Equations 17, 18, and 19.

$$FR = \frac{\% FC_{\text{briquette}}}{\% VM_{\text{briquette}}} \quad (17)$$

$$CI \left[\frac{MJ}{kg} \right] = \frac{HHV_{\text{briquette}}}{FR} \times (115 - \% Ash_{\text{briquette}}) \times \frac{1}{105} \quad (18)$$

$$VI \left[\frac{MJ}{kg} \right] = \frac{HHV_{\text{briquette}} - 0.338(\% FC_{\text{briquette}})}{\% VM_{\text{briquette}} + \% MC_{\text{briquette}}} \times 100 \quad (19)$$

Experimental Design and Statistical Analysis.

In this study, the Minitab® 19 software [35] was used to map out the design of the experiment and subsequent statistical analysis. Three response variables namely: bulk density, higher heating value (HHV) and compressive strength (CS), are of interest in this work. A 32-full factorial experimental design was employed by varying two input factors: compaction pressure (CP) and lignin content (LC); with three levels each: low, intermediate, and high. The runs are done in replicates of two, giving a total of 18 replicates along with 6 replicates of binder-less briquettes used as control. The factors along with their corresponding levels are tabulated in Table 3.

Table 3. Factors and corresponding levels of the 32-full factorial experimental design.

Level	Compaction Pressure (MPa)	Lignin Content (wt. %)
Low	5	4%
Intermediate	8	8%
High	11	12%

An analysis of variance (ANOVA) on the gathered data was also done to identify the significance of the main and interaction effects.

Results and Discussions

Raw Material Characterization.

Physical properties such as bulk density, particle size, and proximate composition of both SCB and lignin, as well as their higher heating values (HHV) are summarized in Table 4. The physical properties such as bulk density and particle size of the biomass are important considerations for transportation and storage purposes. Sugarcane bagasse (as received) was analyzed to have a moisture content of $10.21 \pm 0.28\%$. The samples were sun-dried and milled passing through a 2-mm mesh and physical properties such as bulk density and particle size were determined.

Table 4. Characteristics of sugarcane bagasse and lignin.

Characteristics	Sugarcane Bagasse			Lignin		
	Experimental Values	Literature Values	Reference	Experimental Values	Literature Values	
Bulk Density [g/cm³]						
Loose	0.110 ± 0.000	0.10 ± 0.00	[34]	-	-	
Tapped	0.170 ± 0.000	0.15 ± 0.01	[34]	-	-	
Average Particle Size [mm]	0.61 ± 0.01	0.56 ± 0.01	[34]	-	-	
Moisture (as received)	10.21 ± 0.28	-		-	-	
Proximate Components						
Moisture, M [%]	8.14 ± 0.10	$7.53 \pm .08$	[34]	8.12 ± 0.19		
Volatile Matter, VM^{db} [%]	83.10 ± 0.24	72.08 ± 0.56 80.27 78.60 ± 7.70	[34] [36] [37]	81.60 ± 0.93	59.6^{Kr} ; 72.81^K & 72.08^W	[38] [39]
Fixed Carbon, FC^{db} [%]	14.65 ± 0.90	18.23 ± 0.49 14.72 9.45 ± 2.45	[34] [36] [37]	17.84 ± 0.93	39.6^{Kr} ; 26.19^K & 27.16^W	[38] [39]
Ash^{db} (ash)	2.26 ± 0.11	9.69 ± 0.49 5.01 1.80	[34] [36] [37]	0.56 ± 0.00	0.8^{Kr} ; 1.00^K & 0.79^W	[38] [39]
Higher Heating Value, HHV^{db} (MJ/kg)	18.2 ± 0.3	17.5 16.2 ± 2.0	[34] [37]	22.6 ± 0.1	23.6 - 25.58	[19]

^{db}on dry basis; ^{Kr}value for Kraft lignin; ^Kvalue for Klason lignin; ^Wvalue for Willstatter lignin

Lignin used by this study was extracted using an alkaline extraction process devised by DOST-PTRI and precipitated using an acid precipitation method by Alriols et al. [21]. This is different from the extraction processes used by the cited references in Table 4 which were obtained using Kraft, Klason, and Willstatter lignin extraction techniques. The main difference between Klason and Willstatter hydrolysis extraction methods is that the former uses dilute H₂SO₄ and the latter uses dilute HCl.

Raw Material Characterization: SCB Density and Particle Size.

Bulk densities of SCB as determined by this work is 0.110 ± 0.000 g/cm³ (loose) and 0.170 ± 0.000 g/cm³ (tapped), which is only slightly higher compared to literature with values of 0.10 ± 0.00 g/cm³ (loose) and 0.15 ± 0.01 (tapped) g/cm³[34]. The loose and tapped density are used to characterize the flowability of particles. At large scale productions of briquettes, the ease of handling the raw materials would be a significant factor to efficiency, thus it is important to determine the loose and tapped densities.

The particle size of biomass is important in terms of storage and transportation since fuel with very small particle size could get easily blown by the wind thus needing additional storage equipment and more expensive equipment to transport. For briquetting, the particle size of the feedstock also has an effect on the quality of the produced briquette.

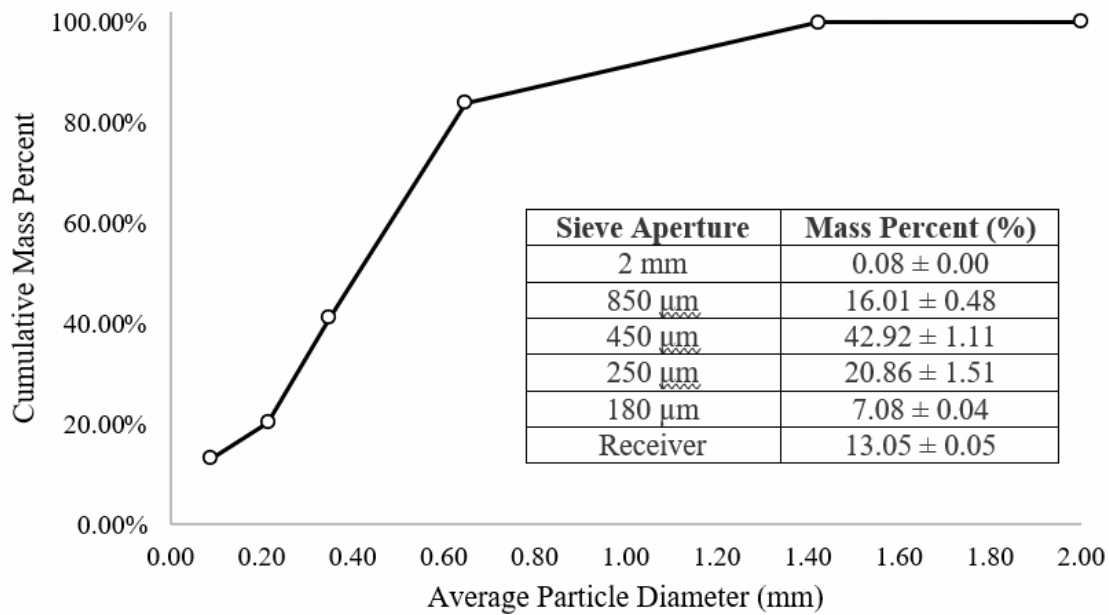


Fig. 3. SCB particle size distribution from sieve analysis.

The milled SCB used for briquetting in this study has an average particle size of 0.61 ± 0.01 mm with a particle size distribution as shown in Fig.3. Particles with sizes ranging from 0.45 to 0.85 mm make up 58.93% of the distribution. Generally, biomass particles of 6 to 8 mm in size give the best briquetting results. However, it is also desirable to have a random distribution of particle size to make sure that there is an adequate amount of small particles that is present for embedding in the larger particles [41]. The longer fiber lengths of the biomass promote mechanical interlocking between the particles increasing the strength of the resulting briquette [42]. The SCB used has a wide particle size distribution, with particles having sizes greater than 0.45 mm making up 59.01% while those less than 0.45 mm making up 40.99%. The presence of particles with different sizes improves the packing dynamics and contributes to a high strength [43], making the SCB feedstock used suitable for producing strong briquettes.

Raw Material Characterization: Proximate Analysis and Higher Heating Value.

After drying and milling, a separate sample of SCB was further characterized for its proximate components. For solid fuels, moisture is an unwanted component as it significantly lowers the heating value of the solid fuel and may result in incomplete combustion of the combustible matter, the deposition of unburnt carbon around the equipment and might cause a difficulty in ignition. Decreasing the moisture content of the biomass before briquetting is also important in complying with briquette standards. The moisture content of as-received SCB decreased from 10.21% to 8.14% after drying and milling, therefore is desirable since the chosen standards for briquette quality allows at most 15% moisture. Also, a study by Grover & Mishra [41] observed that when a feedstock has 8-

10% moisture, the briquettes would be strong, free of cracks and the briquetting process would be smooth. Compared to other biomass used for densification listed in Table 5, moisture of SCB was within the range of values found in literature. Aside from moisture, the amount of volatile matter (VM) and fixed carbon (FC) significantly influences the heating value of the biomass. The VM and the FC represent the components of carbon, hydrogen and oxygen present in the biomass. VM volatilizes when heated, leaving FC as the remaining source of carbon in the biomass. Heating value is proportional to the carbon and hydrogen content of the biomass thus, HHV is assumed to be a function of FC and VM. Compared to other biomass, The FC of SCB falls within the range of values found in literature, however, the VM of SCB is relatively higher compared to literature. This explains why the HHV of SCB has a larger HHV value compared to the mentioned biomass, thus making it a desirable feedstock for solid fuels. Ash is a non-combustible component that lowers the heating value of the solid fuel as its amount (wt%) increases. Also, the ash content of a biomass is an indicator of slagging behavior of biomass. Generally, the greater the ash content, the greater the slagging behavior. In Table 5, SCB has the lowest ash content compared to the other biomass. [18, 19, 37, 44, 45].

In this study, SCB and lignin were found to have heating values of 18.18 ± 0.20 and 22.30 ± 0.44 MJ/kg, respectively. Both materials are found to have HHV values close to their respective literature values in Table 4 [19, 28, 37]. The difference between the raw material HHVs can be traced back to the difference in their respective proximate components. The VM of SCB is only slightly higher compared to that of the lignin samples, however, there is a large difference between their FC and ash contents as observed in Table 4. The FC of SCB is lower than that of the FC of lignin and the ash content of SCB is higher compared to that of the SCB samples. This explains the higher HHV of lignin compared to SCB where it coincides with a study done by Cordero et al. [46] that VM, ash and FC content has increasing significance to the HHV value of lignocellulosic and carbonaceous materials. Lignin contains very low ash content, low MC, and high values of FC and MC, thus making it a desirable feedstock for biomass fuels. In addition, it has good binding properties making it a very suitable fuel additive and binder.

Table 5. Proximate compositions and higher heating values of common feedstocks used in densification.

Biomass Feedstock	Proximate Composition (%wt.)				HHV (MJ/kg)	Reference
	Moisture	Ash	FC	VM		
Sugarcane bagasse	8.14 ± 0.10	2.26 ± 0.11^a	14.65 ± 0.90^a	83.10 ± 0.24^a	18.19 ± 0.30^a	This study
Rice bran	11.40 ± 0.08	16.17 ± 0.71	17.14 ± 0.19	66.69 ± 0.64	17.18 ± 0.14	[13]
Rice husk	-	16.22	16.08	67.70	15.44^a	[20]
Rice straw and Sugarcane leaves ^b	4.2 – 6.2	7.84 – 12.85	9.06 – 13.63	68.18 – 74.67	16.33 – 17.83	[47]
Corn cob	9.71	2.97	71.21	16.11	16.65	[48]
Pigeon pea stalk	5.96	10.62^a	7.25^a	83.13^a	14.81^a	[49]
Cotton stalk	10.2	12.13^a	14.00^a	73.87^a	15.72^a	[49]
Soy stalk	11.84	10.39^a	7.22^a	82.39^a	14.66^a	[49]

^aon dry basis ^b range of compositions from pure rice straw to pure sugarcane leaves.

Briquette Characterization: Proximate Analysis.

Compared to as received loose SCB (MC = $11.09 \pm 0.83\%$), and dried SCB samples (MC = 8.14 ± 0.10), majority of the briquettes had lower moisture contents ranging from 5.06% to 8.25%. This is due to water evaporating from the heating of the mold at 150°C for 30 minutes during the briquetting process. All the briquettes pass the moisture content standard for Ö-NORM M7135 ($\leq 10\%$) and EN 14961-3 ($\leq 15\%$). The other incombustible component of the solid fuel briquette is the ash content. Ash content from the briquettes produced range from 1.83 – 2.50% which are higher than the

prescribed ash content by the above-mentioned standards (< 0.7 wt%). However, the ash content of the briquettes produced, which have an average ash content of $2.28 \pm 0.00\%$, are relatively lower compared to other common biomass fuels ($0.5 - 19.2\%$, average of 4.65%) [51].

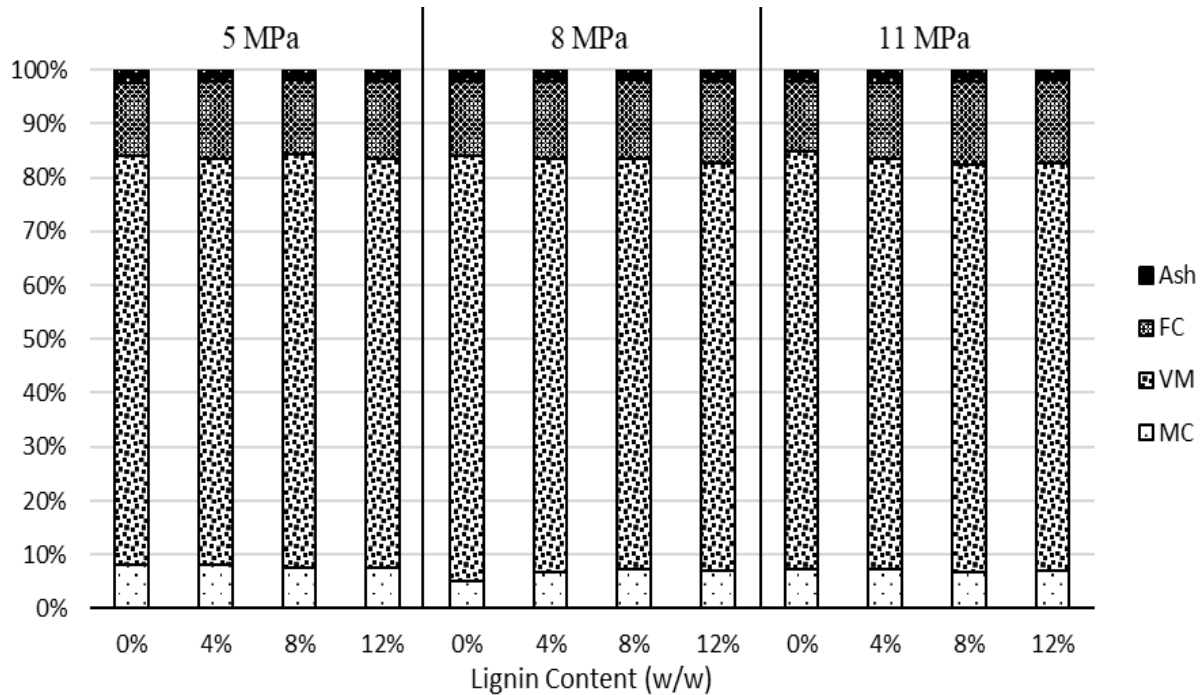


Fig. 4. Proximate composition of solid fuel briquettes with varying compaction pressure (5 MPa, 8 MPa, 11 MPa) and binder content (0%, 4%, 8%, 12%).

Fig. 4 shows the proximate composition of all the briquettes studies. There is no significant change in the proximate composition of the briquettes with the addition of lignin. This may be due to the fact that the SCB and lignin have similar compositions (refer to Table 4). The values of VM and FC range from $75.18\% - 79.23\%$ and $12.63\% - 15.51\%$, respectively. The amounts of VM and FC are responsible for the heating value of the solid fuel. However, it is not enough to determine the total amount of VM and FC that the solid fuel has but it is also rather important to calculate the ratio of FC and VM, also called the fuel ratio, a combustion characteristic of a solid fuel. Further discussions on the combustion characteristics and other implications of the proximate composition are discussed in the previous subsections.

Briquette Characterization: Density.

The density of the briquette is an important property to consider when producing briquettes, it mainly affects the storage requirements of these solid biofuels. Briquettes with high density are desirable since they take up less space when stored and they are more energy dense compared to the loose biomass [7, 49]. Standards for briquettes require their densities to be greater than 0.90 g/cm^3 at the minimum, these are based on European Standards EN 14961-3. Fig. 5 shows the densities of the briquettes at different lignin contents and compaction pressures.

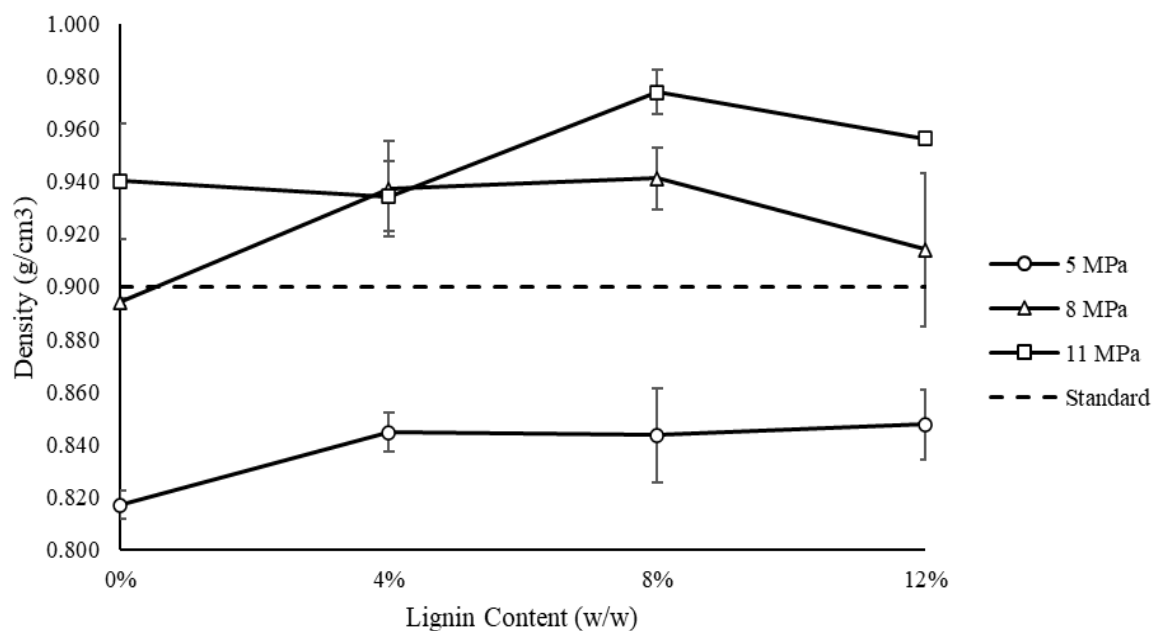


Fig. 5. Briquette density at various binder contents and compaction pressures. Error bars represent standard deviations for two replicates ($n = 2$).

By doing an analysis of variance (ANOVA) with a 95% confidence interval, only the compaction pressure has a significant effect on the density ($p < 0.05$) while lignin content has no significant effect on the density ($p > 0.05$). Effects of interactions between the two factors are also not observed. Increasing the pressure from 5 to 8 MPa for briquettes with 8% binder content increases from 0.843 ± 0.018 to 0.941 ± 0.012 g/cm³ (11.59% increase) while further increasing the pressure to 11 MPa brings the density up to 0.974 ± 0.029 g/cm³ (3.50% increase). Higher pressures exert more force onto the biomass, forcing the particles to fill the void spaces within the briquette and thereby increasing its density [13]. As shown in Fig. 5, only briquettes made with compaction pressures of 8 and 11 MPa meet the standard (0.90 g/cm³) while those made using a pressure of 5 MPa fall short of the standard with densities of around 0.838 g/cm³. Compared to the loose biomass, which has a density of 0.121 ± 0.000 g/cm³ (loose), the densities of the briquettes range from 0.817 ± 0.006 to 0.974 ± 0.029 g/cm³. The SCB was compressed by about 85 to 88% with densities of about 7 to 8 times the density of the raw biomass.

Briquette Characterization: Higher Heating Value.

The HHV determines the energy the biomass gives off when burned which means a high HHV is desirable for briquettes and fuels in general. Fig. 6 shows the HHVs of the briquettes. From the graph, it can be observed that, for the ranges chosen in the experiment, HHV increases as the lignin content is increased while increasing compaction pressure does not have a significant effect on the HHV between briquettes of the same lignin content. For instance, increasing the lignin content from 0% to 12% for a briquette with a compaction pressure of 8 MPa increases the HHV from 18.2 ± 0.2 to 19.4 ± 0.8 MJ/kg (6.59% increase) while increasing the compaction pressure from 5 MPa to 11 MPa for a briquette with 4% lignin content does not affect the HHV much (18.5 ± 0.6 to 18.6 ± 0.0 MJ/kg).

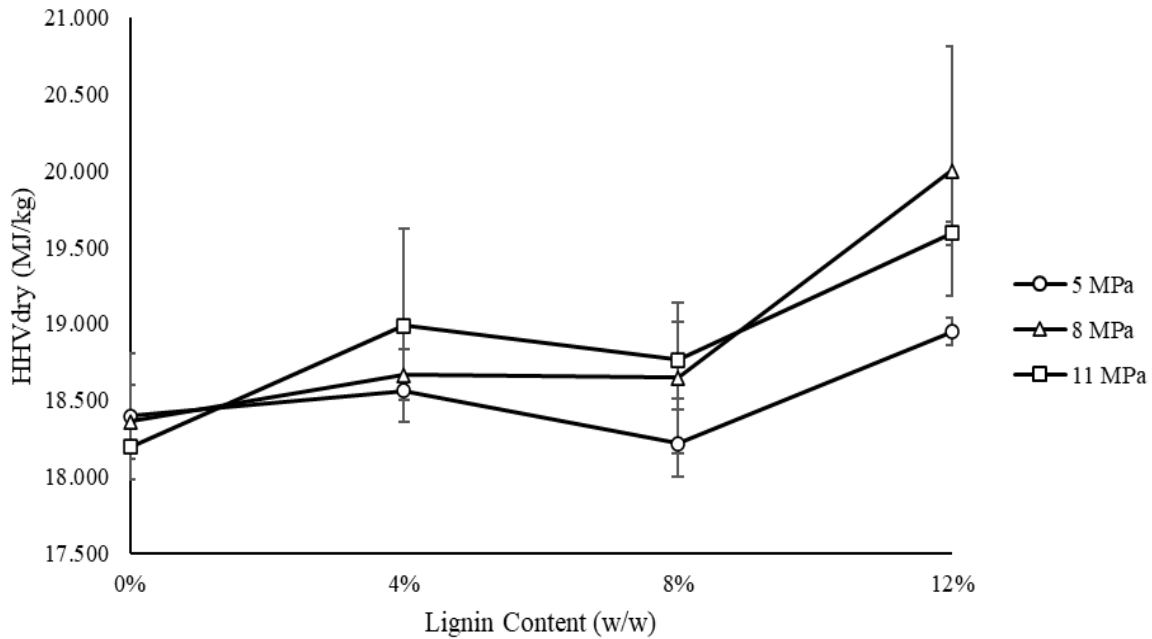


Fig. 6. Higher heating value of briquettes at various lignin contents and compaction pressures. Error bars represent standard deviations for replicates of two ($n = 2$).

By analysis of variance at a confidence level of 95%, only the lignin content significantly affects the HHV ($p < 0.05$) while the effects of the compaction pressure are not found to be statistically significant to the HHV of the briquette ($p > 0.05$). Effects of interactions between the two factors are not observed. Increasing the lignin content would logically increase the HHV since the heating value of the lignin binder is higher than the raw SCB. On the other hand, the compaction pressure would not directly affect the HHV other than the leaching out of the extractives within the biomass [13]. Several studies have shown that the heating value of biomass is proportional to its lignin content [18, 52, 53] since the heating value of lignin is higher than the other two components of lignocellulosic biomass (cellulose and hemicellulose). The higher heating value of lignin compared to the other components is due to its higher carbon content [50], thereby the addition of lignin to the SCB before briquetting would contribute to an increase in the overall HHV of the mixture. Generally, all of the briquettes produced have HHV values that fall between the HHV of raw SCB (18.2 ± 0.3 MJ/kg dry basis) and the HHV of pure lignin (22.6 ± 0.1 MJ/kg dry basis), which is to be expected. The European standard (EN 14961-3) for non-industrial wood briquettes requires the HHV to be a minimum of 15.3 MJ/kg wet basis [54], briquettes produced in this work have a minimum HHV of 16.6 ± 0.4 MJ/kg wet basis and can go up to 18.3 ± 0.1 MJ/kg wet basis which is above the standard, given that it was set for wood briquettes.

Briquette Characterization: Compressive Strength.

The compressive strength is an important metric of mechanical strength in order to estimate the tolerable load a briquette can withstand [11]. In this regard, no standard minimum strength has been determined for commercialized briquettes. However, briquettes with high compressive strength are desirable.

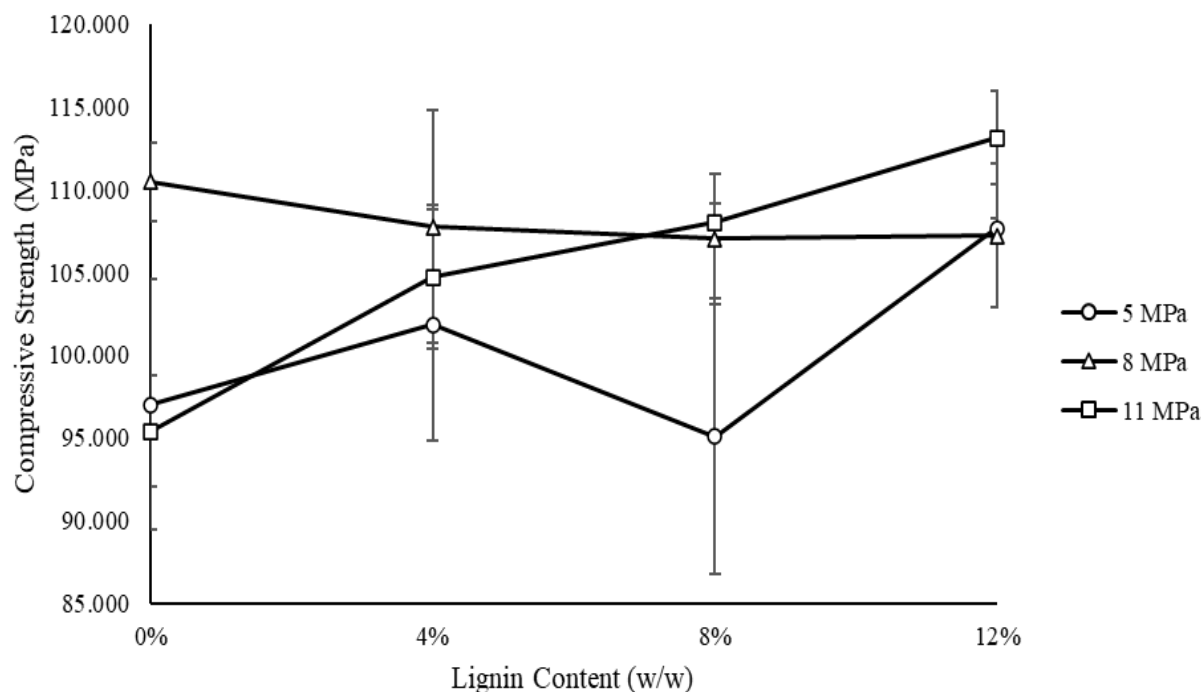


Fig. 7. Compressive strength of briquettes at various lignin contents and compaction pressures. Error bars represent standard deviations for replicates of two ($n = 2$).

Fig. 7 shows the compressive strength of briquettes at different binder contents and compaction pressure. No apparent trend was observed over all the data, however there is a positive trend observed when lignin content is increased for briquettes compressed at 11 MPa. The highest compressive strength obtained was also at 113.13 ± 2.81 MPa by a briquette with 12% lignin content and compressed at 11 MPa. By analysis of variance, both of the factors, compaction pressure and lignin content, do not have significant effects on the compressive strength at a confidence level of 95% ($p > 0.05$). Effects of interactions between the factors were not observed. The results in this work are contrary to that of a previous study with a similar range of pressures [13] and even to another study with a wider range of pressures [55], both of which prove that the compaction pressure significantly affects the briquette strength. Ideally, briquette strength increases as the compaction pressure increases [56], but this may have been affected by the compacting procedure, especially since an external binder such as lignin has been added, which affects the layers distribution in the briquette and its strength [56]. Some studies [57–59] have theorized that the softening of lignin by high temperatures and its subsequent cooling while under pressure is a key factor in improving the briquette strength with lignin. The briquetting procedure employed in this study, which is mainly based on the work of Navalta et al. [13], does not include a cooling step in the total compression time. Lignin is an amorphous thermoplastic material which undergoes plastic deformation at its glass transition temperature range and low compaction pressures [59], at this point the lignin fills the gaps between particles. Cooling under pressure allows the softened lignin to solidify and form stronger solid bridges between the particles in the briquette while preventing it from going back into its relaxed state [57]. In future works, it may be advisable to include a cooling step in the procedure to see if there is any effect on the strength.

The range used in the study for lignin concentrations may have been too low for it to exhibit any significant effect on the strength. However, some studies which also use lignin as an additive in low pressure briquetting/pelletizing makes use of a similar range of lignin concentrations [50, 60, 61] and shows a clear positive effect in the improvement of briquette strength. The main difference between this study and the cited studies is that the other studies make use of wood biomass in their

briquettes/pellets while the biomass used in this study is an herbaceous biomass. Studies on non-woody biomass briquettes with lignin as an additive are scarce. A recent study by Chang and Byung [62] on non-woody biomass pellets made from an energy crop, *Miscanthus sacchariflorus*, with Kraft lignin as an additive had a similar observation with this work. The study concluded that the addition of lignin additive up to 5% w/w showed only a little to no increase in the compressive strength of the pellet which the study attributed to the weak bonding between the lignin additive and the biomass. The studies mentioned, however, did not statistically determine the significance of the effect of lignin as an additive in briquettes/pellets.

Nevertheless, the briquette with the lowest compressive strength (8% lignin, 5 MPa) obtained in this work can accommodate up to ~550,000 stacked 25-g briquettes (diameter = 42.5 mm), which is more than sufficient for practical transport and storage of briquettes. In a previous study by Navalta et al. [13], at least ~300,000 of 25-g briquettes could be stacked even with the lowest compressive strength achieved, also noting that the quantity of stackable briquettes is more than enough for practical transport and storage.

Fuel Characteristics of the Best Briquette.

As a result of the investigation performed, the conditions that produced the best briquette with the highest HHV and CS obtained in this study were found to be at a formulation with 12% lignin content, pressed at a compaction pressure of 11 MPa. Further characterization was performed on the best briquette obtained. The additional briquette characterization steps are summarized in Table 6, and results are then compared across various briquetting systems found in literature.

Fuel Characteristics of the Best Briquette: Moisture Reabsorption Rate and Equilibrium Moisture Content.

Since high moisture content is unwanted in solid fuels, one way to assess a solid fuel quality is by determining the moisture uptake rate and the equilibrium moisture content the fuel can hold at normal conditions. To determine these, a control briquette (0% Lignin, 11 MPa) and the best briquette was exposed to an environment with temperature ranges of $T_{wb} = 26.4 \pm 1.8^\circ\text{C}$ and $T_{db} = 31.9 \pm 1.8^\circ\text{C}$, and relative humidity (RH) of $63.4 \pm 7.8\%$.

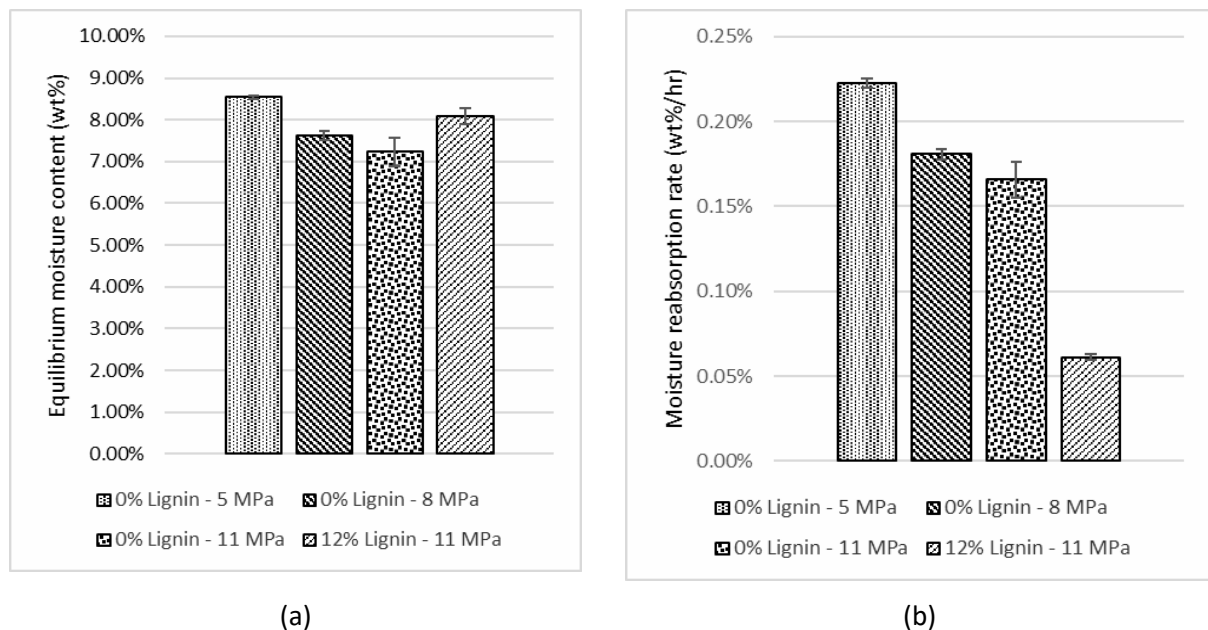


Fig. 8. Equilibrium moisture content (a) and moisture reabsorption rate (b) of binderless briquettes compressed at the different compaction pressures and the optimum briquette (LC = 12% w/w; CP = 11 MPa). Error bars represent standard deviations for replicates of two ($n = 2$).

As presented in Fig. 8, the equilibrium moisture of the best briquette (8.09%) is found to be higher than that of binder-less briquettes compressed at 8 MPa (7.63%) and 11 MPa (7.24%). It was also observed that the addition of a binder (lignin) increased the equilibrium moisture. This is due to the presence of oxygen-containing functional groups of lignin. Comparing the briquette with 12% lignin content compressed at 11 MPa (12% Lignin – 11 MPa) with the binder-less briquette compressed at 11 MPa, the briquette with lignin had a larger equilibrium moisture content by 1.19%. In a similar study conducted by Hu et al. [63], it was observed that increasing the binder (lignin) content of the rice husk bio-char pellets from 5% to 20% increased the equilibrium moisture content from 4.69% to 6.48%.

The control briquette (BC = 0%, CP = 11 MPa) exhibits a moisture reabsorption rate 0.1660% per hour, while the rate of moisture reabsorption is much slower for the briquettes densified at the determined best conditions (BC = 12%, CP = 11 MPa) at 0.0611% per hour. In the study by Hu et al. [63], lignin binder, rice husk bio-char pellets yielded reabsorption rates of 0.2345 - 0.3240% per hour. Compared to values obtained from literature, the produced SCB briquettes have higher values of equilibrium moisture, but lower values of rate of moisture reabsorption. This implies that the briquettes could be stored for a longer time compared to binderless briquettes since the rate of moisture uptake is slow. However, long storage periods would cause swelling and disintegration due to a higher equilibrium MC content compared to the control briquettes. Nevertheless, the equilibrium moisture of the best briquettes still complies with the chosen standards.

Fuel Characteristics of the Best Briquette: Burning Rate.

The most straightforward technique of unlocking the energy potential in biomass feedstock is through combustion. Thus, investigating the solid fuel burning characteristics is essential in trying to describe and understand the behavior of the fuel when subjected to combustion. In this study, the burning characteristics are mainly gauged by the burning rate and the combustion indices.

The burning rate (BR) of the best briquette is in the range of 1.30 - 1.58 g/min, which translates to a normalized burning rate (NBR) of 5.31 - 6.43 % mass loss/min, with typically short ignition times ranging from 0.17 - 0.50 minutes and burning times ranging from 12.33 - 14.83 minutes. A study by Navalta et al. on bagasse-rice bran briquettes yielded similar results with an NBR of 4.28 - 7.75% mass loss/ min, ignition times ranging from 0.17 - 1.83 minutes and burning time of 3.67 - 10.67 minutes [13]. Comparing to different briquettes in literature, starch binder empty fruit bunches briquette (EFB) had a BR of 4.35 g/min and ignition time of 6 minutes with ignition aid [65], and asphalt binder EFB briquettes with BR of 3.7 g/min and ignition time of 13 minutes with ignition aid [65]. The briquettes in this study have lower burning rates and shorter ignition times. However, burning time of coal-biomass briquette systems prove to be longer, spanning from 60-100 minutes for maize cob-coal and groundnut shell-coal briquettes [66]. This implies that the briquettes produced are consumed faster compared to coal-based briquettes and large amounts of fuel are required to sustain burning. This poses a disadvantage in industrial fuels. However, the ease of ignition is a convenient feature for fuels targeted for domestic/household usage.

Fuel Characteristics of the Best Briquette: Combustion Indices.

For the briquette combustion indices, the fuel ratio (FR), combustibility index (CI), and volatile ignitability (VI) are considered in this work. The rationale behind obtaining these indices revolves around the idea that the volatile matter content in the fuel is used as a basis to determine combustibility and eventually select better solid fuel alternatives for boiler applications [32] and ultimately, these indices give an idea as to the potential for industrial applicability of the produced solid fuel briquettes.

In this study, the best briquette bears an FR value of 0.2041 ± 0.0016 . FR ranging from 0.5-3.0 are commonly used in coal-fired power plants [34]. In thermoelectric power plants, the FR of sub-bituminous coal is in the range of 0.5-1.0 [33]. The briquettes produced do not meet the FR values to qualify for usage in power plants. This implies that the solid fuel contains high quantities of volatile matter and that it leads to challenges in energy recovery due to rapid combustion leading to poor boiler efficiencies and unwanted gaseous emissions [13, 67]. This is true for solid fuels with low FR. However, mechanical densification techniques such as briquetting can be used in tandem with other densification techniques in order to improve FR and other combustion indices [13]. An example would be torrefaction, a type of chemical densification technique. In a study by Conag et al. [34], torrefied SCB were determined to have an FR ranging from 0.22 - 1.68. The VI, which is a gauge for the energy imparted by the total volatile matter, must be at least 14 MJ/kg. For the best briquette produced in this study, the VI value is at 15.78 ± 0.09 MJ/kg. This indicates that the briquettes have sufficient volatile matter for ease of ignition, as supported by the FR value, proximate analysis, and the short ignition time observed in burning rate experiments. This is further supported by the fact that biomass is considered as a more reactive fuel than coal, giving a much faster combustion rate during the devolatilization phase [45]. The CI can be used to evaluate the suitability of the material for mixed combustion in coal-fired thermal power plants [33]. The CI appropriate for co-firing must be in the range of 12.56 - 23.03 MJ/kg [33]. The best briquette yielded a CI value of 96.40 ± 1.13 MJ/kg. The CI values of the briquette is comparable to those of raw biomass feedstock. In a study of Conag et al., unprocessed SCB had a CI of 82 MJ/kg. On the other hand, torrefaction of SCB significantly reduced the CI to an acceptable value of 16 MJ/kg, noting that torrefaction at 350°C provided the best results [34]. Ultimately, it is obvious that the best briquette produced in this work is not fit for co-firing in industrial boilers.

Table 6. Fuel characteristics of various densified solid fuels.

Solid Fuel	Briquetting conditions			Binder Content /Ratio		Briquette dimensions		Compressive strength (MPa)		Moisture reabsorption		Combustion indices			
	Compaction rate (MPa/min)	Compaction pressure (MPa)	Compaction temperature (°C)	Biomass particle size (mm)			Briquette diameter (mm)			Equilibrium Moisture (wt. %)	Moisture reabsorption rate (wt. % per hr)	Burning rate (g/min)	FR	VI (MJ/kg)	CI (MJ/kg)
Lignin-binder, SCB briquettes (12% Lignin - 11 MPa)*	0.37	11	150	0.61 ± 0.01	12	43.30 ± 0.04	113.13 ± 2.81	8.0189 ± 0.1473	0.0611 ± 0.0017	1.30-1.58	0.2040	15.91 89	104.0988		
SCB briquettes (0% Lignin - 11 MPa)*	0.37	11	150	0.61 ± 0.01	0	43.38 ± 0.11	95.39 ± 3.36	6.8308 ± 0.3381	0.1660 ± 0.0106	1.56-1.63	0.1700	14.71 59	114.6898		
RB -SCB briquettes [13]	0.27 - 0.40	8 - 12	150	0.22 ± 0.00; 0.54 ± 0.01	0	42.5	50.21 - 122.37	4.2065-6.4919	0.3840-1.0800	1.21-1.80	0.20-0.25	14.1-14.7	59.1 - 87.6		
Mustard stalk briquettes [64]	-	112.34 - 123.42	-	0.352	5 - 20	41.5 - 43.5	6.52	3.56 - 8.50 ^{db}	-	-	-	-	-		
Mixed waste (tree leaves and grasses) briquettes [64]	-	112.34 - 123.42	-	0.378	5 - 20	41.5 - 43.5	6.54	3.09 - 4.85 ^{db}	-	-	-	-	-		
Saw mill wood waste briquettes [64]	-	112.34 - 123.42	-	0.561	5 - 20	41.5 - 43.5	5.43	2.49 - 4.39 ^{db}	-	-	-	-	-		
Lignin-binder, rice husk bio-char pellets [63]	-	95.54	-	-	5 - 20	20	-	4.69-6.48	0.2345-0.3240*	-	3.6470 - 5.5086	13.33 87-17.99	3.31 22-5.00		
Starch-binder, EFB briquette [65]	-	-	-	-	-	-	-	6.01	-	4.35	-	-	-		
Asphalt-binder, EFB briquette [65]	-	-	-	-	-	-	-	10	-	3.7	-	-	-		

* values obtained in this study **assumed 20-hr test period (as stated by Hu et al. [63]) ^{db}expressed on dry basis.

Conclusions and Recommendations

The effects of SCB:lignin ratio and compaction pressure on the quality and fuel characteristics of briquettes were investigated in this work. SCB briquettes were found to have improved bulk density (7 to 8-fold increase), high compressive strength and retarded moisture uptake. Compaction pressure is shown to have a significant effect on the briquette density ($p < 0.05$). On the other hand, both compaction pressure and lignin content do not exhibit significant effects on the compressive strength ($p > 0.05$). While the addition of lignin binder lowers the moisture reabsorption rate, the opposite is true for the equilibrium moisture content.

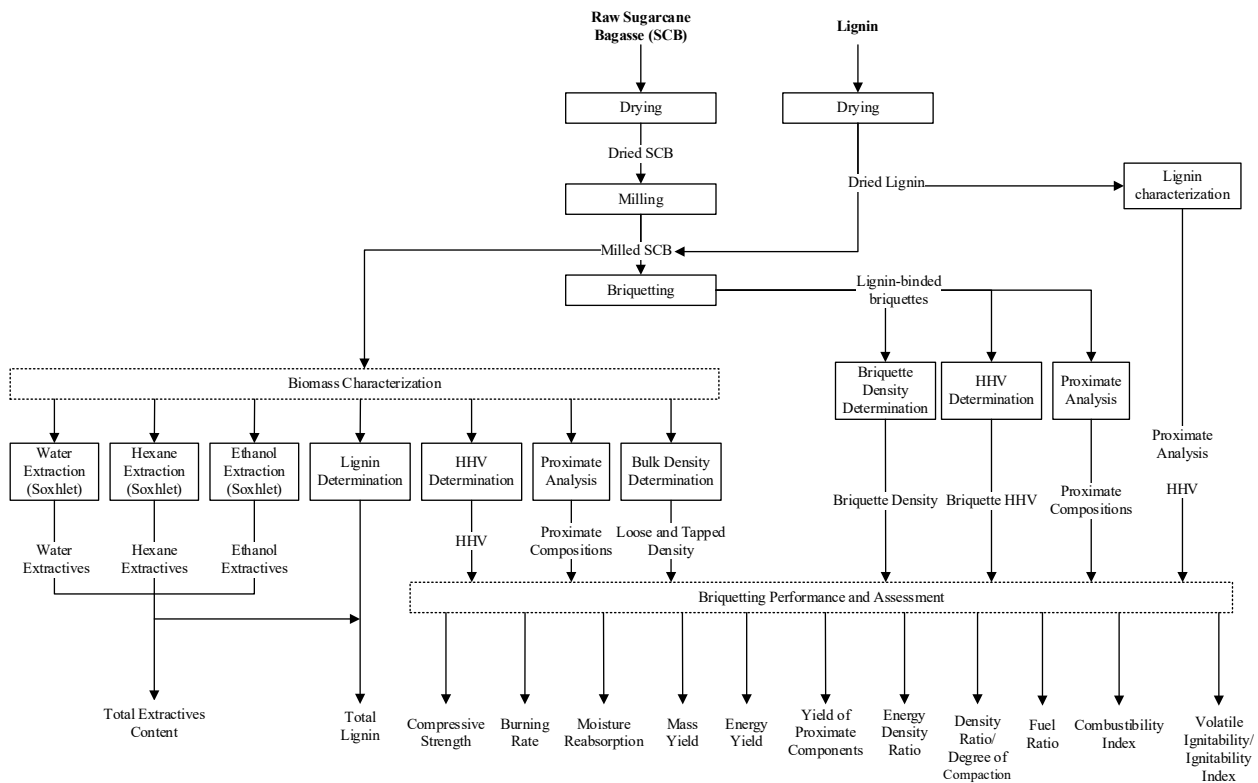
As for the fuel characteristics, the HHV of briquettes are shown to increase with increasing lignin content ($p < 0.05$). Proximate analysis reveals that the briquette and feedstock compositions are identical, showing high levels of volatile matter. Thus, the briquettes exhibit relatively short burning times compared to coal and coal-biomass briquettes. Combustion indices determine that SCB briquettes are not suitable for industrial applications.

To further improve the study, it is recommended that other sources of lignin be explored, and lignin binder range explored in this study could be expanded ($> 12\%$) to better understand its effects on the compressive strength and other briquette characteristics. Process conditions during briquetting could also be altered such as the pressing temperature, time, and pressure to further optimize the briquetting process. Furthermore, various biomass densification techniques can also be explored, such as a combination of briquetting (mechanical densification) and torrefaction (thermochemical densification) techniques.

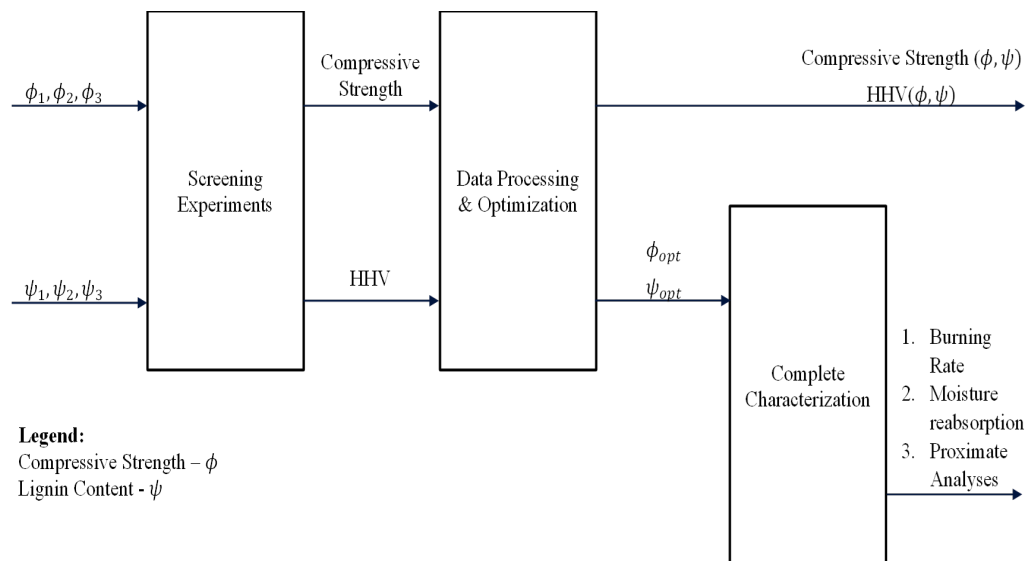
Acknowledgement

The authors would like to acknowledge the support provided by the Bogo-Medellin Milling Company for the allowing us to source sugarcane bagasse from their facility as raw material for this research and the Department of Science and Technology - Philippine Textile Research Institute for the provision of waste degumming liquor and for allowing us to use their research facility for the acquisition of lignin which was used as a raw material for our study. And lastly, to the University of San Carlos – Research Development Extension and Publications Office (RDEPO) for the grant provided to I.D.F Tabañag.

Appendix A: Research Design



Experimental Flow Diagram of This Study



Experimental Design Diagram of This Study

References

- [1] British Petroleum Company, BP statistical review of world energy. British Petroleum Company, 2018.
- [2] J. Orloff, "The Definition of Bioenergy," 2018.
- [3] D. J. Tenenbaum, "Food vs. fuel: diversion of crops could cause more hunger." National Institute of Environmental Health Sciences, 2008.
- [4] "Performance of Philippine Agriculture January-March 2018."

-
- [5] A. W. Go and A. T. Conag, "Utilizing sugarcane leaves/straws as source of bioenergy in the Philippines: A case in the Visayas Region," *Renew. Energy*, 2018.
- [6] Y. R. Loh, D. Sujana, M. E. Rahman, and C. A. Das, "Review Sugarcane bagasse - The future composite material: A literature review," *Resour. Conserv. Recycl.*, vol. 75, pp. 14–22, 2013.
- [7] J. S. Tumuluru, C. T. Wright, J. R. Hess, and K. L. Kenney, "A review of biomass densification systems to develop uniform feedstock commodities for bioenergy application," *Biofuels, Bioproducts and Biorefining*, vol. 5, no. 6, pp. 683–707, 2011.
- [8] C. Karunanithy, Y. Wang, K. Muthukumarappan, and S. Pugalendhi, "Physiochemical Characterization of Briquettes Made from Different Feedstocks," *Biotechnol. Res. Int.*, vol. 2012, pp. 1–12, 2012.
- [9] A. Bauen and M. Londo, "Bioenergy – a Sustainable and Reliable Energy Source," no. April 2015, pp. 1–108, 2012.
- [10] P. K. Toan, N. D. Cuong, N. T. Quy, and P. K. Sinh, "Application of Briquetting Technology To Produce Briquettes from Agricultural Residues and By-products," pp. 1416–1419, 2000.
- [11] M. Ibrahim, U. O. Aroke, and A. Abdulrasheed, "Investigation of Mechanical & Combustible Properties of Briquettes Produced From Rice Husk Using Polystyrene Foam Adhesive As a," vol. 3, no. 2, 2015.
- [12] S. V. Bhagwanrao, M. Singaravelu, and others, "Bulk density of biomass and particle density of their briquettes," *Int. J. Agric. Eng.*, vol. 7, no. 1, pp. 221–224, 2014.
- [13] C. J. L. G. Navalta, K. G. C. Banaag, V. A. O. Raboy, A. W. Go, and L. K. Cabatingan, "Solid Fuel from co-briquetting of Sugarcane Bagasse and Rice Bran Solid Fuel," 2018.
- [14] M. Rahman, K. Khan, M. N. Ali Moral, and A. N. M. M. Rahman, "Biomass Briquetting Technology and Environmental Pollution." 2003.
- [15] N. H. Azim, A. Subki, and Z. N. B. Yusof, "Abiotic stresses induce total phenolic, total flavonoid and antioxidant properties in Malaysian indigenous microalgae and cyanobacterium," *Malays. J. Microbiol.*, vol. 14, no. 1, pp. 25–33, 2018.
- [16] C. Holley, *Binder and Binder Systems for Agglomeration*, vol. 2. Madison: The Institute for Briquetting and Agglomeration, 1983.
- [17] N. Kaliyan and R. V. Morey, "Densification Characteristics of Corn Stover and Switchgrass," vol. 0300, no. 06, 2006.
- [18] A. Demirbaş, "Relationships between lignin contents and heating values of biomass," *Energy Convers. Manag.*, vol. 42, no. 2, pp. 183–188, 2001.
- [19] A. Demirbas, "Relationships between Heating Value and Lignin, Moisture, Ash and Extractive Contents of Biomass Fuels," *Energy Explor. Exploit.*, vol. 20, no. 1, pp. 105–111, 2002.
- [20] Q. Hu, J. Shao, H. Yang, D. Yao, X. Wang, and H. Chen, "Effects of binders on the properties of bio-char pellets," *Appl. Energy*, vol. 157, no. June 2015, pp. 508–516, 2015.
- [21] A. García, M. G. Alriols, R. Llano-Ponte, and J. Labidi, "Energy and economic assessment of soda and organosolv biorefinery processes," *Biomass and Bioenergy*, vol. 35, no. 1, pp. 516–525, 2011.
- [22] ASTM, "ASTM D7481-09 Standard test methods for determining loose and tapped bulk densities of powders using a graduated cylinder," *Annu. B. ASTM Stand.*, no. October, p. American Society for Testing and Materials, West C, 2009.
- [23] ASTM, "Standard Test Method for Chemical Analysis of Wood Charcoal," *ASTM Int.*, vol. 84, no. 2, pp. 1–2, 2011.

-
- [24] A. Sluiter, B. Hames, R. Ruiz, C. Scarlata, J. Sluiter, and D. Templeton, "Determination of ash in biomass," Cole Boulevard, Golden, Colorado, 2008.
 - [25] Parr Instrument Company, "Oxygen Bomb Calorimeter," Combustion, no. 204, 2008.
 - [26] Parr, "Operating Instructions for the 1108 Oxygen Combustion Bomb," no. 205, pp. 1–12, 2007.
 - [27] American Society for Testing and Materials, "ASTM D2015: Standard Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Calorimeter," US Gov., vol. 552, no. 5, p. 5, 1996.
 - [28] A. T. Conag, J. E. R. Villahermosa, L. K. Cabatingan, and A. W. Go, "Energy densification of sugarcane leaves through torrefaction under minimized oxidative atmosphere," *Energy Sustain. Dev.*, vol. 42, no. 6, pp. 160–169, 2018.
 - [29] P. Krizan, *The Densification Process of Wood Waste*. 2015.
 - [30] J. O. Chaney, M. J. Clifford, and R. Wilson, "An experimental study of the combustion characteristics of low-density biomass briquettes," in *Proceedings of 3rd International Conference of Appropriate Technology*, Kigali, Rwanda, 2008, pp. 12–15.
 - [31] E. A. Emerhi, "Physical and combustion properties of briquettes produced from sawdust of three hardwood species and different organic binders," *Adv. Appl. Sci. Res.*, vol. 2, no. 6, pp. 236–246, 2011.
 - [32] N. Oka, S. Shinozaki, M. Shibaoka, C. G. Thomas, C. Division, and P. O. B. N. Ryde, "The Influence of Rank and Maceral Composition on Ignition and Char Burnout of Pulverized Coal 1 . Energy R & D Labo ., Energy Development Dept ., IOI ~| ITSU KOSAN CO ., LTD . Investigation of combustion characteristics for different ranks of coal was con," vol. 15, pp. 213–224, 1987.
 - [33] T. I. Ohm, J. S. Chae, J. K. Kim, and S. C. Oh, "Study on the characteristics of biomass for co-combustion in coal power plant," *J. Mater. Cycles Waste Manag.*, vol. 17, no. 2, pp. 249–257, 2015.
 - [34] A. T. Conag, J. E. R. Villahermosa, L. K. Cabatingan, and A. W. Go, "Energy densification of sugarcane bagasse through torrefaction under minimized oxidative atmosphere," *J. Environ. Chem. Eng.*, vol. 5, no. 6, pp. 5411–5419, 2017.
 - [35] "Minitab LLC © 2019, Minitab Release 19.1.0: Statistical Software for Windows 2010." 2019.
 - [36] B. Dass and P. Jha, "Biomass characterization for various thermo-chemical applications," *Int. J. Curr. Eng. Sci. Res.*, vol. 2, no. 2, pp. 59–63, 2015.
 - [37] J. Parikh, S. A. Channiwala, and G. K. Ghosal, "A correlation for calculating HHV from proximate analysis of solid fuels," *Fuel*, vol. 84, no. 5, pp. 487–494, 2005.
 - [38] E. A. Mousa, H. M. Ahmed, and C. Wang, "Novel Approach Towards Biomass Lignin Utilization in Ironmaking Blast Furnace," *ISIJ Int.*, vol. 57, no. 10, pp. 1788–1796, 2017.
 - [39] D. J. Horst, J. J. Ramírez Behainne, P. P. de Andrade Júnior, and J. L. Kovaleski, "An experimental comparison of lignin yield from the Klason and Willstatter extraction methods," *Energy Sustain. Dev.*, vol. 23, no. 1, pp. 78–84, 2014.
 - [40] Y. H. Leang and H. Y. Saw, "Proximate and functional properties of sugarcane bagasse," *Agro Food Ind. Hitech*, vol. 22, no. 2, Suppl. S, pp. 5–8, 2011.
 - [41] P. D. Grover and S. K. Mishra, "Biomass Briquetting: Technology and Practices. Regional wood energy development programme in Asia GCP/RAS/154/NET, Field document No. 46," *Reg. Wood Energy Dev. Program. Asia*, no. 46, pp. 1–48, 1996.

-
- [42] L. Tabil, P. Adapa, and M. Kashaninejad, "Biomass Feedstock Pre-Processing – Part 2: Densification," *Biofuel's Eng. Process Technol.*, pp. 439–464, 2011.
- [43] C. Ludwig, "How and why solids agglomerate," *Chem. Eng.*, no. January, pp. 156–160, 1994.
- [44] V. Panwar, B. Prasad, and K. L. Wasewar, "Biomass Residue Briquetting and Characterization," *J. Energy Eng.*, vol. 137, no. 2, pp. 108–114, 2011.
- [45] A. Tamilvanan, "Preparation of Biomass Briquettes using Various Agro- Residues and Waste Papers," *J. Biofuels*, vol. 4, no. 2, p. 47, 2013.
- [46] T. Cordero, F. Marquez, J. Rodriguez-Mirasol, and J. Rodriguez, "Predicting heating values of lignocellulosics and carbonaceous materials from proximate analysis," *Fuel*, vol. 80, no. 11, pp. 1567–1571, 2001.
- [47] P. Jittabut, *Physical and Thermal Properties of Briquette Fuels from Rice Straw and Sugarcane Leaves by Mixing Molasses*, vol. 79, no. x. Elsevier B.V., 2015.
- [48] H. Jin, Y. Lu, L. Guo, C. Cao, and X. Zhang, "Hydrogen production by partial oxidative gasification of biomass and its model compounds in supercritical water," *Int. J. Hydrogen Energy*, vol. 35, no. 7, pp. 3001–3010, 2010.
- [49] R. Mythili and P. Venkatachalam, "Briquetting of agro-residues," *J. Sci. Ind. Res. (India)*, vol. 72, no. 1, pp. 58–61, 2013.
- [50] W. T. N. Boschetti, A. M. M. L. Carvalho, A. de C. O. Carneiro, L. C. Santos, and L. de B. Q. Poyares, "Potential of kraft lignin as an additive in briquette production," 2019.
- [51] A. Demirbas, "Potential applications of renewable energy sources, biomass combustion problems in boiler power systems and combustion related environmental issues," *Prog. Energy Combust. Sci.*, vol. 31, no. 2, pp. 171–192, 2005.
- [52] D. A. da Silva, V. C. Almeida, L. C. Viana, U. Klock, and G. I. B. De Muñiz, "Evaluation of the Energy-related Properties of Tropical Wood Waste Using NIR Spectroscopy," vol. 21, no. 4, pp. 561–568, 2014.
- [53] A. Oliveira, A. Carneiro, B. Vital, W. Almeida, B. Pereira, and M. Cardoso, "Quality parameters of Eucalyptus pellita F. Muell. Wood and charcoal," *Sci. For. Sci.*, vol. 38, pp. 431–439, 2010.
- [54] Deutsches Pelletinstitut, "Manual for the certification of wood briquettes for the end-user market," *ENplus Briquettes*, no. January, 2013.
- [55] S. J. Mitchual, K. Frimpong-Mensah, and N. A. Darkwa, "Effect of species, particle size and compacting pressure on relaxed density and compressive strength of fuel briquettes," *Int. J. Energy Environ. Eng.*, vol. 4, no. 1, pp. 1–6, 2013.
- [56] J. Kers et al., "Determination of physical, mechanical and burning characteristics of polymeric waste material briquettes," *Est. J. Eng.*, vol. 16, no. 4, p. 307, 2010.
- [57] S. Eriksson and M. Prior, "The briquetting of agricultural wastes for fuel," *FAO environment and energy paper*, no. No. 11. Food and Agriculture Organization of the United Nations, Rome, p. 131 pp., 1990.
- [58] T. Hardianto, F. F. Pambudi, and A. R. Irhamna, "A study on lignin characteristic as internal binder in hot briquetting process of organic municipal solid waste," vol. 030013, no. July, 2018.
- [59] N. Kaliyan and R. V. Morey, "Natural binders and solid bridge type binding mechanisms in briquettes and pellets made from corn stover and switchgrass," *Bioresour. Technol.*, vol. 101, no. 3, pp. 1082–1090, 2010.

-
- [60] W. T. N. Boschetti, A. do C. P. Lopes, R. A. Ribeiro, R. Q. Reyes, and A. de C. O. Carneiro, "Kraft lignin as an additive in pine and eucalyptus particle composition for briquette production," vol. 43, no. 2, pp. 1–8, 2019.
- [61] B. L. C. Pereira, A. de C. O. Carneiro, A. M. M. L. Carvalho, B. R. Vital, A. C. Oliveira, and W. D. Canal, "Influence of adding kraft lignin in eucalyptus pellets properties.," *Floresta*, vol. 46, no. 2, pp. 235–242, 2016.
- [62] H. M. Chang and H. U. Byung, "Effect of Process Parameters and Kraft Lignin Additive on The Mechanical Properties of Miscanthus Pellets," vol. 45, no. 6, pp. 0–2, 2017.
- [63] Q. Hu, J. Shao, H. Yang, D. Yao, X. Wang, and H. Chen, "Effects of binders on the properties of bio-char pellets," *Appl. Energy*, vol. 157, no. January, pp. 508–516, 2015.
- [64] G. C. Wakchaure and I. Mani, "Effect of binders and pressures on physical quality of some biomass briquettes," *J. Agric. Eng. Res.*, vol. 46(4), no. October-December, 2009, pp. 24–30, 2009.
- [65] K. Ugwu and K. Agbo, "Evaluation of binders in the production of briquettes from empty fruit bunches of *Elais Guinensis*," vol. 2, no. 4, pp. 176–179, 2013.
- [66] T. U. Onuegbu, N. O. Ilochi, I. M. Ogbu, F. O. Obumselu, and I. Okafor, "Preparation of environmental friendly bio-coal briquette from groundnut shell and maize cob biomass waste: comparative effects of ignition time and water boiling studies," *Curr Res Chem*, vol. 4, pp. 110–118, 2012.