Effects of Process Variables on Physico-Mechanical Properties of Abura (Mitrogyna ciliata) Sawdust Briquettes

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Abstract: Efficient utilization of biomass requires conversion into forms that can be optimally applied in energy generation. Briquetting involves the compaction of biomass into solid blocks that are more efficient than raw biomass, and provides ease of transport and handling. These are improved when the briquettes possess a high density, shatter index, and compressive strength. Due to differences in nature and composition, it is imperative to define optimum conditions for the production of quality and durable briquettes for individual biomasses that are compacted into briquettes. This study investigated the effects of process variables on the strength, durability, and density of biomass briquettes produced using Abura sawdust. The lateral compressive strength and drop shatter index were investigated whilst varying the temperature (100–150 °C), pressure (9–15 MPa), and hold time (15–30 min). The compressive strength ranged between 2.06 and 5.15 MPa, whilst the shatter index was between 50 and 600. Briquette density was between 518.8 and 822.9 kg/m³. The pressure was significant to the determination of the compressive strength (p < 0.1) and the shatter index (p < 0.05). The pressure, temperature, and hold time are significant to the briquette density. Physical and mechanical characteristics of the binderless Abura sawdust briquettes can be improved by optimizing the densification variables during the briquetting process when moderate pressures are used for compaction.

Keywords: biomass densification; Abura sawdust briquettes; response surface methodology; shatter index; lateral compressive strength

1. Introduction

The economic development and growth of a nation depends on major factors such as technology because it allows for more efficient production and better goods and services. Energy is a major driver of technology, and its availability determines sustainable and rapid development of a nation’s technological and industrial growth [1]. However, the prevalent use of fossil fuel for energy generation has raised major concerns globally because it results in the release of greenhouse gases that trap heat in the atmosphere and cause global warming. As a result, several alternatives have been exploited for energy generation. One of the alternative sources is biomass energy, which has proven to be viable, renewable, and sustainable.

Biomass exists naturally and abundantly, and hence can serve as an ideal alternative energy resource [2–4]. Biomass can be harnessed to produce a lot of power, and one of its major applications is found locally in cooking and the warming of households. There are studies that have pointed to the potential of generating power regionally and globally from biomass [2,5–8], where it has been noted that there are several methods to harness...
Biomass energy. Biochemical methods of harnessing biomass for various applications have included anaerobic digestion [9], and fermentation [10]. Thermochemical methods include gasification [11,12], pyrolysis [13], torrefaction [14], and carbonization [8]. Mechanical methods include size reduction [15,16] and densification [17].

Biomass in its free form is often not usable directly in combustion grates or in some gasification processes because of its low bulk density, low energy output, and size. To circumvent these problems, loose biomass can be pressed into solid fuels in the form of pellets or briquettes with improved properties. A briquette is a compressed block of biomass material [18,19] which is produced in a convenient shape and can burn like wood. Briquetting is a densification technology that converts loose low-density biomass to briquettes under applied pressure, thereby improving its physical properties [19–21]. During biomass densification, process variables must be taken into consideration; they include hold-time, pressure, and temperature. Other material variables that must be considered are moisture content, particle size, biomass type, and biochemical composition. These variables potentially determine the physico-mechanical and combustion characteristics of manufactured briquettes [8,22,23].

Obi [24] considered the influence of temperature on the biomass briquette properties and showed that temperature significantly affected the physical and combustion behaviour of briquettes. Lela et al. [25] also investigated the physico-mechanical and combustion properties of sawdust briquettes for energy generation. The study reported that pressure did not have much significance on the calorific value, but temperature was observed to significantly affect the briquette properties. Orisaleye et al. [26] studied the influence of densification variables on the density of briquettes, noting the importance of such study for the design of efficient densification equipment. The briquette density was dependent on the applied pressure, die temperature, and particle size of the corncobs. Orisaleye et al. [27] discovered that higher die temperature and hold times improved the water resistance of corncob briquettes. Kpalo et al. [28] evaluated the properties of briquettes composed of paper pulp with Mesua ferrea leaves. The shatter index ranged between 79.18 and 99.9%, with density reaching up to 370 kg/m$^3$

Shuma and Magyira [29] reported the effect of various production variables utilized in the production of briquettes from loose biomass using cactus and cow dung as binders. Ori-\text{abure} [30] investigated briquette properties using Terminalia metalis and Daniela oliveri and found that the biomass material determined the shatter index of the briquettes. Orisaleye et al. [31] utilized a hydraulic briquetting press to produce poplar briquettes and found that the temperature was significant to the density, water resistance, and durability of the briquettes. Jekayinfa et al. [32] developed empirical models from multiple regression analysis for corncob briquettes, which relate densification variables to the physical properties of the briquettes. Zepeda-Cepeda et al. [33] considered how particle sizes of sawdust influenced briquettes made from Pinus durangensis. The best mixture of particle sizes for optimum compressive strength and shatter index were determined using RSM.

Ladla et al. [34] investigated the properties of briquettes produced in a low-pressure briquetting machine and found that the shatter index and water resistance were within the range of 80 to 95%, with compressive strength reaching up to 19.52 MPa. Jekayinfa et al. [35] showed that the quality of densified rice bran pellets was affected by binder utilization, along with die configuration and design. Ossei-\text{Bremang} et al. [36] utilized bentonite clay and cardboard pulp as binders for palm kernel and decanter cake briquettes, and the optimum shatter index of briquettes was determined using Response Surface Methodology (RSM). Orisaleye et al. [37] also conducted investigations on the physical properties of briquettes from Abura sawdust using RSM. Statistically significant variables that were identified to influence density were hold time, temperature, and pressure. For water resistance, only the die temperature was significant. Afrifah et al. [38] found that by varying the composition of materials and pressure, briquettes of the desired quality can be produced with low pressure from sugarcane peels, empty fruit bunch, and sawdust.
Previous studies have noted that each biomass has its own set of characteristics which determine the quality of briquettes produced. This creates a need to investigate and optimize the characteristics of briquettes produced from different biomass sources. There are very few studies on the utilization of sawdust from Abura wood despite its availability and extensive utilization as timber in West Africa. Jekayinfa et al. [2] and Ojolo et al. [7] noted that a large quantity of residues is produced during logging. The forestry residues can be utilized effectively after densification into briquettes or pellets. This study investigates the effects of process variables on some physical and mechanical characteristics of briquettes made from Abura sawdust without the utilization binders and aims to develop empirical models to predict and optimize the properties of briquettes.

2. Materials and Methods

2.1. Acquisition and Properties of Sawdust

Abura (Mitragyna ciliata) is a tropical hardwood species which is largely available in West Africa, with lignin ranging between 19.0 and 21.5% [39]. Sawdust from processing Abura wood was acquired from an industrial sawmill in Lagos State, South West Nigeria. After acquisition, drying of the sawdust was carried out under ambient conditions in the laboratory to reduce the moisture content. The moisture content of the sawdust received at about 46.50% was reduced to 14.75% (wet basis) after drying. Thereafter, sieving was carried out to remove lumps, stones, and other large materials from the sawdust before they were stored in polyethylene bags. From the particle size analysis of the dried sawdust presented in Figure 1, the median particle size ($D_{50}$) was 0.85 mm. Particles greater than 3.35 mm constituted 3.33% of the sample, whilst particles less than 300 microns were 4.83%.

![Particle size distribution of sawdust.](image)

2.2. Experimental Briquetting Press

An experimental-scale uniaxial briquetting press developed at the Mechanical Engineering Department in the University of Lagos, Nigeria, was utilized for the manufacture of the briquettes. The press compacts biomass materials within a heated cylindrical mould using a hydraulic jack. The temperature of the mould was regulated using a thermocouple with a temperature regulator (Paulgrand, China). The pressure used in compaction was determined from a pressure gauge (Econosto, Germany) attached to the hydraulic jack. During operation, the mould was closed on one end and pressed from the other end. After the briquetting operation, the closed end was opened, and the hydraulic jack was used to...
push the compacted briquette out of the mould. Thereafter, the briquette was cooled, and the properties of the briquette were tested.

2.3. Design of Experiment

The investigation of the properties of the briquettes was carried out using RSM. A Box–Behnken design of experiment (DOE) with 3 factors and 3 levels was utilized. The process variables, or factors, that were investigated are compaction pressure, hold time, and temperature. The temperatures used were 100, 125, and 150 °C; hold times were 15.0, 22.5, and 30.0 min; and the compaction pressures utilized were 9, 12, and 15 MPa. The Box–Behnken DOE and the variables that were studied are presented in Table 1.

Table 1. Experimental design using Box–Behnken RSM.

<table>
<thead>
<tr>
<th>Exp. No.</th>
<th>Pressure (MPa)</th>
<th>Temperature (°C)</th>
<th>Hold Time (min)</th>
<th>CS (MPa)</th>
<th>SI</th>
<th>ρ (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9</td>
<td>100</td>
<td>22.5</td>
<td>2.54</td>
<td>50.0</td>
<td>539.5</td>
</tr>
<tr>
<td>2</td>
<td>9</td>
<td>125</td>
<td>15.0</td>
<td>2.27</td>
<td>166.7</td>
<td>555.6</td>
</tr>
<tr>
<td>3</td>
<td>15</td>
<td>125</td>
<td>15.0</td>
<td>3.48</td>
<td>150.0</td>
<td>596.4</td>
</tr>
<tr>
<td>4</td>
<td>12</td>
<td>100</td>
<td>30.0</td>
<td>2.66</td>
<td>100.0</td>
<td>677.3</td>
</tr>
<tr>
<td>5</td>
<td>12</td>
<td>100</td>
<td>15.0</td>
<td>2.71</td>
<td>200.0</td>
<td>518.8</td>
</tr>
<tr>
<td>6</td>
<td>9</td>
<td>150</td>
<td>22.5</td>
<td>2.53</td>
<td>150.0</td>
<td>601.7</td>
</tr>
<tr>
<td>7</td>
<td>15</td>
<td>125</td>
<td>30.0</td>
<td>3.38</td>
<td>600.0</td>
<td>738.1</td>
</tr>
<tr>
<td>8</td>
<td>12</td>
<td>125</td>
<td>22.5</td>
<td>3.76</td>
<td>100.0</td>
<td>669.5</td>
</tr>
<tr>
<td>9</td>
<td>15</td>
<td>150</td>
<td>22.5</td>
<td>4.58</td>
<td>300.0</td>
<td>815.9</td>
</tr>
<tr>
<td>10</td>
<td>15</td>
<td>100</td>
<td>22.5</td>
<td>2.59</td>
<td>150.0</td>
<td>621.6</td>
</tr>
<tr>
<td>11</td>
<td>12</td>
<td>150</td>
<td>15.0</td>
<td>3.74</td>
<td>50.0</td>
<td>680.9</td>
</tr>
<tr>
<td>12</td>
<td>9</td>
<td>125</td>
<td>30.0</td>
<td>3.47</td>
<td>300.0</td>
<td>647.7</td>
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<tr>
<td>13</td>
<td>12</td>
<td>125</td>
<td>22.5</td>
<td>3.01</td>
<td>50.0</td>
<td>675.0</td>
</tr>
<tr>
<td>14</td>
<td>12</td>
<td>125</td>
<td>22.5</td>
<td>2.06</td>
<td>100.0</td>
<td>669.9</td>
</tr>
<tr>
<td>15</td>
<td>12</td>
<td>150</td>
<td>30.0</td>
<td>5.15</td>
<td>50.0</td>
<td>823.9</td>
</tr>
</tbody>
</table>

2.4. Determination of Mechanical Properties of Briquettes

2.4.1. Compressive Strength

The lateral compressive strength of briquettes was determined rather than the longitudinal compressive strength since it is a more consistent measure of briquette strength [40]. Consequently, the ASTM D 3967-95a [41] was adopted for the determination of the lateral compressive strength. An analogue laboratory compression strength tester was used to carry out the compressive strength tests. The load was applied by manually operating the lever if the hydraulic pump of the machine and the value were read from an attached gauge. The load, \( F \), at which the briquette fractured was taken from the compression tester. The geometry of the cross-section of the lateral plane was estimated, which included the length of the briquette, \( L \), and diameter of the briquette, \( D \). The radial, or lateral, rupture or compressive strength (CS) was determined from Equation (1).

\[
CS = \frac{2F}{\pi DL}
\]

2.4.2. Shatter Index

The shatter index is a measure of durability of the briquettes which determines if they would crumble when transported or on impact due to collision. The shatter index tests were carried out on sawdust briquettes to determine the durability of the compacted biomass produced under different conditions. The procedure stated in ASTM D440-86 [42] and methods presented by Richards [43] were adopted to determine the shatter index. The test requires the briquette samples to be dropped onto a solid base from a height of 2 m. Each briquette was dropped repeatedly until it fractured. The number of drops, \( N \), and
the number of pieces, \( n \), the briquettes broke into were noted. The shatter index \((SI)\) was obtained from Equation (2) [40,43].

\[
SI = \frac{100 \times N}{n} 
\]  

(2)

2.4.3. Density

The density is the measure of compaction of the biomass into briquettes. The density of the sawdust briquettes was determined by dividing the mass of each briquette by the estimated volume of the briquette. The mass, \( m \), of the briquette was determined using an electronic weighing scale (WeiHeng, China; accuracy 0.01 g). The diameter, \( d \), and length, \( l \), of the briquette were measured using a vernier calliper. The density of each of the briquettes was determined from Equation (3).

\[
\rho = \frac{4m}{\pi d^2 l} 
\]  

(3)

2.5. Statistical Analysis

The responses from the experimental design were analysed utilizing Minitab (Version 19.1). Response surface models were developed using multiple regression analysis, and the models relate the independent variables to the responses. Analysis of Variance (ANOVA) was carried out to identify variables which influence the mechanical properties of the briquettes. ANOVA was also used to determine if the model was appropriate. Contour plots were utilized to determine the combined influence of the variables.

3. Results and Discussion

In Table 1, alongside the Box–Behnken experimental design, results for the compressive strength and shatter index of sawdust briquettes are shown. From Table 1, the shatter index ranged between 50 and 600. The compressive strength of sawdust briquettes ranged from 2.06 to 5.15 MPa. The range of compressive strength for both sawdust briquettes is lower than briquettes produced by Thliza et al. [44] and Borowski and Hycnar [45], which used binders. Similarly, the shatter index of most of the briquettes is above the minimum range of 50 specified by Richards [43]. The density of the briquettes ranged between 518.8 and 822.9 kg/m\(^3\).

3.1. Compressive Strength Analysis

The results of the compressive strength of sawdust briquettes were analysed using ANOVA. Figure 2 shows that the assumptions of ANOVA were not violated for the analysis of compressive strength of sawdust briquettes. As is required, the normal distribution plot can be fitted with a linear plot, and the points are not distant from the linear plot. Similarly, the residual versus fits plot is well distributed across the entire plot area, whereas the residual versus order plots are random and do not follow any distinct pattern. The ANOVA for compressive strength of sawdust briquettes is shown in Table 2, where backward elimination has been used in the elimination of the least influential terms from the response surface model. It is observed that only the temperature is significant at a level of significance of 5%. However, at a level of significance of 10\%, the pressure significantly affects the compressive strength of the briquettes. The model is, however, observed to be significant \((p < 0.05)\); however, the lack of fit of the model is not significant \((p > 0.05)\), implying that the model could be useful in the investigation of the influence of the variables on the strength of briquettes. Comparable to the observation in this study, Gao et al. [46] found that briquetting load was a very influential factor in the production of briquettes from sewage sludge. This also aligns with the finding of Helwani et al. [47], which showed that the pressure was significantly influential on the compressive strength of briquettes produced from palm oil stems using byproducts of biodiesel as adhesive. Mitchual et al. [48] noted that the compacting pressure was one of the variables which had a significant effect on the compressive strength of briquettes. Khlifi et al. [49] also observed that increasing compaction pressure directly
influenced the compressive strength of cassava residue briquettes. Okot et al. [50] found that the strength of briquettes increased with the increase in temperature and pressure. An increase in temperature aids the release of natural binders within the biomass cell structure which form solid bridges when cooled. This phenomenon has been observed from the observation of the scanning electron microscopy of briquettes from different materials. When pressure is applied at elevated temperature, the natural binders are squeezed out of the particles to improve particle-to-particle bonding. As observed from this study, Bazargan et al. [51] and Oliveira Maia et al. [52] found that the pressing or retention time had little to no significant influence on the strength of briquettes. Wilczyński et al. [53] noted that the pressure had the highest effect on the compressive strength of briquettes, which is contrary to observations from this study, namely that temperature has the highest effect on compressive strength.

![Figure 2](image)

**Figure 2.** Plots to check assumptions of ANOVA for compressive strength: (a) normal probability plots; (b) residual versus fits; (c) residual versus observation order.

**Table 2.** ANOVA for compressive strength.

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Adj SS</th>
<th>Adj MS</th>
<th>F-Value</th>
<th>p-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model</td>
<td>4</td>
<td>2.7665</td>
<td>0.6916</td>
<td>4.66</td>
<td>0.022</td>
</tr>
<tr>
<td>Linear</td>
<td>3</td>
<td>2.3569</td>
<td>0.7856</td>
<td>5.29</td>
<td>0.019</td>
</tr>
<tr>
<td>Pressure (MPa)</td>
<td>1</td>
<td>0.5253</td>
<td>0.5253</td>
<td>3.54</td>
<td>0.089</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>1</td>
<td>1.5312</td>
<td>1.5312</td>
<td>10.32</td>
<td>0.009</td>
</tr>
<tr>
<td>Hold time (min)</td>
<td>1</td>
<td>0.3003</td>
<td>0.3003</td>
<td>2.02</td>
<td>0.185</td>
</tr>
<tr>
<td>Two-Way Interaction</td>
<td>1</td>
<td>0.4096</td>
<td>0.4096</td>
<td>2.76</td>
<td>0.128</td>
</tr>
<tr>
<td>Pressure (MPa) × Temperature (°C)</td>
<td>1</td>
<td>0.4096</td>
<td>0.4096</td>
<td>2.76</td>
<td>0.128</td>
</tr>
<tr>
<td>Error</td>
<td>10</td>
<td>1.4841</td>
<td>0.1484</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lack-of-Fit</td>
<td>8</td>
<td>0.8976</td>
<td>0.1122</td>
<td>0.38</td>
<td>0.866</td>
</tr>
<tr>
<td>Pure Error</td>
<td>2</td>
<td>0.5865</td>
<td>0.2932</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>14</td>
<td>4.2506</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The response surface model derived from the regression modelling, with backward elimination, is presented in Equation (4). The values of $R^2$ and $R^2_{adj}$ are 58.02% and 46.57%, respectively.

\[
\text{Compressive strength} = 5.22 - 0.448P - 0.0337T + 0.00427PT \tag{4}
\]

The comparison between the estimated values for the compressive strength of sawdust briquettes and experimental values are presented in Figure 3. The figure shows that the model is quite useful in the prediction of the compressive strength of briquettes made from sawdust. The contour plots of the compressive strength of sawdust briquettes with the pressure and temperature are presented in Figure 4. From the plots, it is seen that the maximum compressive strength of the briquettes is obtained at the highest values of pressure (15 MPa) and temperature (150 °C). This is likely because at the high temperatures considered, natural components such as lignin are more readily available to bind the biomass particles. With the availability of the binder, a high pressure forces the biomass particles into close proximity, which makes them more compact. The combination of high pressure and temperature ensures that the particles are well bonded and sufficiently compact to resist external forces.

![Figure 3. Comparison of experimental values with predicted values of compressive strength.](image1)

![Figure 4. Contour plot of compressive strength with pressure and temperature.](image2)
3.2. Shatter Index Analysis of Briquettes

The responses for the shatter index obtained for sawdust briquettes were checked to verify that the assumptions of ANOVA were not violated. Figure 5 shows that the assumptions of ANOVA including normality, variance, and independence were not violated. Table 3 presents the ANOVA using backward elimination to check for the significant variables to the shatter index of briquettes. From Table 3, the significant terms influencing shatter index of briquettes are pressure and the square term of pressure ($p < 0.05$). Ossei-Bremang et al. [36] also discovered that pressure was influential in determining the shatter index of decanter cake and charred palm kernel shell briquettes using cardboard pulp as a binder. Similarly, Daniel et al. [54] found that the compaction pressure of composite briquettes of rice husk and bagasse was significant to the shatter index of briquettes using cassava and clay binders. Although the durability of briquettes produced by Nurek et al. [55] from logging residues was insufficient, the study recommended that compaction should be carried out at higher temperatures, having noted that higher durability was obtained with increased temperature. Rahaman and Salam [56] identified the significance of the compaction pressure in the cold densification of rice straw briquettes. A similar observation on the significance of pressure was made by Kimutai and Kimutai [57] for cashew nut shell briquettes. Contrary to findings from this study, Nganko et al. [58] noted that the shatter index of briquettes from carbonized sawdust using a binder was significantly influenced by the hold time, amongst other factors.

![Figure 5](image-url)

**Figure 5.** Plots to check assumptions of ANOVA for shatter index: (a) normal probability plots; (b) residual versus fits; (c) residual versus observation order.
Table 3. ANOVA for shatter index.

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Adj SS</th>
<th>Adj MS</th>
<th>F-Value</th>
<th>p-Value</th>
</tr>
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<tbody>
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<td>Model</td>
<td>5</td>
<td>189,694</td>
<td>37,938.9</td>
<td>3.30</td>
<td>0.057</td>
</tr>
<tr>
<td>Linear</td>
<td>2</td>
<td>106,836</td>
<td>53,418.1</td>
<td>4.65</td>
<td>0.041</td>
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<td>80,697.3</td>
<td>7.03</td>
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<tr>
<td>Hold time</td>
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<td>26,138.8</td>
<td>2.28</td>
<td>0.166</td>
</tr>
<tr>
<td>Square</td>
<td>2</td>
<td>99,871</td>
<td>49,935.6</td>
<td>4.35</td>
<td>0.048</td>
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<tr>
<td>Pressure × Pressure</td>
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<td>79,693.9</td>
<td>6.94</td>
<td>0.027</td>
</tr>
<tr>
<td>Hold time × Hold time</td>
<td>1</td>
<td>26,195</td>
<td>26,194.8</td>
<td>2.28</td>
<td>0.165</td>
</tr>
<tr>
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<td>25,074.7</td>
<td>2.18</td>
<td>0.174</td>
</tr>
<tr>
<td>Pressure × Hold time</td>
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<td>25,075</td>
<td>25,074.7</td>
<td>2.18</td>
<td>0.174</td>
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<td>Error</td>
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<td>11,482.5</td>
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</tbody>
</table>

Based on the terms considered in the ANOVA, the model developed is stated in Equation (5). The model has $R^2$ and $R^2_{adj}$ values of 64.73% and 45.14%, respectively.

$$Shatter\ index = 3647 - 448P - 101.4H + 16.28P^2 + 1.493H^2 + 3.52PH$$ (5)

Figure 6 shows plots comparing the predicted values with actual data obtained from the experimental data. The plots are seen to have a similar trend, which shows the applicability of the model for prediction.

Figure 7 shows the contour plot of the combined effects of the pressure and hold time on the shatter index of the sawdust briquettes. The figure reveals that the maximum value of the shatter index of briquettes is obtained when the pressure and hold time are at the highest setting. The findings on the relationship of shatter index with pressure are similar to observations from the studies of Adu-Poku et al. [59] and Ossei-Bremang et al. [36]. The studies observed that the shatter index of the briquettes produced increased with increases in the compaction pressure. Rajaseenivasan et al. [60] also found that the shatter index increases with increases in compaction pressure for neem and sawdust briquettes. Ibitoye et al. [61] also noted that the durability improves with compaction pressure. Bhowmik and Bora [62] stated that the shatter index of Sal leaves increases with pressure.
Figure 7. Contour plot of shatter index with pressure and hold time.

3.3. Density Analysis of Briquettes

The assumptions of ANOVA were checked for compliance using Figure 8. From the figures, it can be seen that the assumptions of ANOVA are fulfilled. Table 4 therefore presents the results of the ANOVA for density, applying backward elimination to remove the least significant terms. From the ANOVA, all of the linear terms including the pressure, temperature, and hold time significantly influence the density of the sawdust briquettes. The most significant interaction is that of the pressure and temperature. The study on briquettes made from maize cob by Okot et al. [50], Jekayinfa et al. [32], and Orisaleye et al. [26] reveal that pressure and temperature are amongst the most significant factors influencing briquette density. A similar observation was made in a study by Wilczyński et al. [53], where it was noted that compaction pressure had the highest effect on density, followed by temperature. Contrarily, in this study, temperature had the highest effect, followed by hold time and pressure, respectively.

From the ANOVA, the model is significant, and the developed model is shown in Equation (6). The model has $R^2$ and $R^2_{adj}$ values of 95.34% and 92.75%, respectively.

$$Density = 22 + 53.6P - 2.46T + 8.92H - 3.78P^2 + 0.440PT$$  \(6\)

Figure 9 shows plots comparing the predicted values of the density with actual data obtained from the experiments. It is seen that the predicted and experimental values are close, showing that the model is suitable for prediction. The combined effects of the variables are presented in Figure 10. From Figure 10a–c, it can be observed that the highest density is obtained at the highest values of pressure, temperature, and hold time. This signifies that the density increases with increasing pressure, temperature, and hold time. Ibitoye et al. [61] noted that the density increases as the compaction pressure increases. Ekoube et al. [63] noted that the density of rice husk briquettes increased with increases in temperature. Similarly, Kimutai [64] found that pressure was one of the factors that positively influenced the density of cashew nut and cassava binders. Chen et al. [65] noted that temperature is crucial to determine the quality of pellets from bagasse, with high temperatures resulting in homogenous, smooth, and shiny pellets with no cracks. From the study of Bhowmik and Bora [62], the density of briquettes from Sal fallen leaves was found to increase with increasing pressure.
Compaction pressure had the highest effect on density, followed by temperature. Contrarily, in this study, temperature had the highest effect, followed by hold time and pressure, respectively. From the ANOVA, the model is significant, and the developed model is shown in Equation (6). The model has $R^2$ and $R^2_{adj}$ values of 95.34% and 92.75%, respectively.

$$\text{Density} = 22 + 53.6P - 2.46T + 8.92H - 3.78P^2 + 0.440PT$$

Figure 8. Plots to check assumptions of ANOVA for density: (a) normal probability plots; (b) residual versus fits; (c) residual versus observation order.

Figure 9 shows plots comparing the predicted values of the density with actual data obtained from the experiments. It is seen that the predicted and experimental values are close, showing that the model is suitable for prediction. The combined effects of the variables are presented in Figure 10. From Figure 10a–c, it can be observed that the highest density is obtained at the highest values of pressure, temperature, and hold time. This signifies that the density increases with increasing pressure, temperature, and hold time. Ibitoye et al. [61] noted that the density increases as the compaction pressure increases. Ekoube et al. [63] noted that the density of rice husk briquettes increased with increases in temperature. Similarly, Kimutai [64] found that pressure was one of the factors that positively influenced the density of cashew nut and cassava binders. Chen et al. [65] noted that temperature is crucial to determine the quality of pellets from bagasse, with high temperatures resulting in homogenous, smooth, and shiny pellets with no cracks. From the study of Bhowmik and Bora [62], the density of briquettes from Sal fallen leaves was found to increase with increasing pressure.

Table 4. ANOVA for density.

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Adj SS</th>
<th>Adj MS</th>
<th>F-Value</th>
<th>p-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model</td>
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<td>107,250</td>
<td>21,450.1</td>
<td>36.80</td>
<td>0.000</td>
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<tr>
<td>Linear</td>
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<td>98,563</td>
<td>32,854.4</td>
<td>56.37</td>
<td>0.000</td>
</tr>
<tr>
<td>Pressure (MPa)</td>
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<td>22,843.8</td>
<td>39.19</td>
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</tr>
<tr>
<td>Temperature (°C)</td>
<td>1</td>
<td>39,915</td>
<td>39,914.8</td>
<td>68.48</td>
<td>0.000</td>
</tr>
<tr>
<td>Hold time (min)</td>
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<td>35,805</td>
<td>35,804.8</td>
<td>61.43</td>
<td>0.000</td>
</tr>
<tr>
<td>Square</td>
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<td>4327</td>
<td>4326.9</td>
<td>7.42</td>
<td>0.023</td>
</tr>
<tr>
<td>Pressure (MPa) $\times$ Pressure (MPa)</td>
<td>1</td>
<td>4327</td>
<td>4326.9</td>
<td>7.42</td>
<td>0.023</td>
</tr>
</tbody>
</table>
Table 4. Cont.

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Adj SS</th>
<th>Adj MS</th>
<th>F-Value</th>
<th>p-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Two-Way Interaction</td>
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<td>4360</td>
<td>4360.1</td>
<td>7.48</td>
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</tr>
<tr>
<td>Pressure (MPa) × Temperature (°C)</td>
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<td>4360</td>
<td>4360.1</td>
<td>7.48</td>
<td>0.023</td>
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<tr>
<td>Error</td>
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<td>582.8</td>
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<tr>
<td>Lack-of-Fit</td>
<td>7</td>
<td>5227</td>
<td>746.7</td>
<td>79.82</td>
<td>0.012</td>
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<tr>
<td>Pure Error</td>
<td>2</td>
<td>19</td>
<td>9.4</td>
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<td></td>
</tr>
<tr>
<td>Total</td>
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<td>112,496</td>
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<td></td>
</tr>
</tbody>
</table>

Figure 10. Contour plot of density with (a) pressure and temperature; (b) pressure and hold time; and (c) temperature and hold time.

3.4. Relationship between Quality Parameters

The Pearson correlation was utilized to determine if there was a relationship between the compressive strength, shatter index, and density. From Table 5, there is a strong positive correlation between the density and the compressive strength. Weak positive correlation, however, exist between the compressive strength and shatter index, and the shatter index and density. The implication of this is that briquettes with a high density would have high compressive strength. The positive correlation, though slight, of the shatter index with the density, implies that the shatter index increases with the density of the briquettes. Brand et al. [66] obtained a correlation coefficient of 0.47 for the relationship between...
compressive strength and density of bamboo briquettes, whilst Dias Junior et al. [67] obtained a correlation coefficient of 0.60. A strong positive correlation of density with compressive strength was also obtained by Kpalo et al. [68], Rhén et al. [69], Taulbee et al. [70], and Gendek [71].

Table 5. Pearson correlation for quality parameters.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>CS</th>
<th>SI</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS</td>
<td>1.000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SI</td>
<td>0.104</td>
<td>1.000</td>
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</tr>
<tr>
<td>D</td>
<td>0.763</td>
<td>0.217</td>
<td>1.000</td>
</tr>
</tbody>
</table>

4. Conclusions

The effects of process variables on the strength and durability of briquettes produced from Abura sawdust were investigated in this study. The lateral compressive strength of Abura sawdust briquettes was between 2.06 and 5.15 MPa. The shatter index of the sawdust briquettes was between 50 and 600, and the density was between 518.8 and 822.9 kg/m³. From the statistical analysis, the pressure was found to be significant to the determination of the compressive strength (\( p < 0.1 \)) and the shatter index (\( p < 0.05 \)). The pressure, temperature, and hold time were all significant factors impacting the density of the briquettes. The empirical models developed from the RSM were good predictors for the lateral compressive strength and shatter index of the briquettes, but had low values for the coefficients of determination. The optimum compressive strength was obtained at the maximum pressure of 15 MPa and temperature of 150 °C. The highest shatter index was obtained at the maximum pressure and hold time of 30 min. With the proper selection of densification variables, strong and durable briquettes can be produced. Increasing the pressure, temperature, and hold time positively affects the briquette density. Density has a strong positive correlation with compressive strength, but a weak positive correlation with shatter index.


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Conflicts of Interest: The authors declare no conflicts of interest.

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