# Effect of densification variables on water resistance of corn cob briquettes

J.I. Orisaleye[1], S.O. Jekayinfa[2], R. Pecenka[3,*] and T.B. Onifade[2]

[1]University of Lagos, Faculty of Engineering, Department of Mechanical Engineering, PMB 56, Akoka, Nigeria
[2]Ladoke Akintola University of Technology, Faculty of Engineering and Technology, Department of Agricultural Engineering, PMB 4000, Ogbomoso, Nigeria
[3]Leibniz Institute for Agricultural Engineering and Bioeconomy, ATB, Department of Post-Harvest Technology, Max-Eyth-Allee 100, DE14469 Potsdam, Germany
*Correspondence: rpecenka@atb-potsdam.de

**Abstract.** Solid biofuels can be used in heat and power generation applications. The utilization of agricultural residues for this purpose would be of immense benefit to rural communities of developing countries where the resource is being produced. Water resistance is a crucial property for transport and storage of biomass briquettes under moist climate conditions. In this study, the effect of process and material variables on the water resistance property of corn cob briquettes was investigated. The water resistance of briquettes produced ranged between 32.6 and 94.8% for die temperature between 90 °C and 120 °C, hold time from 7.5 to 15 minutes and die pressures between 9 and 15 MPa. A higher die temperature resulted in an increase in the water resistance of the biomass briquettes. Also, increasing the hold time improved the water resistance of the briquettes. Using a particle size less than 2.5 mm resulted in higher briquette water resistance property compared to briquettes produced from particle sizes greater than 2.5 mm. It was also shown that the effect of the interaction of the temperature with particle size on the water resistance of corn cob briquettes was statistically significant ($p < 0.05$).

**Key words:** biomass briquette, temperature, particle size, pressure, hold time, uniaxial compaction.

## INTRODUCTION

Biofuels are being considered for replacement of fossil fuels in existing energy and power technologies due to the ease of finding, low price, carbon neutral feature and very high regional and global potentials (Yokoyama et al., 2000; Kaygusuz & Türker, 2002; Omer, 2005; Moreira, 2006; Haykiri-Acma & Yaman, 2010; Ojolo et al., 2012). The huge potential is yet to be fully utilized for the generation of energy particularly in the rural areas of the developing countries where there is an abundance of agricultural residues (Jekayinfa & Scholz, 2009). Solid biofuels are mainly used for the production of heat and/or electricity (FAO, 2009; Voytenko, 2010). Combustion technologies produce energy from biomass, converting biomass fuels into several forms of useful energy, e.g. hot air, hot water, steam and electricity. Biomass combustion covers basic

energy requirements for cooking in rural households. It also provides heat for processes in a variety of traditional industries in the developing countries. Commercial and industrial combustion plants can burn many types of biomass ranging from woody biomass to municipal solid waste. One of the primary determinants in the selection and design of any biomass combustion system is the characteristics of the fuel. Conversion of biomass into briquettes improves the fuel characteristics and allows for automating its feeding into combustion systems (Bhattacharya, 2003; Rosillo-Calle, 2007; Ojolo & Orisaleye, 2010).

Quality attributes of densified biomass play a major role in the end-user applications (Tumuluru et al., 2010). Quality parameters include the moisture content, unit and bulk density, durability index, compressive strength, percent fines and calorific values. Furthermore, storability and transport behaviour are important characteristics of solid biomass fuels for its availability all over the year. Water resistance of briquettes shows the resistance to absorption of water during transportation and storage. The water resistance property is crucial for biomass briquettes which must be transported through high humidity environment.

Some studies have determined the resistance to water penetration based on the quantity of water absorbed within a specified period when the briquettes are fully immersed in water. Sengar et al. (2012) stated that the resistance to water penetration of cashew shell briquettes was better when compared with grass and rice husk briquettes. Saha et al. (2014) determined the water resistance of coconut coir dust briquettes and rice husk briquettes and stated that the briquettes made from mixed coconut coir dust and rice husk had desirable properties when compared to rice husk briquette. Lindley & Vossoughi (1989) noted that briquettes made from sunflower stalk, wheat straw, and flax straw had average weight gains of 9.9, 32.3 and 38.1% after immersion for 30 seconds in water at room temperature. Davies & Davies (2013a) stated that water hyacinth briquettes produced with small particle sizes had higher water resistance characteristic compared to briquettes produced from larger particle sizes. Davies & Davies (2013b) obtained results between $52 \pm 2.42\%$ to $97.1 \pm 3.39\%$ for the water resistance property of water hyacinth briquettes for five binder levels studied.

Other studies, however, determined the water resistance quality as the time taken for the briquette to disperse in water at room temperature. Križan (2007) stated that when the briquette disperses in water in less than 5 minutes, the briquette is of low quality while high quality briquettes should have dispersion times up to 20 minutes. Mitchual (2015) observed that water resistance quality of briquettes produced from different biomass materials used ranged between 1.01 to 6.63 minutes. Li & Liu (2000) reported that briquettes produced from oak sawdust, pine sawdust, and cottonwood sawdust disintegrated in less than 5 minutes after immersion in water at room temperature.

Pilipchuk et al. (1975) have also stated that high temperature pressing of wood increases the reaction rate and the accumulation of high molecular products, mostly lignin. It was further stated that the accumulation of partly melted and insoluble high-molecular substances in the voids of capillary and submicrocapillary systems will prevent the soaking of water into the cell walls. This inhibits the swelling and secures the water resistance of the wood base laminate (Zandersons et al., 2004).

Some studies have been carried out on the investigation of properties of briquettes produced from corn cobs, but they have either been limited to the use of binders (Oladeji & Enweremadu, 2012; Muazu & Stegemann, 2015; Oyelaran & Tudunwada, 2015), or

composite briquetting with other materials (Ikelle & Ivoms, 2014; Muazu & Stegemann, 2015; Oyelaran & Tudunwada, 2015; Nurhayati et al., 2016). Studies on production of binderless briquettes did not consider the water resistance property of the briquettes (Kaliyan & Morey, 2010a; Okot et al., 2018; Orisaleye et al., 2018). Studies which consider the effects of interactions of densification variables on water resistance of briquettes are also rare. This study, therefore, aims to determine the effects of pressure, temperature, hold time and particle size and their interactions on the water resistance property of binderless briquettes produced from corn cobs.

## MATERIALS AND METHODS

### Materials
Corn cobs were collected from a corn processing firm in Lagos, Nigeria. The corn cobs were then air-dried for three weeks to remove excess moisture within the cobs. Afterwards, the corn cobs were milled in a commercial hammer milling machine. The milled corn cobs were sieved using a 2.5 mm mesh sieve into two particle sizes – less than 2.5 mm and greater than 2.5 mm.

Preliminary experiments for the determination of moisture content were carried out by following procedures outlined in ASTM D 4442-92 standard test methods. The difference in the masses, $\Delta$m, was determined and the moisture content was computed using:

$$Moisture\ content\ (\%) = \frac{\Delta m}{m_i} \times 100 \qquad (1)$$

The initial mass of corn cob sample is $m_i$; $\Delta m$ is the change in mass. The moisture content in the milled air-dried corn cobs was determined to be 9.8%.

### The briquetting press
The uniaxial briquetting press used for the production of briquettes was locally developed and is shown in Fig. 1. The press produces one briquette in a setting and the briquette diameter is 50 mm. The components of the briquetting press are a 5 tonnes hydraulic jack, die, heating band rated at 1,000 W, tie bar, pressure gauge (Econosto, EN 837-1, Germany), cover plate and bottom plate. The hydraulic jack is used to apply the required pressure to the raw biomass and the heating band which is controlled by a temperature controller (Jetec, JTC-903, China) supplies the heat required to raise the temperature of the biomass.
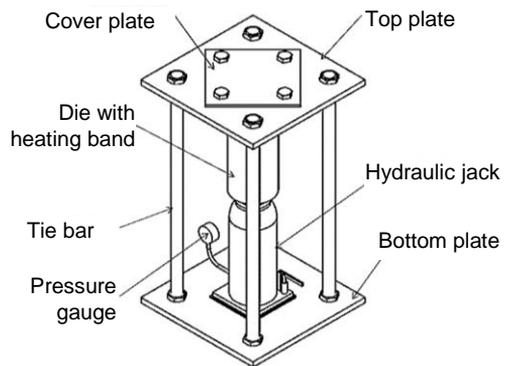


**Figure 1.** The developed experimental biomass briquetting Press.

During operation, the top of the hydraulic jack was set to the bottom dead centre of the die. The electric heater, which surrounds the die, was then powered on and set to the required temperature. At this position of the hydraulic jack, biomass material was loaded into the die until it was filled up. The top plate was then fastened to the die. The hydraulic

jack was operated until the required pressure is reached. At this pressure, the apparatus was left to stand for the required hold time. At the end of the period, the top plate was removed from the die and the hydraulic jack operated until the briquette reached the top dead centre of the die. The briquette formed was picked away from the top of the piston of the hydraulic jack and allowed to cool and stabilize for one hour.

### Design of experiments

The effects of temperature, pressure, hold time and particle size on the density of biomass briquettes were determined during the experiments. The temperatures used were 90 °C and 120 °C. The pressures used were 9 MPa, 12 MPa and 15 MPa. The hold time used were 7.5 minutes and 15 minutes. All possible combinations of values of pressure, temperature, hold time and particle sizes ($< 2.5$ mm and $> 2.5$ mm) were utilized during the experiments. The variables were coded using Table 1. A full factorial experimental design was used which considers all possible combinations of variables being studied. The experimental design is shown in Table 2.

### Determination of water resistance

The water resistance is determined from the percentage of water absorbed by a briquette when immersed in water. The briquettes formed from each experimental run in Table 2 were immersed in water at room temperature for 30 seconds. The procedure follows studies of Birwatkar et al. (2014), Sengar et al. (2012) and Saha et al. (2014). The column of water in bucket used was 150 mm high at room temperature. The resistance to water penetration is determined from:

**Table 1.** Coded values of densification variables

| Variable | -1 (Low) | 0 (Medium) | 1 (High) |
|---|---|---|---|
| Pressure | 9 MPa | 12 MPa | 15 Mpa |
| Temperature | 90 °C | - | 120 °C |
| Hold time | 7.5 mins. | - | 15 mins. |
| Particle size | $< 2.5$ mm | - | $> 2.5$ mm |

**Table 2.** Full factorial experimental design for the study of water resistance of corn cob briquettes using coded variables

| Exp. No. | Pressure | Temp. | Hold time | Particle size |
|---|---|---|---|---|
| 1 | -1 | -1 | -1 | -1 |
| 2 | 0 | -1 | -1 | -1 |
| 3 | 1 | -1 | -1 | -1 |
| 4 | -1 | 1 | -1 | -1 |
| 5 | 0 | 1 | -1 | -1 |
| 6 | 1 | 1 | -1 | -1 |
| 7 | -1 | -1 | 1 | -1 |
| 8 | 0 | -1 | 1 | -1 |
| 9 | 1 | -1 | 1 | -1 |
| 10 | -1 | 1 | 1 | -1 |
| 11 | 0 | 1 | 1 | -1 |
| 12 | 1 | 1 | 1 | -1 |
| 13 | -1 | -1 | -1 | 1 |
| 14 | 0 | -1 | -1 | 1 |
| 15 | 1 | -1 | -1 | 1 |
| 16 | -1 | 1 | -1 | 1 |
| 17 | 0 | 1 | -1 | 1 |
| 18 | 1 | 1 | -1 | 1 |
| 19 | -1 | -1 | 1 | 1 |
| 20 | 0 | -1 | 1 | 1 |
| 21 | 1 | -1 | 1 | 1 |
| 22 | -1 | 1 | 1 | 1 |
| 23 | 0 | 1 | 1 | 1 |
| 24 | 1 | 1 | 1 | 1 |

$$\%WR = 100 - \left[\frac{M_{wet\ briquette} - M_{initial}}{M_{initial}} \times 100\right] \qquad (2)$$

where $\%WR$ – water resistance in percentage; $M_{wet\ briquette}$ – mass of wet briquette; $M_{initial}$ – initial mass of briquette.

# RESULTS AND DISCUSSION

The briquettes produced using the experimental runs in Table 2 had densities ranging between 570 and 1,300 kg m$^{-3}$. This depended on the operating conditions specified in the experimental runs. The results of the graphical and statistical analysis of the effects of the temperature, pressure, hold time and particle size on the water resistance of briquettes are presented in this section.

### Effect of temperature on the water resistance of corn cob briquettes

Figs 2–5 show the effect of temperature on the water resistance of biomass briquettes produced from corn cobs. It is observed that the water resistance, which ranges from 32.6% to 94.8%, increases for all particle sizes, die pressures and hold times with an increase in the temperature from 90 °C to 120 °C. It has been noted from previous work (Kaliyan & Morey, 2009) that increased temperature is required to activate the natural binding components such as lignin, protein, starch and water-soluble carbohydrates. The increased water resistance may likely be the result of improved bonding between particles due to better flow or plastic properties of the binding components at higher temperature. Lignin, which is the primary binding agent, has been identified to be hydrophobic in nature (Anglés et al., 2001; Kaliyan & Morey, 2010b) which would increase the water resistance of the briquettes. Pilipchuk et al. (1975) and Zandersons et al. (2004) have stated that melted lignin prevents soaking of water into cell walls.
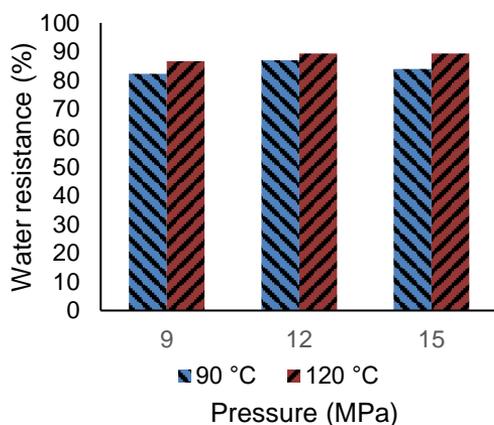


**Figure 2.** Water resistance of corn cob briquettes produced at different die temperature with a hold time of 7.5 minutes and particle size less than 2.5 mm.
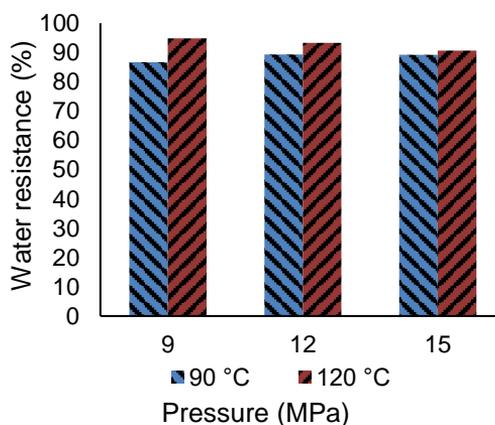
**Figure 3.** Water resistance of corn cob briquettes produced at different die temperature with a hold time of 15 minutes and particle size less than 2.5 mm.
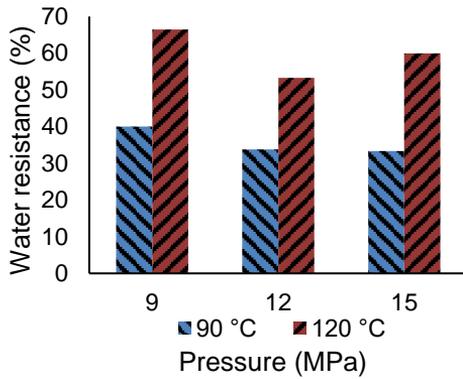
**Figure 4.** Water resistance of corn cob briquettes produced at different die temperature with a hold time of 7.5 minutes and particle size greater than 2.5 mm.
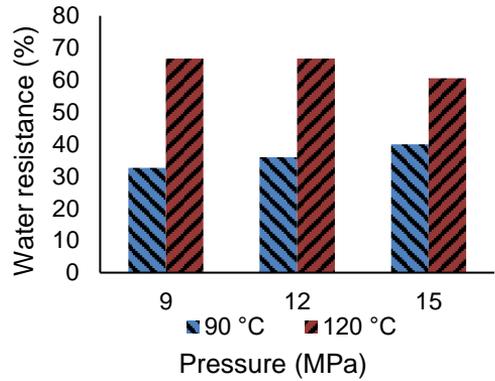
**Figure 5.** Water resistance of corn cob briquettes produced at different die temperature with a hold time of 15 minutes and particle size greater than 2.5 mm.

### Effect of hold time on the water resistance of corn cob briquettes

The influence of hold time on water resistance of the corn cob briquettes is shown in Figs 6–9. It is seen that the water resistance using a longer hold time of 15 minutes was mostly higher compared to when a hold time of 7.5 minutes was used.
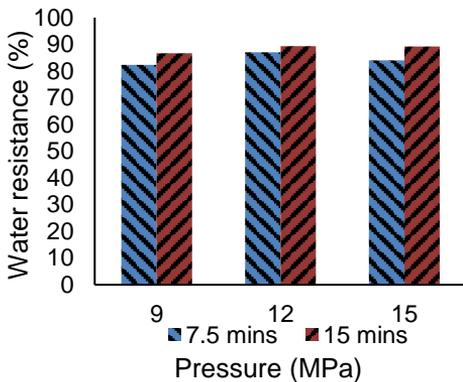




**Figure 6.** Water resistance of corn cob briquettes produced at different hold times with die temperature of 90 °C and particle size less than 2.5 mm.
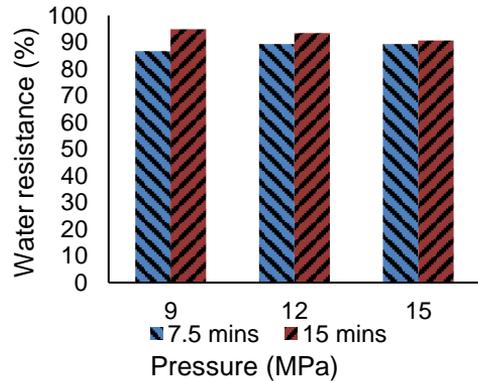
**Figure 7.** Water resistance of corn cob briquettes produced at different hold times with die temperature of 120 °C and particle size less than 2.5 mm.

Fig. 8, however showed that for particle size greater than 2.5 mm, die pressure of 9 MPa and temperature of 90 °C respectively, the water resistance for the hold time of 15 minutes is lower than that obtained using 7.5 minutes. The increase of water resistance with increased hold time is probably due to the lignin and other natural binding materials having time to coat particles and ensure stronger bonds between the particles. He et al. (2011), have however, stated that a longer holding time did not guarantee better water resistance when lignite briquettes had already gained water resistance through the heat treatment process.
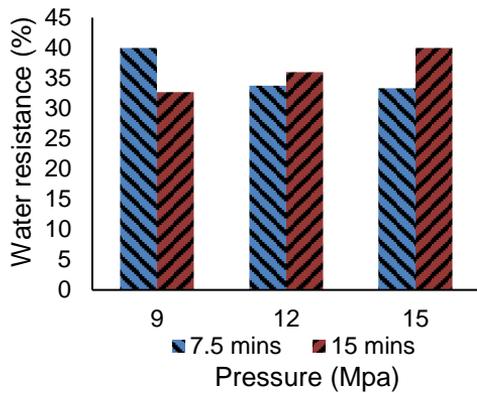
**Figure 8.** Water resistance of corn cob briquettes produced at different hold times with die temperature of 90 °C and particle size greater than 2.5 mm.
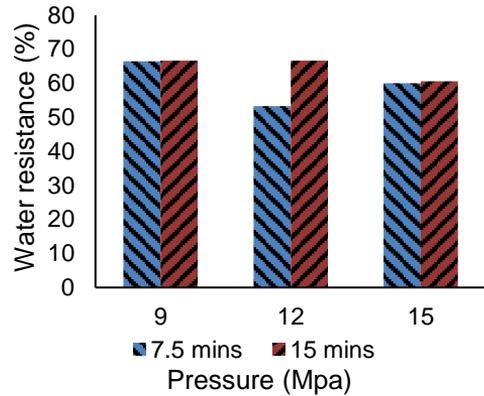


**Figure 9.** Water resistance of corn cob briquettes produced at different hold times with die temperature of 120 °C and particle size greater than 2.5 mm.

### Effect of pressure on water resistance of corn cob briquettes

The effect of pressure on the water resistance of corn cob briquettes is shown in Figs 2–9 and Figs 10–13. The figures show that there is no clear pattern for the effect of pressure on water resistance. This indicates that pressure at the level applied during these experiments is not a significant factor in determining the water resistance of corn cob briquettes. Contrary to results obtained, Mitchual (2015) stated that compacting pressure between 20 and 50 MPa, together with interactions with biomass materials, had significant effect on quality of briquettes produced from sawdust of *Piptadenia Africana* and *Ceiba pentandra* at room temperature. Mitchual (2015) noted that water resistance quality of briquettes produced from the using low compacting pressure was low. Zanjani et al. (2014) also stated that the increase of briquetting pressure showed positive but insignificant effects on water resistance index.
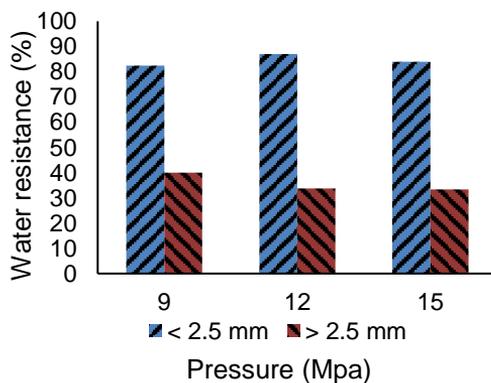


**Figure 10.** Water resistance of corn cob briquettes produced at different particle sizes with die temperature of 90 °C and hold time of 7.5 minutes.
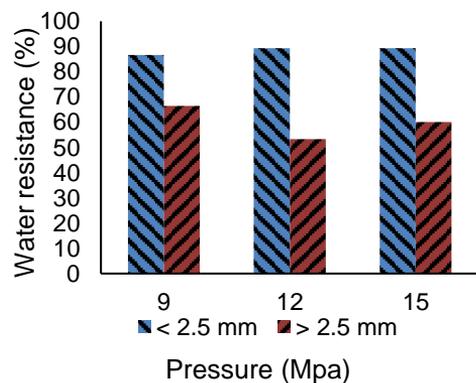


**Figure 11.** Water resistance of corn cob briquettes produced at different particle sizes with die temperature of 120 °C and hold time of 7.5 minutes.
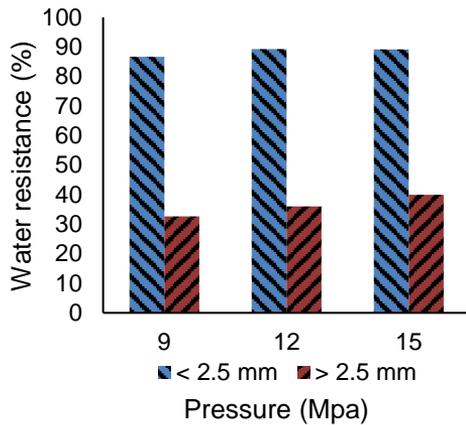
**Figure 12.** Water resistance of corn cob briquettes produced at different particle sizes with die temperature of 90 °C and hold time of 15 minutes.
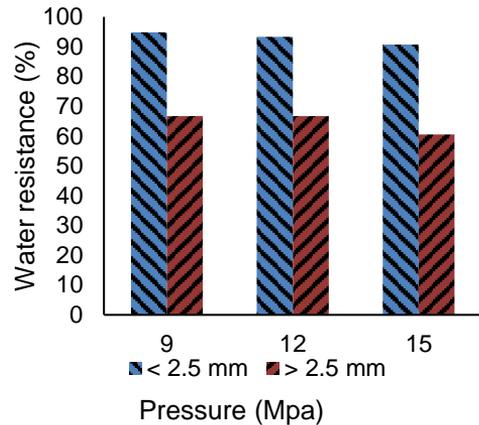
**Figure 13.** Water resistance of corn cob briquettes produced at different particle sizes with die temperature of 120 °C and hold time of 15 minutes.

**Effect of particle size on water resistance of corn cob briquettes**

The effect of particle size on the water resistance of corn cob briquettes is indicated in Figs 10–13. It is shown that for all combination of variables, the water resistance of briquettes produced with smaller particle sizes of corn cob (< 2.5 mm) was higher than those produced with larger sized particles (> 2.5 mm). This is likely due to the better bonding which occurs with smaller particle sizes which results from the increase in the contact points for inter-particle bonding. For briquettes produced with larger particle sized materials, there are wider inter-particle voids which limits the bonding of the particles, thereby reducing the water resistance of the briquettes. In addition, the surface of the briquettes produced using smaller particle sizes was smoother than those produced using the larger particle sizes. This indicates that the pores on the surface of the briquettes produced with larger particle sizes was larger and allowed easier penetration of water into the briquettes. Križan et al. (2018) also observed that the is a positive relationship between particle size and porosity. Davies & Davies (2013a) also observed that the water resistance capacity of briquettes progressively improved with decrease in particle sizes. Some of the briquettes produced using particle sizes greater than 2.5 mm are shown in Fig. 14, (a) while Fig. 14, (b) shows briquettes produced with particle sizes less than 2.5 mm.
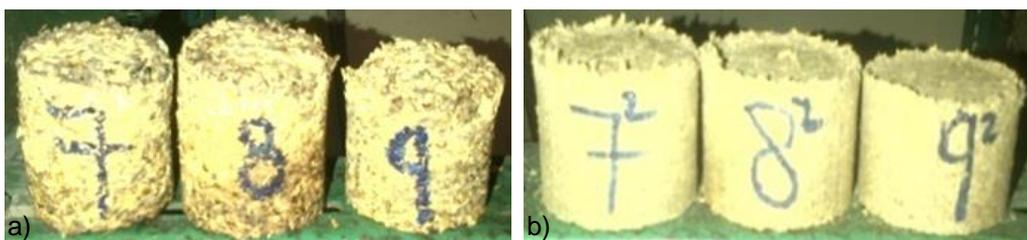


**Figure 14.** Briquettes produced with (a) particle sizes greater than 2.5 mm (b) particle sizes less than 2.5 mm.

**Statistical analysis**

The effects of the densification have also been investigated using statistical methods of analysing factorial experimental designs using the analysis of variance (ANOVA). The assumptions made in ANOVA include normality, constant variance and independence. Normality of the ANOVA implies that the population in each treatment from which the sample is drawn is normally distributed. Constant variance implies that the variance of observations in each treatment should be equal. Independence is the assumption requiring that the observations should be randomly selected from the treatment population. These assumptions need to be checked for the ANOVA before deductions are made from the analysis.

Fig. 15 shows the normality probability plot of residuals for water resistance of corn cob briquettes. The plot shows that the distribution of residuals is normal. Fig. 16 shows the plot of residuals versus fitted means. It is observed that the plot has a random pattern and shows no recognizable pattern which satisfies the constant variance assumption of ANOVA. The independence assumption is checked with the plot of residual versus observation order shown in Fig. 17. The plot shows that the assumption of independence is met since there is no defined pattern for the plot.
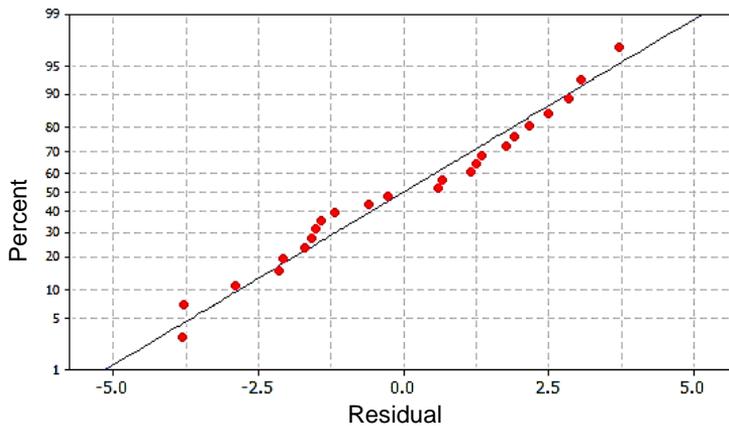


**Figure 15.** Normal probability plot of residual for water resistance of corn cob briquettes.
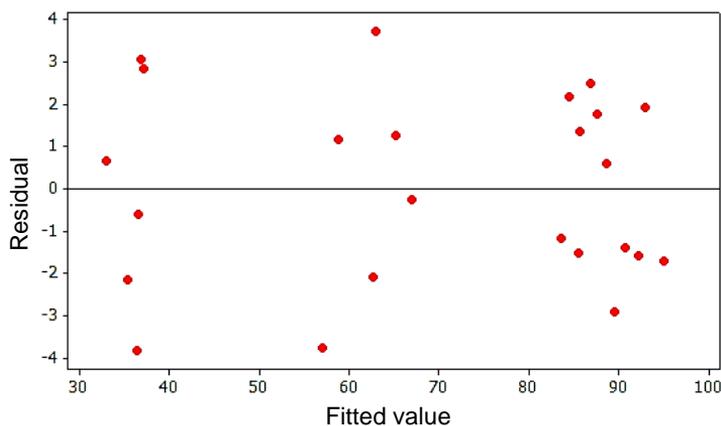


**Figure 16.** Plot of residual versus fits for water resistance of corn cob briquettes.
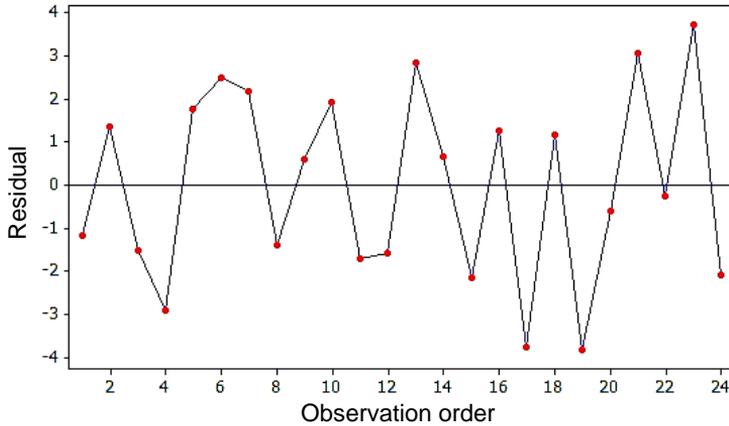
**Figure 17.** Plot of residuals versus fits for water resistance of corn cob briquettes.

Table 3 shows the ANOVA table for the water resistance of corn cob briquettes. It is shown from the tables that the temperature, hold time and particle size have P-values less than a significance level of 0.05. This implies that their effects on the water resistance of corn cob briquettes are statistically significant. It is also observed that the interaction of temperature with particle size also has a statistically significant effect on the water resistance of the corn cob briquettes.

**Table 3.** Analysis of variance (ANOVA) table for water resistance of corn cob briquettes

| Source | DF | Seq SS | Adj SS | Adj MS | F | P |
|---|---|---|---|---|---|---|
| Pressure | 2 | 6.11 | 6.11 | 3.05 | 0.24 | 0.788 |
| Temperature | 1 | 1,403.62 | 1,403.62 | 1403.62 | 112.47 | 0.000** |
| Hold time | 1 | 70.11 | 70.11 | 70.11 | 5.62 | 0.042** |
| Particle size | 1 | 9,326.77 | 9,326.77 | 9,326.77 | 747.36 | 0.000** |
| Pressure *Temperature | 2 | 26.39 | 26.39 | 13.19 | 1.06 | 0.387 |
| Pressure *Hold time | 2 | 17.25 | 17.25 | 8.62 | 0.69 | 0.526 |
| Pressure *Particle size | 2 | 38.00 | 38.00 | 19.00 | 1.52 | 0.269 |
| Temperature *Hold time | 1 | 8.35 | 8.35 | 8.35 | 0.67 | 0.434 |
| Temperature *Particle size | 1 | 729.74 | 729.74 | 729.74 | 58.48 | 0.000** |
| Hold time *Particle size | 1 | 3.95 | 3.95 | 3.95 | 0.32 | 0.587 |
| Error | 9 | 112.32 | 112.32 | 12.48 | | |
| Total | 23 | 11,742.62 | | | | |

**Statistically significant.

The main effects plot for the water resistance is shown in Fig. 18. It shows that the temperature and hold time improve the water resistance of the corn cob briquettes when a higher value is used for each of them. However, a higher particle size will have a negative effect on the water resistance of the briquettes. The interaction plot for the water resistance is shown in Fig. 19 which shows that briquettes with the highest water resistance are produced with higher temperature and lower particle size.
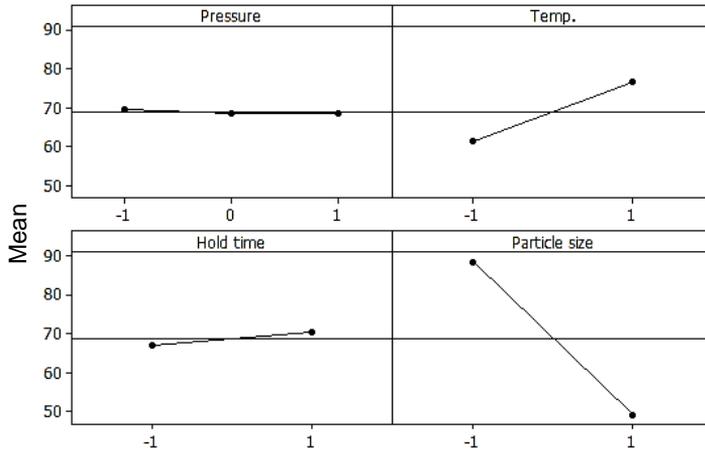
**Figure 18.** Main effects plot using data means for water resistance of corn cob briquettes.
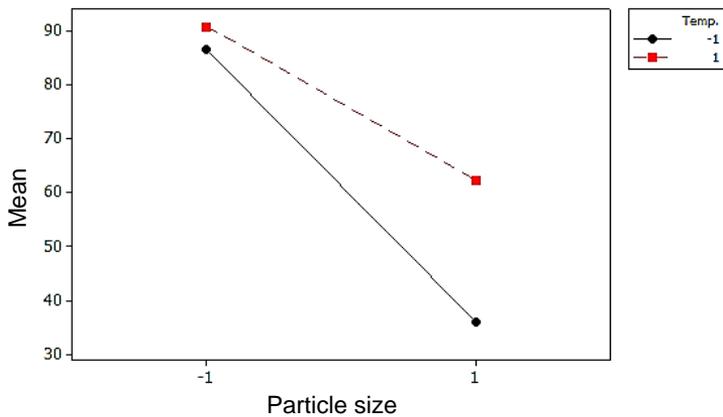


**Figure 19.** Interaction plot using data means for water resistance of corn cob briquettes.

## CONCLUSIONS

In this study, the effect of pressure, temperature, hold time and particle size on the water resistance property of briquettes produced from corn cob was investigated. The water resistance of briquettes produced ranged between 32.6% and 94.8%. Graphical analysis showed that increasing the die temperature from 90 °C to 120 °C resulted in an increase in the water resistance of the biomass briquettes. Also, increasing the hold time from 7.5 minutes to 15 minutes increased the water resistance of the briquettes. Using a particle size less than 2.5 mm resulted in higher briquette water resistance property compared to briquettes produced from particle sizes greater than 2.5 mm. The pressure had no definite effect on the water resistance of the briquettes. Hence, water resistance of corn cob briquettes is largely determined by the temperature, hold time and particle size of the particles. Statistical analysis using ANOVA also showed that the effects of temperature, hold time and particle size on the water resistance of corn cob briquettes were statistically significant. It was also shown that the effect of the interaction of temperature with particle size on water resistance of the briquettes was statistically significant. It can

be concluded that high quality briquettes which have good water resistance can be produced from corn cobs. This is achieved by compacting under high temperature and with small particle sizes although more energy will be required to mill the materials to finer particles.

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