

# Potential of Walnut Shell Flour as a Binder in Briquette Production from Industrial and Garden Wastes

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Briquettes as a product of biomass compaction is considered a renewable product for replacing fossil fuels. Briquettes can be produced from several sources using the development of technology. In this study, the effect of walnut shells as an organic binder on the properties of biofuel briquettes made of industrial and garden wastes was investigated. Cylindrical briquettes from walnut shell flour with weight ratios of 5%, 10%, 15%, 20%, and 25% were prepared from industrial sawdust and ground garden residue with a weight ratio of 50/50. Briquettes were compacted under a temperature of 170 °C and pressure of 150 kg/cm<sup>2</sup> for 30 s. Results indicated that the chemical analyses of compounds, compaction ratio, and density of the briquettes containing different proportions of walnut shells were not significantly different. The lowest fixed carbon was measured for briquettes containing 5% of walnut shell, and increasing the ratio of walnut shell significantly increased the compressive strength of the resulting briquette.

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## INTRODUCTION

Fossil fuels, encompassing petroleum products, coal, natural gas, and others, are widely acknowledged as primary energy sources, constituting approximately 80% of the world's primary energy supply (Sansaniwal *et al.* 2017). The combustion of fossil fuels and deforestation, which are the predominant contributors to greenhouse gas emissions primarily composed of carbon monoxide and carbon dioxide, result in the degradation of the ozone layer (Olaoye and Kudabo 2017). The escalating global demand for fossil fuels, accompanied by the release of greenhouse gases, has yielded adverse environmental repercussions, presenting a substantial threat to human health. The continued use of these finite fossil fuels across diverse industries exacerbates pollution and environmental concerns (Tian *et al.* 2018). Pursuing contemporary, sustainable, and ecologically sound energy sources will mitigate environmental pollution and augment energy production through eco-friendly products (Mitchual *et al.* 2019).

Briquettes represent a form of solid biofuel with the primary aim of generating electricity and heat (Tomen *et al.* 2023). Briquettes can be fabricated from various sources as a byproduct of technological advancement. In certain developing nations, briquettes are derived from agricultural, horticultural, and forestry residues to ameliorate coal extraction,

amplify coal yield, mitigate environmental challenges, and cultivate a sustainable coal industry. Biomass feedstock has complex physicochemical properties such as moisture content, fixed carbon, and ash content that complicate the processing and combustion of densified solid biofuel. Ash content as an undesirable component of biomass for energy production is preferred to be less than 4% (Nikkhah Shahmirzadi *et al.* 2022). The diminishing availability of wood resources and the mounting expense of wood-based briquettes, combined with nations' emphasis on curtailing harvesting and deforestation, have spurred researchers to explore alternatives for briquettes production. The compaction of biomass particles is achieved by applying mechanical force, sometimes with heating for developing sturdy bridges at connection points. Binding agents are pivotal in the briquette production process, influencing resistance, thermal stability, combustion efficiency, and the overall cost of briquettes (Soleimani *et al.* 2017). The plastic and elastic behaviors during the compression of lignocellulosic constituents lead to bonding in the end product, facilitated by natural binding agents like lignin and starch (Afra *et al.* 2021).

The role of lignin as a binding agent in compacting lignocellulosic materials to briquettes is attributed to its capacity for combustion and hydrogen bond formation at elevated temperatures (Tumuluru *et al.* 2011; Mardiyati *et al.* 2021). Van Dam *et al.* (2004) reported that lignin acts as a resin at temperatures above 140 °C, producing more durable pellets. The moisture content of biomass affects the enough temperature for lignin softening, the glass transition temperature of lignin, and the formation of solid bridges between particles (Afra *et al.* 2021), which was not a variable considered in this research.

Walnut shells containing the highest levels of lignin (49.1%) compared to hardwood and softwood residues (Pirayesh *et al.* 2012), could be an organic binding agent in producing biofuel briquettes. Walnuts are cultivated globally on a substantial scale, with production exceeding 3.7 million tons. Walnut shell waste in Iran, with an annual yield of 150,000 tons, has no economic value for industrial use and is usually thrown away or burned (Ebrahimi *et al.* 2009; Pirayesh *et al.* 2012). Accordingly, the aim of this research is to optimize the ratio of using walnut shell as an organic binder containing lignin compounds for biofuel briquettes.

## EXPERIMENTAL

### Materials

Industrial sawdust and ground garden residue were obtained from carpentry plants and grinding residues of peach tree pruning, respectively. The flour of walnut shell waste was supplied from a Mehr Sanat Company (Iran). Industrial sawdust and ground garden residue were sieved to a particle size of 40- to 60-mesh and heated in an oven at 103 °C for 24 h.

### Methods

#### *Briquetting procedure*

Industrial sawdust and ground garden residue were mixed in ratios of 50:50 by weight. Walnut shell flour (5%, 10%, 15%, 20%, and 25% w/w) was used as the binder. The cylindrical briquettes (50 mm outer diameter and 20 mm height) were pressed using a hydraulic piston at 170 °C temperature, 150 kg/cm<sup>2</sup> pressure, and a retention time of 30 s. The details of the hydraulic press are given in Fig. 1.

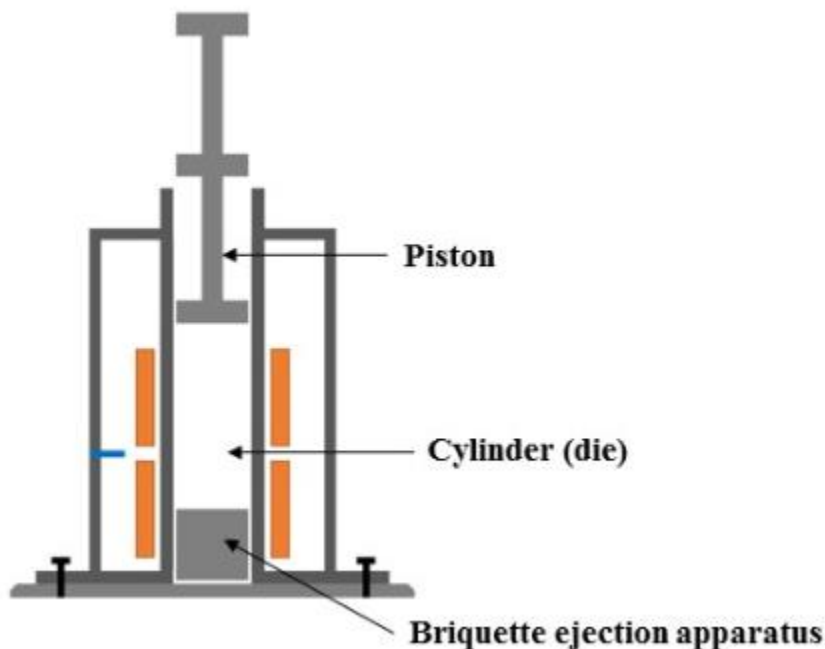


Fig. 1. Schematic plan of hydraulic press

### Chemical Analysis

Chemical analyses of the raw materials were measured following TAPPI T204 cm-97 (ethanol-benzene extractives), TAPPI T203 cm-99 (cellulose), and TAPPI T222 om-11 (acid non-soluble lignin).

### Proximate Analysis

Moisture content was determined by oven-dry methods following ASTM D2867 (2009). Each biomass was weighed and then oven-dried at 103 °C for 3 h. The loss in mass, expressed as a percentage of the final oven-dried mass, was taken as the moisture content of the biomass. The moisture content was calculated by Eq. 1,

$$\text{Moisture content (\%)} = \frac{\text{Wet weight} - \text{weight after drying}}{\text{weight after drying}} \times 100 \quad (1)$$

Volatile matter content was assessed following ASTM D5832-98. Approximately 1.0 g of biomass was placed in a dry crucible with the lid on and heated in a muffle furnace (Nabertherm, Germany) at 900 °C for 7 min. The volatile matter present in the sample on a percentage basis was calculated by Eq. 2,

$$\text{Volatile matter (\%)} = \frac{\text{Loss in weight due to volatile matter removal}}{\text{Weight of sample}} \times 100 \quad (2)$$

Ash content was determined based on ASTM D2866 (2011). About 1.0 g of biomass was heated in a crucible in a muffle furnace (Nabertherm, Germany) to 600 °C for 3 h. The crucible was removed and cooled in a desiccator before weighing. The weight of the residue was reported as the ash content of the sample on a percentage basis and was calculated by Eq. 3,

$$\text{Ash content (\%)} = \frac{\text{Weight of ash left after heating}}{\text{Weight of sample}} \times 100 \quad (3)$$

Fixed carbon content was calculated as the difference between 100 and the sum of

volatile matter, moisture, and ash percentage contents. It was also expressed on a percentage basis, as shown by Eq. 4,

$$\text{Fixed carbon (\%)} = 100 - (\text{Moisture content} + \text{Volatile matter} + \text{Ash content}) \quad (4)$$

### Fourier Transform Infrared Analysis

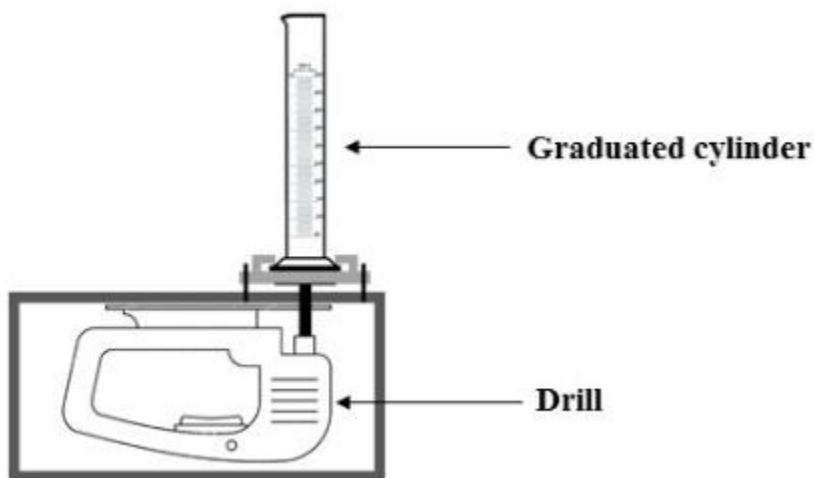
The Fourier transform infrared (FTIR) spectra of sawdust and binder were recorded utilizing the Thermo Nicolet Nexus 670 spectrometer (Waltham, MA, USA) in the attenuated total reflectance (ATR) mode. The measurement of dried samples was conducted at room temperature by co-adding 30 scans at a resolution of  $4 \text{ cm}^{-1}$  within the wavelength range of 400 to  $4000 \text{ cm}^{-1}$ .

### Tapped Density

The tapped density is an increased bulk density achieved after mechanically tapping a graduated cylinder containing the industrial sawdust and ground garden residue and walnut shells as the powder samples. The tapped density is determined by mechanically tapping a graduated measuring cylinder containing the powder sample according to the ASTM D7481 (2018) standard. The apparatus (Fig. 2) consists of a 250-mL graduated cylinder and a drilling apparatus capable of producing, in 1.0 min, nominally  $250 \pm 15$  taps. About 10 g of industrial sawdust and ground garden residue and walnut shell flour were separately introduced into graduated cylinders. After adding the powders, the measuring cylinder is mechanically tapped with a drill, and volume readings are recorded until a little further volume change is observed. The mechanical tapping was achieved by raising the cylinder and allowing it to drop under its mass. This process effectively removes the space between the powder particles. The tapped density ( $\rho$ ) was calculated using the Eq. 5,

$$\rho = \frac{M}{V} \quad (5)$$

where  $\rho$  is the tapped density of the powder ( $\text{g/cm}^3$ ),  $M$  is the mass of the powder (g), and  $V$  is the volume of the powder ( $\text{cm}^3$ ).



**Fig. 2.** The apparatus consisting of a 250-mL graduated cylinder and a drill as a tapper device

### Briquette Density

The density of briquette, as the mass ratio to the briquette volume (Rabier *et al.* 2006), was determined immediately after press using Eq. 6,

$$\rho = \frac{M}{V} \quad (6)$$

where  $\rho$  is density of the compressed briquette ( $\text{g}/\text{cm}^3$ ),  $M$  is mass of the briquette (g), and  $V$  is volume of the briquette ( $\text{cm}^3$ ).

### Compaction Ratio

The compaction ratios were also determined by Eq. 7,

$$\text{Compaction ratio} = \frac{\rho_b}{(\rho_i \times a_i) + (\rho_g \times a_g) + (\rho_w \times a_w)} \quad (7)$$

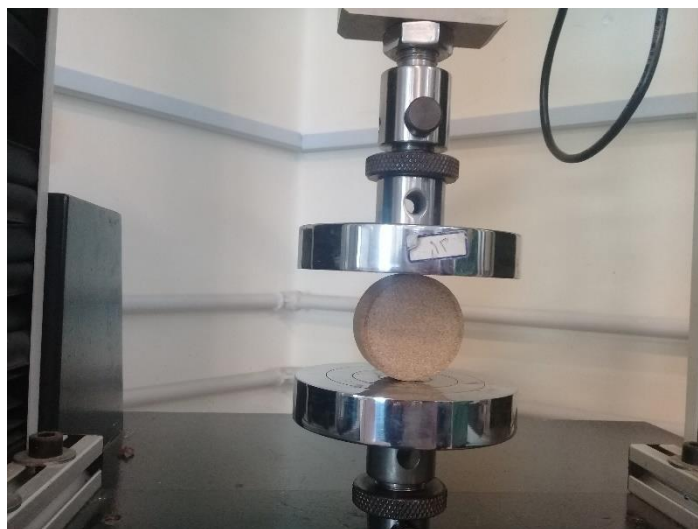
where  $\rho_b$  is the density of the compressed briquette ( $\text{g}/\text{cm}^3$ ),  $\rho_i, \rho_g, \rho_w$  are the densities of the industrial sawdust, ground garden residue, and walnut shell flour (g), and  $a_i, a_g, a_w$  are mass fractions of the industrial sawdust, ground garden residue, and walnut shell flour in the briquette ( $\text{cm}^3$ ), respectively.

### Compressive Strength

The compressive strength of the briquettes was determined following ASTM D2166-85 (2008) using an Instron Universal Strength testing machine (SANTAM-STM-20) with a load cell capacity of 50 kN. The briquette was horizontally positioned in the compression test machine and loaded at a constant rate of 0.305 mm/min until failure or cracking occurred (Fig. 3). The compressive strength was subsequently calculated according to Eq. 8,

$$\text{Compressive strength} = \frac{3 \times \text{the load fracture point}}{[l_1 (\text{mm}) + l_2 (\text{mm}) + l_3 (\text{mm})]} \quad (8)$$

where  $l_1, l_2$ , and  $l_3$  were lengths of briquettes at points one, two, and three, respectively (mm).



**Fig. 3.** Determination of compressive strength of briquette

## RESULTS AND DISCUSSION

### Characterization of Feedstock and Binder

Table 1 shows the chemical and proximate analyses of feedstock and binder. Based on the results, unlike hemicellulose and volatile matter contents, the contents of extractives, lignin, cellulose, moisture, and ash for the industrial sawdust were higher than the ground garden residues. Lignin, as a binding agent in wood structure, is known to significantly contribute to biomass densification by forming a strong bond following plasticization under heat (Lehtikangas 2000). Additionally, lignin, with more combusting energy than cellulose, can be considered as a fuel.

**Table 1.** Characterization of Feedstock and Binder

	Feedstock		Binder
	Industrial Sawdust	Garden Sawdust	Walnut Shell
<b>Component Analysis (wt%):</b>			
Extractives	5.57 (0.37)	4.25 (0.01)	6.3
Lignin	25.90 (0.53)	23.43 (0.17)	47.1
Cellulose	44.15 (0.19)	43.10 (0.17)	25.4
Hemicellulose	24.48 (1.25)	29.25 (0.10)	21.2
<b>Proximate Analysis (wt%):</b>			
Moisture	11.53 (0.12)	10 (0)	8.74 (0.20)
Volatile matter	73.09 (1.29)	75.85 (1.22)	62.55 (1.21)
Ash	3.54 (0.04)	1.78 (0.67)	5.28 (0.21)
Fixed Carbon	11.83 (1.31)	12.38 (0.68)	23.43 (1.43)
<b>Tapped Density (g/cm<sup>3</sup>)</b>	0.25 (0.01)	0.23 (0.01)	0.46 (0.02)

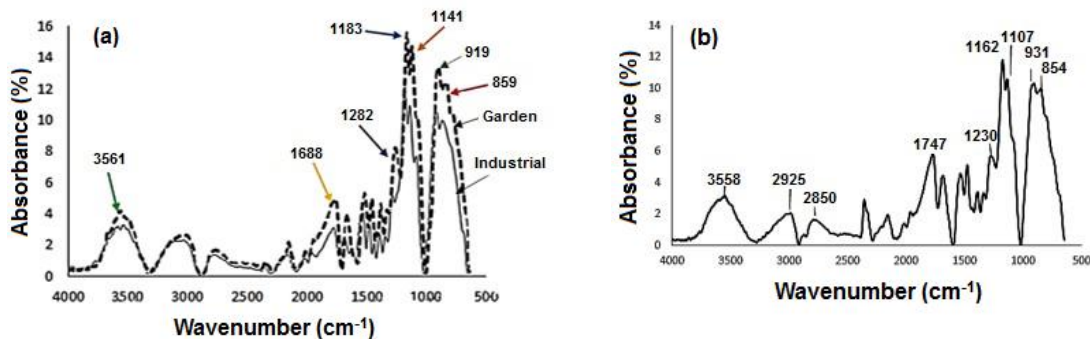
The data in parentheses are standard deviations

In addition to the type of raw materials, moisture, and ash content, the flow characteristics of biomass are also effective in energy production (Chen *et al.* 2009). The role of moisture content is critical in the briquetting process. In the briquetting process, moisture affects the bonding mechanism and enhances the hydrogen bonds in the briquette. The moisture can enhance solid fuel properties by reducing the glass transition temperature of lignin and the bridge formation between the particles. Moreover, the moisture of biomass facilitates protein gelatinization and enhances fiber interlocking during compaction (Miranda *et al.* 2015). High moisture content reduces calorific value, while low moisture content can adversely affect briquette quality, as it burns rapidly without generating sufficient energy (Song *et al.* 2010). Based on previous research findings, the ideal moisture content for briquette production is approximately 8% (Chin and Siddiqui 2004). Mani *et al.* (2006) reported that the moisture of biomass increases the particle contact surface, subsequently inter-particle bonds created by van der Waals forces during compaction. Furthermore, utilizing biomass with a higher volatile matter content provides advantages for combustion (Akowuah *et al.* 2012). However, as an undesirable factor in energy production, ash reduces the calorific value (Nasser and Aref 2014). Biomass with low ash (less than 4%) and high fixed carbon content is preferred as a feedstock for briquette (Chen *et al.* 2009).

### FTIR Analysis

Figure 4 shows the FTIR spectra of feedstock sawdust and walnut shell powder. A vibration peak at 3561 cm<sup>-1</sup> (OH stretching) has appeared in industrial and garden sawdust (Ali *et al.* 2022). The band at 2900 cm<sup>-1</sup> is attributed to aliphatic C-H bond stretching

vibrations (Nikkhah Shahmirzadi *et al.* 2022) (Fig. 4a). The  $1688\text{ cm}^{-1}$  peak corresponds to carbonyl ester groups, which are more numerous in ground garden residue. These carbonyl groups are mainly found in the side chains of hemicelluloses (Sammons *et al.* 2013). The  $1282\text{ cm}^{-1}$  (C-O in guaiacyl and syringyl rings) and  $859\text{ cm}^{-1}$  (out-of-plane C-H) peaks have been observed in both industrial and garden residues (Sammons *et al.* 2013). The peaks at  $1141\text{ cm}^{-1}$  and  $1183\text{ cm}^{-1}$  resulted from the stretching of C-H and C-O bonds, respectively (Singh *et al.* 2011). The peak at  $919\text{ cm}^{-1}$  related to the asymmetric out-of-plane stretching in the *p*-hydroxyphenyl ring (Michell and Higgins 2002) indicates the higher proportion of hemicellulose in ground garden residue.



**Fig. 4.** FTIR spectra of feedstock (a) and walnut shell powder (b)

Figure 4(b) illustrates the results of FTIR spectroscopy of walnut shell powder. The peak at  $3558\text{ cm}^{-1}$  indicates the presence of hydroxyl (OH) group stretching (Tang *et al.* 2017). Peaks at  $2850\text{ cm}^{-1}$  and  $2925\text{ cm}^{-1}$  are ascribed to aliphatic C-H deforming vibration in lignin methyl and methylene structures (Lin *et al.* 2021). The  $1747\text{ cm}^{-1}$  peak is assigned to the vibration of carbonyl (C=O) groups or the presence of carboxylic bonds (Lin *et al.* 2021). The band at  $1230\text{ cm}^{-1}$  is attributed to the stretching vibrations of ester groups (Esteves *et al.* 2018). The peak at  $1162\text{ cm}^{-1}$  could be associated with the deformation of  $\text{CH}_2$  groups (Lin *et al.* 2021). The peak at  $1107\text{ cm}^{-1}$  resulted from the C-O-C groups of polysaccharides (Michell and Higgins 2002). The peak  $931\text{ cm}^{-1}$  was assigned to the stretching of pyranose ring bonds (Lin *et al.* 2021).

### Characterization of Biofuel Briquette

Table 2 shows the proximate analysis of briquettes containing different proportions of walnut shell powder. Based on the results, no statistically significant differences were observed in moisture content, volatile matter, and ash content between briquettes of different proportions of walnut shells. The lowest fixed carbon was measured in briquettes containing 5% walnut shell, which did not show a statistically significant difference with other ratios.

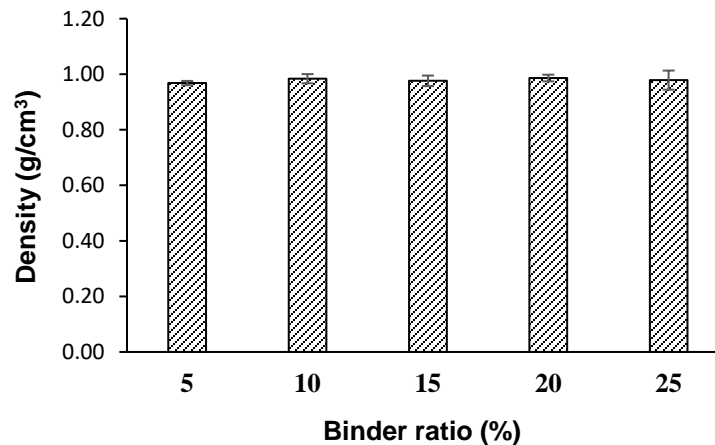
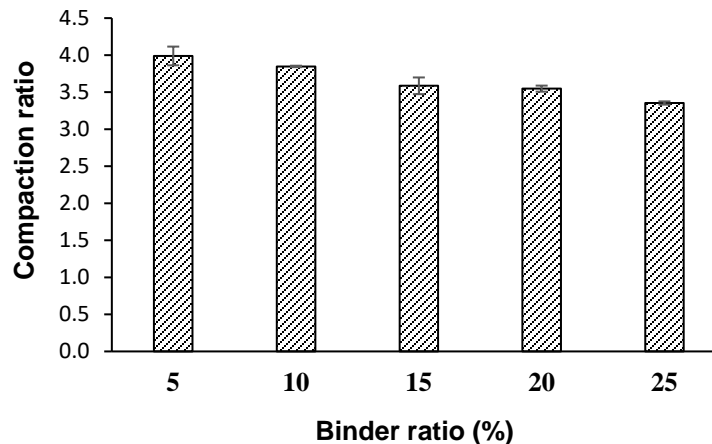
All of the briquettes met the minimum values of ash contents according to ISO 17225-7 (2014) standard (a minimum value of  $\leq 6\%$ ), which indicated good quality briquettes (Sawadogo *et al.* 2018).

**Table 2.** Proximate Analysis of Briquette

Proximate Analysis (wt%):	Walnut Shell Binder (%)				
	5	10	15	20	25
Moisture	1.56 (0.43)	1.34 (0.32)	1.05 (0.21)	1.13 (0.20)	1.29 (0.24)
Volatile matter	77.53 (0.55)	76.34 (0.21)	75.87 (0.79)	75.34 (0.24)	75.04 (0.87)
Ash	1.57 (0.18)	1.92 (0.47)	1.72 (0.10)	1.87 (0.08)	2.01 (0.07)
Fixed Carbon	19.34 (0.98)	20.40 (0.59)	21.36 (0.93)	21.66 (0.05)	21.66 (0.76)

The data in parentheses are standard deviations

The effect of the walnut shell binder ratio on the density of the resultant briquettes is shown in Fig. 5. Based on the results, the density of the briquettes containing different proportions of the walnut shell did not show a significant difference. The results were also confirmed by Font *et al.* (2023), who proposed a density of 951 to 1056 kg/m<sup>3</sup> for good quality in aspect of efficient transportation and safe storage briquettes in their work.

**Fig. 5.** The effect of walnut shell powder ratio on the density**Fig. 6.** The effect of walnut shell powder ratio on the compaction



The compaction ratios of briquettes using various proportions of the walnut shell powder are illustrated in Fig. 6. The apparent differences were not statistically significant.

The effect of walnut shell ratio as a binder on the compressive strength is shown in Fig. 7. Increasing the ratio of walnut shells increased the compressive strength of the resulting briquette. The briquettes containing 20% of the walnut shell binder exhibited the highest compressive strength. No statistically significant differences were observed in the compressive strengths between briquettes containing 15% and 20%, nor between those containing 15% and 10% walnut shells. The results are also consistent with the observation by Sen *et al.* (2016) that the compressive strength increased with increasing binder ratio. Conversely, increasing the walnut shells ratio to 25% reduced the compressive strength.

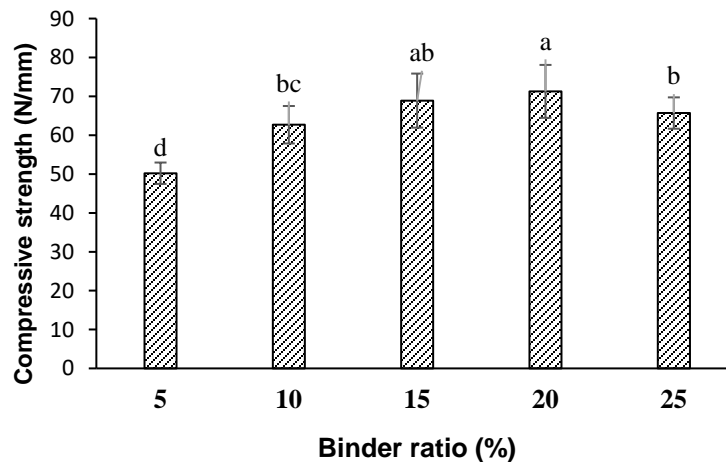


Fig. 7. The effect of walnut shell powder ratio on the compressive strength

It is worth highlighting that walnut shells contain approximately 49.1% lignin (Pirayesh *et al.* 2012), nearly double that of industrial and garden sawdust. As a result, increasing the walnut shell ratio to 20% significantly enhanced the compressive strength due to increased lignin proportion. However, a 25% ratio of walnut shell powder to feedstock sawdust led to a significant reduction in compressive strength, which can be attributed to the higher bulk density of walnut shells compared to the industrial sawdust and ground garden residue, consequently reducing the compaction ratio of resulting briquettes.

## CONCLUSIONS

In this study, the potential of using walnut shells as a binder for manufacturing solid biofuels *via* biomass compaction was investigated.

1. Despite the higher content of lignin and fixed carbon in the powder compared to sawdust, the results of the proximate analysis of briquettes showed no significant difference between various ratios of walnut shell powder as an organic binder.
2. Upon assessing the physical and mechanical properties of the biofuel briquettes containing the industrial and garden wastes (50/50% W/W), the compaction ratio of resulting products declined proportional to the increase of the binder ratio from 5% to 25 wt%.

3. Unlike the compaction ratio, the compressive strength was enhanced by increasing the binder ratio from 5% to 20 wt%. It can be concluded that for producing biofuel briquettes, the properties of briquettes were improved by increasing the ratio of the walnut shell as a binder up to 20%. Organic binder content of 25% resulted in a significant decline of compressive strength in the product.

In general, based on the present study, it can be suggested that walnut shell waste as an organic binder can be substituted for a part of sawdust.

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