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Eggshell and Walnut Shell in Unburnt Clay Blocks

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Abstract: Agricultural residues/by-products have become a popular choice for the manufacturing of building materials due to their cost-effectiveness and environmental friendliness, making them a viable option for achieving sustainability in the construction sector. This study addresses the utilisation of two agro-wastes, i.e., eggshell and walnut shell, in the manufacture of unburnt clay blocks. The experiments were carried out on three series of samples in which first eggshell (10–50%) and walnut shell (5–20%) were incorporated individually and then combined (5% walnut, 10–30% eggshell) in the mixture to assess their influences on the physical and mechanical properties of the unburnt clay blocks. This study performed the following tests: Density, capillary water absorption, linear shrinkage, flexural and compressive strength. The results indicated that eggshell enhanced the strength relative to the control sample when the materials were employed individually, but walnut shell lowered it. Moreover, combining the two materials in the mixer reduced the strength of the samples even further. Nevertheless, the inclusion of the waste materials decreased the density, capillary water absorption coefficient and linear shrinkage of the samples. The findings indicate that eggshell has great potential for unburnt clay block manufacture. However, walnut shell integration needs further research.

Keywords: clay blocks; eggshell; mechanical properties; physical properties; unburnt; walnut shell

Citation: Jannat, N.; Latif Al-Mufti, R.; Hussien, A. Eggshell and Walnut Shell in Unburnt Clay Blocks. *CivilEng* **2022**, *3*, 263–276. https://doi.org/10.3390/civileng3020016

Academic Editor: Aires Camões

Received: 24 February 2022
Accepted: 25 March 2022
Published: 1 April 2022

1. Introduction

It is well acknowledged that the application of building materials has a substantial influence on the environment because of embodied energy and the challenges of disposal of particular materials. Hence, new approaches for the manufacture of building materials are essential. One approach for lowering the negative environmental effect of building materials and reducing the consumption of material resources is to utilise the waste materials to manufacture building materials. Agricultural industries produce a large amount of wastes and by-products that can be an important source of raw materials for different industrial sectors, including building material construction. Walnut shell is an agricultural residue/by-product that is either burnt or disposed of in landfills. According to the report, worldwide walnut production reached around 965,400 tonnes in 2019 [1] and given that the shell accounts for almost 67% of the total fruit weight, this equates to 646,818 tonnes of walnut shells per year [2,3]. Figure 1 shows the leading walnut-producing countries [4]. The hydrophobic elements (lignin, 50.3%) in walnut shells are more abundant than hygroscopic materials (cellulose (23.9%) and hemicellulose (22.4%)) [5,6]. The lower water absorption, higher strength and bio-resistance characteristics of walnut shell have made it popular in the building construction industry. Studies have been conducted using walnut shell in the production of bio-composite [7–13], particleboard [14,15], MDF panels [16,17] and as an aggregate substitute in concrete [18–22]. However, the use of walnut shells in the production of unburnt clay brick is rarely mentioned in the literature. Mirón et al. [23] used different percentages of walnut shell (5–20%) to develop compressed earth blocks in two series of experiments. One series

had 10% cement while the other had 7% lime and 3% gypsum. The results revealed that only the control sample satisfied the Mexican Standard [24] requirement of 6 MPa and both series of samples showed a decreasing trend in strength when walnut shell was added. The reduction was up to 94% for cement-stabilised samples and up to 65% for lime and gypsum samples. Moreover, the control sample had a water absorption value of 30%, which was 9% higher than the standard requirement (21%). In cement-stabilised samples, 5% walnut shell samples exhibited a water absorption of 23.8%, which was only 4% above the standard. However, other percentages (10%, 15% and 20%) of samples deformed in this series after removing from the water. On the other hand, lime and gypsum stabilised samples containing 20% and 10% walnut shell showed comparatively lower water absorption values of 18.55% and 19.39%, respectively. Marte [25] utilised two different granulometric sizes (2 and 4 mm) of walnut shell to substitute gypsum in plaster by 5%, 10%, 15%, and 20%. The findings showed that all walnut shell blended samples had flexural and compressive strength values that were within specifications but lower than the control sample. In both granulometries, the best strength performance was achieved at 5% and 10% of replacement. Furthermore, compressive strengths dropped less in 4 mm than in 2 mm samples. Besides, the weight of the samples decreased in higher percentages of replacement and higher levels of granulometries. In terms of water absorption, the samples with the 4 mm granulometry showed a 29% drop in absorption compared to the control sample.

Figure 1. Global walnut-producing countries [4].

Another potential waste material that is available in abundance is poultry eggshell from sources such as hatcheries, food industries and domestic homes. Around 110 billion eggshells are produced globally [4] (Figure 2), with most of them ending up in landfills [26]. Such waste has a negative impact on the environment because it raises waste management costs and produces a bad smell on the site. The Environmental Protection Agency has listed eggshell waste as the 15th top food sector pollution concern [27]. The poultry eggshells are grainy in texture and constitute about 9–12% of the overall egg weight [26,28]. Eggshells are generally thought to have no economic worth, despite being high in amino acids and minerals. Calcium oxide is reported as the main component of the eggshell (94% to 98%) [29–31]. Eggshell powder can be used as a partial replacement for industrial lime due to its chemical composition, which is similar to lime [32,33]. Furthermore, the calcium content of eggshells is more absorbable than calcium from coral or limestone [34]. As a result, poultry eggshell has become a popular natural reinforcing material in the

construction industry because of these characteristics. Researchers have studied applying eggshell powder in sustainable building material production, such as masonry blocks, clay bricks, soil stabiliser, cement replacement in concrete and mortar [35]. Adogla et al. [36] used eggshell powder (0–40 wt%) in compressed earth brick production and observed that samples containing 30% eggshell powder showed better properties. In another study, Ayodele et al. [37] incorporated eggshell ash and sawdust ash (0–16 wt%) in the lateralised unburnt bricks where samples with 2–4% ash had the maximum compressive strength.

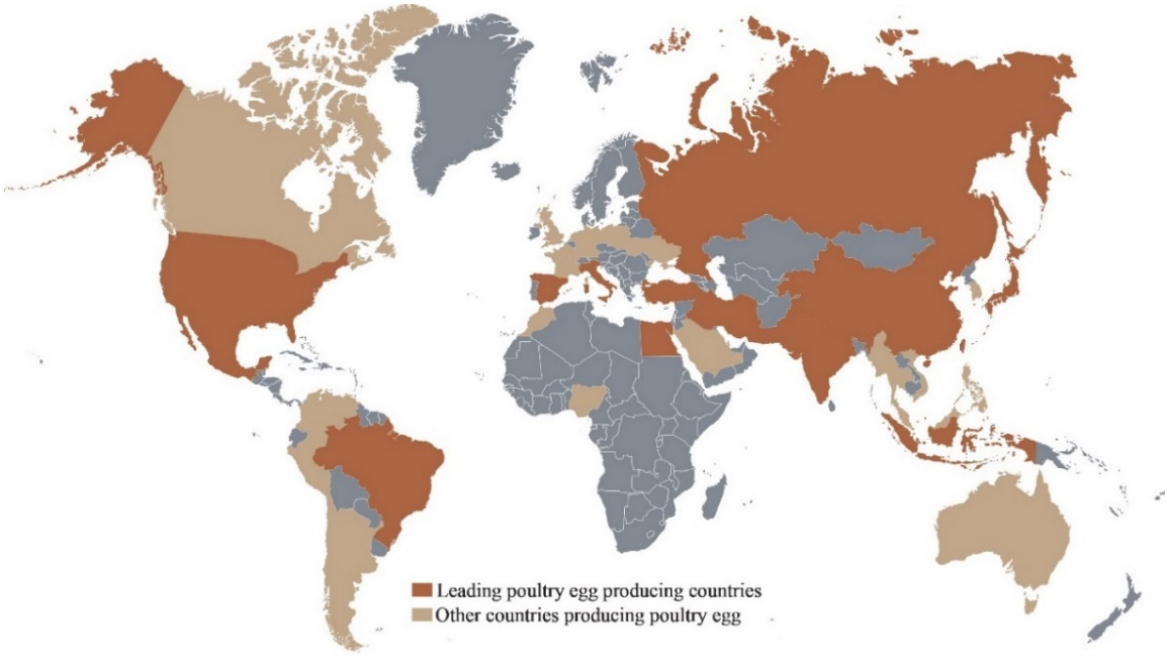


Figure 2. Global poultry egg-producing countries [4].

The above studies show the potentiality of employing walnut shell and eggshell in the manufacture of building materials. However, utilisation of these materials in unburnt clay brick is rarely investigated. Furthermore, no research is available on the combined effect of including walnut shell and eggshell in unburnt clay brick production. Hence, this study investigates the physical and mechanical characteristics of unburnt clay blocks using these two agro-wastes in different combinations. The effect of both individual and combined addition of walnut shell and eggshell are examined in this study.

2. Materials and Experimental Programme

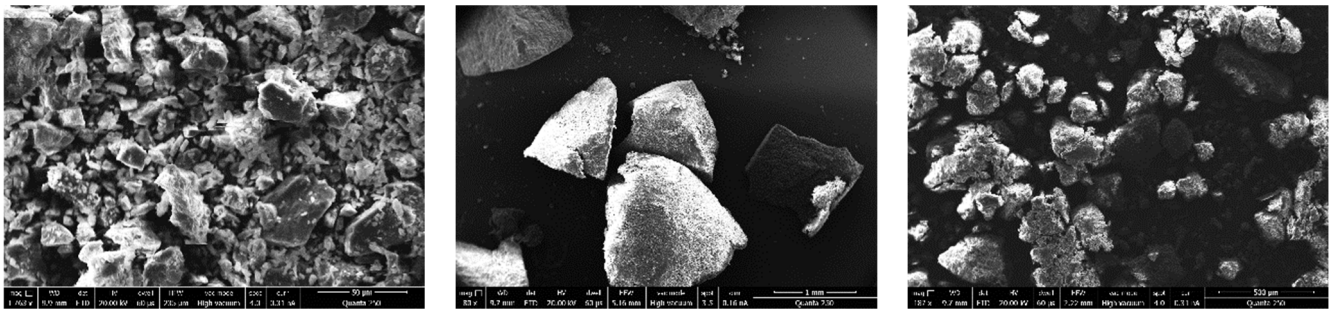
2.1. Raw Materials

This investigation used Red Clay Powder (RCP) which was supplied by the Bath Potters' Supplier, UK. Besides, two agro-wastes were considered as stabilisers: (i) Eggshell Powder (EP); particle size: 150–212 µm and (ii) Walnut Shell Grit (WSG), particle size: 1.18–2.00 mm (Figure 3). The local retailers in the UK provided the EP and WSG utilised in this investigation. All the raw materials used to make the samples were untreated. The surface morphology of the raw materials was performed using Scanning Electron Microscopy (SEM) (Figure 4). Besides, mineralogy composition was estimated by X-ray diffraction (XRD) analysis (Figure 5). Furthermore, X-ray fluorescence (XRF) spectrometry was done in order to determine the most predominant or influential oxides in the raw materials (Table 1). Next, some physical characterisations of the raw materials were conducted, which are presented in Table 2. The SEM image shows that EP has angular and irregularly shaped stone-like particles forming agglomeration (Figure 4a). On the other hand, due to the high lignin concentration, WSG has a very rigid and thick structure in the cell wall (Figure 4b) [38]. The XRD pattern reveals quartz as the primary mineralogical component

in RCP (Figure 5c). Furthermore, haematite (Fe_2O_3) and kaolinite ($Al_2Si_2O_5(OH)_4$) were found in the RCP. All diffraction peaks of EP indicate the existence of calcite ($CaCO_3$) as the major component (Figure 5b), whereas WSG exhibits a considerable degree of amorphosity in the XRD pattern (Figure 5a).

(**a**) (**b**) (**c**)

Figure 3. Photographs of raw materials: (**a**) EP; (**b**) WSG; (**c**) RCP.



(**a**) (**b**) (**c**)

Figure 4. SEM images of raw materials: (**a**) EP; (**b**) WSG; (**c**) RCP.

Table 1. Raw materials' physical properties.

Materials	Properties								
	Plasticity Index (%)	Maximum Dry Density (kg/m^3)	Optimum Moisture Content (%)	Density (kg/m^3)	Specific Gravity	Porosity	Natural Moisture Content (%)	Water Absorption after 24 h under Water (%)	Colour
RCP	12.36 (Medium plastic)	2320	15.50	1430	2.32	0.38	6.47	27.57	Red
EP	-	-	-	1170	1.74	0.56	0.31	39.42	White
WSG	-	-	-	630	1.28	0.92	6.75	29.90	Sandy brown

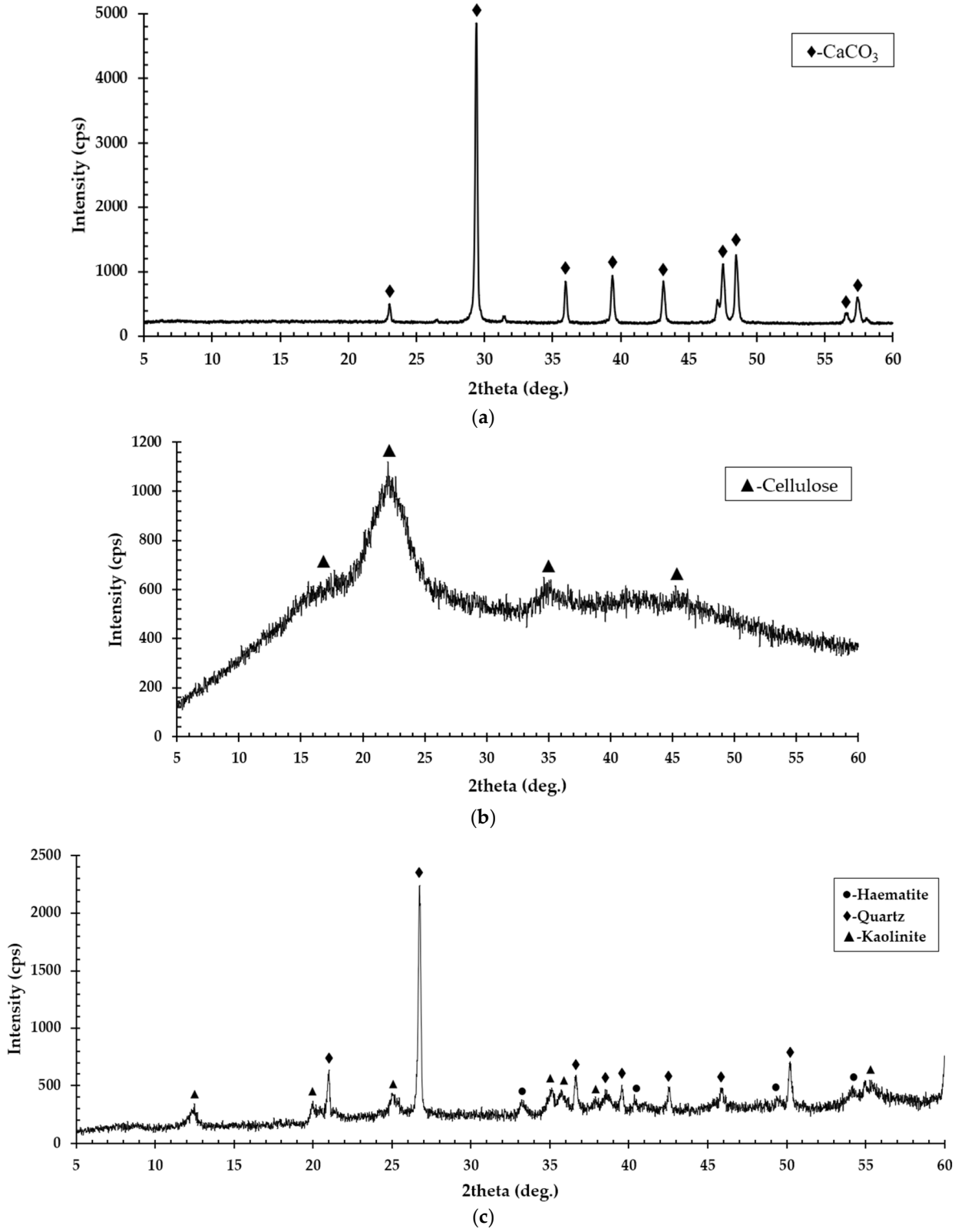


Figure 5. XRD spectrograms of raw materials: (**a**) EP; (**b**) WSG; (**c**) RCP.

Table 2. Raw materials' chemical components.

Chemical Compounds (%)	RCP	EP	WSG
SiO_2	41.454	0.097	1.103
Al_2O_3	15.214	-	0.536
K_2O	1.636	0.155	1.871
MgO	5.114	0.522	0.512
Fe_2O_3	8.104	-	0.062
CaO	0.633	78.111	1.722
Na_2O	1.027	1.423	0.930
TiO_2	1.411	0.096	0.098
SO_3	0.047	0.345	0.057
BaO	0.216	0.189	0.075
ZrO_2	0.035	0.008	0.002
MnO	0.040	-	0.002
SrO	0.011	0.042	0.001
P_2O_5	-	-	0.073

2.2. Sample Preparation

The mix proportions of the samples are given in Table 3. Three series of samples were prepared for the test. The first series of samples (E-10 to E-50) consisted of 10–50% EP, whereas the second series (W-5 to W-20) had 5–20% WSG. Besides, to assess the combined effect of eggshell and walnut shell the third series of samples (WE-5/10 to WE-5/30) were made using 10–30% EP with 5% WSG. Clay blocks with no additives were made for the control samples (C) in this study. The wastes materials were mixed in relation to the dry weight of the clay. The dry raw materials in the specified proportions were first poured into a mechanical mixer and thoroughly combined. After that, water was gradually added to the blend and mixed until it became uniform. Finally, the mixture was put into the prismatic mould of 16 × 4 × 4 cm and hand compacted. The samples were then cured at a laboratory room condition (23–26 °C temperature and 30–34% relative humidity) for 28 days. Three samples were examined to determine the impact of waste content on physical and mechanical characteristics.

Table 3. Mix proportions.

Sample ID	RCP (g)	Waste (%)		Waste (g)	
		EP	WSG	EP	WSG
C	550	0	0	0	0
E-10	550	10	0	55	0
E-20	550	20	0	110	0
E-30	550	30	0	165	0
E-40	550	40	0	220	0
E-50	550	50	0	275	0
W-5	550	0	5	0	27.50
W-10	550	0	10	0	55
W-15	550	0	15	0	82.50
W-20	550	0	20	0	110
WE-5/10	550	10	5	55	27.50
WE-5/20	550	20	5	110	27.50
WE-5/30	550	30	5	165	27.50

2.3. Sample Testing

The samples were examined for density, capillary water absorption, linear shrinkage, flexural strength and compressive strength at the end of the 28-day curing period. Additionally, the finely crushed powder of the samples was subjected to XRD tests to determine their crystal structure. The density of the samples was calculated according to the standard BS EN 771-1 [39]. The sample blocks were weighed, and their dimensions were measured using digital callipers in all three directions. The density was then determined by dividing the weight (kg) by the volume (m^3).

The test for determining the capillarity water absorption coefficient (C_w ($kg/(m^2 \times min^{0.5})$) was carried out on half prismatic samples from the flexural strength test following BS EN 1015-18 (2002) [40]. According to the standard, the half prism samples were oven-dried (60 ± 5 °C, 24 h), and their weights (M_i (kg)) were recorded. They were then immersed in water to a depth of 5 mm in a container. The samples were removed from the water after 10 min and weighed again (M_t (kg)) (Figure 6a). Equation (1) from the standard was used to get the capillary water absorption coefficient,

$$C_w = 0.1 \times (M_t - M_i) \quad (1)$$

(**a**) (**b**) (**c**)

Figure 6. Test photographs: (**a**) Capillary water absorption; (**b**) flexural strength; (**c**) compressive strength.

In order to assess the linear shrinkage of the samples, the lengths of the samples were measured using a digital calliper before and after drying. Linear shrinkage is expressed as a percentage of the change in length compared to the original length.

The BS EN 1015-11 standard [41] specifies the flexural and compression strength test procedures. After 28 days of curing period, full prism samples ($16 \times 4 \times 4$ cm) were used for the flexural strength test. The test equipment was a Tinius Olsen H25KS, which consisted of two rollers separated by 10 cm on which the sample rested. A load was then progressively employed at a rate of 10 N/s to the top roller that was placed on top of the sample at the middle point (Figure 6b). The maximum load was recorded to calculate the flexural strength using Equation (2),

$$f = \frac{1.5FL}{bd^2} \quad (2)$$

where f (MPa) is the flexural strength, F (N) is the maximum load, L (mm) is the space between the rollers, b (mm) is the sample's height and d (mm) sample's width.

The compressive strength test was performed on the half prism samples obtained from the flexural strength test. According to the standard, the samples were positioned centrally between two steel bearing plates of 4×4 cm and 0.40 MPa/s charge velocity was applied until the block fractured at which point the maximum load was recorded (Figure 6c). The compressive strength (C, MPa) is defined by the ratio of the axial force F (N) and the cross-section area A (mm^2).

$$C = \frac{F}{A} \quad (3)$$

3. Results and Discussions

Figure 7 presents the XRD analysis of the composites. The addition of WSG did not result in the creation of new mineral phases, as shown in Figure 7a. However, there was a pozzolanic reaction generated by calcium ions of EP with clay minerals (Figure 7b), which strengthened bonding in the clay matrix but decreased open porosity. It can be noticed that the density of samples lowered with the addition of EP and WSG (Figure 8). However, the density values of all EP incorporated samples and 5–10% WSG samples were higher than 1750 kg/m^3 satisfying the standard requirement [42,43] for load-bearing material. Moreover, the WE-5/10 and WE-5/20 samples met the standard with densities of 1811 and 1755 kg/m^3, respectively. Density decreased around 21% for 20% WSG content and 14% for 50% EP content compared to the control sample. When WSG and EP were combined in the mixture, the decrease was about 20%. The lower specific gravity of EP and WSG than the clay particle (see Table 1) resulted in a reduction in sample density. Figure 9 shows that capillary water absorption in EP samples decreased until it reached 40% content, after which it marginally increased. This can be explained by the reduction in open porosity in the samples due to the pozzolanic reaction in the mixture. On the other hand, the non-absorbent nature of WSG particles caused a reduction in capillary water absorption of the samples. Moreover, capillary water absorption further decreased in combined mix samples. Concerning the linear shrinkage, a decreasing trend was observed with the addition of both types of wastes separately and together (Figure 10). The decreases were up to 44% for 20% WSG, 30% for 50% EP and 37% for the combined mixture relative to the control sample.

Figures 11 and 12 present the mechanical strength test results. Both oven-dried and air-dried samples were tested for compressive strength. The results reveal that all the samples satisfied the standard requirements of compressive strength (1–2.80 MPa) [43–46] and flexural strength (0.25–0.50 MPa) [42,44,45]. Figure 11 shows that the mechanical strength of the EP samples improved progressively by increasing EP content from 10 to 40%, then decreased for 50% EP. The improvement in strength is attributed to the pozzolanic reaction of the minerals in RCP with calcium in EP in the mixture. When calcium oxide (CaO) in EP came into contact with water, it produced portlandite ($Ca(OH)_2$). Then, portlandite reacted with silica (SiO_2) in the RCP to form the cementitious compound calcium silicate hydrate ($CaSiO_3 \cdot 2H_2O$), which enhanced the strength. On the other hand, the mechanical strength of the WSG samples declined by increasing WSG from 5 to 20%, which can be explained by the poor bond between the clay particles and WSG. Moreover, strength showed a decreasing trend when WSG was combined with EP. In this case, when clay was replaced by WSG, available silica content for reaction with portlandite reduced, and unreacted portlandite caused a negative impact on strength [47]. Oven-dried samples exhibited better strength than air-dried samples for all the mixtures. The percentages of increase/decrease in strength of different samples relative to the control sample are presented in Figure 12.

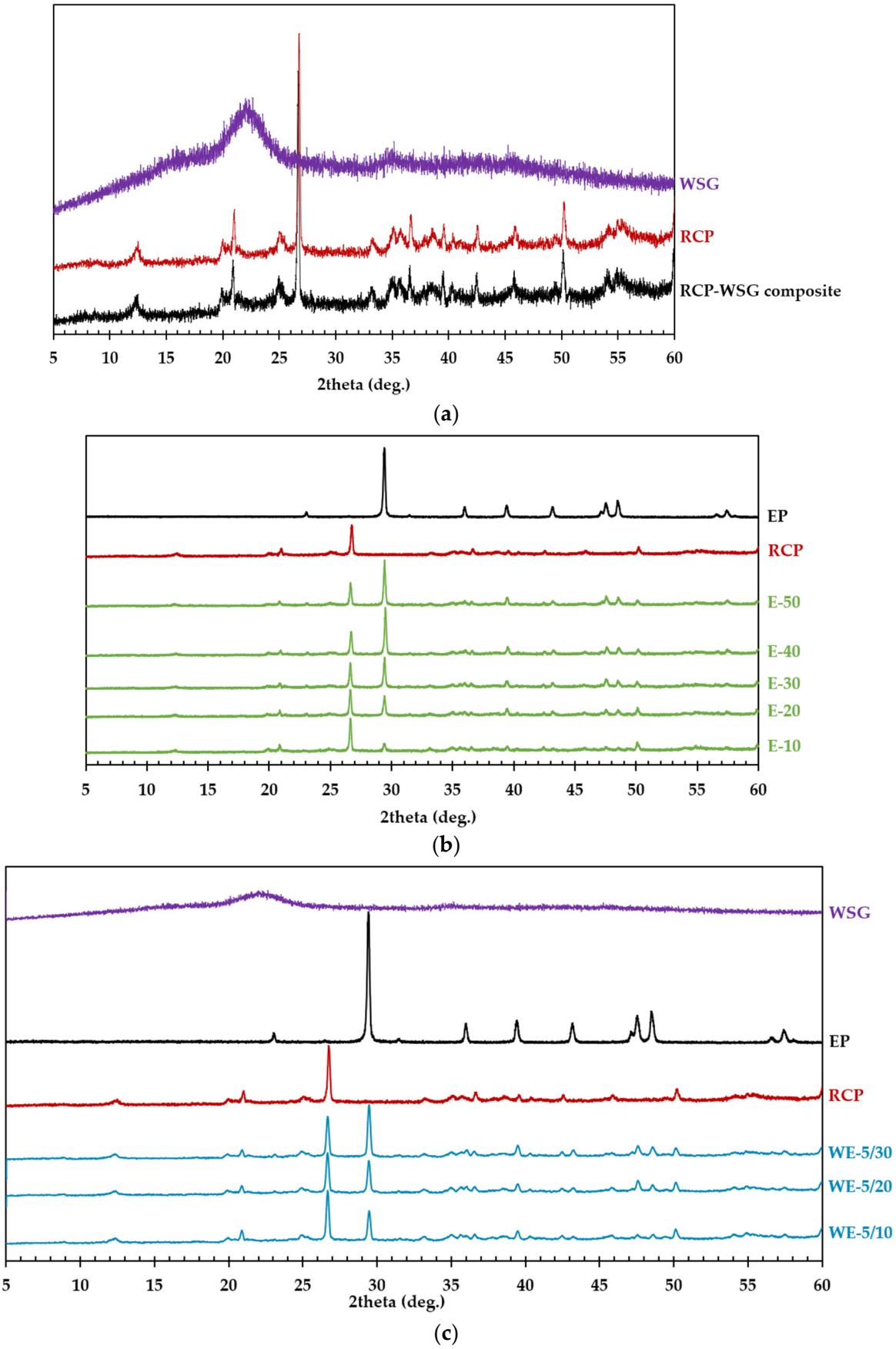


Figure 7. XRD spectrograms of composites: (**a**) WSG samples; (**b**) EP samples; (**c**) WE samples.

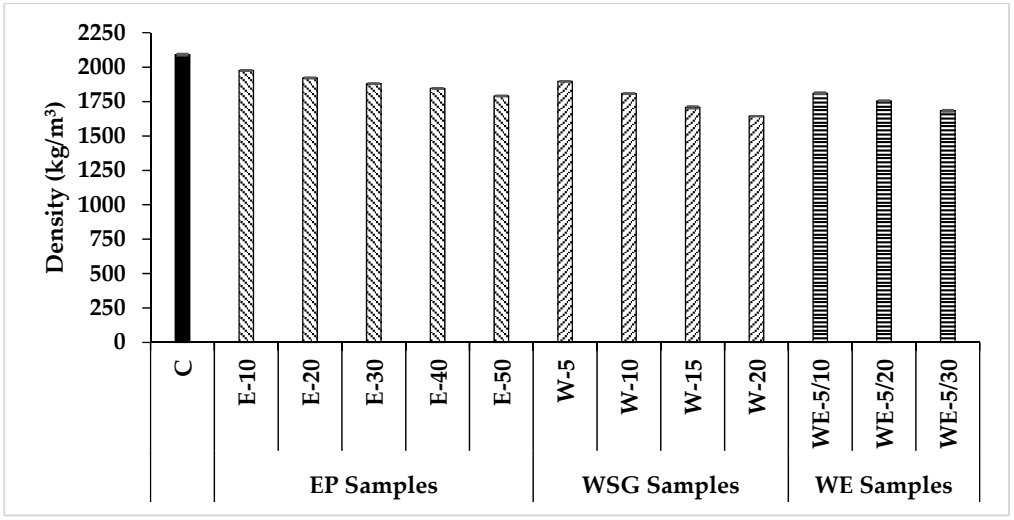


Figure 8. Density results.

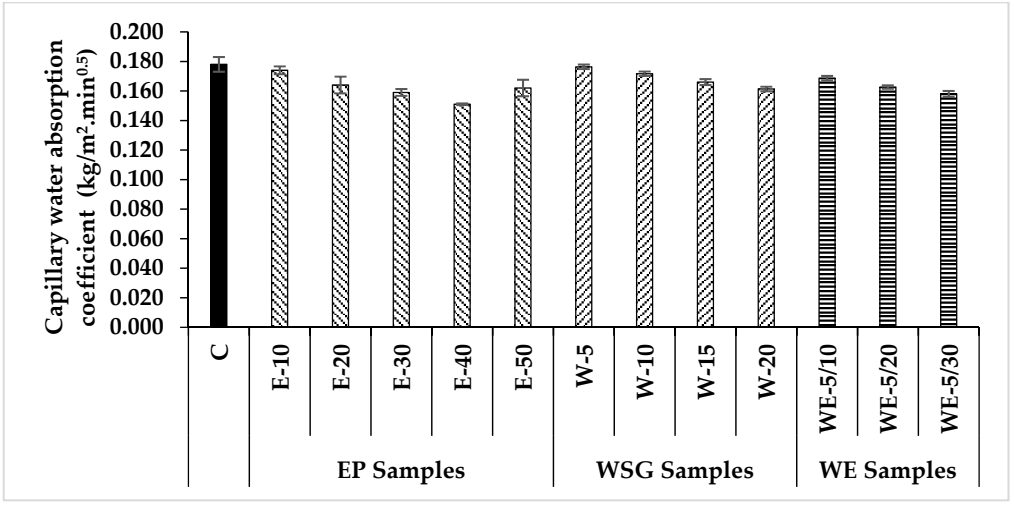


Figure 9. Capillary water absorption results.

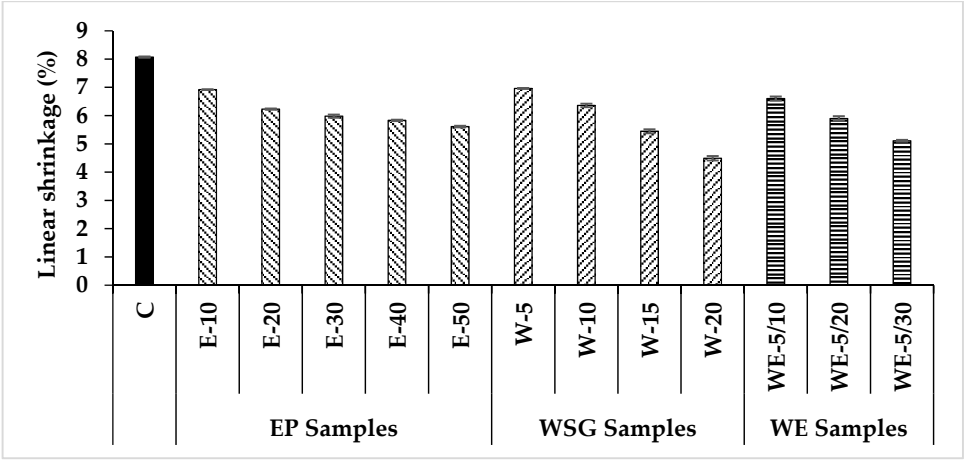


Figure 10. Linear shrinkage results.

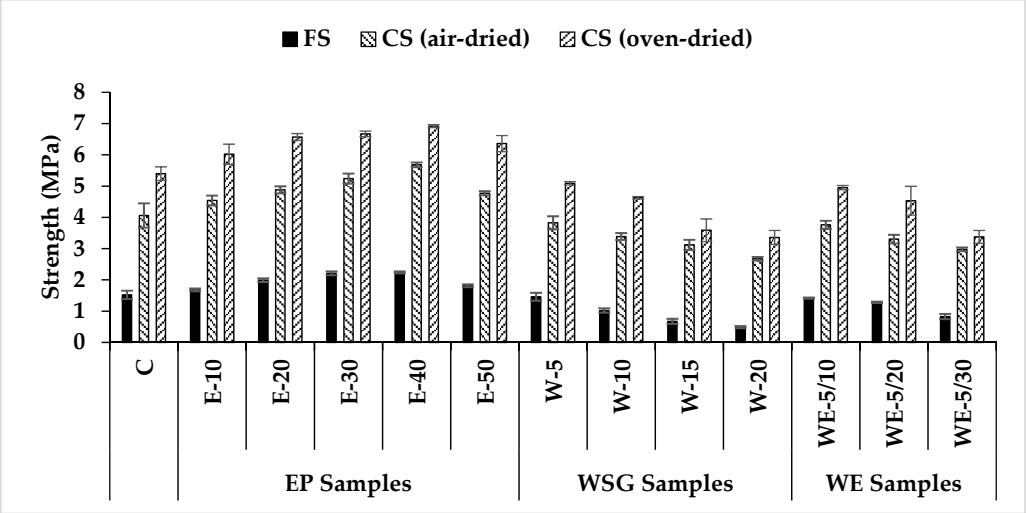


Figure 11. Test results of flexural strength (FS) and compressive strength (CS).

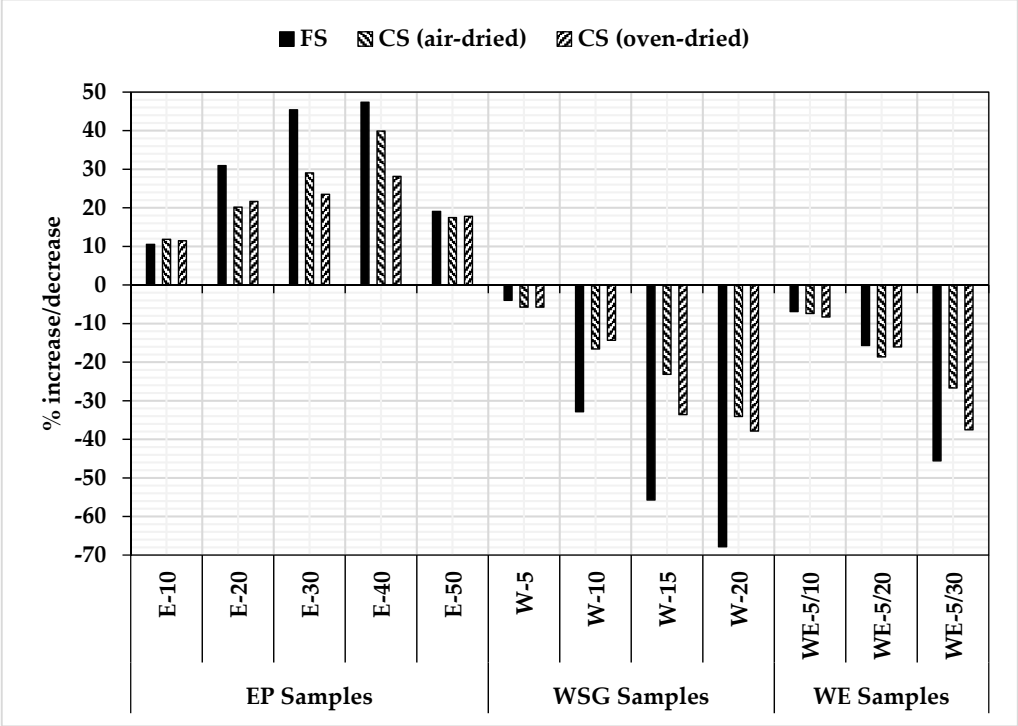


Figure 12. Comparison of flexural strength (FS) and compressive strength (CS) between the control and waste-incorporated samples.

4. Conclusions

The efficacy of incorporating eggshell and walnut shell as natural stabilisers in unburnt clay mixtures was investigated in this study. The test findings lead to the following conclusions:

- The addition of both types of agro-wastes reduced the density of the samples because of their lower specific gravity compared to the clay particles used.
- The linear shrinkage values decreased for increasing both types of wastes separately and combinedly.
- In terms of capillary water absorption, the EP sample showed a decreasing trend for increasing content from 10 to 40%. Furthermore, for WSG samples, capillary water

absorption value slightly decreased as the percentage increased. Moreover, for the combination of EP and WSG, capillary water absorption decreased gradually.

- The mechanical strength of the EP samples increased with incorporating EP from 10 to 40%, and then the values decreased for 50% EP content. The samples with 40% EP showed peak compressive (5.68 MPa) and flexural strength (2.30 MPa). On the other hand, the addition of WSG has an adverse effect on the strength of the samples. The optimal compressive (3.83 MPa) and flexural (1.45 MPa) strength values for WSG samples were found at 5% content, which was lower than the control sample's strength values. Besides, the strength decreased further when WSG was combined with EP. For all the samples, oven-cured samples had higher compressive strength than the laboratory environment cured samples.

It is noticed that eggshell has great potential to be used for manufacturing unburnt clay blocks. The test findings of walnut shell samples, on the other hand, revealed that while it enhanced several physical properties of the samples, it had a detrimental impact on their strength. Hence, the utilisation of walnut shell in unburnt clay materials production needs further investigation with other clay types and additives. Moreover, future research in this area can investigate the effects of different particle sizes of agro-wastes materials on the properties of unburnt clay blocks.

Author Contributions: Methodology, N.J. and R.L.A.-M.; Investigation, N.J.; Supervision, R.L.A.-M.; Writing—original draft, N.J.; Writing—review & editing, R.L.A.-M. and A.H. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: Not applicable.

Acknowledgments: The authors acknowledge the financial and laboratory support provided by the School of Civil Engineering and Built Environment at Liverpool John Moores University, United Kingdom.

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

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