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Utilization of Eggshells Waste as an Additive to Improve the Mechanical Properties of Clays

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Abstract

In this study, we investigated the reuse of eggshell powder waste to improve the mechanical properties of clay bricks as a cement replacement material. The eggshell powder was used as a potential and environmentally friendly and economical solution for strengthening clay structures. Specimens were prepared with eggshell powder content varying from 0%, 5%, 10%, 15% and 20% by dry weight in order to determine the optimal percentage and observe the strength of the bricks to produce a good cementing material using eggshell powder. Chemical and mineralogical characterization by XRF, FTIR, SEM/EDS and Atterberg limits, respectively, was carried out to assess the physicochemical properties of the raw materials and the final product, while compression and flexural tests were performed to determine the mechanical properties. The results showed that adding 10% by weight of ESP resulted in a flexural strength of 1.062 MPa for Tchicky clay. Adding 15% by weight of ESP resulted in a compressive strength of 4.212 MPa for clay 1 (Tchicky).

Keywords

Clays, Eggshells, Physicochemical Properties

1. Introduction

Cement is one of the main materials used in construction in Senegal. However, as the cost of producing cement remains high, its selling price can be a barrier to its use for some low- and middle-income households. The search for alternatives to cement has therefore been of particular interest to scientists in recent years. Clay is an obvious alternative to cement, particularly because of its abundance. It also has the advantage of being non-toxic and recyclable. Clay has been used for several decades in certain rural areas of Senegal for the construction of dwellings. However, its use in construction poses a major problem due to its poor mechanical properties compared to cement. To solve this problem, several researchers have undertaken the design of clay-based composites. In these materials, clay serves as the matrix, while the reinforcement can be polymers, biopolymers, wood or domestic waste. Eggshells, which are mainly composed of calcium carbonate ($CaCO_3$) [1]-[4] are waste from the food industry or poultry farming. Eggshell powder has a composition similar to that of limestone and Portland cement [3] [5]. The use of eggshell powder as an additive to improve the mechanical properties of clays is becoming increasingly common in various fields of engineering and construction it contains large amounts of calcium carbonate [6]-[9], a chemical compound known to strengthen materials. Adding eggshell powder to clays can increase their compressive strength, improve their durability and reduce their permeability. Numerous studies have been conducted to evaluate the effects of incorporating eggshell powder into clays. For example, research conducted by S. Pornsimma *et al.* studied the incorporation of eggshell as a flux in porcelain production to reduce the firing temperature through an extrusion process. In this study, porcelain clay products were prepared by extrusion. The eggshells incorporated into the porcelain clay products reduced the firing temperature from 1250˚C to 800˚C, as well as the firing time [10]. Similarly, a study by J. P. Leclair and Lucas H. investigated another approach that seeks to reduce the consumption of virgin plastic resources by replacing them in part with eggshell powder. This helps divert waste from landfill sites by reusing it, given that large quantities of eggshells come from cracking plants [11]. In addition, B.H. Ngayakamo and colleagues at the University of Science and Technology in Abuja, Nigeria, studied the effective reuse of granite waste and eggshell powder for the production of clay bricks as an alternative method of waste disposal while improving the physical and mechanical properties of fired clay bricks. Their study showed that fired bricks incorporating 20% of granite and 10% of eggshell powder achieved the highest compressive strength 3.24 MPa, a bulk density of 1.76 g/cm^3 and water absorption of 12.2% to 900˚C. This is considered as an energy-saving process for the manufacture of fired clay bricks [12]. Eggshell powder has also been used as an additive in fired clay bricks as a bio-filler and flux in earthenware clay compounds through an extrusion process. This study showed that the optimum firing temperature for clay bricks was 1000˚C for a period of 5 h. The best physical, mechanical and thermal properties were obtained by adding 20% of eggshell powder to the fired clay brick [13]. Furthermore, Thomas McGauran *et al.* studied the incorporation of poultry shells and slag ash as high-load polymer fillers in polypropylene. Both were added to polypropylene in loads of up to 55% by weight and were successfully compounded into consistent polymer granules. Characterization showed that the eggshell and litter ash adhered to the polypropylene, exhibiting full contact between the particles and the matrix, verifying the potential as a polymer filler material [14]. In addition, eggshell powder has been used in the development of biphasic bone cement. The objective of their work was to obtain biphasic calcium phosphate biochemistry from chicken eggshells. These physical and mechanical properties, as well as its apparent porosity, were evaluated and compared to commercial cement [15]. R. P. Munirwan and his colleagues examined the performance of adding eggshell powder to clay soil to stabilize it. Their study showed that the use of eggshell powder for stabilization in this experiment can improve bearing capacity, making it beneficial for construction in the field [16].

The aim of this study is to design a clay/eggshell powder (ESP) material with very good mechanical properties. The raw materials (clay and eggshell powder) were characterized using X-ray fluorescence (XRF) analysis, Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) coupled with energy dispersive X-ray analysis (EDX). The microstructure of the test specimens was observed. The physical and mechanical properties of the clay/ESP test specimens were also examined by measuring their flexural and compressive strengths, bulk density and linear shrinkage.

2. Materials and Experimental Methods

2.1. Materials

The clay samples were collected from the open-cast quarry at the Tchicky clay site, due to its good cohesion. The village of Tchicky is located 4 km south of the National Road 1 (RN1) between Diass and Sindia (Thies region, Senegal) and the Ndioudiouf-Ndiob backwater (Fatick region, Fatick Department) see **Figure 1(a)** and **Figure 1(b)**. Eggshell waste was collected free of charge from a cafeteria at Cheikh Anta Diop University of Dakar (UCAD), Senegal.

Figure 1. Image of (a) clay 1 (b) clay 2 soil and (c) eggshell waste (d) eggshell powder.

The eggshell samples were first subjected to pre-treatment such as drying in a HERAEUS OVEN scientific oven at a temperature of 40˚C for 24 hours. The eggshell and clay samples were then crushed using a pestle and mortar until a fine powder was produced see Figure 1(c) and Figure 1(b). The powder was sieved through a 125 µm mesh sieve. After pre-treatment, 1.2 g of each sample was weighed separately using an S-Artoruis balance, then mixed with 0.12 g (10% of the sample mass) of Licowax binder. In addition, each mixture was compacted separately using a compressor, where it was trapped between two cylindrical stainless steel discs to form pellets as shown in Figure 2. These pellets were used for physicochemical and mineral characterization. The raw materials, clay and eggshell powders, were carefully selected based on their availability, purity and physicochemical characteristics.

Figure 2. (a) clay pellet 1Tchicky, (b) clay pellet 2 Ndioudiouf and (c) eggshell powder pellet.

The bricks were made by incorporating four different percentages 5, 10, 15 and 20 wt% of PCO, which was labeled as: B-1, B-2, B-3 and B-4 respectively. To allow a comparison of properties, bricks without PCO were also manufactured and labeled B-0, see Table 1. The bricks were prepared by varying the quantities of eggshell powder and clay soils as shown in Table 1.

Table 1. Formulation of raw materials as a percentage by weight %.

Batch compositions	Raw Materials		
	Clay 1 (%)	Clay 2 (%)	Eggshell powder (ESP) (%)
B-0	100	100	0
B-1	95	95	5
B-2	90	90	10
B-3	85	85	15
B-4	80	80	20

Five different formulations were prepared for each type of clay, with increasing levels of ESP - B-0: 0% ESP (control sample) - B-1 to B-4: Increasing ESP levels (within a margin of 5%) Each mixture was homogenized, molded into parallelepiped brick boxes measuring (4 × 4 × 16) cm^3, see Figure 3, then dried at room temperature for a curing period of 21 and 28 days.

2.2. Experimental Methods

2.2.1. Physicochemical and Mineral Characterization of Raw Materials

The main physical-chemical and geotechnical characterization tests were carried

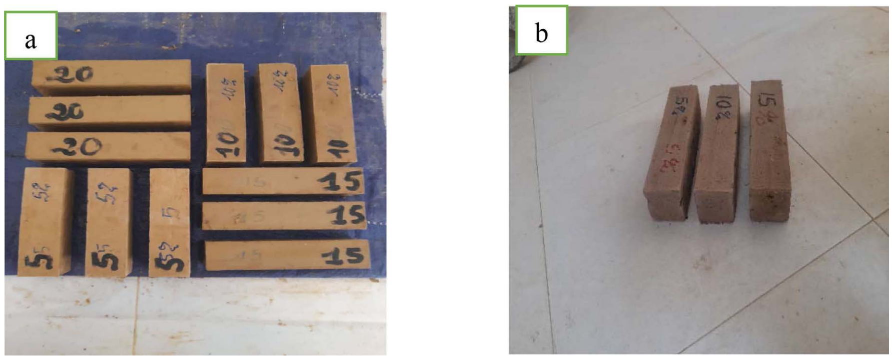

Figure 3. Clay brick (a) Tchicky and (b) Ndioudiouf.

out on the samples in accordance with international recommendations and standards. The water content of the various samples was determined using the oven drying method, in accordance with the experimental standard [XP CEN ISO/TS 17892-1, 2005]. This involves determining the mass of free water removed by drying in an oven at a temperature of 105˚C [17]. The Atterberg limits are the water contents that represent the limits of transition from one state to another: the liquidity limit and the plasticity limit. The plasticity limit (LL) is the water content corresponding to the transition from the solid state to the plastic state. However, the liquidity limit (LP) represents the transition from the plastic state to the liquid state. These two limits were determined in accordance with standard [NF P94-051, 1993] [18]. The chemical composition of the clay and eggshell powder samples was determined by X-ray fluorescence using a Niton XLT 400s portable thermoscientific spectrometer (P-XRF) with a silver anode excitation source and filters as a secondary excitation target [19]. Fourier transform infrared spectroscopy (FTIR) analysis of the clay and eggshell powder samples allows the functional groups of the mineral species they contain to be identified based on their specific vibration frequencies [20].

2.2.2. Physical-Mechanical Characterization

The compression test is performed on a TECH CYBER PLUS Progress hydraulic press, shown in the figure below. The test piece is placed in the center of the press plate. The contact surface between the test piece and the plate and the compression plate is 16 cm^2: (4×4) cm^2. The compression force is increased automatically. This test is carried out in accordance with standard MT-6 by applying a compression load rate of 10 kN/s to a test piece in the form of a cube measuring $(4 \times 4 \times 8)$ cm^3, positioned in the center of the lower plate of the compression testing machine, until it breaks. With a displacement speed of 10 kN/s and a starting load of 2 kN. The study aims to evaluate the effect of adding eggshell powder (ESP) on the mechanical strength of clay bricks for a curing period of 21 and 28 days. Two types of bricks (Tchicky Clay (1) and Ndioudiouf Clay (2)) were tested with varying amounts of ESP, ranging from 0% to 20% with a margin of 5%.

3. Results and Discussion

3.1. Characterization of Raw Materials

The water content of a material A is the ratio of the weight of water (W_w) in that material to the weight of the same dry material (W_d) (dried in an oven at 105˚C for 24 to 48 hours). A 24-hour drying period in an oven at 105˚C for Tchiky clay at 12.09 g resulted in a moisture content of 1.85%.

Table 2 shows the Atterberg limits, as well as the plasticity index, liquidity index and consistency index for the different samples tested. The plasticity index (PI) is the difference between the liquid limit (LL) and the plastic limit (LP), showing the extent of the plasticity range and defining the clay content of soils.

Table 2. Atterberg limits of the samples.

	W_L	W_P	PI	CI
Clay 1	33.86	18.14	15.72	2.04
Clay 2	33.61	17.51	16.10	1.97

After repeated tests to limit margins of error, we obtained the results shown in Table 2. The results show that our clay samples are plastic clays, as their plasticity indices (PI) are greater than 15. In general, good building soil has a plasticity index (PI) between 16% and 28% and a liquidity limit (WL) between 32% and 46% [15]. We can conclude that our two clays are good building materials (bricks, concrete, mortar, etc.), as their plasticity indices and liquidity limits are within these ranges, 16% to 28% and 32% to 46%, respectively.

The chemical composition of the clay and eggshell powder samples was determined by X-ray fluorescence using a Niton XLT 400s portable thermoscientific spectrometer (P-XRF) with a silver anode excitation source and filters as a secondary excitation target.

Table 3. Chemical composition of clays 1 and 2 and eggshell powder in %.

Raw Materials	Oxides										
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	ZnO	K_2O	TiO_2	Cl_2O	ZrO_2	SO_2
Clay 1	50.39	10.55	9.73	0.93	LOD	0.01	0.03	0.95	0.03	0.09	0.12
Clay 2	60.54	13.98	7.73	0.88	LOD	0.005	3.02	1.03	0.03	0.12	0.15
ESP	12.14	8.14	0.12	48.46	25.95	0.001	0.14	0.01	0.02	LOD	0.54

ESP: Eggshell powder.

The chemical composition of clays 1 and 2 and eggshell powder was measured using XRF, and the data is presented in Table 3. The chemical composition of clay soils (clay 1 and clay 2) consists mainly of silica (SiO_2), which is 50.39% for clay 1 and 60.54% for Clay 2, while the amount of alumina (Al_2O_3) is 10.55% for Clay 1 and 13.98% for clay 2. In addition, iron oxide (Fe_2O_3) is also present with the amounts of 9.73% and 7.73% for Clay 1 and Clay 2, respectively, along with other oxide compounds [21]-[23]. The main composition of eggshell powder used as a mineral additive is 48.46% calcium oxide (CaO), 12.14% silica, 8.14% alumina and other oxidized compounds in small quantities [24].

Figure 4 shows the surface morphology and EDS spectra of the clay and eggshell powder samples. SEM observation (Figure 4(a) and Figure 4(d)) shows a clear distinction between the morphology of the clay and the eggshell powder. The eggshell powder has a fibrous, tubular morphology, in contrast to the hexagonal, spheroidal morphology of the clay particles. However, the morphology of the clay can be attributed to the difference between the dimensions of the b-axis of the tetrahedral layer and the octahedral layer. This creates tensions within the structure, causing the layers to curl up. The resistance of the layers adjacent to this tension is greatly weakened by the presence of the water layer between the sheets. The EDS analysis in Figure 4(b) shows that the peaks for silicon (Si), aluminum (Al) and oxygen (O) are high compared to the other elements. The silicon and aluminum contents in the form of oxide and fluxing metal such as potassium (K) in clay soil are essential for the formation of the mullite phase known for exceptional strength, thermal shock resistance, and chemical inertness. On the other hand, the SEM micrograph of eggshell powder in Figure 4(c) shows non-agglomerated particles in the form of several interlocking fibers, which are different from those observed in clay soil. This shows that eggshell powder does not have plastic properties. However, the EDS in Figure 4(d) revealed high peaks of calcium (Ca), carbon (C) and oxygen (O), confirming the presence of calcium carbonate ($CaCO_3$) in the eggshell powder [25].

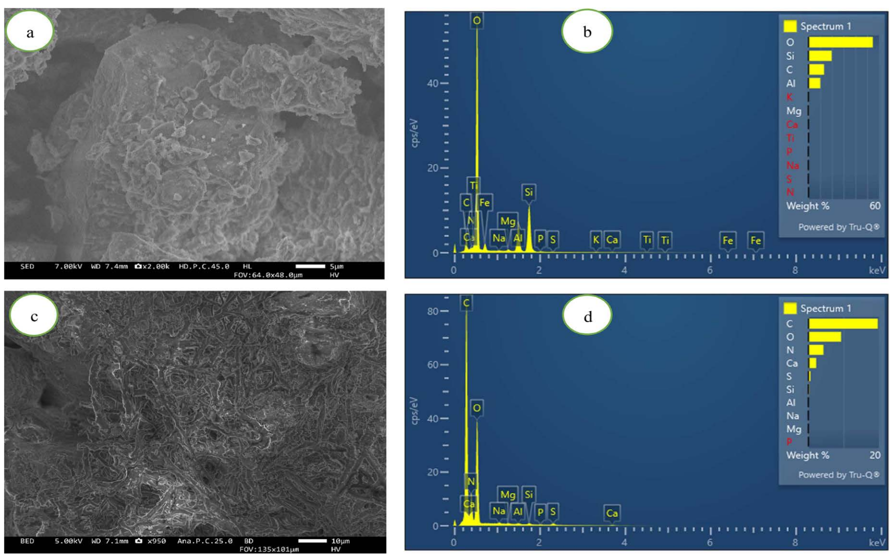

Figure 4. (a) (b) SEM micrograph and EDS spectrum of clay soil (c) (d) SEM micrograph and EDS spectrum of eggshell powder.

The FTIR spectra of the clay samples were recorded in the range of 450 - 4000 cm^{-1}, as shown in Figure 5 and Figure 6.

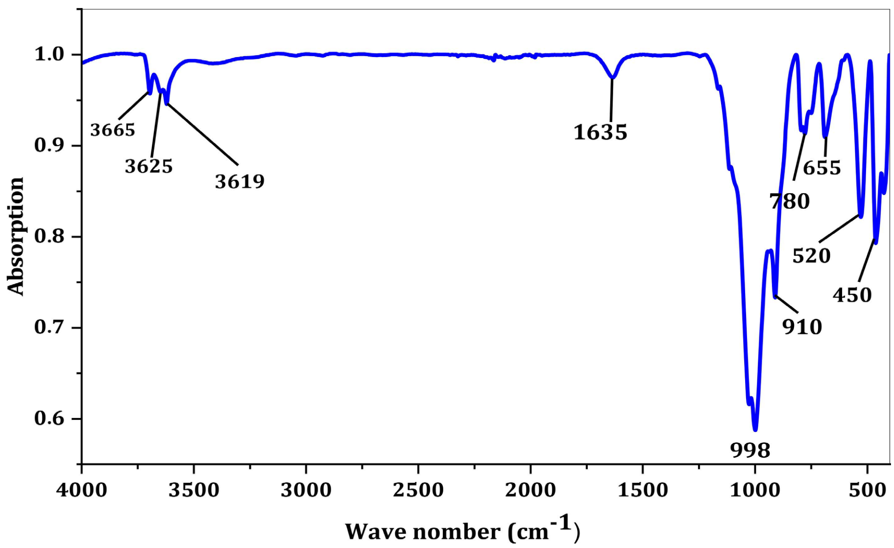

Figure 5. Fourier transform IR spectrum of clay 1.

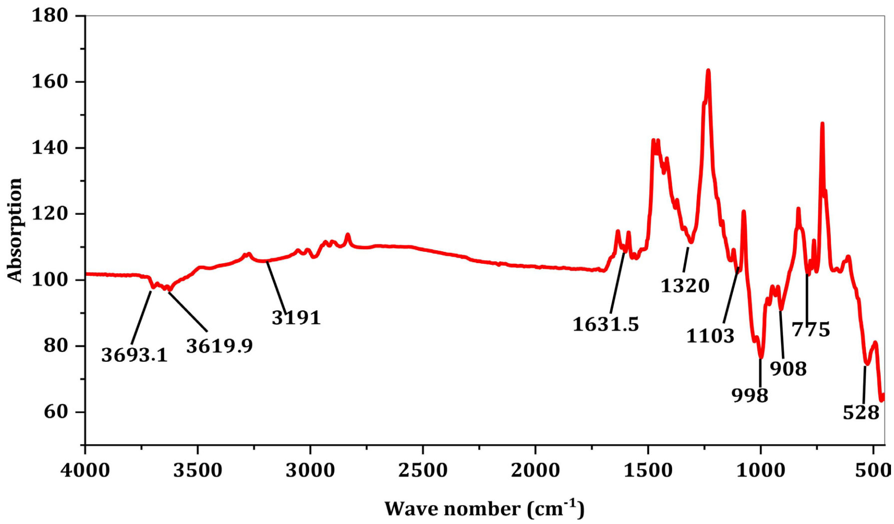

Figure 6. Fourier transform IR spectrum of clay 2.

The FTIR spectra of clays 1 and 2 shown in **Figure 5** and **Figure 6**, respectively are in good agreement with the literature on clay materials [25]-[27]. By comparing the observed bands of clays (1 and 2) with those predicted by S. CAILLERS *et al.*, we confirm that the main dominant component of our clays is kaolinite, with a small amount of quartz and illite. In the high frequency range, the spectra of clays 1 and 2 show the characteristic vibrations of hydroxyl groups at 3625.15 cm^{-1} and 362,369 cm^{-1} respectively in the valence vibration range. Three other bands, 906 cm^{-1}, 934.1 cm^{-1} and 998 cm^{-1}, correspond to angular deformation bands. These hydroxyls correspond to the characteristic vibrations of dioctahedral minerals, in particular kaolinite. The shoulder of a weak band at 3210 cm^{-1} in both clays, as well as the appearance of bands at 647 cm^{-1} and 678 cm^{-1} respectively, confirms the dominance of kaolinite [28]. In addition, absorption bands can be distinguished at 1552.73 cm^{-1}, 1601.49 cm^{-1} and 1697.64 cm^{-1} corresponding to the deformation vibrations of water (H_2O) hydrating the interlayer cations. The band around 2987 cm^{-1} is due to the presence of aliphatic groups. Two medium bands are also observed at 1459.16 cm^{-1} and 1308.97 cm^{-1} due to the presence of calcite [24]. The absorption bands around 1103.82 - 1129.2 cm^{-1}, 998 - 1040 cm^{-1} and 997.9 - 1042 cm^{-1} correspond respectively to the deformation vibrations of the Si-O and symmetric Si-O-Si and asymmetric Si-O-Al bonds [25]. The bands at 934.1 - 909.73 cm^{-1}, as well as those between 788.13 cm^{-1} and 774 cm^{-1}, are attributed to vibrations of the bonds Al-O-OH and Al-OH (*Al* is tetracoordinated) in kaolinite and halloysite. The bands at 647cm^{-1} for clay 1 and 678.84 cm^{-1} for clay 2 are attributed to the vibration of the bond Si-O-Si in kaolinite [26]. The shoulders around 464.4 cm^{-1} and 525.71 cm^{-1} for clays 1 and 2, respectively, are attributed to the deformation vibrations of the Si-O-Si and Si-O-Al bonds [27]. However, elemental chemical analysis has helped to further this information by providing the oxide content of each chemical element.

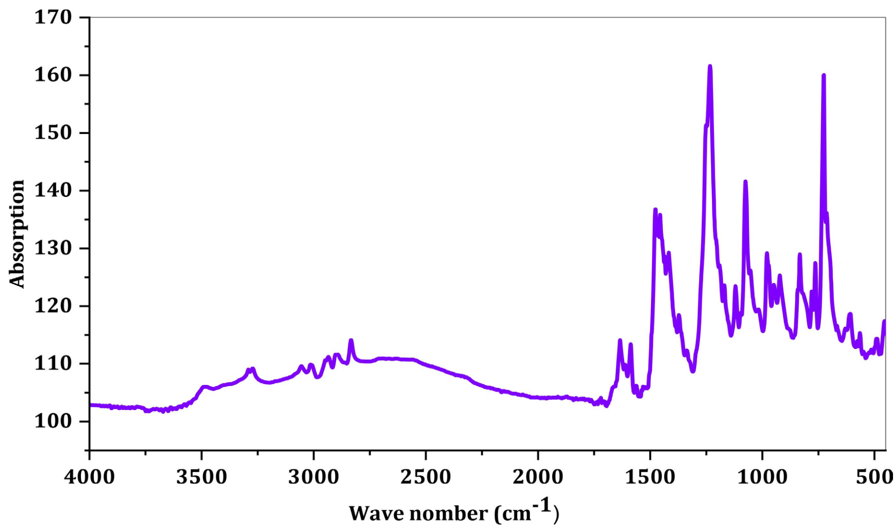

Figure 7. Fourier Transform IR spectrum of eggshell powder.

The FTIR spectrum of eggshell powder was recorded in the range of 450 - 4000 cm^{-1}, as shown in **Figure 7**. In the high frequency range, two broad bands are observed at 3450 cm^{-1} and 3198.93 cm^{-1}, which can be attributed to the hydroxyl groups linked (O-H) [28]. Indeed, the absorption bands at 2989.72 cm^{-1} and 2870.37 cm^{-1} are the harmonic vibrations of the bond stretching modes (C-O) [29]. In addition, the vibration bands ranging from 1696.96 cm^{-1} to 1546.6 cm^{-1} are attributed to the (O-H-O) bonds of water molecules. Similarly, the absorption bands at 1459.11 cm^{-1}, 1430.49 cm^{-1} and 1380.25 cm^{-1} correspond to the (C=O) bonds from the ion carbonate (CO_3^{2-}). Likewise, the absorption bands between 773.03 cm^{-1} and 762.96 cm^{-1} correspond to the elongation modes of the C O bonds of calcite. The band at 872.07 cm^{-1} is due to asymmetric stretching (C=O) for carbonate species [30]. This is mainly due to the exposure of the highly active surface of CaO to atmospheric air and the absorption of CO_2 during dehydration, which ultimately absorbed water and carbon dioxide on the surface of the catalyst and converted CaO to $(OH)_2$ and CaO_3. Finally, the bands around 588.8 cm^{-1} and 477.16 cm^{-1} are attributed to the bonds Ca-O and O-Ca-O, respectively [31].

3.2. Mechanical Behaviour of Unfired Clay Bricks

3.2.1. Flexural Strength

Flexural strength is defined as the stress in bricks or any other material just before it fails in a flexural test. The flexural strength of raw clay bricks with different proportions of eggshell powder is illustrated in **Figure 8**. It can be seen that raw bricks with 5%, 10% and 15% eggshell powder added have a higher flexural strength than the control brick after 21 days of curing. This may be due to the formation of amorphous phases that ensure the bonding of clay and ESP particles and making the bricks more compact and more resistant, improved compactness and cohesion between the clay grains. A more co-pact density and hydration chemistry between silica and alumina. Silica reacts with alumina under the action of water to form aluminosicate [32]. In addition, the clay brick with 10% eggshell powder by weight had the highest flexural strength with 0.674 MPa. The same constants were also observed for a 28-day curing period, with a maximum strength of 1.062 MPa for the brick with 10% eggshell powder added. However, this strength is lower than the value of the control sample when 20% eggshell powder by weight is added after 21 and 28 days of curing. Low-content eggshell powder (≤10%) fills pores by acting as a binder or filler, reducing porosity and increasing flexural strength, which is particularly noticeable at 28 days when reactions are more advanced [33]. This increase may be due to the good structure of the bricks with the addition of ESP, which leads to stress concentration and reinforcement of the interfacial bond between the bricks. Whereas the addition of high-content ESP (>10%) leads to matrix saturation: excess ESP can create non-reactive or poorly bonded areas. Similarly, an increase in porosity due to poor dispersion or differential shrinkage can induce microcracks. The chemical incompatibility of certain ESP components can inhibit the formation of cementitious phases [23].

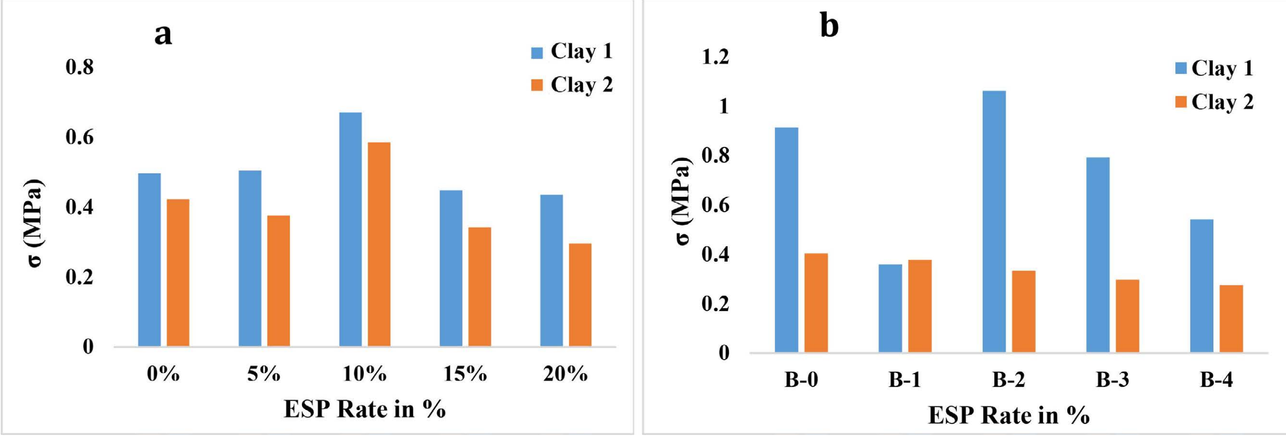

Figure 8. Flexural stress as a function of % ESP by weight in clay bricks after 21 (a) and 28 days (b) of curing.

3.2.2. Compressive Strength

Compressive strength, as undoubtedly the most important mechanical property of bricks, is shown in **Figure 9**. At 21 days of curing, the compressive strength measured on the bricks produced from clays 1 and 2 shows a slight decrease from B-1 to B-3 of ESP compared to the control sample B-0. In B-4, there is a significant increase (4.054 MPa), the highest value in the series. This could indicate that 20% ESP improves strength, unlike the other rates. In clay 2, strength decreases overall with increasing ESP content, except at 10%, where it increases sharply (2.442 MPa). This peak at 10% could indicate optimal interaction between the components at this specific rate. However, the behaviour of clay 2 indicates increased sensitivity to ESP content, with an optimal formulation around 10%. Beyond this, strength decreases, which may be due to an excess of non-reactive material or poor dispersion. However, the 28-day compressive strength for clay 1 increases overall with the addition of ESP. The maximum is reached for sample B-3 (4.212 MPa), indicating a significant improvement over the control sample B-0 (3.658 MPa). A slight decrease is observed in B-4 (4.041 MPa), suggesting that an excess of ESP could lead to saturation or a decrease in the beneficial effect. Eggshell powder probably acts as a binder or reinforcing agent, improving the cohesion of clay particles up to an optimal threshold [10] [34]. Unlike clay 1, the strength of clay 2 bricks initially decreases from B-1 to B-2 compared to the control sample B-0, reaching a minimum at B-2 (1.175 MPa). A clear improvement is observed at B-3 (2.060 MPa), followed by a decrease at B-4 (1.308 MPa). Clay 2 used in brick making appears to be less reactive to the addition of ESP, or requires a more precise dosage to benefit from its effects. Clay 2 seems less reactive to the addition of ESP, probably due to its chemical composition. Silica (SiO_2) and alumina (Al_2O_3) are the main components of clay bricks. Clay 2 contains a high level of silica, at 60.5%, which could explain its low reactivity. Excess silica can destroy cohesion between clay particles, making bricks brittle and weak. Good quality clay bricks usually contain between 50% and 60% of silica and 10% to 20% of alumina [35]. It is possible that clay 2 has an imbalance in its composition, which affects its mechanical properties. To improve its reactivity, the proportion of silica and alumina may need to be adjusted. The non-linear behaviour suggests a different chemical or physical interaction compared to clay 1. The addition of eggshell powder can improve the compressive strength of clay bricks, but the effect depends heavily on the type of clay used and the ESP content. An optimal dosage (as an inappropriate dosage can reduce the effectiveness of the material Clay 1.

4. Conclusion

In this study, clay collected from the Tchicky quarry was used as the raw material and eggshell powder as a mineral additive to produce green clay bricks. Clay bricks with different levels of eggshell powder added were made in rectangular parallelepiped moulds using the adobe method. The brick samples were dried at room temperature for curing periods of 21 and 28 days. The results of the laboratory

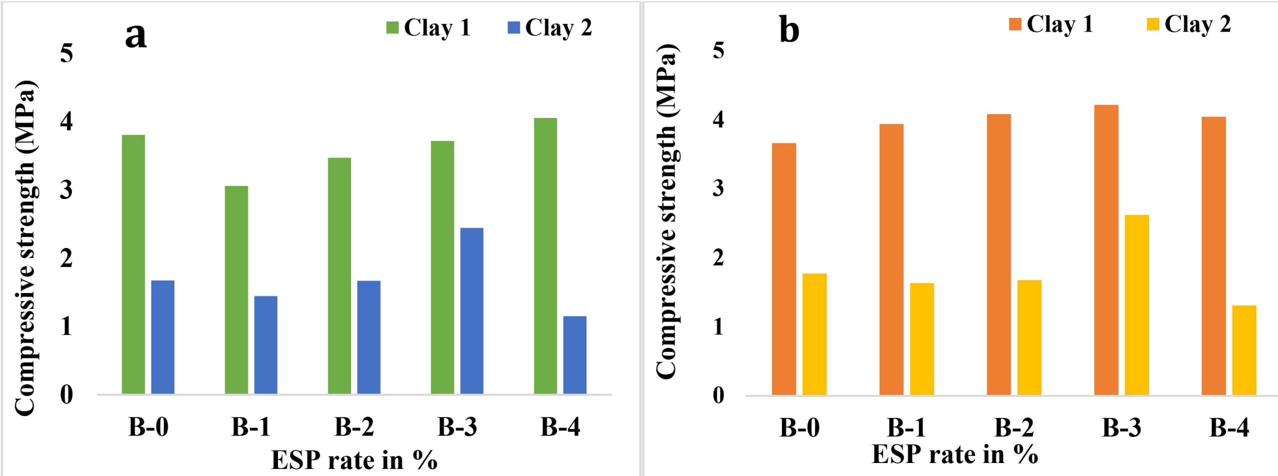

Figure 9. Compressive strength of clay bricks 1 and 2 as a function of ESP content at 21(a) and 28 (b) days of curing.

tests led to the following conclusions: The addition of eggshell powder to clay bricks improves the mechanical properties of the clay, particularly after 28 days of curing. The sample with 15% eggshell powder had the highest compressive strength (4.212 MPa). Meanwhile, the sample with 10% ESP performed best in terms of flexural strength. This is due to densification and improved cohesion. This suggests that an optimized formulation could be considered for practical applications in sustainable construction. These results are consistent with certain values obtained in the literature, with some authors recommending 15% and 20% ESP. The use of ESP in this study is considered an effective waste disposal method and an environmentally friendly approach to waste management in the food processing industry. It also demonstrates the potential for recycling organic waste (eggshells) in the manufacture of building materials. Additional studies are needed to understand the mechanism of interaction between eggshell powder and clay. To do this, we will start by firing our clay bricks with different PCO rates, and then study the technological properties of the final product (porosity, bulk density, mechanical property...).

Acknowledgements

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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