Reuse of livestock waste for the reinforcement of rammed-earth materials: investigation on mechanical performances

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Abstract

Agricultural wastes as an additive within raw earth materials could improve the mechanical and physical properties of new sustainable construction materials and enhance waste management from a circular economy perspective. This study intends to fill the lack of knowledge considering the mechanical effects of animal fibres on rammed-earth materials. The effects of livestock waste, *i.e.*, sheep wool fibre (SWF), as a reinforcing element in building components produced using raw earth and lime-free mortars have been evaluated. The samples were made by varying the wool content (0.25% or 0.50% weight) and the length of the fibres (from 10 mm to 40 mm). Linear shrinkage, flexural strength, compressive strength, and fracture energy were evaluated on samples incorporating SWF, to assess the effects of this waste addition on the mechanical performances of new bio-composite material. The best result of the flexural strength was 1.06 MPa, exhibited by samples made with the longest and highest percentage of fibres, 40 mm, and 0.50%, respectively. The average compression strength was about 3.00 MPa. The average energy fracture of the composite was 806.38 (N/mm).

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Introduction

Today, the construction sector is the leading cause of environmental degradation, global warming, and climate change, with 50% of carbon emissions, 20% to 50% of energy and natural resource consumption, and 50% of the total production of solid waste (Vasilca *et al*., 2021). For this reason, researchers and technicians' attention to new alternative construction materials derived from renewable sources is constantly growing (Bonoli *et al*., 2021).

In this context, the interest in earthen construction materials is increasing for the restoration of existing historical and cultural built heritage and new constructions. Several advantages come from using raw earth-based materials, all related to a significant decrease in environmental pollution and CO_2 emissions. Raw earth-building components, if made without chemical additives (*i.e*., only by physical and mechanical stabilisation), are totally recyclable (Achenza and Sanna, 2009; Parlato *et al*., 2021). Generally, raw earth-based building components are extracted and worked directly or close to the building site, so their use significantly reduces logistic and transportation costs and related gas emissions. Compared to standard building materials (*i.e*., concrete, steel, glass), raw earth-based materials are suitable to balance and control indoor acoustic and thermal comfort (Fagone *et al*., 2019).

To improve earthen materials' behaviour, additives or stabilisers are often used to design raw mixes, such as mineral binders (cement, alginate, bitumen) (Rivera-Gómez and Galán-Marín, 2017; Turco *et al*., 2021), animal and vegetal stabilisers (oil, casein, animal glue, latex) (Medvey and Dobszay, 2020), reinforcement fibres, synthetics, or natural (Ramesh, 2016; Eliche-Quesada *et al*., 2017). Several studies focused their attention on agricultural waste (AW) and their high potential use in different construction applications, all aiming to minimise their production and promote environmental sustainability (Reif *et al*., 2016; Liuzzi *et al*., 2017; Barreca *et al*., 2019). Furthermore, when considering their significant mechanical and physics performances, AW are suitable alternative materials in the building sector and are considered the most sustainable, economical, and energy-efficient resource (Jannat *et al*., 2020). Many scientists have evaluated the use of AW as natural additive in the field of unfired earth materials (Serrano *et al*., 2016; Vatani Oskouei *et al*., 2017; Araya-Letelier *et al*., 2018). These studies focused mainly on agro-waste fibres (*e.g*., straw fibres, *Hibiscus cannabinus* fibres, ground olive stones, and wheat straw fibres) (Salih *et al*., 2020). The main advantage of fibre reinforcement is to improve the mechanical properties, shrinkage rate, and ductility of the composite (Vega *et al*., 2011; Parisi *et al*., 2015; Laborel-Préneron *et al*., 2016).

The major part of the paper is related to adding vegetable fibres to rammed earth; fewer researchers have evaluated the mechanical effects of reinforcements of animal fibres (*e.g*., pig hair, sheep wool) (Araya-Letelier *et al*., 2018; Statuto *et al*., 2018).

In the sustainable use of natural resources framework, raw sheep wool is reconsidered as a renewable resource by converting difficult waste into value-added material. In Europe in 2011, the estimated production of raw sheep wool based on sheep number was about 260,000 tons. In Italy, the estimated annual production of raw wool is around 14,000 tons, of which only a small part, around 5%, is suitable for the textile industry and has a commercial value (Rajabinejad *et al*., 2019).

A large amount of raw sheep wool that is unsuitable for the market represents a relevant problem for sheep farmers due to the complexity and difficulty of the disposal management. Moreover, the increasing waste landfill fees are often the main reason for the illegal disposal of raw sheep wool (Saxena and Sewak, 2016). In accordance with European Environmental Regulations [EC Regulation 1069 (2009), EU Regulation 142 (2011)], raw sheep wool must be sent to specialised sites for incineration or landfill, and only if it is previously washed or disinfected it can be buried or burned without a permit. The valorisation of this livestock waste, the complex disposal, as components of building elements, is, in turn, sustainable both from the point of view of reducing CO_2 emissions in the production process and reducing energy costs for managing the construction. Furthermore, wool fibres, obtained from raw wool generally washed with natural soap in the form of soft mats or lose wool, could be used as thermal and acoustical insulation of buildings, as reinforcement fibre for composite materials, as sorbent materials for the treatment of water pollution, *etc*. (Dénes *et al*., 2019).

This study refers to the possible reuse of raw sheep wool, trying to partially fill the lack of knowledge about the effects of animal fibre reinforcement on rammed-earth materials.

This paper proposes using short wool fibres as reinforcing elements of non-structural building components in combination with eco-sustainable materials such as raw earth and lime-free mortars.

The research gap that this paper attempts to address and, therefore, the study's novelty, lies in the combination of animal fibres with binders that do not use cement or lime to reduce the impact of the production processes. In addition, results involve comprehensive examination through cross-comparison of mechanical performance to obtain in-depth information to propose these new materials as an alternative to other more common earthen products reinforced with plant fibres. The attempt is to evaluate the effects derived from adding this waste to unfired materials made with clay soil present in Sicily, which was formerly used to produce bricks and mixed up with pyroclastic sand. This pyroclastic sand is typical of the Etna volcano area in Sicily (Italy) and is commonly used to increase the mechanical strength of mortar and concrete products. The design of the raw earth mix was prepared by performing only a physical stabilisation, without chemical additives, by obtaining a material that is totally recyclable at the end of its useful life (Parlato *et al*., 2021). The elements that we propose to develop could be used as closing elements.

Then, to avoid chemical additives, the present work investigates the application of a promising natural fibre, *i.e*., sheep wool fibre, for rammed earth stabilisation, including their influence on the material's mechanical properties. The main purpose is finding the most performant design for fibre length and percentage. Then, physical and mechanical tests were carried out to obtain information related to earthen material. Other interesting aspects that could be investigated in the future are concerning material's durability, its thermal properties, and interaction between fibres and the matrix soil by SEM analyses.

Finally, the results have been compared with other studies investigating other agricultural waste additives.

Materials and Methods

Experimental tests on the physical and mechanical behaviour of raw earth-based materials were reported under flexural and compression tests. Since the mechanical behaviours of the raw earth building components are sensitive to both soil composition and fibre content, the length and percentage of the SWF were changed to evaluate the best mix design. According to the results, the reinforcement by fibres is essential to confer the due ductility to the bio-compound. First, test samples were performed using the same soil mix design and varying only the fibre content (0.25% or 0.50%) and the length of the fibres (from shorter fibres of 10 mm to longer fibres of 40 mm).

Clayey soil and volcanic sand

Experimental tests were carried out on a soil mix previously investigated by authors (Parlato *et al*., 2021); this soil, chosen among five different soil designs for its best performances, was embedded with raw sheep wool. The base material is composed of kaolinite soil called *Terra di Floridia* (FS) (extracted close to Syracuse in Sicily and traditionally used to produce bricks) modified through a physical stabilisation process (Achenza and Sanna, 2009) to improve its mechanical behaviour.

The particle size distribution of FS has been changed by adding clay, in the proportion of 58% FS soil and 42% clay, in weight. In literature, good results have been achieved with a similar soil composition (Galán-Marín *et al*., 2013). Clay improves plasticity, mechanical characteristics, and cohesion, reducing water absorption and enhancing erosion resistance to wind and waterproofing toward capillarity water.

The clay used for the stabilisation process comes from a pit near Misterbianco, in the province of Catania (Italy). The basic components used for casting the samples were chosen for their chemical and mechanical properties and favouring local materials with a consequent reduction in logistic and transport costs. Subsequently, to improve its mechanical resistance and prevent shrinkage and cracking problems, the modified *Terra di Floridia* (FS^M) has been mixed with typical pyroclastic sand, sieved to 2 millimetres of the Etna volcano area (Sicily) and commonly used to produce mortars and concretes. Although the rate was 45% of sand and 55% of FS^M on a dry weight basis, the final design, including water, was 45% FS^M, 35% sand, 20% water. In a previous work by authors, this mix obtained the best mechanical performances among five different mixes (Parlato *et al*., 2021). This characteristic sand called '*azolo'* is formed on the surface of the lava by crushing glassy materials generated by the rapid cooling of the magma. Table 1 shows the chemical composition of clay and volcanic sand added to FS.

Sieve analyses were carried out in an earlier study (Parlato *et al*., 2021) according to ASTM D7928 - 17 requirements to determine the particle size distributions of FS and clay (Figure 1A and B). Figure 1C shows the particle size distribution of the soil mix FS^M, and Figure 1D the particle size of the final mix (including additive sand).

Livestock waste as reinforcement fibers: sheep wool

The livestock waste used in this investigation is typical raw wool from Sicilian sheep of the *Valle del Belice* race, widespread in this region, whose thick and medium length fleece is rejected by the textile industry. In the recent past, these fibres were suitable for mattress and/or pillow production, but currently, they are only considered special waste with high disposal costs for breeders.

Sheep wool fibres used as reinforcement fibres, that is, *Valle del Belice* fleece, is a very coarse wool with a diameter ranging from around 70.0 μm. The authors physically and mechanically characterised these fibres in a recent study (Parlato *et al*., 2022). A selection of about 180 fibres were measured (length and mass), and tensile tests were performed on selected fibres.

Due to the high hydrophilic content of wool, three different conditioning programs (wet, dry, and intermediate conditions) were carried out and compared, to acquire useful information about the maintenance of the mechanical properties in wet environments like those present in lime mixes. Three different test settings were studied: wet condition, controlled environment, and oven-dried condition SWF tensile performances. Secant stiffness modulus (*Es*), stress (σ_y and strain ε_y) at the yield, elongation at break (E_b), and stress at break (σ_b) are the mechanical properties evaluated from each test. Average values μ_i and standard deviations σ_i for these quantities are reported in Table 2, separately for the three testing conditions and the whole population. Although the tests produced

Table 1. Chemical composition of clay and volcanic sand.

Chemical components	Clay (%)	Volcanic sand (%)
SiO_2	53.15	45.9
Al_2O_3	14.42	20.43
TiO_2	0.85	1.44
Fe_2O_3	6.09	9.99
MnO	0.10	0.15
MgO	2.13	4.71
CaO	7.21	10.22
Na_2O	1.17	4.02
K_2O	2.08	1.35
S	0.03	-
P_2O_5	0.16	0.48

Table 2. Characteristics of the fibers used as reinforcement (SWF 'Valle del Belice') (Parlato *et al*., 2022).

	σ_b (MPa)		E_b (%)		σ_y (MPa)		ε_y		E_s (MPa)	
	μ	σ	μ	σ	μ	σ	μ	σ	μ	σ
1. Saturated samples	134.57	34.10	42.00	0.11	75.37	21.92	0.04	0.02	2057.06	584.53
2. Normal conditioning	144.02	41.61	43.00	0.11	84.70	23.31	0.05	0.02	1903.59	621.32
3. Dry samples	133.65	47.22	43.00	0.19	85.97	33.59	0.07	0.03	1367.38	381.40
Entire population	137.31	41.37	42.00	0.14	81.44	27.15	0.05	0.02	1739.41	755.44

μ, average values; σ, standard deviation.

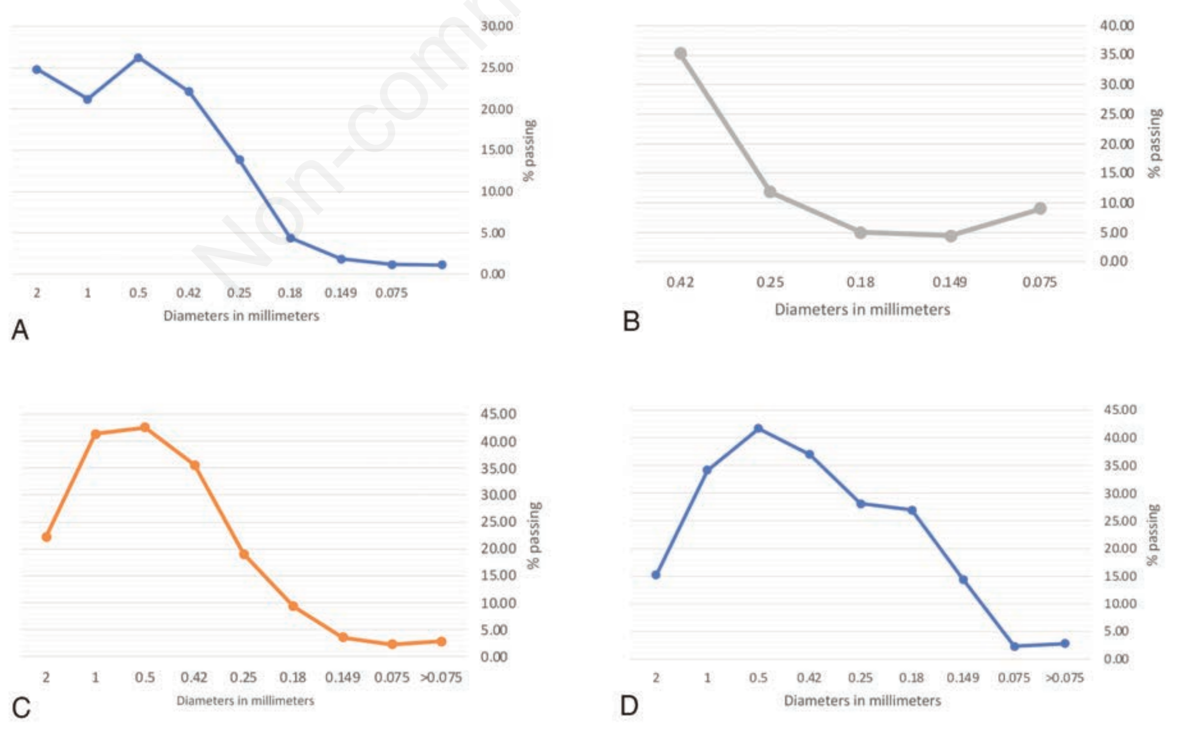

Figure 1. Particle size distribution of Floridia soil (FS) (**A**), clay (**B**), the size distribution of FSM (**C**), the particle size of the final mix (including sand) (**D**).

very similar results, better results have been obtained for the fibres tested under the second experimental condition, that is, in a controlled environment (SWF immersed in distilled water for ten minutes and left to dry for 24 h in ambient with temperature and humidity monitored). The results appear to support the use of SWF as a reinforcing material because this second conditioning procedure is close to the real condition of fibres inside a mixture of an earth-based composite. The average tensile strength obtained was 137.31 MPa, with elongation at break 42.00%. These values are comparable with wool tensile strength found in literature, ranging between 120 and 174 MPa, and higher respect the elongation at break ranged between 25 and 35% (Cheung *et al*., 2009). Compared to other natural fibres, for example, jute and sisal, with an average tensile strength of 249 MPa and 484 MPa (Alves Fidelis *et al*., 2013), respectively, wool exhibits a lower strength, while the elongation at break is higher than the most common natural fibres used as reinforcement material (Ku *et al*., 2011).

The percentage of fibres used in the mix ranged in weight between 0.25% and 0.50%. This low percentage in weight corresponds to a larger volume of fibres due to the low density of this kind of wool (the average density of 0.94 gr/cm^3).

The length of the fibres varies between 10-40 mm to evaluate the possible effects caused by the different lengths on the mechanical behaviours of the samples.

Figure 2 (A and B) shows a scanning electron microscopy (SEM) analysis of the wool fibre surface and transversal section. SEM analysis was carried out at *Torre Biologica* of the University of Catania.

Adobe mix sample preparation

The preparation of the samples began with adding fibres to the homogeneous soil mix, that is, FS^M and sand. All specimen preparation and compaction processes have been executed manually (Figures 3 and 4). Since manual compaction was adopted, the compaction energy was not monitored, but the manufacturing water content of the soil was controlled while mixing. The same amount of mix was cast

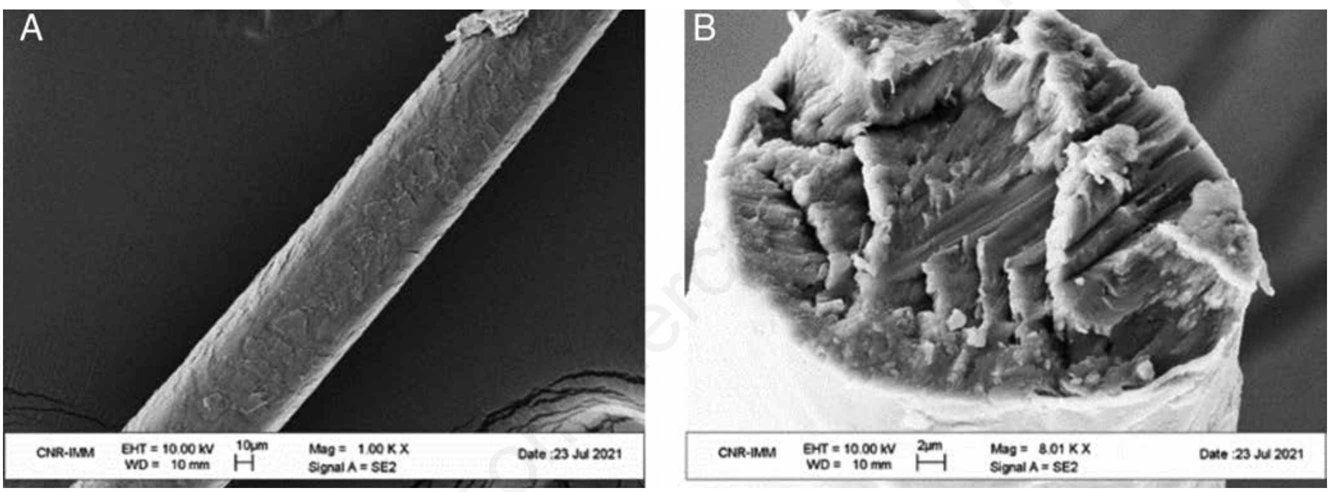

Figure 2. Scanning electron microscope analysis of sheep wool fibre surface (**A**) and transversal section (**B**).

Figure 3. Specimens tested in this study.

for each formwork to obtain the design density of 1800 kg m^{-3}.

SWFs were slowly and carefully added to the clay soil to reduce the formation of fibre bundles. Finally, once the fibres were fully incorporated into the mixes, water was added in four steps, manually stirring between each step. The samples were cast in consecutive layers and compacted by hand, applying sufficient pressure. Three days after casting, the samples were demoulded and put in a dry and aired storage area (AT=26°C, ARH=60%) for 28 days to cure before testing. Moreover, after this time, the specimens 'weight difference of two consecutive readings (24 hours) was constant (±0.01 g). A similar curing procedure is in accordance with the New Zealand Code (NZA 4298, 1998) and was used in previous research, also with non-cement stabilised earth samples (Parlato *et al*., 2021). After curing time, the weights and dimensions of each sample were measured, and then the densities were evaluated. As stated above, to perform a sensitivity analysis on the impact of different fibre lengths and dosages on the performance of adobe mixes, SWF was cut in length (from 10 mm to 40 mm) and mixed in dry soil at a 0.25% or 0.50% per dry weight of soil. Nine combinations of mixes, with six repetitions for each mix, were tested to assess the influence of SWF addition on the design of the adobe mix. Table 3 reported the different mixes tested within this study identified by the related ID. For example, mix identified with ID 0 was the control sample, mix identified with ID XX-YY incorporated fibre XX mm length at a rate of 0, YY%.

Mechanical and physical performances

Flexural and compressive test

According to European standards (EN 1015-11:2019), prismatic samples (160×40×40 mm) were prepared for the mechanical testing of moulded mortar specimens. To prevent adherence, the standard steel moulds used were previously moistened.

Nine combinations of mixes, named and listed in Table 3, with six repetitions for each mix, were tested to assess the influence of SWF addition on the design of the adobe mix. The samples were manufactured by changing both the length and percentage of fibre.

First, mechanical assessment began with the flexural tests. The test was performed using a universal testing machine (UTM) connected with a Load Cell Hottinger Baldwin; Catman Software for Tests with Huge Channel Counts implemented data acquisition. Applying a single point load at the mid-span performed flexural tests, the load speed was 10 N/s. Load values were recorded from the start until the sample failure. Next, the Prismatic specimens were placed on two lower roller supports (100 mm wheelbase). Using Eq. 1, the flexural strength of specimen (σ_f) was evaluated.

$$\sigma_f = \frac{3\,F\,L}{2\,b\,d^2}\,[MPa] \quad (1)$$

where *F [N]* is the maximum applied load, *L [mm]* is the span between supports (100 mm), *b [mm]* is the width of the specimen at the mid-section, and *d [mm]* is the average depth of the specimen at the fracture section. After failure, the compressive test was performed on the two remaining prismatic parts obtained after the flexural fracture. In order to avoid the concentrations of applied forces through particular points of the specimens due to the presence of irregularities on the surface of the samples (Ciancio *et al*., 2013), the two remaining parts tested under compression have been positioned on the side part with a level surface. This level and even end surface was obtained by the previously moistened steel mould used for the casting process. Samples kept the same flexural ID by adding numbers 1 or 2. To determine compressive strength value (σ_c), Eq. 2 was applied:

$$\sigma_c = \frac{F}{S}\,[MPa] \quad (2)$$

where *F [N]* is the maximum applied load, and *S [mm²]* is the surface of the loaded section. In both cases, *i.e*., flexural and compressive tests, the breaking loads were determined in correspondence with the maximum load reached during the tests.

Figure 4. A sample ready for the flexural test.

Table 3. Sample composition used for mechanical tests.

Mix	Wool length (mm)	Wool (%)	Soil* (%)	Water (%)
ID -0	-	-	80	20
ID 10-25	10	0.25	79.75	20
ID 10-50	10	0.50	79.50	20
ID 20-25	20	0.25	79.75	20
ID 20-50	20	0.50	79.50	20
ID 30-25	30	0.25	79.50	20
ID 30-50	30	0.50	79.50	20
ID 40-25	40	0.25	79.75	20
ID 40-50	40	0.50	79.50	20

*Soil, FS^M (55%) and volcanic sand (45%).

Linear shrinkage

Linear shrinkage test was carried out according with the testing method ASTM C326-09 suitable for earthen materials. The dimensions of the prism mould length (Li, mm) were measured to evaluate linear shrinkage. After 28 days, corresponding to the drying period, the dimensions of the prism sample length (Ld, mm) were recorded using a digital calliper.

The linear drying shrinkage (Sd) is calculated by using Eq. (3):

$$Sd = \frac{Li-Ld}{Li} \times 100 \quad (3)$$

where *Li [mm]* is the drying length of the specimen, measured by using a digital calliper, and L_d *[mm]* is the internal length of the mould.

Fracture energy

In this study, to determine the fracture energy of raw earth samples reinforced with sheep wool fibres, a uniaxial flexural test was carried out with displacement control and load speed of 0.5 N/s. The fracture energy was calculated as the area under the load-displacement curve and was considered as the energy absorbed by the material until the deflection at the final fracture of the beam and considering the Petersson correction (Petersson, 1982) to this area under the load-displacement curve two areas were added evaluated considering the force energetically equivalent to the weight of the samples. Figure 5 shows a typical flexural test on a sample.

$$G = \frac{W0 + W1 + W2}{Alig} \, [\mathrm{Nmm^{-1}}] \quad (4)$$

Figure 5 shows a diagram to evaluate the energy fracture. To calculate the fracture energy has been applied equation 4. Where *G* is the energy fracture, W_0 is the area under the experimental load-displacement curve, W_1 and W_2 are the areas evaluated considering the weight of the sample between supports. In detail, W_1 equals F_0 δ_0, *i.e.*, F_0 is the sample mass (between supports), δ_0 is the last displacement, and W_2 is supposed to equal $W_1/2$. *W3* is considered negligible and equal to zero. A_{lig} is the surface area of the notch; in this study, A_{lig} was assumed to equal the lower surface of the sample because they were realised without a notch. Flexural tests have been carried out on mixes ID 0, ID 10-50, ID 30-50 (SWF 0.50% - 30 mm), and ID 40-50 (SWF 0.50% - 40 mm), made by keeping constant the percentage of wool and varying its length.

Experimental results and discussion

Effect of sheep wool addition on flexural strenghts

Figure 6 shows the average flexural strengths of each of the nine-mix tested after casting, including the minimum and maximum values measured. Again, as usually happens with natural materials, the results are quite dispersed, with average flexural strength values ranging from 1.06 MPa to 0.50 MPa.

In general, the average flexural strength of adobe mixes decreased when sheep wool was introduced into the mix. These reductions respect ID 0 were 23% for ID 10-25, 12% for both mix ID 10-50 and ID 20-25, 24% for ID 20-50, 43% for ID 30-25, of 21% for ID 30-50. Samples realised with ID 40-25 obtained a result comparable with the strength of the control samples, respec-

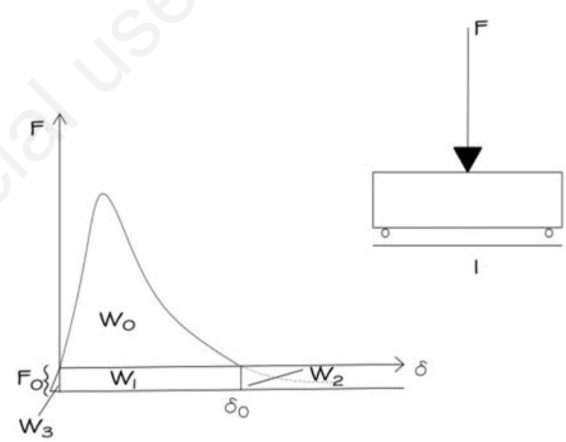

Figure 5. Schematic force-displacement diagram.

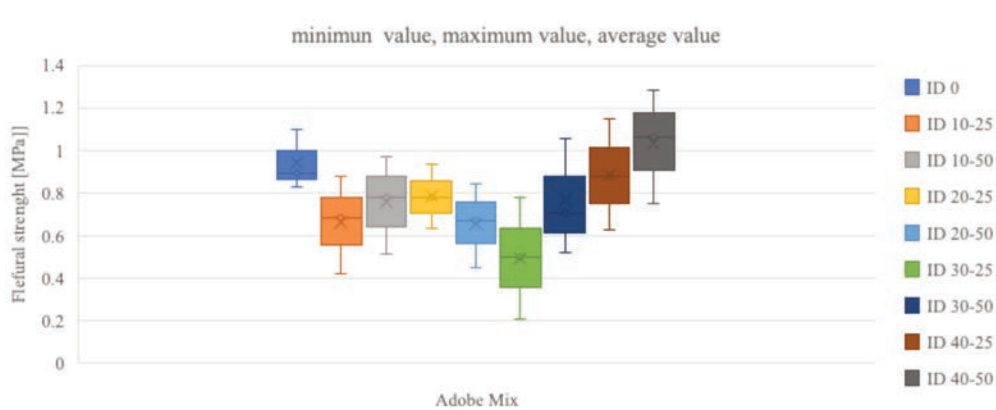

Figure 6. Average values of flexural strength of the adobe mix, including minimum and maximum values for each mix.

tively 0.88 MPa and 0.89 MPa, and mix ID 40-50 registered the highest average flexural strength of 1.06 MPa, with an increment of 16% respect mix ID 0. The standard deviation values varied from 0.16 MPa (ID 10-50) to 0.23 MPa (ID 20-50).

These results have been compared to those previously reported for unfired reinforced adobe, despite in literature there are few studies regarding the flexural strength of raw earth materials. Araya-Letelier *et al*. (2018) in their study carried out investigations on the mechanical properties of unfired adobe reinforced with pig hair. They found a flexural strength ranging from 0.34 MPa to 0.49 MPa.

In their study, Galan-Marin *et al*. (Galán-Marín *et al*., 2010b) evaluated the flexural strength of adobe realised by clay soil incorporating 0.25% of raw wool and 10 mm long fibres. The average flexural strength obtained was 1.10 MPa.

In the present work, the reduction in average flexural strength as sheep wool is introduced into the mix is explained by the generation of fibre clusters inside the composite matrix. This same trend was registered in other research (Baeza *et al*., 2013; Araya-Letelier *et al*., 2018) that ascribes this negative effect of cluster formation inside the mix. Clusters avoid a complete adhesion between fibres and the matrix by affecting the average strength of the composite material. On the contrary, the mix realised with longer fibre exhibits a flexural strength comparable (ID 40-25) or higher (ID 40-50) than the flexural strength of the control sample. The reason is the higher contact surface among the longer fibres and the matrix, as already explained in the literature (Aymerich *et al*., 2012; Oliver-Ortega *et al*., 2018). Moreover, as the fibre is added by weight, by considering the same percentage of fibre, it is necessary to underline that the number of fibres inside the specimens increases for shorter fibre; it is assumed that the 10 mm fibres are 4 times the number of 40 mm fibres. The higher the number inside the mix, the higher the possibility of cluster formation.

In any case, all adobe mixes exhibited an average flexural strength higher than the minimum values required by the worldwide used raw earth regulations, *e.g*., the New Mexico Earthen Building Materials Code, which foreseen an average minimum flexural strength of 0.35 MPa.

Effect of sheep wool addition on compressive strengths

The compressive strength of raw earth materials is considered a fundamental mechanical parameter depending on various parameters, such as compaction energy, manufacturing water content, dry density, and stabilisation process (Agostino and Galipoli, 2015; Arrigoni *et al*., 2018). Figure 7 shows the average results values for the compressive test, including minimum and maximum values and standard deviation. Each value represents the average of a total of 12 specimens.

Compared to flexural strength values, the compressive strength values are less dispersed, with an average ranging from 2.58 MPa to 3.67 MPa, not considering specimens made with mix ID 30-25 that registered a value of 1.42 MPa lower of the 53% respect ID 0. The lowest values of flexural and compressive strength registered by mix ID 30-25 could be explained by some irregularities during the specimens' manufacturing process, so the authors have decided to discard this batch in the conclusions.

Despite the manual specimen preparation process, it could be important to reach a minimum compression of soil by using a by hand press to compact the material better, control its final behaviours, and avoid irregularities of the samples.

In this case, the introduction of fibre inside the mix does not cause a decrease in the average compressive strength, except for mix ID 30-50 and ID 40-25, which registered a reduction with respect to the control mix of 15% and 3%. The best average compressive strength was obtained by mix ID 20-50, with an increment of almost the 15% if considering the control mix. The standard deviations varied from 0.13 MPa (ID 30-25) to 0.62 MPa (ID 10-50). Although the values obtained for the mechanical properties of the unfired adobe considered in this study compare well with those reported in other scientific papers on similar fibres (Parlato *et al*., 2021) with the same mix of ID 0, so without fibre addition, found higher strengths values, *i.e*., 6.74 MPa for compressive strength and 1.65 MPa for flexural strength. The reason for this difference is due to the different conditions of temperature and humidity. In the previous study, the samples were produced during the winter season, with a temperature ranging between 13.1-15.5°C and air humidity between 76.7-80%; in this study, specimens were moulded during the summer season, with a peak of temperature above 40° and a high thermal excursion during the day. So, the drying process was accelerated by this weather condition compromising the final results. Galan-Marin investigated the effect of adding sheep wool and Lignum Sulfonate to raw-earth-based specimens and reported an

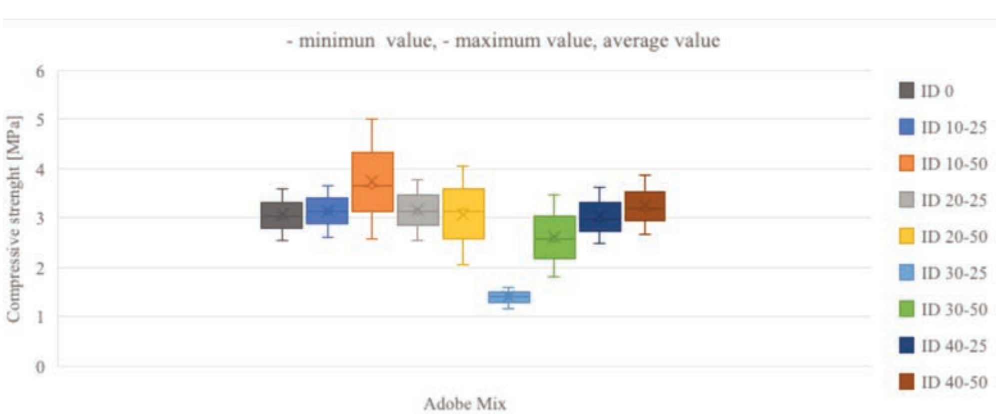

Figure 7. Average values of compressive strength of the adobe mix, including minimum and maximum values for each mix.

increase of compressive strength of the 37% respect control sample, 2.23 MPa and 3.05 MPa, respectively (Galán-Marín *et al*., 2010a).

Statuto *et al*. (2018) evaluated the compressive strength of clay adobe brick reinforced by sheep wool fibres. They found that clay bricks mixed with 3% by weight of sheep wool exhibited an average compressive strength of 4.32 (MPa) (Statuto *et al*., 2018).

In any case, all compressive values found exceed the minimum compressive resistances foreseen by New Zealand Code and New Mexico Building Code, respectively 1.3 MPa and 2.1 MPa (New Mexico, 2009; NZA 4298, 1998).

The high values obtained in the trials carried out within this study are mainly due to the soil mix incorporating '*azolo*', *i.e.*, a siliceous inert, commonly used in the concrete industry, gives high resistance to compression.

The resistance increment is due to the chemical reaction of the pozzolanic material *Azolo*, before inert, induced by water addition into the mix. This chemical reaction produces calcium silicate hydrate (CSH), a crystalline compound responsible for the material's strength, and calcium hydroxide (CH), which acts as a filler and lines pores within the matrix in a manner like that of binding aggregates in concrete (Hossain *et al*., 2007).

In Table 4, mechanical results concerning the adobe mix tested in this study including the dry density, are summarized.

The correlation between dry density and average compressive strength has also been investigated by excluding the outlier values referred to as mix ID 30-25. An almost horizontal linear as shown in Figure 8. The higher the density, the higher the compressive strength obtained by the test.

The direct correlation between density and compressive strength is widely accepted in literature (Kouakou and Morel, 2009; Pelé-Peltier *et al*., 2022). When fibres are introduced into the mix, they replace a quantity of soil. As expected, the bulk density of wool fibres is lower than that of the soil; the addition of fibres led to a decrease in the earth content and thus in the composite dry density. Moreover, when fibres are added, the dry density is lower and the material more porous. Several studies linked mechanical properties with porosity; compressive strength decreases with increasing porosity (Al Rim *et al*., 1999; Ghavami *et al*., 1999; Sutcu *et al*., 2016). Furthermore, compressive strength decreases due to the weak adhesion among fibres and clay matrix. Water absorption by fibre, occurs during the first 24 hours after moulding, and pushes away the soil. After drying, the volume of fibres decreases, and voids are created around them and the soil matrix. Fibres could slip easily, reducing the homogeneity of the composite material (Khedari *et al*., 2005; Quagliarini and Lenci, 2010; Rivera-Gómez *et al*., 2014).

Statistical analysis of test results

The mechanical resistances obtained through the performed trial were validated by a ONE-way analysis of variance (ANOVA) to determine if there is a statistically significant difference among the corresponding data means. A check of normality (by computing the Shapiro-Wilk test) and homogeneity of variance (by computing Levene's test) preceded ANOVA. Statistical analysis was performed using RStudio (https://www.rstudio.com/), a free access software. The ANOVA test was performed both for flexural strength and compressive strength results.

Tables 5 and 6 summarise results concerning the validation of flexural and compressive tests, respectively.

In both cases P-value is above the significance level P=0.05, *i.e.*, 0.95 and 0.71, so not statistically significant. One-way ANOVA test revealed that the null hypothesis could be retained; there was no statistically significant difference among the mean of the data set groups.

Effects of fibres addition on linear shrinkage

Shrinkage is a physical phenomenon that refers to the drying process of the soil mixture caused by the evaporation of moisture content; it determines the cracking of the material, which can increase the penetration of water, loss of strength, and material decay (Sangma and Tripura, 2020). Araya *et al*. (2018) determined

Table 4. Average flexural and compressive strength with correlated standard deviation.

Mix	Average flexural strength (MPa)			Average compression strength (MPa)			Average dry density (kg/m^3)		
	μ	σ	Coeff. of variance (%)	μ	σ	Coeff. of variance (%)	μ	σ	Coeff. of variance (%)
ID -0	0.89	0.18	21.30	3.05	0.43	37.8	1960.0	923.4	49
ID 10-25	0.68	0.16	23.09	3.14	0.29	9.30	1904.3	933.5	49
ID 10-50	0.78	0.16	20.91	3.67	0.63	17.00	1904.4	948.5	49
ID 20-25	0.78	0.17	22.33	3.13	0.35	11.10	1890.0	941.28	49
ID 20-50	0.67	0.23	34.97	3.14	0.62	19.00	1841.3	918.20	49
ID 30-25	0.50	0.20	40.00	1.42	0.13	9.00	1678.0	837.44	49
ID 30-50	0.70	0.19	27.35	2.58	0.47	18.00	1844.5	919.37	49
ID 40-25	0.88	0.19	21.44	2.97	0.35	11.10	1844.5	918.69	49
ID 40-50	1.06	0.20	19.70	3.20	0.40	12.00	1883.0	938.46	49

Table 5. One-way analysis of variance (ANOVA) of flexural strength test results.

	Sum of squares	df	Mean square	F	P
Between groups (SSB)	0.30	7 (dfB)	0.04(MSB)	0.03	0.95
Sum within groups (SSW)	66.53	40 (dfW)	1.66 (MSW)		

SSB, regression sum of the square; SSW, the total sum of squares (SSW=SSB+SSx); dfB, regression degrees of freedom (dfB=k–1); dfW, error degrees of freedom (dfW=n–k); k, total number of groups; n, total observations; MSB, regression mean square (MSB=SSB/dfB); MSW, error mean square (MSW=SSW/dfW); F, F test statistic (F=MSB/MSW); P-value corresponds to FdfB, dfW.

shrinkage rate using a different test procedure; tests were carried out on two flat specimens and by a grid to control crack distribution. As a result, they observed a reduction of crack width for both fibre dosage and fibre length increment (Figure 9). Prismatic specimens tested within this study did not exhibit cracks on the surface; only reductions in linear measures and volume were detected.

The linear shrinkage test is a suitable tool for obtaining information about the shrinkage behaviour of raw earth materials, especially structural components, to correctly anticipate the joint design of space construction (Ciancio *et al*., 2013). The requirements for the maximum shrinkage of rammed earth are identified, although the threshold values are discordant; for example, the New Zealand Standard (NZA 4298, 1998) is 0.05%, and the German Lehmbau Regeln (Volhard, 2009) 2%. The linear shrinkage rate for the specimens concerning the different adobe mixes was evaluated by applying Equation 3. Samples made with soil mix ID 0 exhibited the highest shrinkage rate, corresponding to 6.25%.

The general trend is that the addition of fibres to the mix causes a decrease in the shrinkage rate, as already demonstrated in other research with comparative analysis among reinforced and unreinforced samples (Vega *et al*., 2011).

Influence of reinforcement on the structural response of the material

The quantity of energy necessary to generate and propagate a crack through a material is called fracture energy. Fracture energy is the measure of how resistant a material is to crack formation, considering both instantaneous and long-lasting stresses.

However, fracture energy evaluation is not regulated by universal norms or laws, but only recommendations are introduced by materials testing organisations (*e.g*., RILEM, ASTM); the most common experimental procedures use prismatic samples with or without notch (Volhard, 2009). To investigate the structural response of the material in terms of first-crack resistance, post-cracking performance, and energy absorption capability, flexural tests have been carried out on five different mixes, *i.e*., ID 0, ID 10-50, ID 30-50, ID 40-25, and ID 40-50, the mix with the higher percentage of wool. The material's fracture energy was determined using Eq. 4. In Table 7, the average results obtained for the different mixes investigated in this study are reported. The peak load (F_{peak}) ranges between 296.97 (N) and 529.00 (N). The higher energy fracture was 959.89 (Nmm^{-1}) exhibited by mix ID 30-50; the lower was 706.47 (Nmm^{-1}) of mix ID 30-50. By considering mix ID 0, it was observed that the last displacement δ_0 is equal to the peak displacement (δ_{peak}). Furthermore, it was not possible to determine its fracture energy because the unreinforced samples showed a rapid load drop with a complete loss of residual strength after the peak load.

The maximum value of energy fracture and peak load was reached by mix ID 40-50, F_{peak} 529.00 (N) and G_f 959.89 (N/mm), the average value of energy fracture was 806.38 (N/mm).

Figure 10 shows a demonstrative set of Load-deflection curves (F-δ) related to mix ID 40-50.

The plot of this figure shows that reinforced samples have an almost linear initial response characterised between origin and peak load.

All reinforced specimens present the same mechanical response to load; an initial linear phase until the peak load, and a second phase characterised by a decreasing load and high displacements values; a falling load segment for large deflection value characterises this second phase.

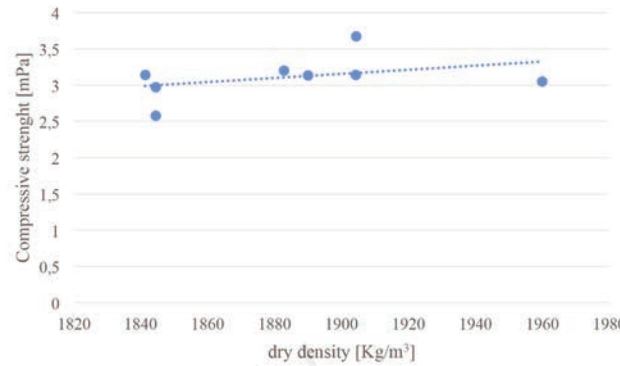

Figure 8. Correlation between dry density and compressive strength.

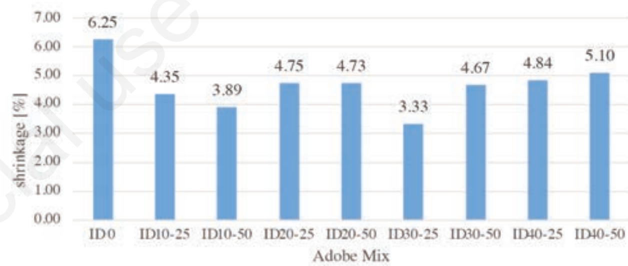

Figure 9. Shrinkage rate for prismatic samples.

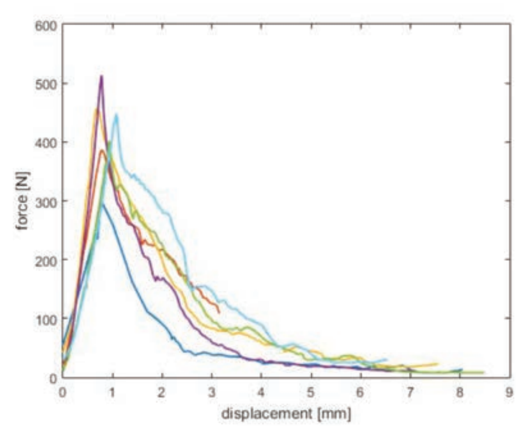

Figure 10. Typical set load-displacement curve for reinforced samples (F-δ).

Table 6. One-way analysis of variance (ANOVA) of compressive strength test results.

	Sum of squares	df	Mean square	F	P
Between groups (SSB)	16.64	7 (dfB)	2.38 (MSB)	0.66	0.71
Sum within groups (SSW)	319.12	88 (dfW)	3.63 (MSW)		

SSB, regression sum of the square; SSW, the total sum of squares (SSW=SSB+SSx); dfB, regression degrees of freedom (dfB=k–1); dfW, error degrees of freedom (dfW=n–k); k, total number of groups; n, total observations; MSB, regression mean square (MSB=SSB/dfB); MSW, error mean square (MSW=SSW/dfW); F, F test statistic (F=MSB/MSW); P-value corresponds to FdfB, dfW.

However, experimental results demonstrated that the addition of sheep wool fibre improves the ductility of the specimens; after failure, reinforced specimens exhibit the two parts linked together, while adobes realised without reinforcing fibres registered a sudden drop in load because of the formation of unstable macroscopic crack after the maximum load. Addition of fibres in the mix determines a redistribution of the internal forces. During the trial, it was observed that the failure mode under the flexural test was at the central axis; this means that samples were homogeneous with little discontinuities in the mass of the material. The failure mode occurs when the imposed load exceeds the flexural capacity of the material, *i.e.*, flexural failure. The observed cracks were vertical and were located in the middle of the sample; these cracks were produced on the tension side of the prismatic specimens, which further extended to the compression side. The flexural tension failure happened gradually, *i.e.*, ductile failure. On the contrary, the sample performed with mix ID 0 exhibited a fragile failure mode; after the peak load was observed, a total loss of load-carrying capability. The two parts of the specimen appear completely separately after failure (Figure 11).

A limited number of studies have explored the influence of fibrous reinforcement on ductility, fracture resistance, and post-fracture behaviour of earthen material (Clementi *et al*., 2008). Aymerich and co-workers (2012) investigated the improvements in strength and post-fracture performances by introducing wool fibre reinforcement in earthen material (Aymerich *et al*., 2012). They performed flexural tests on notched samples (prepared with two fibre weight fractions (2% and 3%) and different fibre lengths (1, 2, and 3 cm) to compare the mechanical response in terms of first-crack resistance, post-crack performance, and energy absorption capability. The results demonstrated that the fibrous reinforcement improved residual strength, ductility, and energy absorption (Aymerich *et al*., 2012). Corbin *et al*. (2014) investigated the fracture energy of rammed earth by using the wedge splitting test, a test for calculating the fracture energy of concrete. As per obtained results, they found that adding cement increases the material's fracture energy but adding wool up a critical amount decreases it. The energy fracture evaluated was 0.71 N/m and 0.68 N/m for control samples made without cement and with wool addition of 1% and 2%, respectively. They used wool fibers from a carpet manufacturer cut into 30-50 mm lengths with an average tensile strength of 69.2 N/mm^2 (Corbin and Augarde, 2014). Recently, Muzíková *et al*. (2021) evaluated the fracture energy and strain-stress curve of three different sets of illite rammed earth. The highest value of the fracture energy found was of 4.858±0.002 J/m^2 (Mužíková *et al*., 2021).

One of the significant effects of the inclusion of natural fibres in the soil matrix is related to the failure mode of the specimen; in the case of the composite material, after the ultimate load was reached, the specimens were still deformed, and fine cracks could be seen on the surface of the specimens, on the contrary, control specimen made without fibre addition broke quickly and almost without warning. Under compression, failure was characterised by the gradual formation of diagonal cracks on the lateral sides of the samples without an immediate failure after the peak load. The natural fibres held together significant parts of the soil matrix by delaying failure. Additionally, there was no rupture of the sheep fibres, although a loss of bond between the fibres and the soil matrix was recorded in the proximity of the cracks (Figure 12).

Under the flexural test, the stress-strain curve obtained is linear for all the test series up to maximum load. For the natural soil, the

Figure 11. Failure mode of the control sample (ID 0).

Table 7. Average results for energy fracture for the different mix.

Mix	F_{peak} (N)	δ_{peak} (mm)	δ_0 (mm)	G_f (N/mm)
ID 0	398.00	4.28	4.28	-
ID 10-50	417.70	3.79	9.12	706.47
ID 30-50	296.97	4.22	8.58	752.79
ID 40-50	529.00	4.66	11.04	959.89
Average	410.41	4.24	8.25	806.38

Figure 12. Composite specimens after compression test.

final failure occurs immediately after the ultimate load, and for this reason, it was not possible to evaluate the material's energy fracture, which is estimated as the area under the stress-strain curve.

Mix ID 10-25 and ID 20-25 obtained the best average compression strength, 3.14 (MPa) and 3.13 [MPa], respectively; the best flexural strengths were obtained by the no fibrous mix with 0.89 (MPa) (mix ID 0), and by mix ID 40-25 with 0.88 (MPa). In general, the average flexural strength of adobe mixes decreased when sheep wool was introduced into the mix; a different trend was obtained when the length and concentration of fibres increased (ID 40-50); the explanation found in literature is on the higher contact surface between fibres and clay matrix (Aymerich *et al*., 2012; Oliver-Ortega *et al*., 2018).

Another important aspect to be considered deriving by fibre addition is the prevention of visible shrinkage cracks due to the drying process. The linear shrinkage rate decreases by fibre addition from ID 0 with a rate of 6.25% to the minimum rate of 3.89%, registered by ID 10-50.

Adding fibres to the mix determines the decrease in dry density from 1960.0 (kg/m^3) to around 1845.00 (kg/m^3) for mix ID 30-50 and ID 40-25, and 1883.0 for mix ID 40-50.

Conclusions

The agricultural sector produces a tremendous amount of waste, by-products, and co-products, whose disposal constitutes a severe financial and ecological concern.

This work assessed the effectiveness of incorporating livestock waste, sheep wool fibre, as a reinforcement fibre in raw earth specimens. The purpose was to valorise local building materials, contemporarily reduce a considerable quantity of waste, and realize a totally recyclable material suitable for non-structural materials.

The final aim was to evaluate the most performant mix by considering fibre rate percentage and length. Based on the mechanical trials, the best results have been exhibited by specimens realised with mix ID 40-50. However, all samples showed significantly higher mechanical strength values than the minimum values required by the most relevant international regulation on raw earth material.

The high values obtained in this study are mainly due to the soil mix incorporating a pozzolanic inert, 'azolo', which gives increased resistance to compression and is traditionally used to improve mechanical strength in concrete production and mortar. *Azolo* soil reacting with clay minerals shapes a variety of cement-like compounds binding soil particles together while reducing water absorption by clay particles.

Future research must fill the gap of this first investigation by exploring the acoustical and thermal behaviour, the moisture absorption and desorption, and the durability of these raw earth materials reinforced with sheep wool fibres.

Moreover, a further detailed study should be able to evaluate, using scanning electron microscope (SEM) and energy dispersive X-ray (EDX) techniques, the fibre-matrix bonding for a better understanding of its mechanical performance.

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