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### **Research** Article

## Ultrasonic, Molecular and Mechanical Testing Diagnostics in Natural Fibre Reinforced, Polymer-Stabilized Earth Blocks

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The aim of this research study was to evaluate the influence of utilising natural polymers as a form of soil stabilization, in order to assess their potential for use in building applications. Mixtures were stabilized with a natural polymer (alginate) and reinforced with wool fibres in order to improve the overall compressive and flexural strength of a series of composite materials. Ultrasonic pulse velocity (UPV) and mechanical strength testing techniques were then used to measure the porous properties of the manufactured natural polymer-soil composites, which were formed into earth blocks. Mechanical tests were carried out for three different clays which showed that the polymer increased the mechanical resistance of the samples to varying degrees, depending on the plasticity index of each soil. Variation in soil grain size distributions and Atterberg limits were assessed and chemical compositions were studied and compared. X-ray diffraction (XRD), X-ray fluorescence spectroscopy (XRF), and energy dispersive X-ray fluorescence (EDXRF) techniques were all used in conjunction with qualitative identification of the aggregates. Ultrasonic wave propagation was found to be a useful technique for assisting in the determination of soil shrinkage characteristics and fibre-soil adherence capacity and UPV results correlated well with the measured mechanical properties.

#### 1. Introduction

The development of building systems has been inextricably linked throughout history with the evolution of construction materials and the technological advancements related to harvesting and exploiting our planet's natural resources [1]. In recent years, the construction sector has been under increasing pressure to reduce its  $CO^2$  emissions and the volume of natural resources which it is responsible for consuming. Environmental concerns relating to the specification of contemporary materials which often involve energy-intensive and oil-dependent processes have become increasingly recognised [2] and with buildings, cities, and their associated infrastructure playing such a significant role in depleting our global resources, it is vital that material utilisation within buildings is specified with care in order to reduce the impact on our planet's resources and delicate ecosystems.

The purpose of this research was to explore the potential for developing a low embodied energy construction materials

obtained where possible from natural, renewable resources. The main barrier to the use of natural materials at present, particularly in developed countries, is their perceived poor mechanical properties and durability in comparison with synthetic materials such as steel, concrete, and other ceramics. This study therefore explores the mechanical properties of an innovative, natural, unfired, composite brick designed to reduce both embodied energy values and CO<sup>2</sup> emissions.

Earth construction is not only cost effective, as a result of the inclusion of low-cost raw materials, but it also uses locally sourced, benign materials. As a building system, it is considered to be highly energy efficient due to the excellent thermal properties which earthen materials exhibit and it also possesses a low embodied energy when raw clay is utilised [3]. Adobe blocks, for example, do not undergo any energyintensive firing processes since they are simply sun-dried and therefore harness solar energy directly. To put this in context, the energy required to produce an adobe block is only 5 (kWh)/cubic meter compared to about 1000 (kWh)/cubic metre for a fired brick and 400–500 (kWh)/cubic metre for concrete [4].

Earth construction is therefore becoming an increasingly valued natural building material and its durability benefits and minimization of pollution and waste characteristics particularly in industrial countries—are also being progressively recognised [5]. With regards to unfired earth construction, Heath et al. [6] have recently shown that there is structural potential for utilizing commercially manufactured unfired bricks but concluded that additional research needs to be carried out into structural behaviour and methods for minimizing moisture susceptibility. This project therefore examines an innovative, sustainable, natural earth product to assess its initial performance against an extensive series of mechanical and analytical laboratory tests [7].

Chemical soil stabilization involves changing the properties of a soil by adding chemicals or additives. This occurs either by creating a matrix, which binds or coats the grains, or by means of a physiochemical reaction between the grains and the additive materials. Cement is one of the most widely used chemical stabilizers for compressed earth blocks (CEBs) and adding it before compaction improves the characteristics of the material, particularly its resistance to water [8–13]. A proportion of at least 5-6% of cement is generally needed to obtain satisfactory results [14]. When compaction of moist soil is used in combination with cement stabilisation, it not only improves compressive strength and water resistance compared to earth construction techniques such as "adobe," but also improves dimensional stability and tolerances improving construction quality and integrity [15].

Another method of stabilizing soil is with nonhydraulic lime (quicklime or slaked lime). This technique is commonly used for road construction, although it is mainly adopted in temporary roads. The use of this type of stabilizer is not recommended, however, for the manufacture of CEBs as these bricks require a fairly low moisture content and a soil with a relatively high sand content. For stabilization purposes the amounts generally used range from 6 to 12% that is equivalent to the proportion of cement used [16]. The disadvantage of using lime alone is its negative impact on durability as described in [17].

Cementing and waterproofing cohesive soils can be achieved with small amounts of natural or synthetic polymers proportionally less than 2% by dry weight of soil. Typical polymers used in soils comprise cement-resin mixes such as polymer cements or organic resins. These range from epoxy, acrylic, polyacrylate, polyurethane, polymers derived from tomato pulp to alginate, which is an extract from seaweed [18]. There are other recently researched methods relating to the stabilization of clay bricks described in [19] and a variety of techniques and compositions currently under investigation relating to fired and unfired bricks. This research work, however, focuses on 100% natural material ingredients, namely, clay, lignin, wool, and alginate.

#### 2. Materials and Methods

The main objectives of this research were to analyse the effect on the mechanical properties of alginate added to

TABLE 1: Physical characteristics, grain size, and Atterberg limits of the three soils.

Physical characteristics	Errol	Ibstock	Raeburn
Sand content	22.50%	27.50%	35.00%
Silt content	45.00%	47.50%	40.00%
Clay content	32.00%	25.00%	25.00%
Classification I.S.S.S.	Silty clay loam	Silt loam	Loam
Liquid limit	34.8%	25.9%	25.9%
Plastic limit	19.1%	16.4%	16.8%
Plasticity index	15.7%	9.5%	9.1%



FIGURE 1: Photograph of the three soil types used.

hand-moulded bricks stabilized with natural fibre and to determine an optimal ratio for wool and alginate within three different soil types. The samples and methods that were selected are described in this section. X-ray diffraction (XRD), energy dispersive X-ray fluorescence (EDXRF), ultrasonic pulse velocity (UPV), and compression and bending tests were all conducted to provide a wide spectrum of data for analysis.

2.1. Soil. The materials used in these experiments were three different types of clay soils, alginate, wool, and lignin. The physical properties and Atterberg limits of the three different types of alluvial soils used in this experimental investigation are described in Table 1. All the soils were supplied by Scottish brick manufacturers; Errol (from the East Coast of Scotland) and Ibstock and Raeburn from Glasgow (see Figure 1). All three soils had different colours and textures but importantly their particle-size distributions were all within the maximum limits specified for utilisation within CEBs.

The moisture content (in mass percentage) at which clays and silts pass from semisolid into plastic states and then into a liquid state is defined by the Atterberg limits, which are empirical divisions between the solid, plastic, and liquid limits of a clay. The upper and lower limits of the range of water content over which soils exhibit plastic behaviour are defined by liquid and plastic limits and the water content range between these values is termed the plasticity index [20]. The Errol soil has a much higher liquid limit compared to the other soils as can be seen in Table 1. The clay in each soil sample acts like cement in concrete, binding all the larger particles in the soil whereas the silt and sand particles behave as filters in the soil matrix in a similar manner to aggregates.

Errol soil is described as a silty clay loam and contains a significantly higher proportion of clay compared to either an Ibstock or Raeburn soil. The Ibstock soil is classified as a silt loam and the Raeburn soil is classified as a loam [20]. With regards to their plasticity indexes, it is interesting to note the quite remarkable variation (see Table 1). All soils were additionally analysed and characterized by utilizing Xray diffraction (XRD) and X-ray fluorescence (EDXRF) tests.

2.2. Alginate. Seaweed is abundant within the coastal waters of countries across the globe and during the last two centuries has been used for a wide variety of products from food and medical products to soda ash production for soap and glass production. In terms of its chemical composition, alginic acid, also called algin or alginate, is a polysaccharide or carbohydrate molecule and it is obtained by extracting alginate salts from the cell walls of brown seaweeds. These alginate salts make up between 20 and 60% of the dry matter of the algae and take the form of different compounds including sodium alginate, calcium alginate, and magnesium alginate. The physical and chemical properties of alginates are nowadays being increasingly investigated and the polymer composition of this natural molecule is increasingly being understood to have a structural function within the cell walls and intercellular mucilage of seaweed [21]. The alginate matrix therefore contributes to the flexibility and mechanical strength of algae [22] in a similar manner to the way that cellulose and pectin components affect land-based plants [23]. Different algal species as well as geographical and environmental conditions influence alginate matrices which gives rise to the variations in properties that different alginates can exhibit.

Alginates are extremely versatile and exhibit important gelling properties as well as high water holding characteristics. Importantly in this research, they have the ability to act as a natural binding matrix within composite systems. Their natural propensity for improving viscosity and stabilizing emulsions has facilitated their widespread use today within the medical, pharmaceutical, and food industries where they are widely used as dental impression materials and gelling agents. Their colours range from white to yellowish brown and they are sold in a variety of forms including filamentous, granular, powdered, or gel forms.

Within the geotechnical engineering sector it, patents have been approved for the use of alginates within in situ stabilization of contaminated and non-contaminated soils [24] and a few previous tests such as those of Friedemann et al. [25] and Galán-Marín et al. [18, 26] have also been carried out incorporating alginate into building materials.

The initial selected proportions of the composite materials was derived from work previously carried out at the Laboratory of the Building Construction Department at the University of Seville and has been the subject of a patent [27]. The alginate used in our research was supplied by FMC Biopolymer, Girvan, Scotland (UK), under the name of seaweed extract and contained sodium alginate, sodium carbonate, and inorganic salt. In these experiments, we used an alginate paste (gluey, brown liquid), which is a product of the first stage of alginate extraction from seaweed.

2.3. Fibre. Traditionally, natural fibres have been used as soil reinforcement where available, to improve certain engineering properties of the soil. Vegetal fibres, derived from plants such as coir, jute, sisal, bamboo, wood, palm leaf, coconut leaf truck, cotton, hemp, and grass, have been tested as reinforcing materials not only for soils, but also within various polymer matrix composites [28] for utilisation within various industries.

Vegetal fibres such as coir can come in different varieties and the individual fibre cells are narrow and hollow, with thick walls made of cellulose. Coir is a relatively waterproof fibre and is one of the few natural fibers resistant to damage by salt water. Jute, in contrast, is a long, soft, shiny vegetable fibre similar to industrial hemp and flax (linen) and can be spun into coarse, strong threads and when woven is called hessian or burlap [29]. Another natural fibre that has recently been utilised in CEB research has been produced from cassava peels [17] and sugarcane bagasse ash [30] so there are a wide variety of vegetal fibres currently being examined in clay composites with regards to strength and flexural properties. Organic products containing cellulose fibres do however have several drawbacks such as an incompatibility with hydrophobic polymer matrices [31] and a propensity to show little resistance to prolonged moisture. For this reason this project has examined the behaviour of animal fibres which to date have tended to be overlooked as a constituent in unfired brick reinforcement.

Wool fibre is composed of a protein known as keratin. Generally, wool fibres measure 40-127 mm in length and 14- $40 \,\mu$  in width. Their cross-sectional shape is oval in form and the fibre grows in the form of a wave with a certain amount of twist. Its mechanical properties include a tensile strength 120-174 MPa, an elasticity component of 25-35% elongation at break and Young's modulus of 2.3-3.4 MPa [32]. Alternative figures listed in CES Edupak (2012) [29] give a tensile strength of between 40 Mpa and 200 Mpa and Young's modulus values between 3.9 MPa and 5.2 Mpa. Different species of sheep produce quite different types of wool with varied fibre length, diameter, and other differing physical characteristics. The molecular structure of wool fibres is interesting in that they comprise two different types of cell. Internal cells are referred to as the cortex and then outside these cells are external cuticle cells (or scales) that form a sheath around the fibre, overlapping like roof tiles. This structure gives wool its uniqueness compared to the variety of other fibres being used within natural composites today.

For the tests carried out in this project, wool fibre was added as the natural reinforcement within earth blocks, totally untreated and taken straight from the animal fleece so that no artificial additives were introduced. In addition, the wool was hand-cut, by trimming the top 10 mm strand of fibre, as longer fibres would have been too long to create a homogenous mix. All the specimens for this study were prepared and manufactured with the addition randomly oriented of a small amount (0.5–0.25%) of this raw, unprocessed wool according to recommendations from previous

Soil mix no.	Proportions	Soil	Alginate	Lignum	Wool	Water
1	Unstabilized soil	80.0%	—	0.5%	—	19.5%
2	Soil + alginate	79.5%	19.75%	0.5%	_	0.25%
3	Soil + 0.25% wool	79.5%	_	0.5%	0.25%	19.75%
4	Soil + alginate + 0.50% wool	79.0%	19.5%	0.5%	0.50%	0.50%
5	Soil + alginate + 0.25% wool	79.5%	19.5%	0.5%	0.25%	0.25%

TABLE 2: Mixes used (by weight).

TABLE 3: Mass percentages of each mineral and clay proportions.

Soil	Calcite	Quartz	Phyllosilicates	Feldspars	Illite	Kaolinite	Chlorite
Errol	<5	41	52	Traces	50	38	12
Ibstock	_	39	59	Traces	36	64	Traces
Raeburn	—	34	62	<5	27	69	4
Raeburn	_	34	62	<5	27	69	4

research which looked at the impact of various proportions [18].

2.4. Lignin. Lignin is a treacle-like resin extracted from wood during the production of cellulose. Lignosulfonate and lignin products are therefore derived from a natural raw material. In all three mixes 0.5% of lignin sulfonate (under name of Additive A, Traffaid 45 by Borregaard LignoTech) was added to improve the workability of the soil mixture because it greatly facilitates the mixing of clay with low proportions of water. This additive is commonly used in the manufacturing processes for unfired bricks.

#### 3. Sample Manufacturing Process

Material preparation was carried out manually in the laboratory of the University of Seville. Machine mixing was carried out using hand compaction and no extra compression was added. The mixtures adopted a proportion of close to 80:20 soil: (water + stabilizer) ratio after making necessary adjustments to previously carried out corrective dosages [26]. It was decided to choose a water/soil ratio of 19.5/80% (adding 0.5% of lignum) to get a normal consistency and low total shrinkage for mixes without the addition of alginate. Ratios of 0.25-0.5% of wool were added to mixes, where alginate was added to the mix and 0.25% only where no alginate was present (see Table 2). Soil Mixes numbered 1, 2, and 3 were used as contrast dosages to compare the effect of either the fibre or the polymer alone. Soil Mixes numbered 4 and 5 were manufactured to detect the appropriate quantity of fibre reinforcement. Reinforcing wool fibres were cut to the required length, soaked in water for 24 hours, to improve mixing, and then added randomly, but in a homogeneous way, to the moist soil using a 5 litre mixer until a completely homogeneous composite was achieved. All the brick samples used in this study were prismatic specimens (160  $\times$  40  $\times$ 40 mm) in accordance with the European standards format for the mechanical testing of mortar tests for masonry [33].

In this research study three different soils were tested and five different mix combinations. For each batch, seven specimens were tested. All specimens were first placed in an oven at 50 °C to dry for 24 hours and subsequently dried at room temperature for 48 hours before unmolding. Specimens were cured in the uncontrolled laboratory environment at  $20-25^{\circ}$ C and about 65% RH. A different consistency and workability was observed during the manufacturing process, for the Errol mixes. This was due to the higher percentages of illite within the Errol soil compared to Raeburn or Ibstock, which allowed more water to be absorbed within the crystal matrix.

3.1. X-Ray Diffraction (XRD). In order to identify and quantify the presence of the different mineral components within the clays and the phyllosilicates in each soil, the researchers followed a standard protocol, set out in a standard preparation protocol procedure, entitled PNT07LRX0044 [34]. The experiments were carried out in the laboratory within the University of Seville and the protocol determined the percentage composition of the small illite, kaolinite, and chlorite grains within each sample, using the oriented aggregates method which led to the proportions described in Table 3.

The oriented aggregates study included dissolving the samples in ethylene glycol and then subjecting them to a heat treatment between 350 degrees and 550 degrees. Figures 2, 3, and 4 show the phyllosilicate and quartz fractions over the majority of each soil type and the percentage and proportion of each mineral is shown in Table 2. Testing and analysis was performed in this way relating to all the different groups of clays prevailing within each soil.

*3.2.* X-Ray Fluorescence Spectroscopy (XRF). XRF is mostly a quantitative technique—the peak-height for any element being directly related to the concentration of that element within the sampling volume. However, extreme care must be taken because two or more elements can interact with each other, resulting in contamination and thus skewing results. XRF tests showed (see Table 4) the chemical composition of three soils (samples dried at 110°C).

3.3. Energy Dispersive X-Ray Fluorescence (EDXRF). Energy dispersive X-ray fluorescence is one of two general types of X-ray fluorescence techniques used for elemental analysis



FIGURE 2: XRD patterns of the Errol soil oriented aggregates.



FIGURE 3: XRD patterns of the Ibstock soil oriented aggregates.

applications. These tests show the chemical composition of the three soils (samples dried at 110°C), their main elements, and traces (see Table 5).

3.4. Ultrasonic Pulse Velocity Testing (UPV). Application of ultrasonic methods for the testing of materials including polymer composites has a long-lasting tradition. In this context, ultrasound's physical nature as a mechanical wave is used and knowledge of sound wave propagation characteristics in a tested medium allows for a theoretical analysis of a phenomenon.

On the basis of wave parameters on the boundary of an area, conclusions can be drawn concerning geometric



FIGURE 4: XRD patterns of the Raeburn soil oriented aggregates.

TABLE 4: Chemical composition of three s	oils.
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		Errol	Ibstock	Raeburn
SiO <sub>2</sub>	%	56,53	56,93	51,72
$Al_2O_3$	%	17,02	19,57	20,19
Fe <sub>2</sub> O <sub>3</sub>	%	7,09	4,81	6,51
MnO	%	0,10	0,06	0,10
MgO	%	2,71	1,21	1,50
CaO	%	2,07	0,60	0,74
Na <sub>2</sub> O	%	1,53	0,23	0,28
K <sub>2</sub> O	%	3,18	2,31	2,22
TiO <sub>2</sub>	%	0,95	1,02	0,95
$P_2O_5$	%	0,15	0,12	0,18
SO <sub>3</sub>	%	0,03	0,02	0,03
PC	%	6,02	10,59	13,34
TOTAL	%	97,39	97,47	97,77

properties and the distribution of physical properties within a medium—in this case unfired natural bricks.

Ultrasonic tests were carried out with the ultrasonic model brand BPV Krautkramer. This equipment gives the delay time from when a transmitted wave leaves the probe until the wave is received back by the probe. It is measured by a liquid crystal five digit display which measures the reading time in microseconds. The accuracy is  $\pm 0.1 \,\mu$ s. Measurements were recorded by cylindrical transducers and the emission frequency of the probes was 50 KHz. A single ultrasonic head was used and the time of a sound wave transition through tested samples ( $\tau$ ), was expressed in microseconds. The sound wave velocity (V) through a sample was then calculated using the following formula:

$$V = \frac{h}{\tau},\tag{1}$$

	Cl	Sc	V	Cr	Со	Ni	Cu	Zn	Ga	Ge	As	Se	Br
	ppm	ppm	ppm	ppm	ppm	ppm	ppm						
Errol	32	15	106	111	16	53	38	86	22	N.D.	14	N.D.	N.D.
Ibstock	35	12	83	102	15	51	28	61	23	N.D.	4	0	N.D.
Raeburn	34	12	83	101	17	57	29	68	24	N.D.	6	0	N.D.
	Rb	Sr	Y	Zr	Nb	Мо	Ag	Cd	Sn	Sb	Te	Ι	Cs
	ppm	ppm	ppm	ppm	ppm	ppm	ppm						
Errol	106	152	24	178	15	1	N.D.	N.D.	4	2	N.D.	N.D.	3
Ibstock	111	184	28	188	17	1	N.D.	N.D.	4	3	N.D.	N.D.	5
Raeburn	102	217	28	167	15	1	N.D.	N.D.	4	4	N.D.	N.D.	5
	Ba	La	Ce	Nd	Sm	Yb	Hf	Та	Tl	Pb	Bi	Th	U
	ppm	ppm	ppm	ppm	ppm	ppm	ppm						
Errol	582	40	72	33	3	1	4	N.D.	0	22	N.D.	11	1
Ibstock	400	45	78	35	6	3	4	N.D.	0	23	0	13	2
Raeburn	402	41	81	37	7	2	4	0	N.D.	24	N.D.	14	2

TABLE 5: Chemical composition of three soils (main elements and traces).



FIGURE 5: Ultrasonic tests.

where *h* was the sample thickness. All the UPV tests were carried out on the prism specimens as per the guidelines of UNE-EN 583-1/A1 [32]. Test pieces with dimensions  $160 \text{ mm} \times 40 \text{ mm} \times 40 \text{ mm}$  were tested perpendicularly to the  $40 \text{ mm} \times 40 \text{ mm}$  plane as shown in Figure 5 and the results are shown in Figure 6. A comparison between a series of mechanical and UPV test results for all soils and mixes is shown in Table 6.

#### 4. Mechanical Test Results

4.1. Compression Tests. After breaking samples roughly in half, three-point bending strength tests were used to determine compressive strength and a total of 210 compressive strength tests were carried out (see Table 6 and Figure 7).

4.2. Flexural Tests. Bending strengths were determined by carrying out a three-point bending test on the specimens, in agreement with the specifications of UNE-EN 1015-11:2000 European standards. This standard is for the determination of bending strength of mortars used for rough castings and mortar linings, but it was decided to adopt this standard in the absence of other specific regulations. Several papers including



FIGURE 6: Graphical comparison of the UPV results on the five different mixes of the three types of soils.

the study carried out by Raut et al. [19] have illustrated the wide range of testing and curing regimes currently being carried out in different laboratories as well as the range of sizes of earth brick samples. Furthemore and Heath et al. [6] have additionally discussed the current limitations in Eurocode 6: Design of masonry structures (BS EN 1996-2:2006) and the fact that it does not currently have a section referring to earth masonry. Within this context, it was felt that UNE-EN1015-11:2000 was an appropriate standard to adopt.

All the flexural tests were conducted at room temperature  $(20^{\circ}C)$  on a Codein S.L., MCO-30/139 machine (maximum

Mix code	01	02	03	04	05	
Mix code	Unstabilized soil	d soil Soil + alginate Soil + 0.25% wool		Soil + alginate + 0.50% wool	Soil + alginate + 0.25% wool	
Errol soil						
Compressive strength (MPa)	2,23	3,77	3,05	4,37	4,44	
Flexural strength (MPa)	1,12	1,06	1,1	1,08	1,45	
Ultrasonic testing (m/s)	1182	1637	1416	1798	1818	
Ibstock soil						
Compressive strength (MPa)	2,06	2,49	1,89	3,43	3,59	
Flexural strength (MPa)	0,97	0,98	0,96	1,28	1,61	
Ultrasonic testing (m/s)	1298	1413	1222	1720	1769	
Raeburn soil						
Compressive strength (MPa)	2,44	2,24	1,88	2,69	3,75	
Flexural strength (MPa)	1,12	1,1	0,93	1,11	1,24	
Ultrasonic testing (m/s)	1240	1153	1075	1280	1604	



Flexural strength (Mpa) 1.0 0.8 0.6 0.4 0.2 0.0 1 2 3 4 Soil mix code 🖾 Errol soil 🔝 Ibstock soil

1.6

1.4

1.2

FIGURE 7: Graphical comparison of the compressive strength results on the five different mixes of the three types of soils.

FIGURE 8: Graphical comparison of the flexural strength results on the five different mixes of the three types of soils.

5

Flexural strength (Mpa)

load 10 kN) in a three-point bending configuration. The support length was adapted to the size of sample, and at least seven specimens were tested for each mix under study. A total of 105 tensile tests were therefore conducted both with the fibrous and non-fibrous samples and the mechanical properties determined from these tests included the flexural modulus, the ultimate stress, and the ultimate strain (see Table 6 and Figure 8).

#### 5. Results and Discussion

📕 Raeburn soil

It is generally accepted that incorporation of fibres increases flexural strength. Our tests have not shown the expected improved flexural strength, indeed sometimes just the opposite, as is shown in Figure 8 for the Ibstock soil. In fact the addition of just fibre to soils, without the presence of alginate (as indicated in the comparison between mixes 03 and 01), does not increase flexural strength. Additionally, compressive

TABLE 6: Mechanical and UPV tests results of three soils.

strength in samples with no alginate and only fibres is increased only in the soil with the high plasticity index, namely, Errol. Indeed, both the flexural and compressive strengths of the Raeburn and Ibstock soils decreased in the mixture only containing wool fibres without the polymer.

In contrast, adding wool into the soil mixed with alginate always increased flexural resistance, especially if a proportion of 0.25% of wool was added. The incremental strength improvement was particularly significant (65%), for the Ibstock soil specimens, where flexural resistance changed from 0.97 MPa (plain soil specimens) to 1.60 MPa (0.25% wool + soil + alginate mixes).

As the XRD analytical results show, higher percentages of illite can be found in the Errol soil (compared to Raeburn or Ibstock). This phyllosilicate appears to allow more water to be absorbed within the crystal matrix giving rise to a drier mix and this finding is consistent with the plasticity indexes obtained for the different soils showed in Table 1. The consequences of this drier and therefore stiffer consistency can be observed in the mechanical results.

UPV measurements (see Figure 6) demonstrated that Errol specimens, in most of the mixes tested, provided much higher compactness and with regards to mechanical tests, Errol specimens reached higher resistance values in the compression tests compared with the other soils. Flexural tests, however were less conclusive, with Errol providing higher flexural strengths in mix 3, Raeburn in mix 2, and Ibstock in mixes 4 and 5. Flexural strength values were equal in the Raeburn and Errol soils in mix 1 and the UPV results showed a pulse velocity increase for mixes 4 and 5 compared to mixtures 1, 2, and 3. Specimens of any type of soil tested for mix 5 (the mix with the lower quantities of wool reinforcement) showed higher UPV values than mix 4. This was particularly the case with the Raeburn soil which had the lowest plasticity index. This compactness decrease could be caused by the fact that the shrinkage values for the fibre were much higher than the soil shrinkage.

Lower compressive and flexural resistance values were obtained when larger quantities of fibre were used in mix 4. This could be explained as follows: the development of strength properties in the fibre/soil mixes mostly depends on the formation of fibre-matrix bonds as has been shown in previous studies [35]. Bonding is affected by fibre dimensions, surface textures, and the number of fibres present in a given volume of material. Increasing wool fibre quantities gives rise to fibre agglomeration and folding of fibres (balling) and this can result in a decrease in the bond strength within the specimens, which in turn leads to lower compressive strength values. In addition to the XRD, XRF, and EDXRF results, the ultrasonic results were incorporated into this study's testing regime in order to indicate the prevalence of voids and compare the relative material densities. The results of these tests within each sample confirm that mix 5 had the best overall engineering properties and it is suggested that this was due to the quality of bonding within the composite matrix and the overall homogeneity of the mixture.

The highest compressive strength, in all three soil types, was obtained with the composite specimens including both alginate and wool reinforcement and better results were obtained with the reduced quantity of wool, that is, the 0.25% mix 04. By adding wool to the mix, the Ibstock soil specimens stabilized with alginate improved their compressive strengths by 74% whilst the Raeburn soil specimens improved their compressive strengths by 54%.

Various research papers have shown that hygrometric shrinkage and its associated cracking of earth-based materials can be greatly reduced by introducing fibres into the mixture and this study confirmed these findings by demonstrating that shrinkage due to the drying process was significantly reduced with the inclusion of natural fibres into the soil mix. As would be expected, specimens of plain soil had a very quick (and almost without warning), brittle failure mode. In contrast, fibre-reinforced mixes deformed, after the ultimate load was reached and fine cracks could be seen on the surface giving warning before failure.

#### 6. Conclusions

Soil characterization through XRD, XRF, and EDXRF tests has proved to be very important in order to understand the different mechanical behaviour of the different stabilised soils (with the same fibre and stabilizer content) and therefore the effect of the stabilization itself.

Mixes 2, 4, and 5 (those that included the alginate polymer as a stabilizer) showed better results in UPV tests in every type of soils, especially though the Errol soil and these results are consistent with the compression test results. As would be expected, flexural resistance values generally increased in mixes 4 and 5, where alginate and fibre were used simultaneously.

The use of UPV, in this study, has added an interesting additional data set with results which closely align with the mechanical compressive strength results. Errol soil, due to its higher content of illite, contains a crystalline structure that facilitates a higher level of water absorption compared to the Raeburn and Ibstock kaolinite crystalline structures. Improved results were therefore obtained with the Errol soil due to the higher plasticity index related to a higher proportion of illite within the clay fraction.

The addition of short wool fibres (10 mm long) randomly oriented to the mixes leads to a decrease in bulk density which correspondingly decreases the compressive strength of the specimens. Therefore UPV measurements were useful in determining the resultant final porosity of the dried mix, after the shrinkage process.

Fibre water adsorption and soil-fibre surface friction, due to the drying shrinkage characteristics of a fibre, depend on the available water and this in turn depends on the characteristics of the soil plasticity for different types of clay. The higher plasticity index obtained for the Errol soil seems to be responsible for its different behaviour observed in the various tests compared with the Ibstock and Raeburn soils, in mixtures with similar water proportions.

As a result of the range of data presented within this paper, it has therefore been shown that it is possible to prepare 100% green composites from natural fibres and natural polymers with mechanical performance results within ranges compatible with producing unfired bricks. Further research is currently under development to investigate methods for improving composite bonding and interaction and ultimately durability.

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