A COMBINATION OF SEM AND EDX STUDIES ON A CLAY-BASED NATURAL COMPOSITE WITH ANIMAL FIBRE AND ITS MECHANICAL IMPLICATIONS

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Keywords: Natural Fibres, Stabilized Soil, SEM, EDX

Abstract

A variety of natural fibres are nowadays being utilized as soil reinforcement. Test results demonstrate the positive effects of adding natural fibres to soils, in that they decrease shrinkage, reduce curing time and enhance compressive, flexural and shear strength if an optimum reinforcement ratio can be utilised. This paper describes a study which uses a Scanning Electron Microscope (SEM) and an Energy Diffraction Analysis of X-rays (EDX) technique on clay-based composites stabilized with natural polymer and fibres. Different dosages of fibres and several types of soils have been used in this study with the aim of determining advantageous properties for building material applications. SEM and EDX test results reveal the degree of bonding between the particles of soil and the natural fibers. This has enabled a better understanding of the micro-morphology of the natural fibers and their effect on the overall composite material structure. Microscopic analysis was combined with mechanical tests to establish the different strength characteristics of every soil.

1 Introduction

One of the most important characteristics of swelling soils, such as clays, is their susceptibility to volume change due to swelling and shrinkage. These swell–shrink movements and total and differential volume changes can result in considerable structural and non-structural damage to overlying structures such as low-rise buildings, highways, roads and buried pipelines and therefore it is important to find mechanisms that will improve the stability of such soils. Natural fibres [1-3] are potentially important by-products of mainly plants or animals and can be used as reinforcement in eco-friendly composites suitable for the building industry. These fibres have been tested as reinforcement in cement [4] and polymer matrix composites [5-7] and comprise plant fibres such as jute, coir, sisal, bamboo, wood, palm leaf, coconut leaf, coir dust, cotton, hemp, grass, etc. These fibres have also, to various degrees, been used as a reinforcing material in order to improve the engineering properties of a given soil. Current research is focusing on materials such as sisal [8], bamboo, jute, hemp, coir and few other natural, plant-derived fibres. However, animal fibres have been relatively neglected in the search for improving soil reinforcement properties. Wool fibers have a large surface area relative to their volume and surface properties play a critical role in applications such as fiber reinforcement in soil. This property is also critical in their ability to form linkages with polymers such as in treatments conduced to be shrink-resistant. This shrinkage is due to the drying effect, but there are chemical treatments to counteract this absorption of water [9]. In any case, the structure of the fibre surface is therefore crucial in order to advance wool processing and finishing technology and examine the interaction within soil structures.

In this research project, a new approach has been applied to try to understand behavior in relation to understanding the natural drying speed on the swelling behavior of natural polymer-stabilized soils. Three types of soils all exhibiting different ranges in plasticity index values and two different dosages of wool fibres were selected and have been analyzed. Specimens were dried to their initial water content to evaluate partial shrinkage and the nature of this change with different amounts of natural fiber. Microscopic structural changes of stabilized specimens were then studied with a Scanning Electron Microscope (SEM) and the results from this analysis were compared with Energy Diffraction Analysis of X-rays (EDX). In addition physical changes were compared with the plasticity index of each soil and the results of mechanical testing on each sample.

2 Materials

2.1 Soil

Three different types of soils were used for the tests. Soils were all supplied by Scottish brick manufacturers, namely Errol (from Perthshire), Ibstock (from Glasgow), and Raeburn (from Glasgow). Alluvial soil from the Tay estuary (on the East Coast of Scotland) has been used by Errol to manufacture unfired and fired bricks since 1850. Indeed, the estuarine clays used to manufacture Errol bricks were laid down after the last Ice Age. Both the Ibstock and Raeburn brick manufacturers, who are based in Glasgow, have used soils sourced in the West Coast of Scotland to manufacture fired bricks. All three soils have a different colour, texture and particle sizes. Soil samples were naturally dried and sieved before they were used in the mixes and the composition was measured by chemical precipitation as illustrated in Table 1. A higher content of Calcium and Magnesium Oxide was observed in Errol soil compared with two other soil types.

Composition (%)	Errol	Ibstock	Raeburn
SiO_2	54.70	62.83	60.32
TiO_2	0.97	0.98	0.96
Al_2O_3	19.70	18.49	18.30
Fe_2O_3	8.63	5.93	5.87
CaO	0.93	0.38	0.32
MgO	3.55	1.86	1.81
K_2O	3.90	3.41	3.47
Na_2O	1.78	0.38	0.45
P_2O_5	0.17	0.12	0.13
Cr_2O_3	0.02	0.01	0.01
Mn_3O_4	0.12	0.07	0.06
ZrO_2	0.03	0.05	0.05
ZnO	0.03	0.01	0.01
BaO	0.08	0.06	0.04
Loss at 1025 ºC	5.04	5.57	5.53

Table 1. Chemical composition of three soils (samples dried at 110ºC)

Clay, just like cement in concrete, essentially acts as a binder for all the larger particles within the soil. The silt and sand constitute the filters as they are non-cohesive soils lacking in binding forces. Soil nomenclature is based on which of the three soil types is dominant in the mix. For instance, geotechnical literature describes clayey, silty and sandy soils as well as various combinations such as a silty clay. Errol soil is classified as a silty clay loam and has a significantly higher clay content than either an Ibstock or Raeburn soil. Ibstock soil is classified as a silt loam and Raeburn soil is classified as a loam (Table 2). The plasticity of each soil type is defined by Atterberg Limits. As can be seen below, the three soils showed a remarkable variation in their index of plasticity (Table 2) with the drier Errol soil having a significantly higher plasticity index.

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%

Table 2. Physical characteristics, grain size and Atterberg Limits of the three soils

Clay soils exhibit quite large ranges in bearing capacity between approximately 75 and 300 KN/m^2 and their particle sizes are less than 0.002mm. In order to identify and quantify the presence of different compounds of clays and phyllosilicates in each soil, the researchers followed a standard protocol, set out in a Spanish Technical Regulation entitled PNT07LRX0044. The experiments were carried out in the CITIUS laboratory within the University of Seville and the protocol determined the percentage composition of the small Illite, Kaolinite and Chlorite grains using the oriented assembly method finding the proportions described in Table 3 [10].

Soil	Illite	Kaolinite	Chlorite
Errol	50	38	12
Ibstock	36	64	traces
Raeburn	27	69	4

Table 3. Study of the fraction size <2µm

2.2 Fibre

Natural fibres, acting as reinforcement within composites, offer many advantages including good strength properties, low cost, low density, high toughness, good thermal properties, biodegradability, non abrasive behaviour and widespread availability. However organic products containing cellulose fibers, have several negative characteristics, such as an incompatibility with the hydrophobic polymer matrix [11] and a tendency to show little resistance to moisture longer. Finite natural lengths and large diameters also limit their potential applications. Most studies of natural fibres concentrate on cellulose-based/vegetal fibres obtained from renewable plant resources such as wood, flax, sisal, or jute. There are very few studies detailing composites made with protein (keratin) fibres. Natural protein fibres are generally obtained from animal hairs and animal secretions. Barone and Schmidt [12] reported on the use of keratin feather fibre as short-fibre reinforcement within LDPE composites. The keratin feather fibre they used had been obtained from chicken feather waste which is generated by the USA poultry industry each year. Protein fibers generally have a greater resistance to moisture and heat than natural cellulosic fibers and other vegetal fibers. Proteins within the fibers, however, have little resistance to alkalis, but are mechanically resistant and have good elastic recovery. Another natural protein fibre containing keratin is wool, which grows outwards from the skin of sheep. Different species of sheep produce different types of wool with varied fibre length, diameter and other differing physical characteristics. Generally, fine wool fibres are 40–127mm in length and 14 - 40µ in width. They are roughly oval in cross-section and grow in a wavy type of form which gives rise to a certain amount of twist. Wool fibres are essentially composed of two types of cell: internal cells known as the cortex and external cuticle cells that form a sheath around the fibre. The cuticle cells (or scales) overlap like tiles on a roof and this characteristic makes wool unique amongst textile fibres. The complex physical structure of cuticle cells is shown in Figure 1 and Figure 2 shows separation of individual cortical cells in a fibre. The cortex component of the wool comprises approximately 90% of the fibre and consists of overlapping spindle shaped cells known as cortical cells, shown schematically in Figure 3.

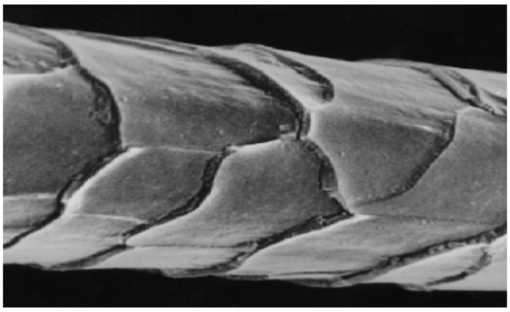


Figure 1. SEM of wool fibre (x1200)

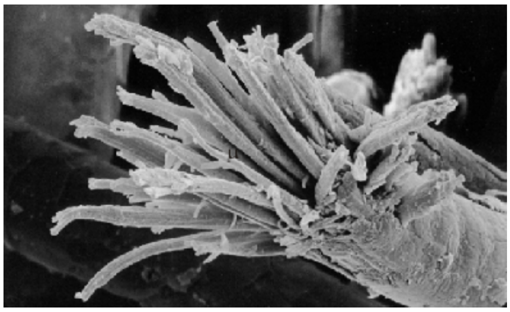


Figure 2. SEM showing fibre fibrillation (x1050)

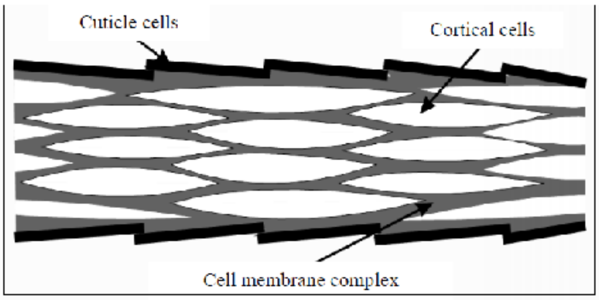


Figure 3. Schematic of a wool fibre showing cuticle and cortical cells.

Wool is a hygroscopic fiber taking up moisture in vapour form. Tiny pores in the cuticle make the fibre semi-permeable, allowing vapor to pass through to the heart of the fiber which means that wool can easily absorb up to 30% of its weight in moisture without feeling damp or clammy. There is generally a two-phase structure for wool fibers which consists of a water-absorbing matrix which contains embedded within it non-water-absorbing cylinders. One of the main objectives of using fibres as reinforcing elements within soil structures is to prevent cracking of the soil which results from shrinkage. Tensile shrinkage cracks in soil are mainly due to rapid and non-uniform drying and reinforcing fibres within the soil structure prevent cracking by adhesion or bonds with the soil particles. The main factors, which affect the adhesion between the fibres and soil are: (a) the cohesive properties of the soil; (b) the compression friction forces appearing on the surface of the reinforcing fibre due to shrinkage of the soil and (c) the shear resistance of the soil, due to the surface form and roughness of the fibres. The dimensional changes of natural fibres due to moisture and temperature variation have an influence on all three of these adhesion characteristics. This is because during the mixing and drying of the soil, the fibres absorb water and expand. This swelling of the fibres pushes away the soil (at the microscopic level) and then at the end of the drying process, the fibres lose the moisture and shrink back almost to their original dimensions leaving very fine voids around themselves. [13-14]. This implies an increased level of porosity of the material and a degree of friction loss fiber-soil.

2.3 Alginate

Alginates are used in a wide range of applications, particularly in the food, industrial and pharmaceutical industries, because of their capacity to hold water, form gels, and form and stabilize emulsions. One of the most important and useful properties of alginates is their ability to form gels by reacting with calcium salts. Alginic acid, also called algin or alginate, is an anionic polysaccharide distributed widely in the cell walls of brown algae, where it, through binding water, forms a viscous gum. In an extracted form it absorbs water quickly and is capable of absorbing 200-300 times its own weight in water. Its colour ranges from white to yellowish-brown and it is sold in filamentous, granular or powdered forms. The chemical formula of the alginic acid is $(C_6H_8O_6)_n$; the two most common alginates being sodium alginate $(C_6H_7NaO_6)_n$ and potassium alginate $(C_6H_7KO_6)$n. Alginate gels, which reproduce the characteristics of a solid when the jellification process concludes, retain its shape and resist stress and are composed of water near 100% (typically 99.0 to 99.5% water and 0.5 -1.0% alginate).Within the engineering and construction industries it has been reported and patents have been approved to use alginates for in-situ stabilization of contaminated and non-contaminated soils [15]. A few previous tests such as Friedemann et Al. [16] and Galán et Al. [17-18], have been carried out incorporating alginate into building materials.

3 Results and discussion

3.1 SEM-EDX analysis

The soils utilized in this study showed different plasticity indexes and different consistencies in the mixture due to the relative water absorption of the three soils selected for SEM-EDX analysis. Specimens from these soils were prepared in the two following dosages (Table 4).

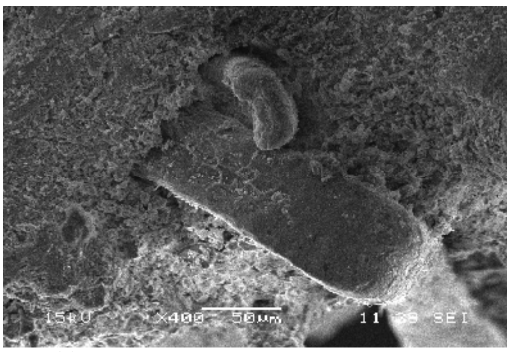
Proportion	Soil	Alginate*	Lignum**	Wool	Water
01_Soil	79.0%	19.5%	0.5%	0.50%	0.50%
02_Soil	79.5%	19.5%	0.5%	0.25%	0.25%

* Wet alginate.

** Lignum Sulfonate is a resin extracted from wood that was added as a dispersing agent (to improve the workability of the soil).

Table 4. Proportions used (by weight).

The selected specimens were left to dry slowly at laboratory temperature for about two weeks. Then, they were mechanically tested. Direct handling of the specimens was kept to a minimum at all stages to avoid contamination (Figures 4-9).



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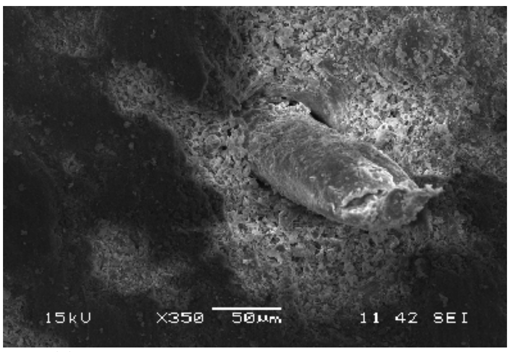

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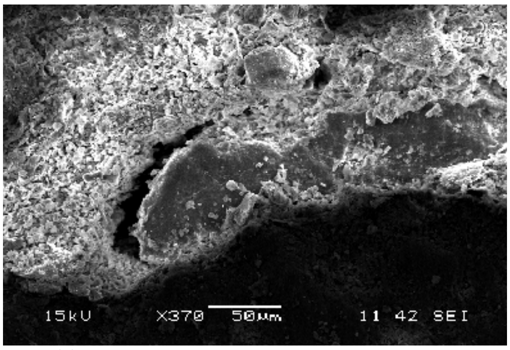

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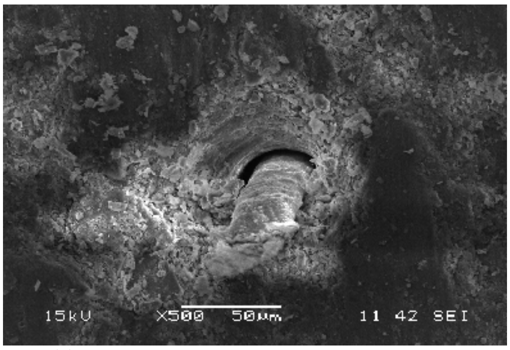

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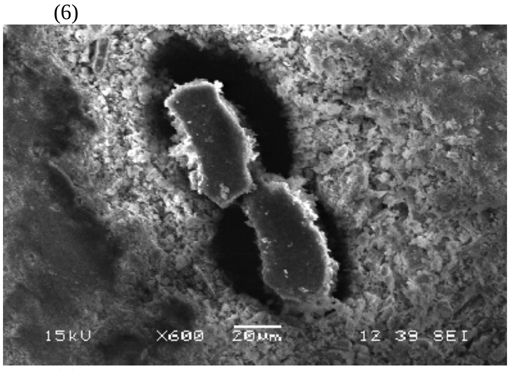

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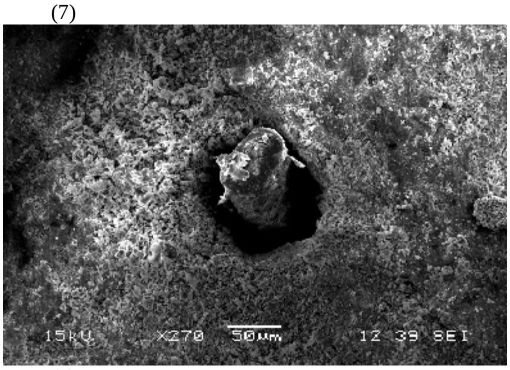

(9)

Figure 4-9. SEM pictures: (4) 01_Errol, (5) 02_Errol, (6) 01_Ibstock, (7) 02_Ibstock, (8) 01_Raeburn and (9) 02_Raeburn.

The EDX test confirmed, by a semi qualitative analysis, the chemical composition of soils and also determined the high level of alginate microscopic dispersion in the samples analyzed, (Figures 10-11), (Tables 5-6).

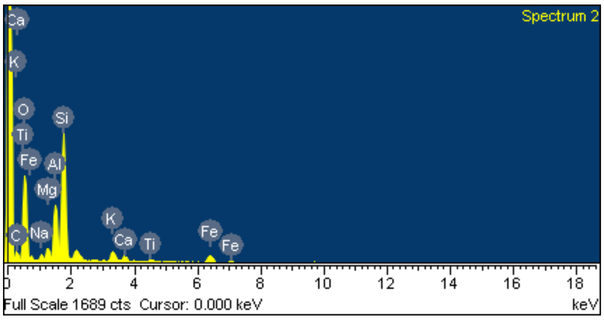

Figure 10. EDX spectra of a sample (mainly soil)

Element	Weight%	Atomic%	Compd%	Formula
C K	0.00	0.00	0.00	CO2
Na K	1.67	1.54	2.25	Na2O
Mg K	2.78	2.42	4.60	MgO
Al K	10.47	8.24	19.79	Al2O3
Si K	27.91	21.08	59.71	SiO2
K K	2.79	1.52	3.36	K2O
Ca K	1.57	0.83	2.20	CaO
Ti K	0.83	0.37	1.39	TiO2
Fe K	5.21	1.98	6.70	FeO
O	46.77	62.02		

Table 5. Chemical composition of the sample.

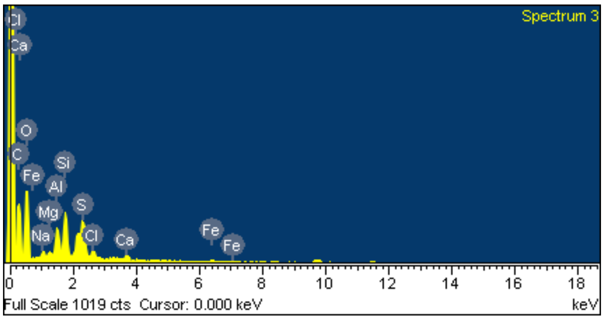

Figure 11. EDX spectra (soil and alginate particle)

Element	Weight%	Atomic%	Compd%	Formula
C K	22.20	28.74	81.33	CO2
Na K	0.77	0.52	1.04	Na2O
Mg K	0.43	0.28	0.72	MgO
Al K	1.78	1.02	3.35	Al2O3
Si K	3.14	1.74	6.72	SiO2
S K	1.91	0.92	4.76	SO3
Cl K	0.68	0.30	0.00	
Ca K	0.47	0.18	0.65	CaO
Fe K	0.58	0.16	0.74	FeO
O	68.05	66.14		

Table 6. Chemical composition of the sample.

3.2 Mechanical test

Mechanical tests showed average values for the three-point bending tests and compressive tests for all three soil types used in the production of test samples. Note that the same procedure was repeated for all three soil types tested in our laboratory. Each value represents the average of a total of 7 (flexural test) and 14 (compression test) specimens. According to the European Standards [19] the number of different mixes (proportions) tested were a minimum of seven specimens per batch (Table 7)

Mix code	**01_Soil** (0.50 % wool)	**02_Soil** (0.25 % wool)
Compressive strength (MPa)		
Errol	4.37	4.44
Ibstock	3.43	3.59
Raeburn	2.69	3.75
Flexural strength (MPa)		
Errol	1.08	1.45
Ibstock	1.28	1.60
Raeburn	1.11	1.24

Table 7. Mechanical tests of the different mixes of the three types of soils.

3.3 Influence of the Atterberg Limits and the fibre absorption

In the manufacturing process a different consistency and workability was observed, which was much drier for Errol mixes. This was due to the ability to introduce water inside the crystalline structure. The consequences of the drier consistency can be observed within the mechanical properties, showing Errol specimens, of any proportion, providing a much higher resistance in compression test than other soils. The water available for the fibre absorption was in direct relation to the plasticity index of each soil due to the soil absorption. That explains why SEM pictures show higher shrinkage around the fibers in the samples made with Ibstock and Raeburn soils.

3 Conclusions

This paper reviews the influence of the water absorption of wool fibres randomly distributed in different types of soil. On the basis of mechanical testing, microscopic analysis and normal geotechnical experimental measurements presented in this paper, it is clear that only compressive strength of fibre reinforced soil increases for soils of higher plasticity index. This is due not only to the fibre content itself but also because of the water absorption of the fibers themselves.

Fibre water adsorption and soil-fibre surface friction, due to the drying shrinkage of a fibre, depends on the available water and this in turn depends on the characteristics of the soil plasticity. A greater amount of freely available water in the mixture reduces strength, especially with regard to compression strength, but not so much for flexural strength. This reduced compressive strength is basically due to the higher porosity of the mix and not due to the interaction between the soil fibre. For all three types of soils tested the effectiveness of the fibre reinforcement is shown not only for shrinkage reduction but also because of better flexural results. These benefits are independent of the plasticity index of the soil itself. SEM images of Raeburn and Ibstock specimens show a bigger space around the fiber due to shrinkage. Wool fibres show a better behavior than other vegetal fibres due not only to the texture of the fibre surface but also to the slow process of absorption and desorption of vapour water.

The changes in this soil-fibre surface interface are clearly visible in the SEM pictures. A higher fibre proportion makes it more difficult to compact the samples and that has a clear influence in the mechanical results both for flexural and compressive strength. The SEM-EDX tests were useful in determining the degree of alginate micro-dispersion. In most of the samples analyzed, it was really difficult to locate alginate particles within the soil matrix which show that the alginate had achieved good levels of integration into the soil mass.

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