Recovery of Critical Raw Materials from Abandoned Mine Wastes: Some Potential Case Studies in Northwest Italy

PHYSICAL AND MECHANICAL CHARACTERIZATION OF NATURAL FIBRES AND FABRICS AS REINFORCEMENT FOR COMPOSITE SYSTEMS

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ABSTRACT

In recent years, the increasing interest in environmentally friendly materials has brought the attention of material technologists and civil engineers to natural fibres and their potential use in composite materials. Although several research activities have shown the good mechanical properties of these fibres, their use is still limited due to both the absence of standards and guidelines and some open durability issues highlighted in the literature.
The present work is a contribution to identifying the main physical and mechanical properties of flax fibres and fabrics intended as internal reinforcement in composite materials with mineral matrix. Due to the lack of standardization on vegetal fibres as constituent of building materials, the experimental research moves from characterising the geometry of bundles. The diameter of single yarn and twisted bundles has been measured by means of a large number of measurements obtained by a microscope: specifically, six values have been recorded for each one of thirty specimens. A comparison with a most accurate SEM analysis has been performed as well. Tensile tests on single yarn, double yarn twisted bundle and textile strip specimens have been carried out. Tensile tests on flax textile have been performed after a specific curing period in controlled environment in order to evaluate the durability of the textile and its sensitivity to alkali agents. The results of the experimental activity showed that the diameter values assessed by means of a microscope are affected by the irregularities of the flax bundles, due to their twisted arrangement. Future developments will target the mechanical behaviour of Textile Reinforcing Mortar (TRM) composite systems based on a bidirectional grid fabric made of the aforementioned flax fibres: assessing the potential of these sustainable and reversible composites systems as a technical solution for in seismic strengthening of existing masonry members and structures is the final goal of this research.

KEYWORDS

Strengthening and repair, Experimental study, Eco-composite & bio-sourced composite materials, FRCM and cement composite materials, Flax fibres.

INTRODUCTION

Nowadays, the increasing ecological awareness has resulted in a rising interest for reducing environmental impact due by the construction sector. In fact, the use of more sustainable materials is deserving more and more attention, among the newly available eco-friendly technological solutions. As the production processes of vegetal fibres require low energy, both for processing and transportation, they certainly represent a “green” material. In recent years, several studies have been devoted to the investigation of these materials, describing them as a valid alternative to the most traditional industrial fibres, such as carbon, glass or plastic ones, used in composite applications in construction industry (Faruk et al. 2012).
Natural fibres, such as flax, jute, hemp, sisal, coir, have shown a mechanical performance, in terms of tensile strength and strain, not so different from those exhibited by the most common synthetic fibres (Torgal & Jalali 2011).
Although natural fibres represent a promising reinforcing material in composite systems, their use is still limited by several drawbacks. Firstly, they present an important variability of the mechanical properties due to the non-uniformity of their structure. Many factors, such as humidity content, structure, irregularities, the morphology of the plant, may affect their mechanical, physical and geometrical properties (Codispoti et al. 2015). Secondly, vegetal fibres present durability issues.
Composite materials produced with Ordinary Portland Cement matrix register an important loss of strength due to the fibre mineralization and alkali attack related to the variation of humidity (Toledo Filho et al. 2000). Moreover, the adhesion between natural fibre and cementitious matrices is still an open issue (Ferreira et al, 2016).
That said, further investigations are still needed with the aim to assess the potential of vegetal fibres to be used as internal reinforcement in cementitious composites.

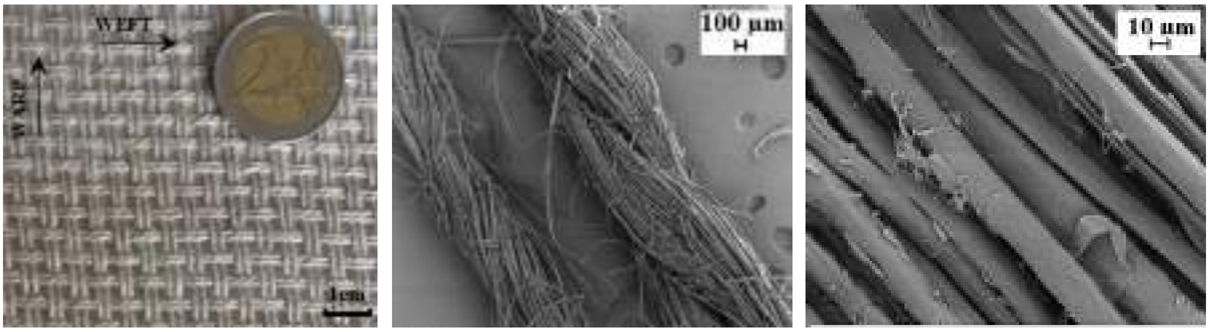
This study aims to analyse both the physical and mechanical properties of flax textile used as reinforcement in cement-based composite systems. Different measurement techniques have been implemented and compared each other in order to assess geometric parameters of the textile. In order to mechanically characterize the textile, tensile tests have been performed on differently sized flax coupons.

Furthermore, with a view to future applications of the material as reinforcement in cement-based composite systems, the response of flax single bundle coupons subjected to an ageing protocol has been observed, giving information about the durability of the material.

MATERIALS AND METHODS

Materials

The material subject of study is a bi-directional woven flax fabric with plain weave in which the warp and the weft directions are arranged forming a simple cross pattern (Figure 23a).
The fabric is characterized by 4.3 threads per centimetre, each one of them consists of a double-twisted flax yarn (Figure 23b), and each single yarn in turn, at the micro-scale, is composed by vegetal filaments (Figure 23c).



(a) bi-directional arrangement (b) single yarn and double-twisted yarns (c) filaments

Figure 23:Flax fabric arrangement

The relevant physical properties of the flax textile under consideration are the *apparent thread diameter*, the *filament diameter,* the *density* and the *effective area* of the bundles. The apparent diameter of the flax bundles has been evaluated by means of an optical microscope: 15 double-twisted yarn samples 15cm long have been analysed and 6 values of the diameter have been recorded for a total of 90 measurements.
A Scanning Electron Microscope (SEM) analysis has been performed in order to scrutinise the geometric and physical properties at a more detailed scale. The investigation of the SEM image in Figure 23b, representing a double-twisted yarn with a magnification of 50x, shows an apparent diameter of the bundle consistent with the assessment performed by means of the optical microscope.
Four SEM images detecting 4 different samples, with a magnification of 1000, have been investigated providing a geometric identification of the filaments constituting the flax threads by means of 28 measurements (Figure 23c). As shown by the SEM images, due to the presence of voids among the flax filaments, the apparent diameter cannot give an effective value of the cross-section to be used for mechanical investigations. The effective cross-section area of the flax bundle has been obtained by means of indirect measurements. Specifically, the density of five 15 cm long double-twisted flax yarns samples has been assessed by using a hydrostatic balance. As known, the evaluation of the density in water is strongly affected by the presence of voids within the sample.
Therefore, only saturated specimens can give reliable values. In order to make sure having saturated samples, the absorption rate of the fibres has been studied. Five flax threads, having a length of 15 cm, have been weighed and then immersed in deionized water. The weighing of the samples has been repeated at an interval of 1 or 2 days up to the achievement of a negligible weight gain. The water absorption curve, whose values have been obtained by dividing the water weight gain for the initial dry weight, is reported in Figure 24 and shows a water absorption rate equal to 222%, with a coefficient of variation of 19%, achieved in 96 hours.

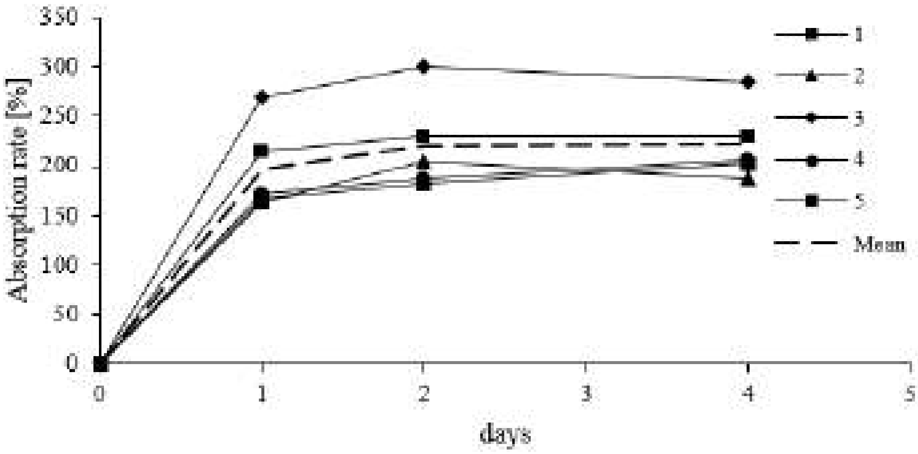


Figure 24:Flax thread water absorption rate

The specific gravity of the 5 flax saturated samples subjected to the water absorption test has been assessed. An indirect estimate approximation of the effective cross-section area has been obtained by dividing the weight of the samples for their density and length. The mean value and its respective coefficient of variation of all the physical properties assessed in this study are reported in Table 11.

Table 11:Physical and mechanical properties of flax fabric

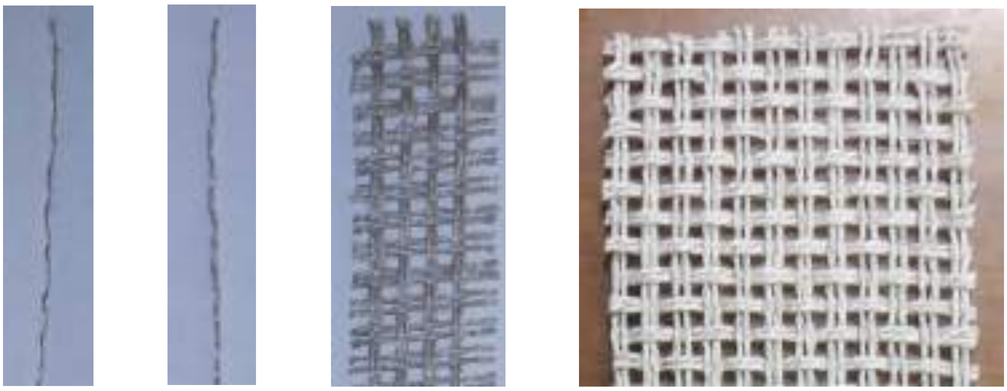
	Mean	Co.V. (%)
filament diameter (μm)	16.78	29.64
density (g/cm^3)	1.19	3.29
linear density (Tex)	302	15.27
n° bundles*/cm	4.3	-
Apparent Diameter* (mm)	0.88	5.27
Effective Area* (mm^2)	0.25	16.62
Young's Modulus* (Gpa)	9.36	10.67
Strain to failure* (%)	3.85	12.94
Tensile strength* (Mpa)	353.72	11.53

**Values refer to flax double twisted yarn samples*

Methods

Tensile tests have been performed on different sized flax samples, in order to provide a mechanical characterization of the material and to monitor the variability of the properties of the fabric. The following series of specimens have been considered:

- *Flax-1Y*: it consists of 15 cm long single yarn samples obtained by separating each other the two yarns constituting the main thread of the fabric (Figure 23a);
- *Flax-2Y*: it represents the main thread of the textile, and it is characterised by double-twisted yarns. Specimens having a length of 15 cm have been obtained from the flax roll both in warp and weft directions (Figure 25b);
- *Flax fabric-2cm*: it consists of flax fabric strips 2 cm wide and 15 cm long, characterised by 8 “2Y-threads” (Figure 25c);
- *Flax fabric-6cm*: it consists of flax strips 6 cm wide and 30 cm long, characterised by 24 “2Y-threads” (Figure 25d).



(a)Flax-1Y (b)Flax-2Y (c)Flax fabric-2cm (d)Flax fabric-6cm

Figure 25:Flax tensile test specimens

The specimens having a length of 15 cm have been tested by using a SANS Universal Testing Machine having a capacity of 10 kN. Tensile tests have been performed by using a 1 kN load cell, with a displacement rate of 4mm/min, adopting a gauge length of 100 mm (Figure 26a).
The mechanical characterization of the "Flax fabric-6cm" specimens has been carried out according to the ISO 13934-1. Tensile tests have been carried out by means of a Zwick Roell Schenck Hydropuls S56, with a maximum capacity of 630 kN. The tests have been executed in displacement control at a rate of 4mm/min and a gauge length of 20 cm. Steel plates have been glued to the specimens ends by means of epoxy resin in order to avoid slipping during the tests (Figure 26b).

(a) Flax 2Y (b) Flax fabric-6cm

Figure 26:Tensile test on different sized flax samples

The ageing protocol adopted in this study to analyse the durability of the flax textile has been chosen in view of the application of these fibres as reinforcement in cement-based composite systems. According to the AC434-1011-R1, the mechanical properties of the so-called Textile Reinforced Mortar (TRM) composite systems are investigated after conditioning periods in specific water environments (Nobili & Signorini 2017). Depending on the conditioning environment the following series of specimens have been considered:

- *Control*: it is represented by the "Flax-2Y" series in warp direction;
- *W1000*: Flax-2Y specimens have been immersed in deionised water for a period of 1000 h (~42 days);
- *S1000*: Flax-2Y specimens have been immersed for a period of 1000 h (~42 days) in a saline environment consisting of 3.5% weight sodium chloride (NaCl) aqueous solution, that should reproduce the world's ocean seawater average salinity;
- *Alk1000*: Flax-Y2 specimens have been immersed for a period of 1000 h (~42 days) in an alkaline environment with 9.5 PH level, reproduced by adding sodium hydroxide (NaOH) to the deionised water.

RESULTS AND DISCUSSIONS

Table 12 reports the main mechanical properties deriving from the tensile tests performed on different flax samples.

Table 12:Mechanical properties of different sized flax samples

	Directior	Number of tests	P_u		f_t		E		ε_u	
			Mean (N)	Co.V. (%)	Mean (MPa)	Co.V. (%)	Mean (GPa)	Co.V. (%)	Mean (%)	Co.V. (%)
Flax-1Y	Weft	15	37.39	15.54	293.40	15.54	11.61	10.03	2.55	9.65
Flax-2Y	Weft	12	87.95	12.75	345.04	12.75	8.53	6.44	4.19	12.37
Flax-2Y	Warp	14	92.05	10.47	361.15	10.47	10.07	6.95	3.56	6.31
Flax fabric-2cm	Weft	14	476.11	8.60	233.49	8.60	4.39	8.40	5.96	9.33
Flax fabric-6cm	Weft	5	1758.81	7.50	287.52	7.50	8.25	9.93	3.61	4.79
Flax fabric-6cm	Warp	5	1743.71	3.91	285.05	3.91	9.81	3.14	2.93	7.66

P_u = Maximum Load; f_t = Tensile Strength; E = Young's Modulus; ε_u = strain at failure

The results are expressed in terms of maximum load, tensile strength, elastic modulus and strain at failure. For each series of values, the coefficient of variation is reported as well. Figure 27 shows the stress-strain curves of Flax fabric-6cm specimens belonging to both the warp and weft directions. The comparison between the two series of specimens shows that the textile is characterized by the same tensile strength in both directions, while a slight change in the Young's Modulus and in the strain at the failure is recorded.
The results represented in Figure 27 fall within the range variation of the same quantities reported in other similar studies already published in the literature (Asprone et al. 2015; Codispoti et al. 2015; Olivito et al. 2014; Pickering et al. 2016).

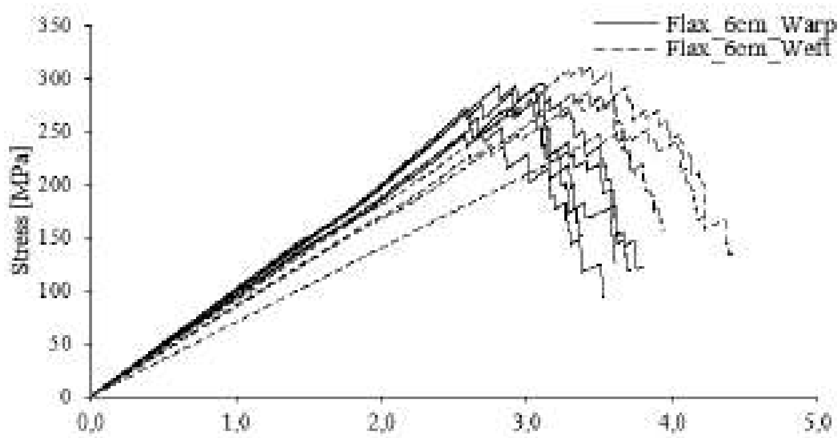
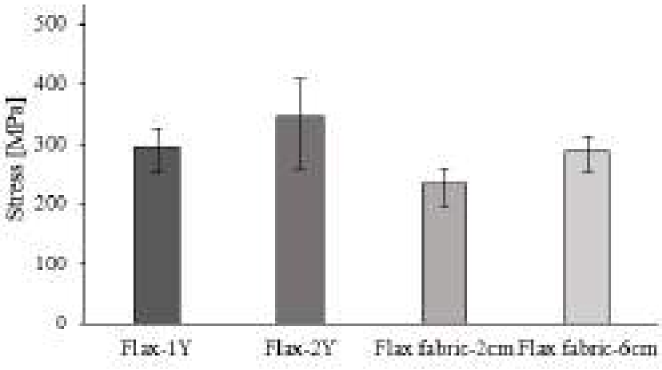
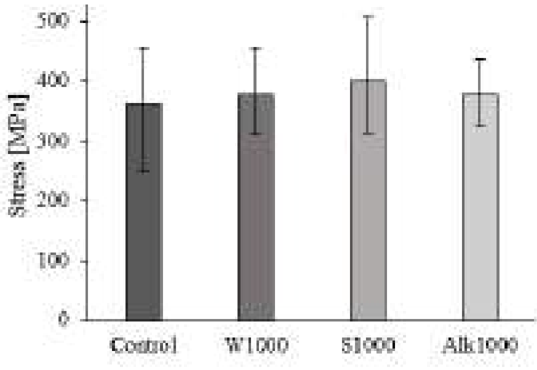


Figure 27:Stress-strain curve of Flax-6cm specimens in warp and weft directions

The main values of the tensile strength referring to the different sized samples are represented in Figure 28. It is clear that the maximum value of the stress is recorded in the double-twisted yarn samples "Flax 2Y". The reduction of the peak stress in the strip samples is due to a non-uniform distribution of the stress among the threads. Figure 28b shows that the main value of the tensile strength of the control series of specimens is even slightly lower than the one recorded in the aged series of specimens. This phenomenon may be attributed to the natural variability of the mechanical properties of the textile.
Table 13 reports the results deriving from the tensile tests of the specimens subjected to the ageing process. It clearly highlights that the adopted ageing and environmental exposure protocol does not result in any reduction of the relevant mechanical property, neither in terms of strength, nor in terms of stiffness. The invariance of mechanical properties after those protocols does not only deal with the average values, as the corresponding Coefficient of Variation is generally about in the order 10%.
The stress-strain curves represented in Figure 29 further confirm this experimental observation.



(a) Mechanical characterization



(b) Durability

Figure 28:Comparison between the tensile strengths of different series

Table 13: Mechanical properties of samples subjected to the durability protocol

	Ageing environment	Number of tests	P_u		f_t		ε_u	
			Mean (N)	Co.V. (%)	Mean (MPa)	Co.V. (%)	Mean (%)	Co.V. (%)
Flax-Y2	Control	12	92.05	10.47	361.15	10.47	3.56	6.31
Flax-Y2	W1000	12	96.17	10.75	377.30	10.75	4.37	10.63
Flax-Y2	S1000	13	102.27	13.19	401.23	13.19	4.40	6.57
Flax-Y2	Alk1000	13	95.92	8.74	376.34	8.74	4.54	7.10

P_u = Maximum Load; f_t = Tensile Strength; ε_u = strain at failure

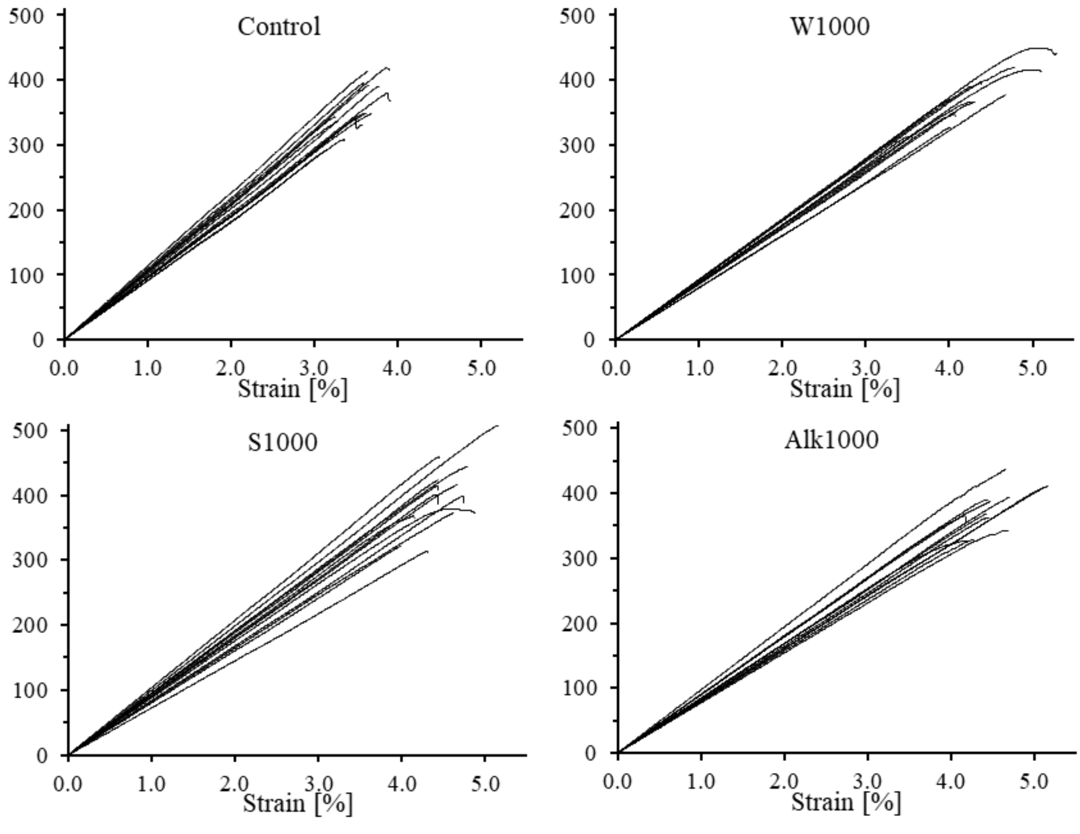


Figure 29:Stress-strain curves of series of specimens subjected to the durability protocol

Finally, the images reported in Figure 30 derive from the SEM analysis and depict the surface of four representative samples of the four series of fibres considered in this study with a magnification of 10000. As can be seen, they do not evidence any relevant damage on the filament surface of the samples subjected to the environmental exposure protocol. This confirms that a period of 1000h under the aforementioned conditions does not affect the mechanical behaviour of the flax fabric under consideration in this study.

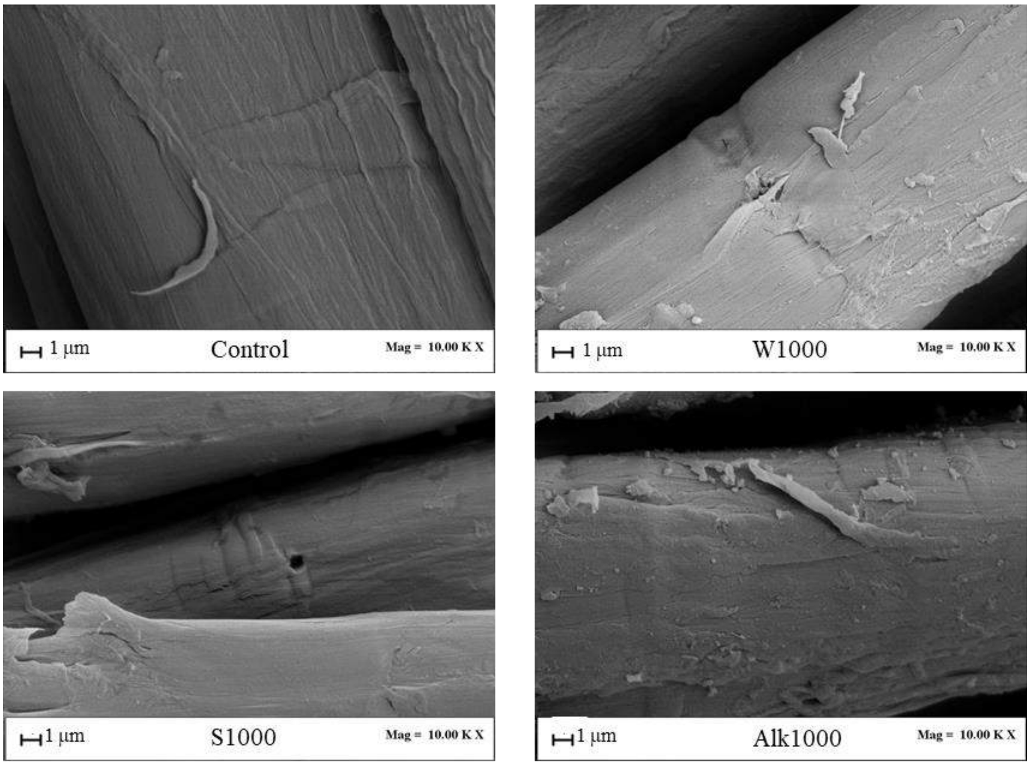


Figure 30:SEM images of samples subjected to the durability protocol

CONCLUSIONS

The experimental study, carried out to mechanically characterise the flax textile and to consider its effectiveness in strengthening composite systems, has led to the following conclusions:

- the geometric investigation by means of an optical microscope cannot provide reliable values to be used for mechanical purposes;
- the main values of the mechanical properties, consisting in a tensile strength of 353.72 MPa, a Young's Modulus of 9.36 GPa, and a strain at the failure of 3.85%, are in line with what is present in the literature;
- the durability protocol chosen is not aggressive enough to trigger the degradation of the natural textile;
- the SEM analysis is an effective technique for both the investigation of the fibres filaments, and the monitoring of the degradation status of the textile.

In order to check the effectiveness of the use of natural fibres, experimental analysis has to be conducted directly on composite systems reinforced by this textile. Moreover, a more challenging durability protocol should be considered with the aim to provide a more representative environmental exposure condition for vegetal fibres under consideration.

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REFERENCES

AC 434-1011-R1 (2011). “Acceptance Criteria for Masonry and Concrete Strengthening Using Fiber-reinforced Cementitious Matrix (FRCM) Composite Systems”.

D. Asprone, M, Durante, A. Prota, G. Manfredi (2011). “Potential of structural pozzolanic matrix-hem fiber grid composites”, Construction and Buildings Materials 25 2867-2874.

R. Codispoti, D.V. Oliveira, R.S. Olivito, P.B. Lourenco, R. Fanguiero (2015). “Mechanical performance of natural fiber-reinforced composites for the strengthening of masonry”, Composites Part B 74-83.

O. Faruk, A.K. Bledzki, H. Fink, M. Sain (2012). “Biocomposites reinforced with natural fibres: 2000-2010”, Prog Polym Sci 37:1552-96.

S.R. Ferreira, E. Martinelli, M. Pepe, F. de Andrade Silva, R.D. Toledo Filho (2016), Inverse identification of the bond behavior for jute fibers in cementitious matrix, Composites Part B: Engineering 95, 440-452

ISO 13934-1 (2013). “Textiles-Tensile properties of fabrics- Determination of maximum force and elongation at maximum force using the strip method”.

R.S. Olivito, O.A. Cevallos, A. Carrozzini (2014). “Development of durable cementitious composites using sisal and flax fabric reinforcement of masonry structures”, Materials and Design 57 258-268.

K.L. Pickering, M.G. Aruan Efendy, T,M, Le (2016). “A review of recent developments in natural fibres composites and their mechanical performance”, Composites: Part A 83 98-112.

R.D. Toledo Filho, K. Scrivener, G.L. England, K. Ghavami (2000). “Durability of alkali sensitive sisal and coconut fibers in cement based composites”, Cement Concr Compos; 6(22):127-43.

F.P. Torgal, S. Jalali (2011). “Cementitious building materials reinforced with vegetable fibres: A review”, Constr. Build Mater; 25(2):575-81.

A. Nobili, C. Signorini (2017). “On the effect of curing time and environmental exposure on impregnated Carbon Fabric Reinforced Cementitious Matrix (CFRCM) composite with design considerations”, Composites Part B 112 300-313.