PERFROMANCE EVALUATION OF PALM STEMS FIBERS IN CEMENT-BASED MATERIALS

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Abstract

Natural fibers, including cellulose, bamboo, hemp, have good mechanical and thermal properties, and they are renewables and recyclables. Recent advances regarding the use of natural fibers in cement composite showed that their behavior is depended on their structure, chemical composition, and their position in the plant. Therefore, the use of natural fibers in cement composite presents important challenges. For example, their hydrophilic character and vulnerability in alkaline environment have to be solved. The objective of this investigation is to characterize and evaluate the performance of date palm stems (pedicel) fibers in cement-based materials. Two different types of natural fiber, including hemp and date palm stems (pedicel) fibers, were evaluated. Both types of fiber have been subjected to several chemical treatments to reduce their high hydrophilicity and increase their durability. The obtained results showed that palm stems fibers have good mechanical properties. Furthermore, the chemical treatment of fiber using hydrophobic resin reduced the hydrophilic character of the fibers, hence resulting in improving the bond between fibers and matrix.

Keywords:

Cement-based materials; chemical treatments; durability; natural fibers; rheology.

1 INTRODUCTION

The use of natural fibers in concrete is one of the most technical advance in recent years. Natural fibers have good mechanical and thermal properties, and they can reduce shrinkage of cement-based materials. For example, their use in cement-based materials is very effective in controlling plastic shrinkage [Boghossian 2008]. For example, the use of 0.3%, by volume, of hemp fibers reduced plastic shrinkage and crack width by 99% [Kawashima 2011]. Also, they are renewables and recyclables. In addition, their incorporation in cement matrix helps preserve the environment. Indeed, the use of hemp fibers helps to produce eco-friendly composite materials that can sequester significant quantities of CO_2. The experimental wall made with lime-hemp concrete (LHC) was found to have a total carbon sequestration of 275.7 kg of CO_2 per 1 m^3. On the other hand, in the context of ecological and sustainable developments, natural fibers present a good opportunity, because cellulose alone presents 40% of the biosphere.

Recent advances regarding the use of natural fibers showed that their behavior in cement composite is depended on their structure, chemical composition, and their position in the plant, i.e. internal part of the stem or the external one. On the other hand, the behavior of these fibers is affected by the interactions between the fibers and the cement matrix, because of their important hydrophilic character, which increase their vulnerability in alkaline. For example, the natural fibers, especially cellulose, hemicellulose, lignin and pectin, can degrade in high pH medium, such cement-based matrix, which results in their fragmentation [Romide 2000]. This degradation can be due to the crystallization of the portlandite inside the lumen, hence leading to the loss of tensile strength of fibers. In addition, the hemp fibers may contain high amount of pectin that can fix calcium and hydroxyl ions on their surfaces. This fixation can be responsible of delaying setting time and hindering the CSH formation [Sedan 2008]. The objective of this investigation is to characterize and evaluate the performance of palm stems (PS) fibers in cement-based materials. Rheological measurements and microstructural observations were carried out. A commercial hemp fiber is also considered for comparisons purposes. Both types of fibers have been subjected to several chemical treatments to improve their durability.

2 EXPERIMENTAL PROGRAM

2.1 Extraction of PS fibers

PS fibers are extracted from date palms. First, the stems (pedicels) are collected and cut to different lengths. The stems were then stored in a controlled room temperature for 48 hours before the extraction process. The stems are placed in a disk mill (Photos 1-4) and subjected to different preparation processes to optimize the geometry of fibers. After several trial and error tests, the best quality of fibers was obtained by using 30 g of PS and grinding for 60 sec.

2.2 Infusion of PS and hemp fibers in water

For both PS and hemp fibers, a given quantity of PS and hemp fibers is immersed in 1.5 L and boiled during 45 min. Two different concentrations of fibers of 10 g and 20 g were used for the first infusion test. After 45 min of immersion, a filtration process was carried out to collect the filtrated liquid. The fibers were kept at room temperature for 48 hours. A second infusion was carried out on the infused fibers and the filtered liquid was collected (Photo 5). The water collected from the two infusions was used separately to proportion cement paste mixtures.

2.3 Fibers treatments

Different fiber treatments were carried out on PS and hemp fibers. The treatment methods are 1. Thermal treatment by infusing fibers in boiling water, 2. Immersing fibers in a hydrophobic resins type, and 3) Combined treatment that consists in immersing fibers in varathane hydrophobic resin followed by imbibition in silica fume suspension. Before the impregnation of the fibers in the resin, their absorption was determined. The water absorption rate of the fibers was calculated using Eq. (1).

$$A\,(\%) = \frac{M_f - M_I}{M_f} \tag{1}$$

Where, A is the percentage of absorption (%), M_i and M_f are the initial and final masse values of fiber, respectively.

The fibers were then dried and placed in a controlled room temperature. After 48 hours, the dried fibers were impregnated in the hydrophobic resin for 10 minutes and then dried in air for 2 hours. In the case of combined treatment, the PS and hemp fibers are impregnated in the hydrophobic resin for 10 minutes. The fibers are then removed from the resin and impregnated directly into the silica fume for 24 hours.

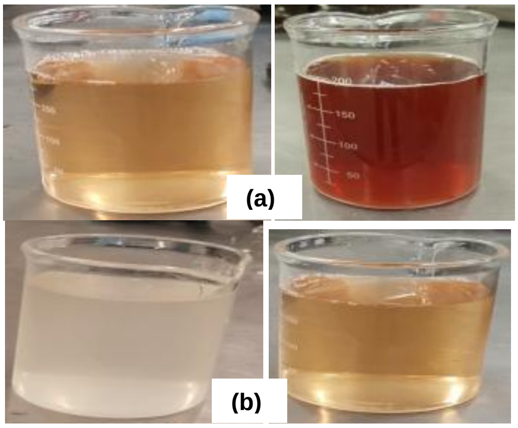

Photo 5: Liquid filtered after infusion of a) PS fibers and b) hemp.

Photo 1: Salvaged date pedicels.

Photo 2: Cut palm stalk.

Photo 3: Mill components.

Photo 4: Palm stem fibers.

2.4 Characterizations of PS fibers

The morphology of fibers was determined using a scanning electron microscope (SEM) equipped with an energy spectroscopy (EDS). Furthermore, the tensile strength and elongation of 6-cm length fibers are determined according to [ASTM 412] Standard using a ProLine test machine (Photo 6).

Photo 6: Tensile tests on fibers

2.5 Cement paste mixtures

The various cement paste mixtures were prepared using general use cement (GU). The physical and chemical compositions of GU cement and silica fume are summarized in Tab1. The paste mixtures were proportioned using a water-to-cement ratio (W/C) of 0.45. In addition to the calorimetry evaluation [ASTM C1702], rheological measurements were carried out on cement paste mixtures using a coaxial cylinders viscometer with serrated surfaces. The diameters of the outer and inner cylinders are 28.911 mm and 26.660 mm, respectively, hence providing a shear gap of 1.126 mm. The rheological measurements were carried out at 10 minutes and 1 hour after the first contact between liquid and cement. All rheological measurements were made at a temperature of 25°C. The flow curves were determined using the protocol presented in Fig.1. The duration of each shear step was optimized to ensure a complete breakdown. The yield stress and plastic viscosity values were estimated using the modified Bingham model (Eq. 2):

$$\tau = \tau_o + \mu_p \dot{\gamma} + C\dot{\gamma}^2 \quad (2)$$

Where, τ_o is the shear stress (Pa), μ_p is the plastic viscosity (Pa.s), and □ is the shear rate (s^{-1}).

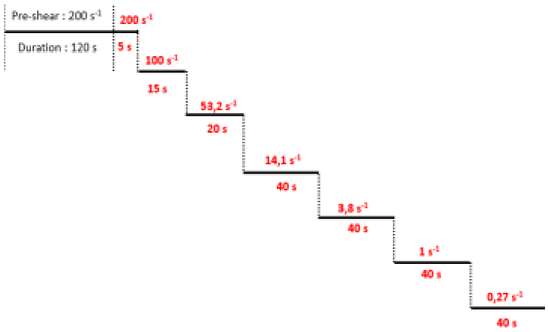

Fig. 1: Experimental protocol for rheological measurements.

3 RESULTS AND DISCUSSIONS

The obtained results will be discussed in terms of fiber characteristics, calorimetry measurements and rheology of cement paste prepared with filtered liquid from fibers.

3.1 Fiber characteristics

The images obtained using the SEM scanning electron microscope (Fig. 2) showed that the PS fibers have a rough and porous structure. On the other hand, the absorption tests revealed that both PS and hemp fibers showed high absorption of around 150%, which is consistent with literature (Figs. 3 and 4). However, the impregnation of fibers in the hydrophobic resin (treated PS) reduced their absorption (reduction between 62 and 82%), regardless of the time of immersion. The evolution of absorption with time of treated fibers can be attributed to the presence of micro-cracks between fibers and resin, which increases the rate of absorption of the fibers with time.

3.2 Tensile strength of fibers

Tensile strength and Young modulus were determined on PS fibers having diameters varying between 200 and 426 μm. The PS fibers showed excellent tensile strength (Tab. 2) value of up to 800 MPa. The Young's modulus of fibers is varying between 20 and 36 GP. It is worthy to mention the high variation observed in measuring tensile strength of fiber, which is probably due to the variation of fiber diameters.

3.3 Calorimetry and rheological measurements

Calorimetry and rheological measurements were carried out on cement paste mixtures proportioned using liquid obtained from the first and second filtration processes. The instantaneous heat and cumulative heat values are presented in Figs. 5 and 6, respectively. As can be observed, the use of liquid filtered from PS and hemp fibers resulted in higher instantaneous hydration peak (Fig. 5) compared to reference mixture. This phenomenon may be due to the alkaline hydrolysis of polysaccharide and impurity of the fibers, which increases the heat. Generally, this trend is common in high-purity nano-cellulosic and bacterial nanocellulose medium [Gomez 2013], [Onuaguluchi 2014]. Indeed, it is reported that the alkaline hydrolysis of cellulose, which is an exothermic reaction, can promote hydration of cement.

The calorimetry results obtained on paste mixtures prepared with first and second filtered liquids obtained after infusion of 20 g of fibers are summarized in Fig.7. On the other hand, the results of mixtures prepared using the first filtered liquid using 10 and 20 g of fibers are presented in Fig. 8.

As can be observed (Fig. 7), cement paste mixture prepared with filtered liquid from PS fiber (first infusion) resulted in higher dormant period compared to those made with hemp fiber and reference mixtures. However, the use of filtered liquid from the second infusion resulted in lower dormant period. This phenomenon may be due to the presence of a large amount of polysaccharide and impurity in the filtered liquid (from the first infusion). The presence of polysaccharides contained in fibers can form a protective layer around cement particles, which contribute in delaying cement hydration and setting. For example, the pectin in fibers can delay cement hydration [Sedan 2008]. The use of PS fibers may results in higher dormant period than hemp fibers. Results presented in Fig. 8 revealed that the increase in dosage of fibers leads to a significant delay in setting of cement paste (Fig. 8). Boiling fiber in water during an optimum time seems to be efficient in extracting polysaccharides from PS fibers and reduce their impact on cement hydration.

The flow curves and variation of apparent viscosity presented in Figs. 9-12 revealed higher stress (flow resistance) of cement paste mixtures proportioned with filtered liquids from fibers, especially after 60 min of age. However, all the investigated mixtures showed a comparable yield stress, regardless of the type and dosage of fibers. On the other hand, the use of liquid from the infusion of PS and hemp fibers induced a relatively higher apparent viscosity at low shear rate and pseudoplastic response, regardless of the duration of infusion. The presence of polysaccharide particles contribute in increasing the contact points between particles, hence increasing the shear stress. Further analysis, such as liquid chromatography, are necessary to determine the chemical composition of filtered liquids.

Tab. 1: Chemical composition of cement and silica fume.

(%)	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3	Other oxides
Cement GU	20.4	0.2	4.4	2.5	2.1	62.0	0.0	0.8	3.8	0.2
Silica fume	94.2	0.0	0.3	0.1	0.3	0.8	0.1	0.5	0.0	0.5

Tab. 2: Properties of PS fibers.

-	Diameter (um)	E (GPa)	ΔL	Tensile (MPa)	Standard deviation (MPa)
PS fibers	Min - Max 200 - 426	Min - Max 20.2 - 35.8	Min - Max 1.64 - 2.60	Min - Max 229 - 804	105

3.4 Effect of fiber imbibition in silica fume

As can be observed in Fig. 13, the incorporation of PS fibers in cement matrix resulted in the formation of portlandite inside their lumens and cause their degradation. On the other hand, the images of fibers taken after their soak in silica fume are shown in Fig. 14. It is worthy to mention that EDX analysis were performed to identify the chemical composition of the constituents. The obtained images showed that after their imbibition in silica fume suspension, large amounts of C-S-H are deposited on their surfaces, and no portlandite was found. The immersion of fibers in silica fume suspension create a thin protective layer of silica on their surfaces, which can promote the formation of C-S-H, hence reduce hydrophilicity and improve durability of fibers.

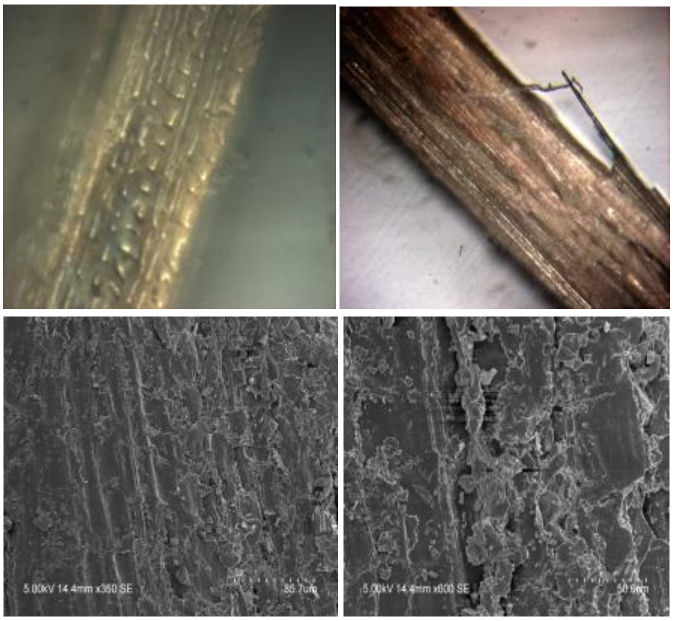

Fig. 2: Microscopic images of PS fibers under different magnifications.

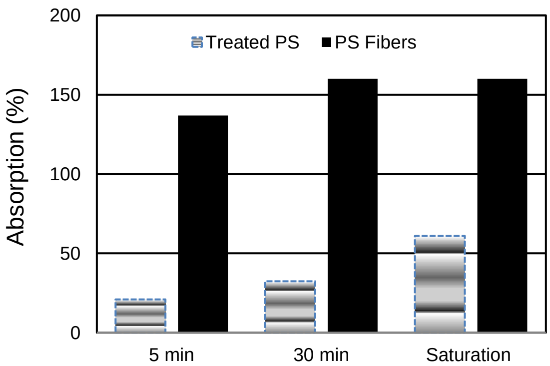

Fig. 3: Absorption PS fibers.

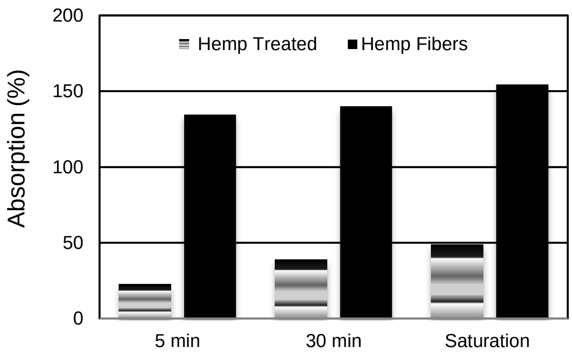

Fig. 4: Absorption *of hemp fibers.*

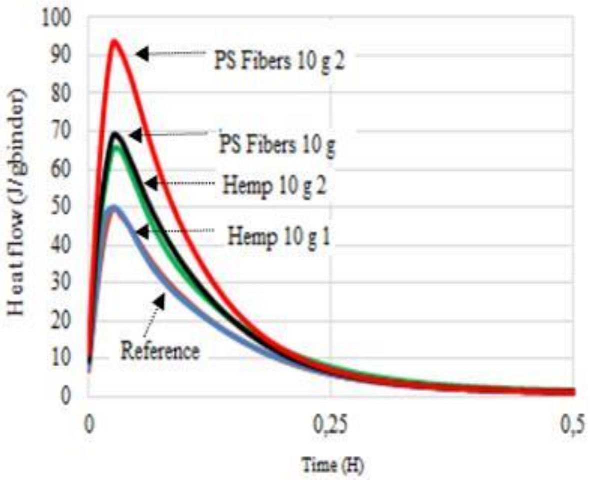

Fig. 5: Heat flow of different cement paste mixtures.

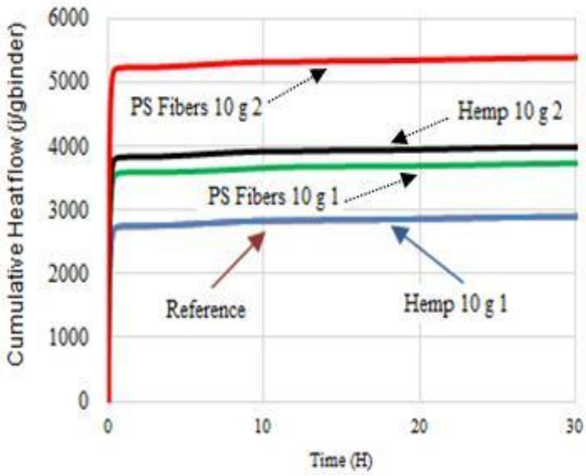

Fig. 6: Accumulated heat of different cement paste mixtures.

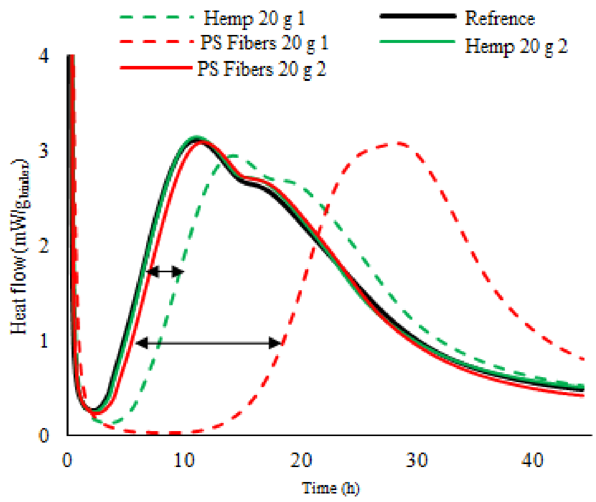

Fig. 7: Heat of cement paste mixtures made with the 1st with the filtered liquid (1st and 2nd infusions.)

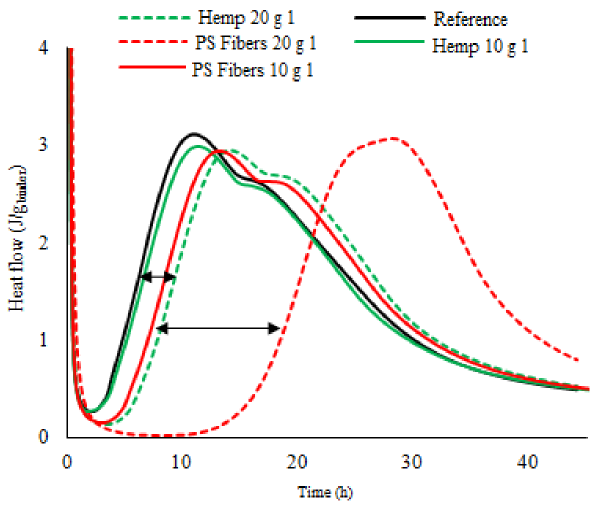

Fig. 8: Heat of cement paste mixtures made with filtered liquid (1st infusion) and different dosages of fibers.

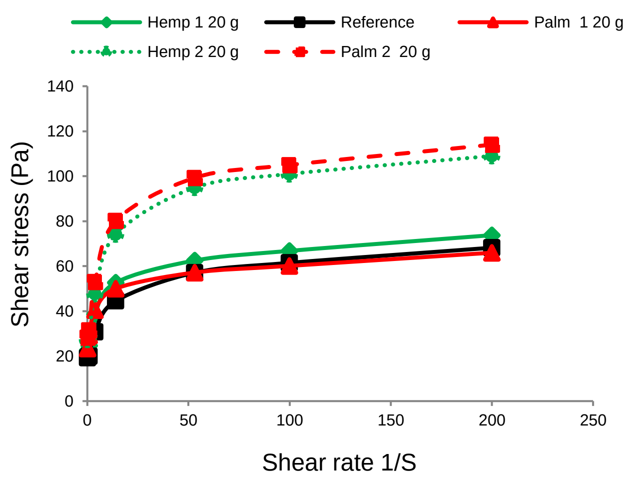

Fig. 9: Flow curves of cement paste mixtures at 10 minutes.

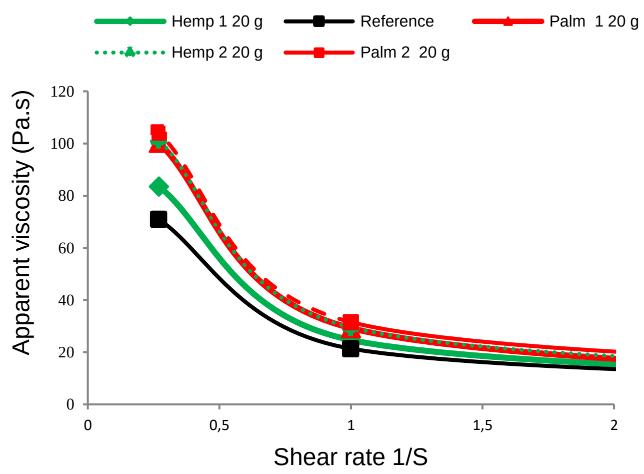

Fig. 10: Variation of apparent viscosity at 10 minutes.

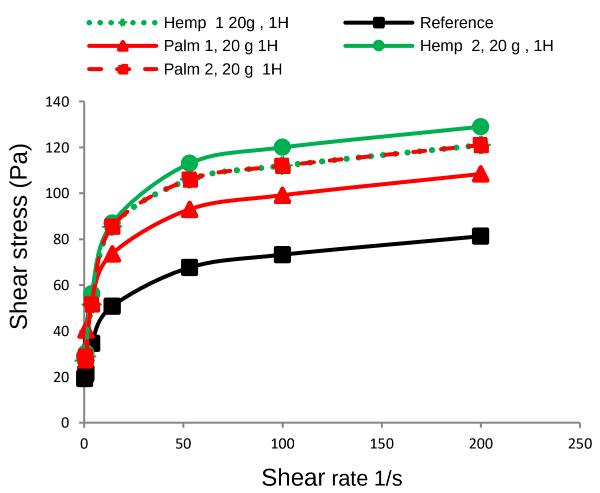

Fig. 11: Flow curves of cement paste mixtures at 60 minutes.

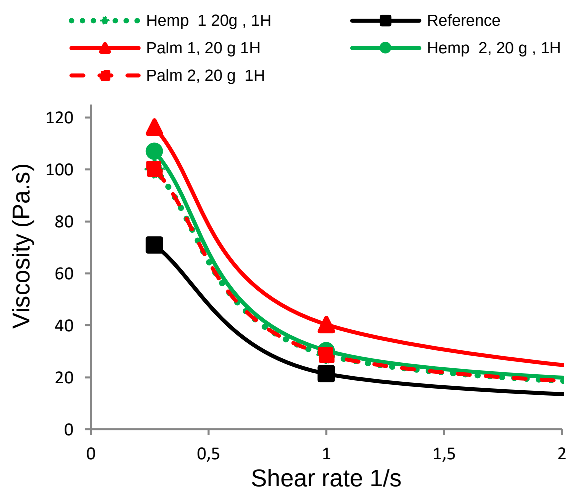

Fig. 12: Variation of apparent viscosity at 60 minutes.

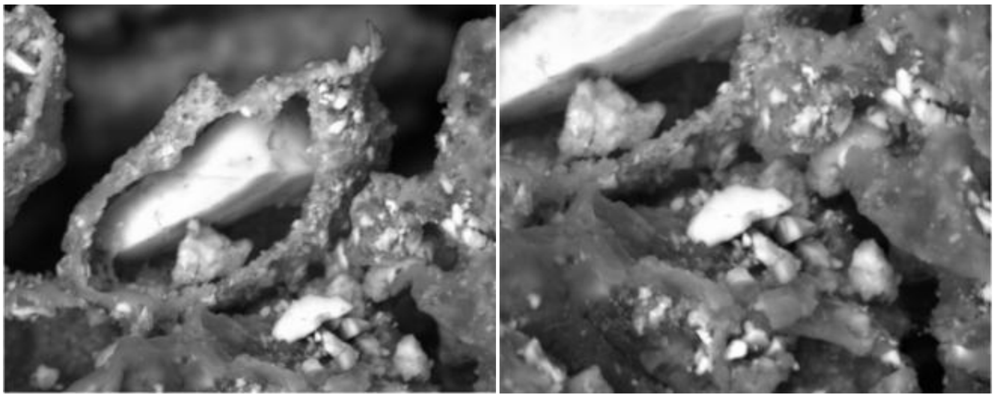

Fig. 13: Portlandite inside the lumen of PS fibers.

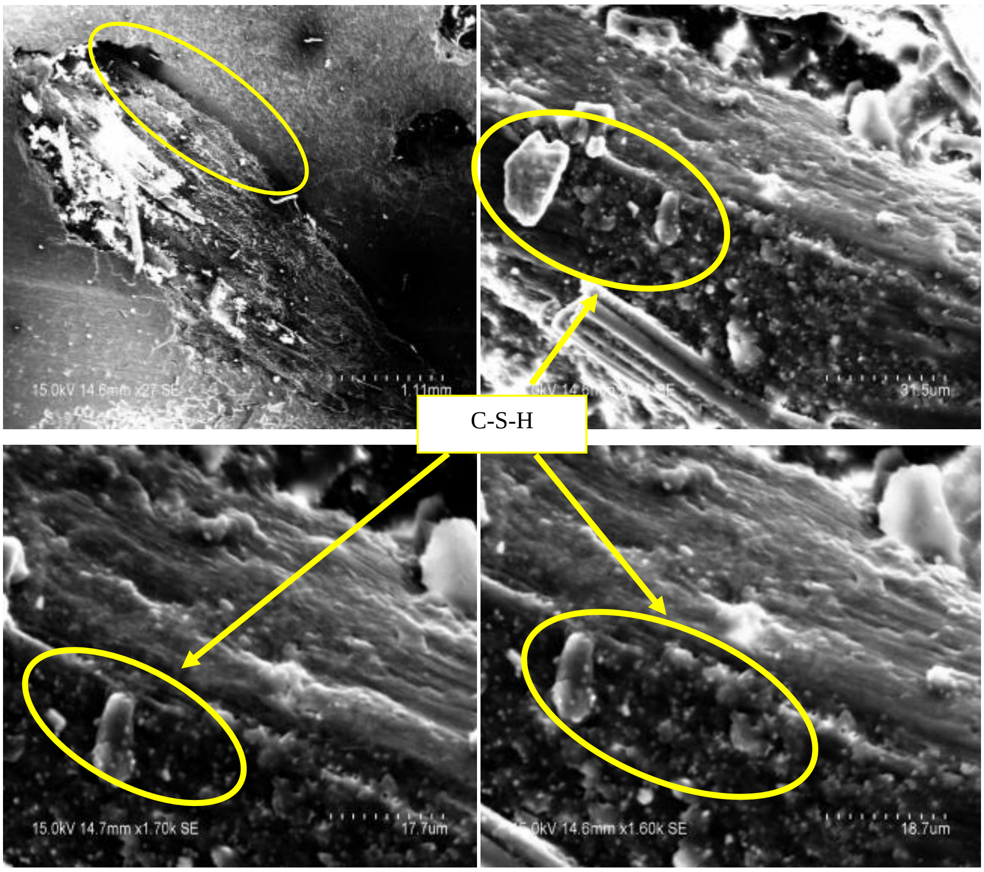

Fig. 14: Microscopic image of date palm fibers soaked in silica fume.

4 SUMMARY

Based on the results presented in this paper, the following conclusions can be pointed out:

1. Palm stem fibers were successfully extracted and their characteristics were determined. These fibers showed high water absorption, but good tensile strength.
2. The impregnation of palm stem fibers in hydrophobic resin is efficient to reduce their water absorption by 62 to 82%.
3. The infusion of natural fibers in boiled water is efficient way to extract the various constituents of fibers causing delay of cement hydration and setting.
4. The imbibition of natural fibers in silica fume is a good way to avoid the formation of portalndite in inside the lumen of fibers, hence improve their durability in cement-based materials. The imbibition of fibers in silica fume promoted the formation of CSH on their surfaces, which can contribute in enhancing their bonding with matrix.
5. The use of filtered liquids from the natural fibers increased the dormant period, apparent viscosity, and pseudoplastic response of cement paste mixtures.

5 ACKNOWLEDGMENTS

I wish to express my gratitude to everyone who contributed to this work.

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