



Received: 03-02-2020
Accepted: 17-02-2020

Anales de Edificación
Vol. 6, Nº2, 82-99 (2020)
ISSN: 2444-1309
Doi: 10.20868/ade.2020.4500

Estudio de los efectos de diferentes combinaciones de materiales repelentes de agua y fibras de piassaba (*Aphandra natalia*) en morteros de cemento.

Study of the effects of different combinations of water-repellent agents and supplementary cementing materials on piassaba fibers (*Aphandra natalia*) in cement mortars.

Cristian Balcázar Arciniega^{a,b}, Eduardo Aguirre Maldonado^{a,b}, Javier Pinilla Melo^a, Francisco Hernández Olivares^a

^a Universidad Politécnica de Madrid (España, c.balcazar@alumnos.upm.es; e.aguirre@alumnos.upm.es; javier.pinilla@upm.es; f.hernandez@upm.es), ^b Universidad Técnica Particular de Loja (Ecuador, cabalcazar@utpl.edu.ec; ebaguirre@utpl.edu.ec)

Resumen— Esta investigación trata sobre el desarrollo y caracterización de un nuevo mortero de cemento reforzado con fibras de piassaba. El objetivo es obtener un material con una cantidad reducida de compuestos de hidróxido de calcio en las superficies de las fibras. Se evaluaron fibras no tratadas, fibras tratadas con hidróxido de calcio y fibras tratadas con combinaciones de agentes hidrófobos y materiales cementantes suplementarios (SCM). La morfología de las fibras se estudió mediante microscopía electrónica de barrido (SEM). Las propiedades químicas de SCM se analizaron mediante la técnica de espectroscopia de fluorescencia de rayos X (FRX). Además, se estudió la degradación de las fibras con y sin tratamientos en una solución de hidróxido de sodio. Se discutieron las propiedades físicas (densidad aparente, porosidad abierta, coeficiente de absorción de agua por inmersión y capilaridad) y las propiedades mecánicas del mortero endurecido (resistencia a la flexión y módulo de elasticidad). Después de la prueba de flexión, se retiraron las fibras de los tubos de ensayo y se analizaron mediante microscopía óptica. Los resultados muestran, en primer lugar, que el látex de caucho natural es mejor adherente y tiene mayor compatibilidad con el SCM que el aceite de semilla de lino, y por otro lado, las cenizas de cáscara de arroz (RHA) tienen mayor compatibilidad con el polímero y el aceite. Y finalmente, que el efecto combinado del látex de caucho natural y los finos en polvo de mortero reciclado quemado (BRMF) sobre la superficie de la fibra mejoran el comportamiento del mortero a los 28 días, y pasado ese tiempo se ve comprometida la durabilidad.

Palabras Clave — Piassaba; *Aphandra Natalia*; Mortero de cemento; Látex de caucho natural; Aceite de semilla de lino; Cenizas de cáscara de arroz; Humo de sílice; Polvo fino de mortero reciclado quemado.

Abstract— This research is about the development and characterization of a new cement mortar reinforced with piassaba fibers. The aim is to obtain a material with a reduced amount of compounds of calcium hydroxide on the fiber surfaces. Untreated fibers, calcium hydroxide treated fibers, and treated fibers with combinations of hydrophobic agents and supplementary cementitious materials (SCM) were evaluated. Fiber morphology was studied through scanning electron microscopy (SEM). The SCM chemical properties were analyzed through the technique of X-ray fluorescence spectroscopy (FRX). Furthermore, the degradation of the fibers with and without treatments was studied in a sodium hydroxide solution. The physical properties (apparent density, open porosity, water absorption coefficient by immersion and capillary), and the mechanical properties of the hardened mortar (flexural strength and modulus of elasticity) were discussed. After the test of flexing, the fibers were removed from the test tubes and they were analyzed by light microscopy. The results show, first that the natural rubber latex is better adherent and has greater compatibility with the SCM than the flax seed oil, and on the other hand, the rice husk ashes (RHA) have greater compatibility with the polymer and oil. And finally, that the combined effect of natural rubber latex and the burned recycled mortar powder fines (BRMF) on the fiber surface improve the performance of the mortar after 28 days, and after that time the durability is seen compromised.

Index Terms— Piassaba; *Aphandra Natalia*; Cement mortar; Natural rubber latex; Flax seed oil; Rice husk ashes; Silica fume; Burned recycled mortar powder fines.

I. INTRODUCTION

The materials traditionally used as reinforcement in cementitious matrix materials (steel, fiberglass and carbon fibers) are nonrenewable resources with a high consumption of energy during its manufacturing process. The natural fibers show several advantages over synthetic fibers and reinforcing steel, for example, abundance, biodegradability, recyclable, easy to use, nontoxicity, low density, no abrasiveness, good thermal properties, good toughness properties and good values of elasticity modulus. However, they exhibit high absorption capacity of water and a poor compatibility with the matrix, which leads unpredictable consequences on the concrete properties (Abiola, 2017; Kılınç, Durmuşkahya, & Seydibeyoğlu, 2017).

The reinforced cement compounds with natural fibers, as they get old, suffer significant losses in their mechanical performance (Barra et al., 2015). The mechanical nuance of the compounds is because, of the one hand, to the water absorption capacity of the fibers during the curing step, responsible for volume changes and loss of fiber- matrix adhesion (Huu, Ogihara, Huy, & Kobayashi, 2011). On the other hand, the fibers deterioration in the cement matrix, caused by the alkaline hydrolysis of amorphous components (lignin and hemicellulose) and the precipitation of the cement hydration products on the fibers cell walls (Rocha et al., 2017; Toledo Filho, Scrivener, England, & Ghavami, 2000). As degradation progresses, calcium silicate hydrate (C-S-H) and the portlandite (CH) gradually infiltrate into the cell walls by causing mineralization and brittleness of fibers (Wei & Meyer, 2015).

In the present investigation, are distinguished four effective methods for restrict the fiber degradation in alkaline media:

- Alkalinity decrease of the concrete, by replacing partially the portland cement by pozzolans (Wei & Meyer, 2015), or

by combining cement with siliceous materials and clay minerals (Wei & Meyer, 2016, 2017).

- Fibers modification through different techniques, such as polymerization with methane's cold plasma (Li, Zhou, & Guo, 2017; Ridzuan, Majid, Afendi, Azduwin, & Aqmariah, 2015), hornification (Claramunt, Ardanuy, García-Hortal, & Filho, 2011), or hydrothermal treatment (Sawsen et al., 2015).
- Water- repellent treatments (Abdullah & Lee, 2017; Basta, Sefain, & El-Rewainy, 2011).
- Hybrid treatments or combinations, for example: waterproofing-pozzolans (Jose et al., 2017), or hornification-polymeric impregnation (Rocha et al., 2015).

From the above, on the one hand, the alkaline treatment is considered the most economically viable (Das & Chakraborty, 2006), on the other hand, the hydrophobe treatment with flax seed oil decreases the fibers water absorption, but the hybrid treatment (natural rubber latex- pozzolans) also reduces the alkalinity of the cement paste, improving the performance and durability of cement-based composite materials.

Likewise, the sustainable development of the construction aims to reduce the extraction and use of conventional materials, through renewable natural resources from waste and by-products that can be recycled. In recent decades, the use of pozzolans as alternative to portland cement have been commonly used for economic, environmental and performance advantages. According to (Nair, Fraaij, Klaassen, & Kentgens, 2008), of all plants residues, the rice husk ashes (RHA) contains the highest proportion of silica, when burnt to temperatures between 500 ° C and 700 ° C, its nature of silica becomes predominantly amorphous and therefore reactive in alkaline conditions as the cement paste.

In addition, the search for new options to take advantage of construction waste, leads to analyze the potential of recycled

aggregates. A special case are burned recycled mortar powder fines (BRMF) (Aguirre-Maldonado & Hernández-Olivares, 2017), one raw material rich in silicon oxide, which can allow new uses in construction materials.

Complementary to this, piassava or piassaba, are rigid brown fibers, considered non-wood forestry product. These fibers come from three palm species *Aphandra natalia*, *Attalea funifera Mart*, and *Leopoldinia piassaba Wallace*. *Aphandra natalia* is a native palm to the western Amazon, it can find from the foothills of Ecuadorian Andes, in the northern part of the Peruvian Amazon, to the state of Acre in Brazil. The piassaba fibers of *Aphandra natalia* (harvested from the leaf sheath and the petiole), represent a resource of great economic importance for the broom industry of Ecuador, Peru, and Brazil. The sustainability of obtaining its fibers depends on the harvest methods developed in each geographical region: In Ecuador and some parts of Peru the extraction is sustainable, but in Brazil and other parts of Peru, the methods are destructive (Balslev, Knudsen, Byg, & Kronborg, 2010; Boll et al., 2005; Kronborg, Grández, Ferreira, & Balslev, 2008; Pedersen, 1996).

Attalea funifera mart and *leopoldinia piassaba wallace*

fibers have characterized and used experimentally as reinforcements of polymer matrices, finding applicability in materials that require good impact resistance and greater hardness (Sergio N Monteiro et al., 2006; Shimazaki & Colombo, 2010). Nevertheless, piassaba from *Aphandra natalia* has not been used experimentally in composite materials (Balcázar-Arciniega & Hernández-Olivares, 2017).

This research is consistent with the promotion of the use of renewable natural resources, to reduce the detrimental effect of synthetic materials on the environment, meeting with the challenge of turn the agricultural and construction waste into useful products to improve performance and durability of cement mortar composites reinforced with vegetable fibers.

II. EXPERIMENTAL PROGRAM

A. Materials

The Fig. 1 shows the adherent substances, and Fig. 2 shows the SCMs. "IMSSA Industries" provided the natural rubber latex, extracted from rubber trees (*Hevea brasiliensis*). The refined flax seed oil, correspond to the employed one for the wood treatment, and oil painting, available in the markets of the town. "Sika Ecuatoriana SA" provided the silica fume (SF).

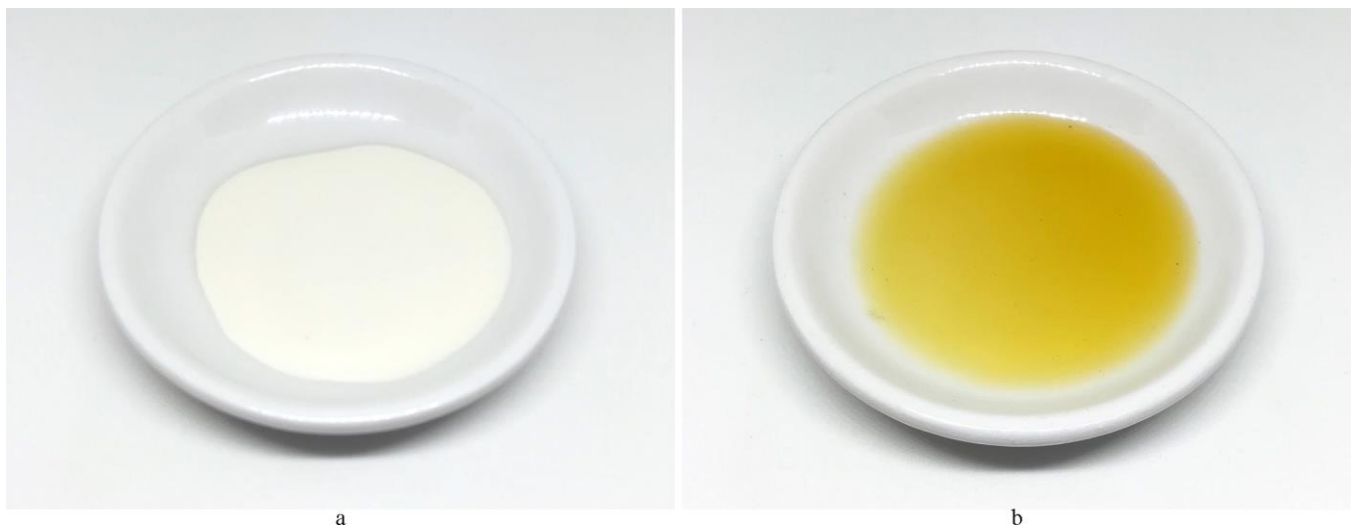


Fig.1. Adherent substances: (a) Natural rubber latex, and (b) Flaxseed oil.

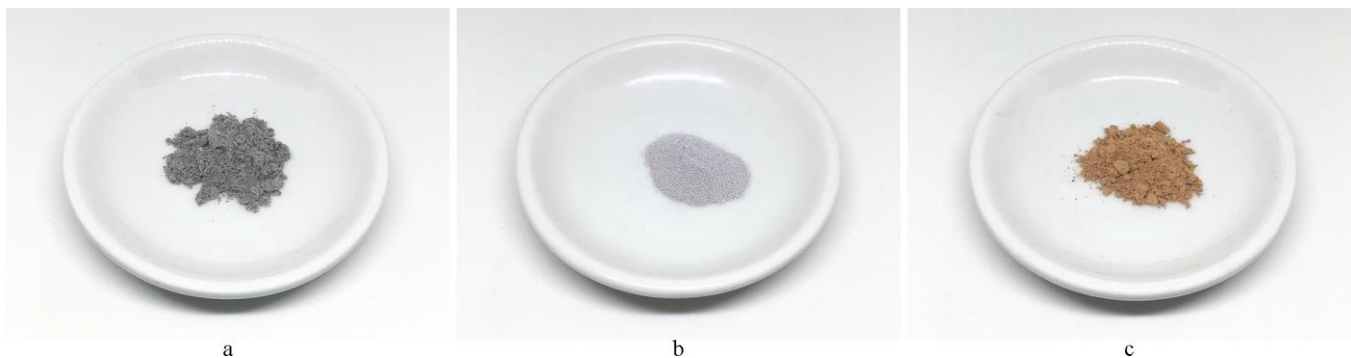


Fig.2. Supplementary cementitious materials (SCM): (a) Rice husk ash (RHA), (b) Silica fume (SF), and (c) Burned recycled mortar powder fines (BRMF).

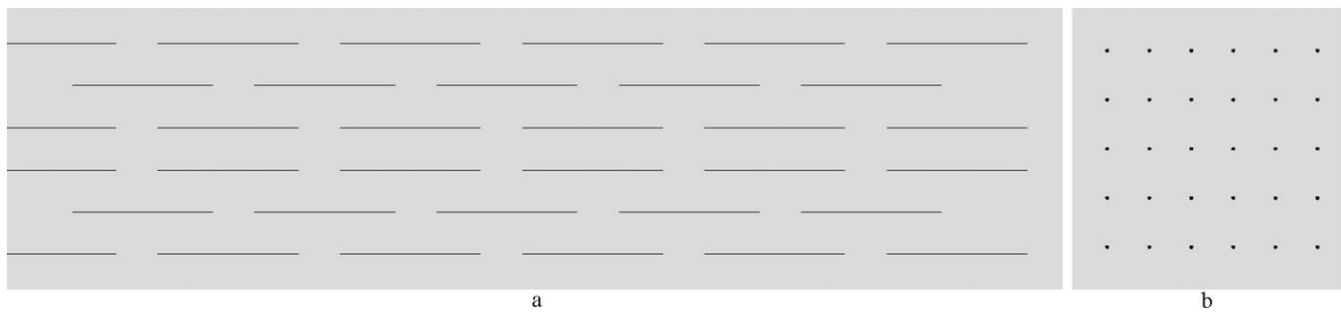


Fig. 3. Disposición de las fibras en las probetas de concreto: (a) vista en sección de planta, y (b) vista en sección transversal.

“UNACEM Ecuador” provided the Portland cement type II. DISENSA provided the calcium hydroxide (INDAMICAL PREMIUN). “Solvesa Ecuador SA” provided the sodium hydroxide (98% purity). Finally, the Loja (Ecuador) broom manufacturers provided the piassaba fibers.

B. Collection and preparation of BRMF

The burned recycled mortar powder fines (BRMF) were obtained according to the process of (Aguirre-Maldonado & Hernández-Olivares, 2017). The raw material comes from demolition process of spoilage buildings. The sample pass for a dried process in order to proceed with two crushing stages and one of spraying. The first crushing stage was done manually to release the coarse aggregate of the concrete samples, and then the mechanical crushing was performed with a grinder, to

finally use a disk sprayer. Then, part of the sandy compounds (approximately 20%) were separated to obtain a finer raw material. Subsequently, dehydration processes were followed in a temperature of 800 ° C for 2 hours, after that the samples were rapidly cooled and placed in airtight containers for storage in a dry environment.

C. Collection and preparation of RHA

The rice husk ashes comes from the Macará canton (province of Loja - Ecuador). In first place, there were burned for 30 minutes on a hot plate, in order to grind it with a ceramic mortar for 5 minutes , after that the ash was treated thermally in a muffle programmed at 500 ° C for 2 hours, obtaining as a result rice husk ashes (RHA) which are rich in silica .

TABLE 1
APPLIED TREATMENTS TO THE FIBERS

Nomenclature	Alkaline treatment	Adherent solution	Coating (SCM)	Details
T00	-	-	-	Untreated fibers.
T01	Calcium hydroxide	-	-	A solution of 5% concentrated Ca(OH) ₂ in distilled water was prepared. The solution was allowed to stand for 24 hours, then the fibers were inserted into the solution for 24 hours, when removed, they were allowed to dry at room temperature.
T02	-	Natural rubber Latex	Rice husk ashes	The fibers are inserted into the polymer for 30 seconds, subsequently impregnated with the SCM.
T03	-	Natural rubber Latex	Silica fume	The same procedure as T02 was used.
T04	-	Natural rubber Latex	Burned recycled mortar powder fines	The same procedure as T02 was used.
T05	-	Flaxseed oil	Rice husk ashes	The fibers are inserted in the oil for 30 seconds, subsequently impregnated with the SCM. No heat treatment was used.
T06	-	Flaxseed oil	Silica fume	The same procedure as T05 was used.
T07	-	Flaxseed oil	Burned recycled mortar powder fines	The same procedure as T05 was used.

D. Treatments

First, each fiber was immersed in one of the adherent solutions for 30 seconds, in this stage, the adhering solution goes around the fiber and generate a bonding layer, after this, pozzolanic materials were used as coating agents. In order to evaluate the effectiveness of treatment untreated fibers (T00) and the ones treated with calcium hydroxide (T01) were inserted. The different combinations are shown in Table 1.

E. Mortar Preparation

One mortar dosed by weight was prepared 1:5, and the relation water / cement 0.6. The cement and water were mixed at low speed for 5 minutes; after the aggregate was incorporated, it was mixed for about 10 minutes more. In the molds of 40 mm x 40 mm x160mm, the fresh concrete paste was layered compact and 170 fibers (Fig.3). The specimens were desmolded after 24 hours and were cured for 28 days in water.

F. Tests performed

1) Study of fibers morphology

Before the study, the fibers were coated with palladium by

sputtering, and then the morphology was analyzed with a scanning electron microscope "Nova NanoSEM 450".

2) Study of the chemical composition of SCMs

The chemical composition of the SCM was studied through X-ray fluorescence spectroscopy (FRX), with the portable XRF analyzer "S1 TITAN model 800".

3) Studies of the physical and mechanical properties of hardened mortars

The following tests were conducted: water absorption, porosity and density according to the UNE standard 83980 (Asociación Española de Normalización y Certificación, 2014), capillarity according the standard UNE-EN 1015 to 1018 (Asociación Española de Normalización y Certificación, 2003), flexural strength according to EN 12390-5 5 (EN, 2009). The universal test machine "SHIMADZU" at a tension speed of 0.8 mm / min was used. Finally, the following equations were applied:

Flexural strength:

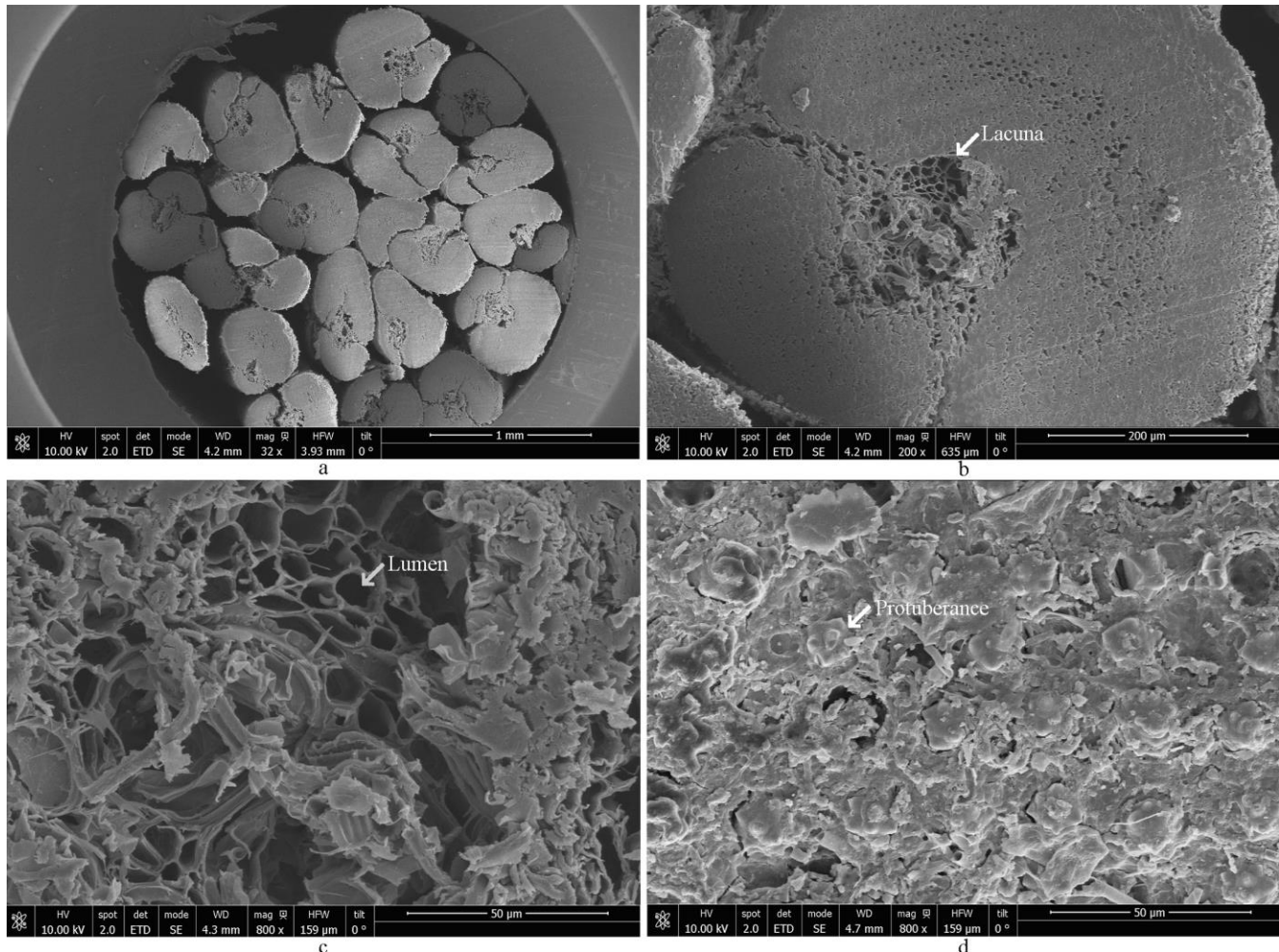


Fig.4. Piassaba morphology (*Aphandra natalia*) through scanning electron microscopy (SEM). Sample: (a) Cross sections, (b) Central fiber lagoon, (c) Lumen seen with greater magnification than b, and (d) Fiber surface.

TABLE 2
 PIASSABA'S PHYSICAL AND MECHANICAL PROPERTIES

Average length	Average diameter	Tensile strength	Elasticity	Aspect Ratio	Moisture content	Moisture absorption	Density at 12,63%	Density at 18.35%
(mm)	(mm)	(MPa)	Modulus (GPa)		(%)	(%)	(g/cm ³)	(g/cm ³)
20	0.35 – 0.40	88.4 – 183.2 (Balcázar-Arciniega & Hernández-Olivares, 2017)	1.1- 4.6 (d'Almeida, Aquino, & Monteiro, 2006a)	57.14 - 50	12.63	18.35	0.94297	1.16084

$$\sigma_f = \frac{3 \cdot F \cdot L}{2 \cdot b \cdot h^2} \quad (1)$$

Elastic modulus:

$$E_f = \frac{L^3 \cdot m}{4 \cdot b \cdot h^3} \quad (2)$$

Where σ_f is flexural strength (MPa), F is la maximum load (N), L is amplitude of the specimens in the three-point bending test (mm), E_f is elastic modulus (GPa), b y h are the width and thickness of the sample a (mm).

4) Study of the degradation of fibers in alkaline media

According to the procedure of (Jose et al., 2017), the fibers degradation that can occur in the concrete during the curing time is simulated. A sodium concentrated hydroxide solution to 1.7% with similar PH to the cement compounds (pH ~ 12.4) was used.

The treated and untreated fibers are immersed in the alkaline solution for about 98 days and these were weighted every 7 days, measurements are made with the analytical balance "OHAUS DV214C, 210g / 0.1mg".

5) Study of the effects of treatments on the fiber surface

To observe the damages suffered on the surface and in the cross-section of the fibers by the concrete alkalinity an optical

microscope was used.

III. RESULTS AND DISCUSSION

A. Fiber morphology

Table 2 shows the physical and mechanical characteristics of the fibers. The average length of the fibers is 20 mm, with diameters between 0.35 and 0.40 mm, and an aspect ratio between 57.14 and 50. In order to achieve an adequate adhesion between the fibers and the matrix, the aspect ratio around 60 is preserved (Tonoli, Joaquim, Arsne, Bilba, & Savastano, 2007), avoiding the excessive bonding that tends to break the fibers, instead of pulling out (Soroushian, Shah, Won, & Hsu, 1994).

According to (Ferrara et al., 2017), all natural fibers share a similar morphology:

- Each fiber consists of several fiber cells (microtubules).
- Each fibers cell is composed of the following parts : primary wall (the fibrils have a reticulated structure), secondary wall (the fibrils are arranged in spirals), tertiary wall (more internal and thin , has a fibrils structure parallel and encloses the lumen).
- The fiber cells are linked together by means of middle lamellae (hemicellulose and lignin).
- The cell walls are arranged in several fibrillated layers (lignin-bound fibrils).
- The fibrils are constructed of microfibrils. Are composed by cellulose chain coatings and bound by hemicellulose.

TABLE 3
 CHEMICAL COMPOSITION OF SUPPLEMENTARY CEMENTITIOUS MATERIALS

Chemical composition (mass %)	RHA	SF	BRMF
Silicon oxide (SiO ₂)	88.90	84.70	64.70
Aluminum oxide (Al ₂ O ₃)	-	-	12.50
Iron oxide (Fe ₂ O ₃)	0.13	1.16	4.34
Calcium oxide (CaO)	1.15	0.77	11.50
Magnesium oxide (MgO)	-	-	0.74
Potassium oxide (K ₂ O)	4.02	1.25	2.11
Sulfur (S)	0.24	0.13	0.44
Magnesium oxide (MnO)	0.32	0.11	0.11
Zinc oxide (ZnO)	0.01	0.41	-
Tin oxide (SnO ₂)	0.59	0.26	0.25
Phosphorus oxide (P ₂ O ₃)	0.90	-	-
Lead oxide (PbO)	-	0.14	-
Titanium oxide (TiO ₂)	-	-	0.52
Antimony oxide (Sb ₂ O ₃)	-	-	2.47
Σ (SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃)	89.03	85.86	81.54

TABLE 4
 PARTICLE SIZE DISTRIBUTION OF SUPPLEMENTARY CEMENTITIOUS MATERIALS.

Sieve (Nº)	Percentage that passes		
	RHA	SF	BRMF
35	0.76	0.25	0.21
45	1.70	2.52	2.33
60	5.18	18.84	8.90
70	16.19	15.64	72.07
120	20.50	38.88	7.77
200	49.48	16.69	6.00
230	6.12	5.22	2.18
325	0.07	1.95	0.54

The Fig.4 shows several cross sections of the fibers with

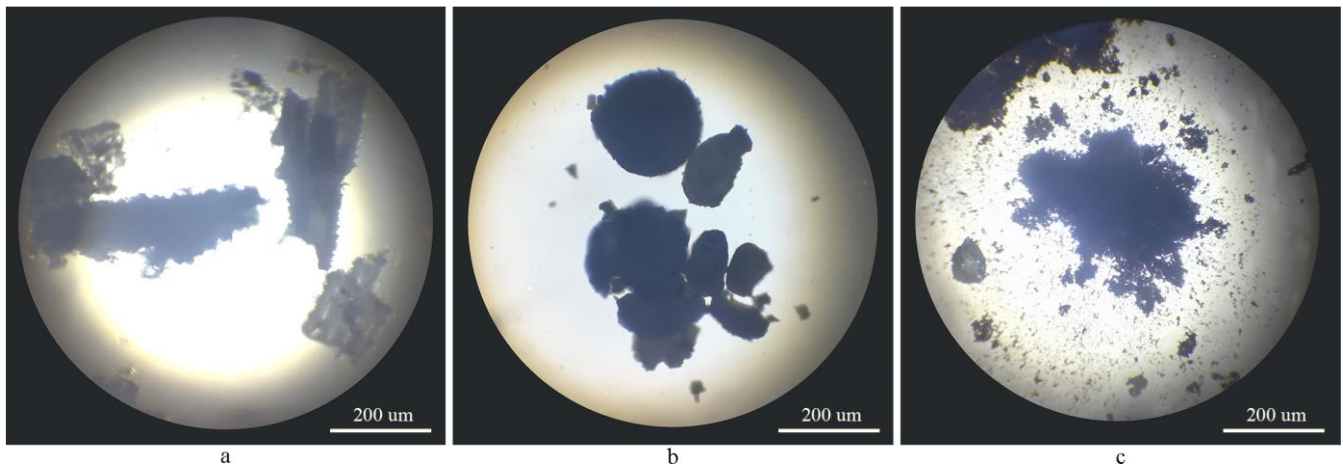


Fig.5. Optical microscopy of the SCM: (a) RHA, (b) SF, and (c) BRMF.

differences in size and shape. The Fig. 4b shows the transverse section of one of the fibers, it discloses in the central region a cavity called lacuna. The Fig. 4c shows channels (lumen) where nutrients and water flow along the fiber. The Fig. 4d shows the fiber surface covered with parenchymal cells along with protruding bodies that are shaped like a spinulase sphere (Nascimento, Ferreira, Monteiro, Aquino, & Kestur, 2012). The spinulases protuberances, which are rich in silica, cannot perfectly adhere to the fiber surface, and can be removed by abrasion, chemical attack or mechanical constraints (Sergio Neves Monteiro, 2009). Finally, the cross section of the piassaba fiber of *Aphandra natalia* is similar to the piassaba cross section of *Attalea funifera mart* (Aquino, Monteiro, & D’Almeida, 2003; Elzubair, Chagas Bonelli, Miguez Suarez, & Biasotto Mano, 2007).

B. SCM chemical composition and morphology

The Table 3 shows the oxide composition of the SCM: rice husk ashes (RHA), Silica fume (SF), and burned recycled mortar powder fines (BRMF). The presence of silicon oxide is greater than 70% in the SCMs. At the same time, the amount of

$SiO_2 + Al_2O_3 + Fe_2O_3$ exceeds 70% in SCMs, in fact, they have similar pozzolanic characteristics to Class F fly ash (ASTM C618 and EN 450-1) (ASTM, 2012; EN, 2012). On the other hand, BRMF unlike RHA and SF contains Al_2O_3 , MgO, TiO_2 , and Sb_2O_3 .

Table 4 shows the particle sizes of the SCMs: RHA shows smaller size particle than SF and BRMF. The Fig.5 shows the morphology of SCM: RHA present flat forms angular, SF have spherical shapes, and BRMF present flat forms amorphous.

C. Physical properties of hardened mortars

1) Calcium hydroxide treatment

Fig.6a and Fig.6b show the density values: T00 and T01 have higher values than TES. Fig.9a shows the values of flexural strength: T00 and T01 have lower values than TES. This is due to the low proportion of fibers in the mixture (0.3%), consequently to a high compaction between concrete and fibers (MA Ismail, 2007).

The Fig.7b shows the values of the water absorption coefficients: T01 has a lower value than TES and T00. The alkaline treatment imparts hydrophobicity to the fibers, when a

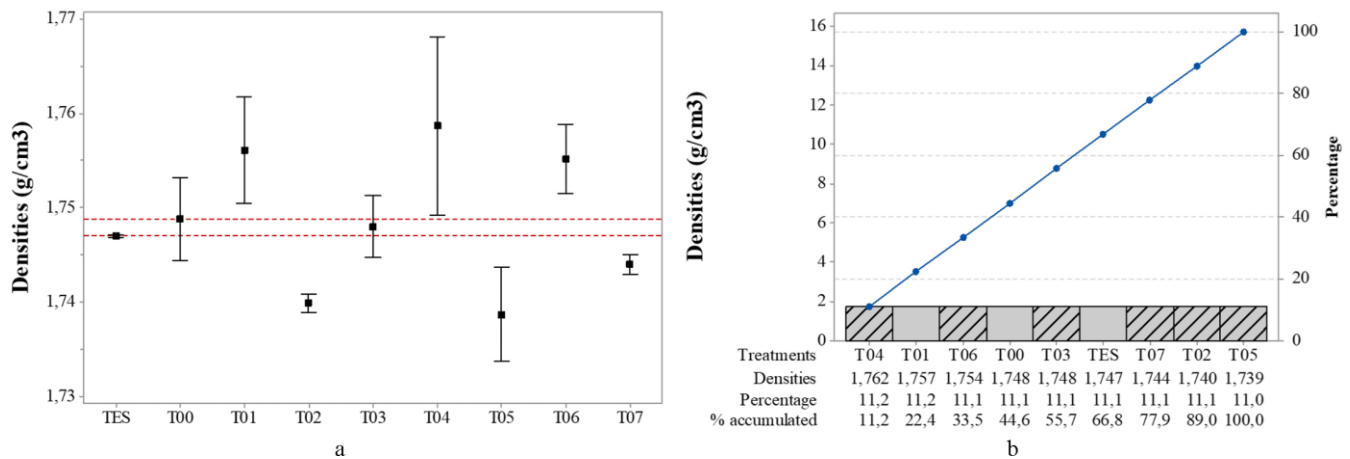


Fig.6. Densities of hardened mortars: (a) Interval graph, and (b) Pareto diagram.

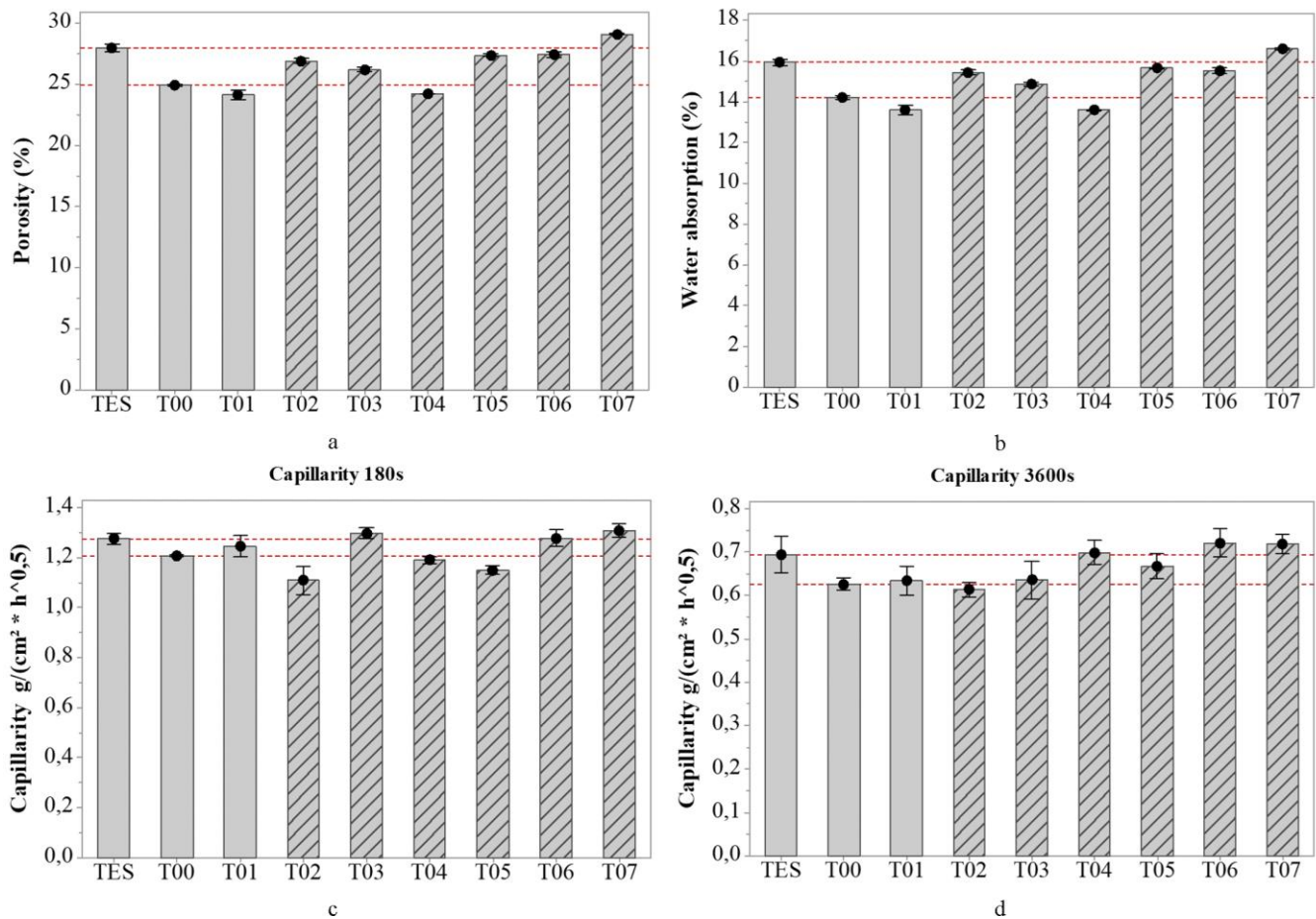


Fig.7. Physical properties of hardened mortars: (a) Porosity, (b) Water absorption, (c) Capillary at 180 seconds, and (d) Capillary at 3600 seconds.

part of lignin and hemicellulose is deleted (Ozerkan, Ahsan, Mansour, & Iyengar, 2013). In addition, it eliminates impurities and breaks down the fiber bundle, into smaller fibers, and increasing the effective area of the fiber surface (Onuaguluchi & Banthia, 2016).

Fig.7c and the Fig.7d show the capillary values to 180 seconds and 3600 seconds, respectively: T00 and T01 have lower values than TES. The alkaline treated fibers and not treated slows the capillary ascending speed of concrete by the high compaction between the matrix and the fibers.

2) Treatments with natural rubber latex- SCM

Fig.7b shows the water absorption coefficients by immersion: T02, T03 and T04 have lower values than T05, T06 and T07, respectively. This implies that the natural rubber latex is better waterproofing for the fibers than the flaxseed oil.

The Fig.6a and the Fig.6b show the density results. On the one hand, T02 has the same density as T05 and lower density than the rest of samples, this is due to the large amount of RHA in the mixture (Obilade, 2014; Yuzer et al., 2013). On the other hand, T04 has a higher density than the rest of the samples, this is because part of the holes that are not occupied by the sand are

filled with BRMF (which are smaller), as the amount of BRMF increase in the mixture, less voids will be occupied by water. Causing a more compact mortar (Braga, De Brito, & Veiga, 2012). Finally, due to the nature of the treatment, it is very difficult to control the amount of SCM attached to natural rubber latex.

The Fig.7c and the Fig.7d shows the capillary results to 180 seconds and 3600 seconds. On the one hand, T02 has a lower value than T00, T03 and T04. On the other hand, T04 has a higher value than T00, T03 and T04. In complement: Fig.8a shows that the RHA particles adhere homogeneously to natural rubber latex, some of these particles (in lesser amount than BRMF) when they are separated from the polymer have sizes and colors similar to the s RHA dries or reactive. Likewise, the Fig.8b shows the SF particles adhere evenly to the natural rubber latex, the color of the adhered particles is similar to the SF dry or reactive. Finally, Fig.8c shows that BRMF particles adhere homogeneously to natural rubber latex, some of those particles (in greater quantity than RHA) when detached from the polymer have similar sizes and colors to the fine of BRMF dry or reactive. This implies:

- The impregnated fibers with natural rubber latex and



Fig.8. Combined treatments: (a) Natural rubber latex- RHA, (b) Natural rubber latex- SF, (c) Natural rubber latex- BRMF , (d) Flaxseed oil- RHA, (e) Flaxseed oil- SF, (f) Flaxseed oil- BRMF.

RHA reduce the capillary of the concrete, because of the RHA fines that are separated from the natural rubber latex, fill the holes that the sand doesn't, causing an effect of pore filling that decreases the demand for water (Alex, Dhanalakshmi, & Ambedkar, 2016; Rukzon, Chindaprasirt, & Mahachai, 2009).

- The impregnated fibers with natural rubber latex and BRMF increase the concrete capillary due to the BRMF are very porous (Corinaldesi & Moriconi, 2009). That porous nature is responsible for the high capacity of water absorption that they present (Narud, n.d.), which will increase in proportion to the decrease in particle size (Poon, Shui, & Lam, 2004), and the amount of cement paste bonded to the surface of the particles (Butler, West, & Tighe, 2011).
- The adhesion homogeneity and the SCM color over the natural rubber latex entails the polymer and SCM compatibility.

3) Treatment with flaxseed oil- SCM

The Figures 7b, 7c and 7d show the results of water

absorption and capillary: the T05 value is greater than T02, the value of T06 is greater than T03, and the value of T07 is greater than the rest of samples. In addition: Fig. 8d shows that RHA particles adhere irregularly to flaxseed oil (to a greater extent than SF and BRMF), some particles that separate from the oil, are attached and darker in color than RHA dry or reactive. Likewise, Fig.8e shows that SF particles irregularly adhere to flaxseed oil, some particles that separate from the oil, are attached and darker in color than dry or reactive SF. Finally, Fig.8f shows that BRMF particles irregularly adhere to flaxseed oil, some particles that separate from the oil, are attached and darker in color than dry or reactive BRMF. This implies:

- Flaxseed oil waterproofs the fibers in the concrete (Sellami, Merzoud, & Amziane, 2013), to a lesser extent than the natural rubber latex by its slow polymerization process.
- Chemical reactions between flaxseed oil and SCMs, in greater intensity with SF and BRMF, damaging the particles quality, thus increasing its porosity and water absorption.

D. Mechanical Properties of hardened mortars

The Fig.9a and 9b show the mechanical properties of hardened mortars after 28 days of curing. After the laboratory

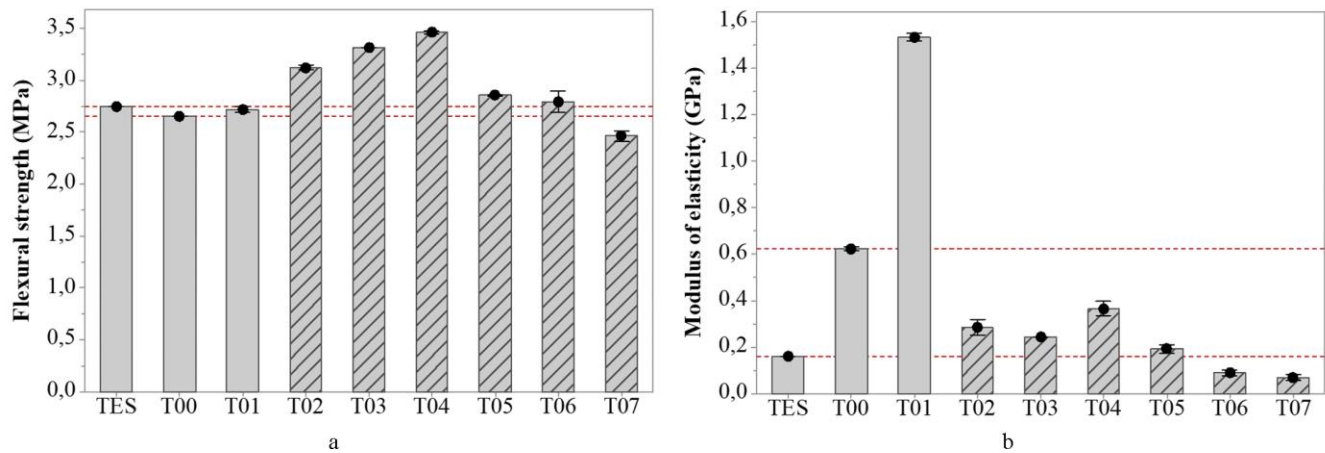


Fig.9. Mechanical properties of hardened mortars of 28 days of curing: (a) Flexural strength, (b) Modulus of elasticity.

tests, the flexural strength and elastic modulus were calculated.

1) Calcium hydroxide treatment

T00 and T01 have lower flexural strength values than TES. This is because, for one part, to the chelating effect between the pectins of the fibers and calcium ions, which decreases the amount of calcium (main hydration product of the cement) to form calcium silicate hydrates (C-S-H), which delays the hardening of the composite material (Sawsen et al., 2015). On the other hand, the water soluble sugars, consequential of the alkaline hydrolysis of lignin and partial solubilization of the hemicellulose contained in the vegetal fibers, causing the delay of the setting time and the reducing of the hydration heat of concrete (Bilba, Arsène, & Ouensanga, 2003).

T01 has greater flexural strength and greater modulus of elasticity than T00. Alkaline treated fibers increases the flexural strength and modulus of the hardened mortars (Hussain & Ali, 2019; Sabarish, Paul, Bhuvaneshwari, & Jones, 2019). This is due, on the one hand, to what it causes in vegetal fibers: rougher surfaces, diameter decrease and decrease in tensile strength (Suardana, Piao, & Lim, 2011). On the other hand, the treatment with calcium hydroxide, leads to the nodules deposit that contains calcium on the surfaces of the fibers, resulting in a strong interaction between calcium ions and pectin molecules, this interaction causes elimination of most of the amorphous components, leaving molecules with greater organization and heat resistant (Le Troedec et al., 2008).

2) Treatments with natural rubber latex- SCM

T02, T03 and T04 have higher values of flexural strength than TES and T00. Treatments with natural rubber latex and SCM maintain the mortar elasticity modulus between the values of TES and T00. This is due to the greater load capacity and the low deformation obtained by the vegetal fibers with a coating layer formed by the interaction of the latex film and the pozzolan inside the composite material (Jose et al., 2017).

T02 has lower flexural strength and greater elasticity

modulus than T03. The decrease in flexural strength is due to the greater amount of RHA fines in the mixture and the greater RHA porous structure compared to SF. The increase in the elasticity modulus is due to the effectiveness of RHA fines in pore filling (Foong, Alengaram, Jumaat, & Mo, 2015). Although the studies have shown that the addition of RHA in the concrete can match, and even overcome the concrete resistance with additions of SF, and this is conditioned by:

- (1) The amount of RHA in the mixture: the 5% of replacement of cement can increase strength, being the 10% of replacement, which contributes most to the reduction of large crystals of calcium hydroxide, and ettringite result of the C-S-H additional phases generated by the pozzolanic reaction. In contrast, additions of 15% and 20% can weaken the concrete by the role of porous microaggregates that perform the abundant RHA particles (Le, Nguyen, & Ludwig, 2014).
- (2) The particle size: to actively participate in the pozzolanic reaction, it is required a particle size less than 45 μm (Nehdi, Duquette, & El Damatty, 2003). To achieve an index of 100% pozzolanic activity, a medium particle size of approximately 8 μm is required (Bouzoubaâ & Fournier, 2001). It does not achieve a significant pozzolanic activity with particle size less than 5.6 microns (Nguyen, 2011).
- (3) The porous structure: the thicker RHA particles with larger pore volume influence in the cement hydration process. At young age, they absorb large amount of water and reduce the cement dilution effect (Le, 2015). At later ages, the released water from the particles maintains the cement continuous hydration (Van Tuan, Ye, Van Breugel, Fraaij, & Dai Bui, 2011).

T04 has greater flexural strength and greater elasticity modulus than T02 and T03. The increase in flexural strength is

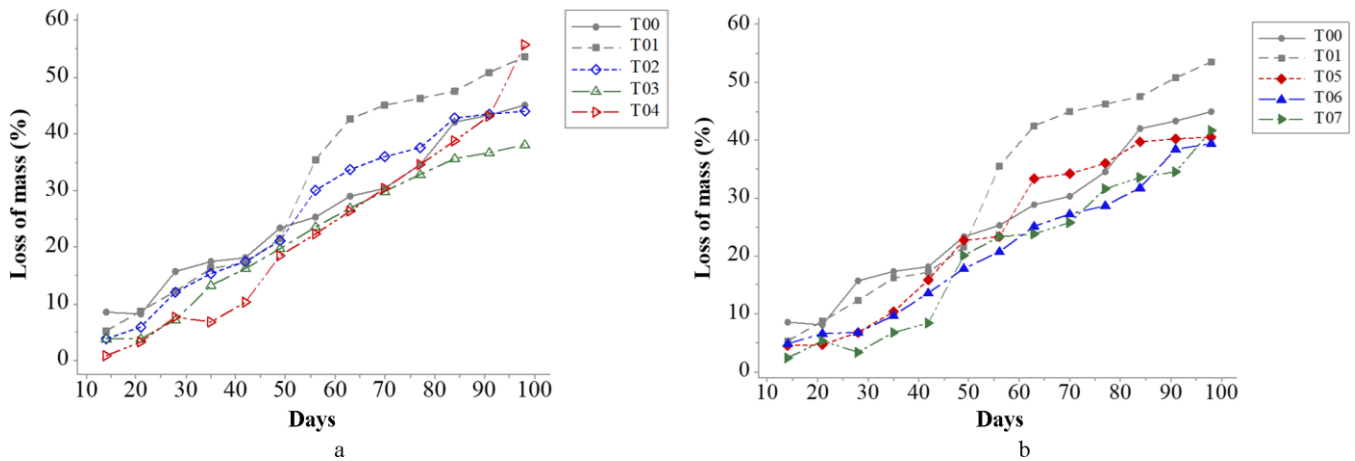


Fig.10. Loss of fiber mass with various treatments: (a) Natural rubber latex- SCM, (b) flaxseed oil- SCM.

due to the amount of BRMF fines incorporated into the mixture (Braga et al., 2012). The increase in the modulus of elasticity is due to the greater amount of recycled aggregates in the mixture (Chakradhara Rao, Bhattacharyya, & Barai, 2011), and its lower quality (Behera, Bhattacharyya, Minocha, Deoliya, & Maiti, 2014a).

3) Treatment with flaxseed oil- SCM

T05, T06 and T07 show greater flexural strength and lower elasticity modulus than T02, T03 and T04. Chemical interactions between flaxseed oil and SCMs decrease or mitigate the pozzolanic effects of SCMs. On the one hand, T05 shows greater flexural strength and greater elasticity modulus

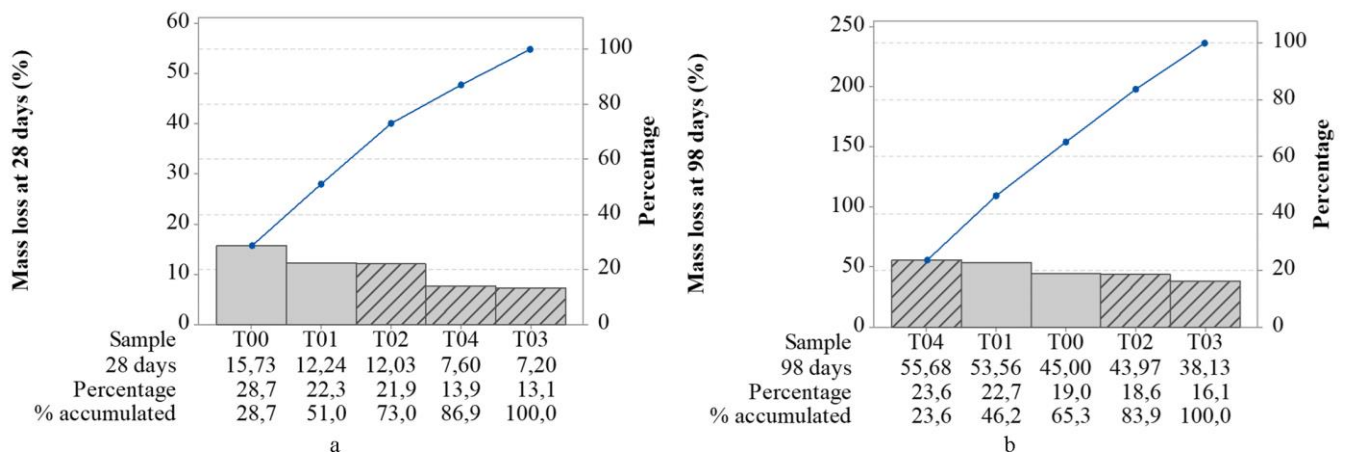


Fig.11. Pareto diagrams of mass losses of fibers treated with natural rubber latex- SCM: (a) 28 days trial, and (b) 98 days trial.

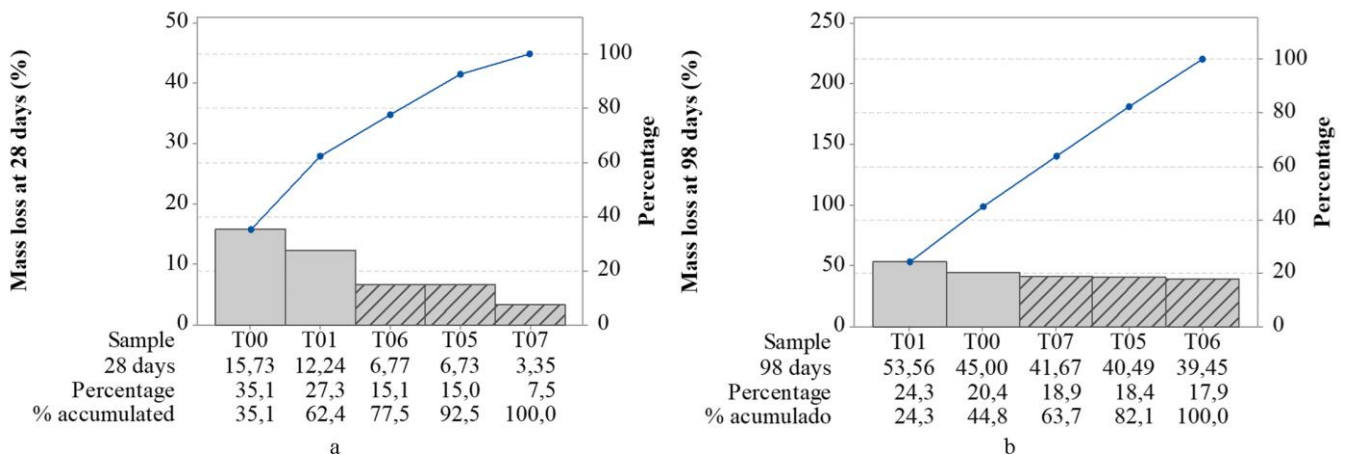


Fig.12. Pareto diagrams of mass losses of the fibers treated with flaxseed oil- SCM: (a) 28 days trial, and (b) 98 days trial.

than T06 and T07, caused by the greater amount of RHA's silicon oxide and low reactivity with the oil. Moreover, T07 shows less bending strength and lower elasticity modulus than other treatments by the amount of cement paste bound the surfaces of BRMF particles, chemically reactive with oil, and inhibiting the BRMFs pozzolanic effects.

E. Degradation study

1) Calcium hydroxide treatment

Fig.10a and Fig.10b shows the fibers mass loss with various treatments at 28 and 98 trial days. They were used control fibers (untreated) exposed to the same alkaline solution that other treatments. T01 has less mass loss at 28 days and greater mass loss at 98 days than T00. The calcium hydroxide treatment when remove some of the fibers amorphous components, improves the hydrophobic condition, stops the degradation process, but accelerates the mineralization (Ardanuy, Claramunt, & Toledo Filho, 2015).

2) Treatments with natural rubber latex- SCM

Fig.11a and Fig.11b show the mass losses of the combined natural rubber latex- SCM treatments at 28 days and 98 days of testing.

At 28 days: T02 has a lower degradation degree than T00, greater than T03 and T04, and similar to T01. At 98 days: T02 has a lower degradation degree than T00, T02 and T04, and greater than T03. I.e., the combination of RHA natural latex and SF natural latex, decrease the fibers degradation process in alkaline media, when it is import the hydrophobicity and decreases of the alkalinity. This results in a fibrous concrete with RHA additions whit better initial flexural strength and greater durability, than matrices with thick RHA additions and fly ash (Wei & Meyer, 2016).

At 28 days: T04 have a lower degradation degree than T00, T01 and T02, and greater than T03. At 98 days: T04 has a higher degradation degree than T00, T01, T02 and T03. I.e. T04 at the same like T01 reduce the fibers degradation, but accelerates the mineralization process. This results in a fibrous cement with BRMF additions of better resistance to initial

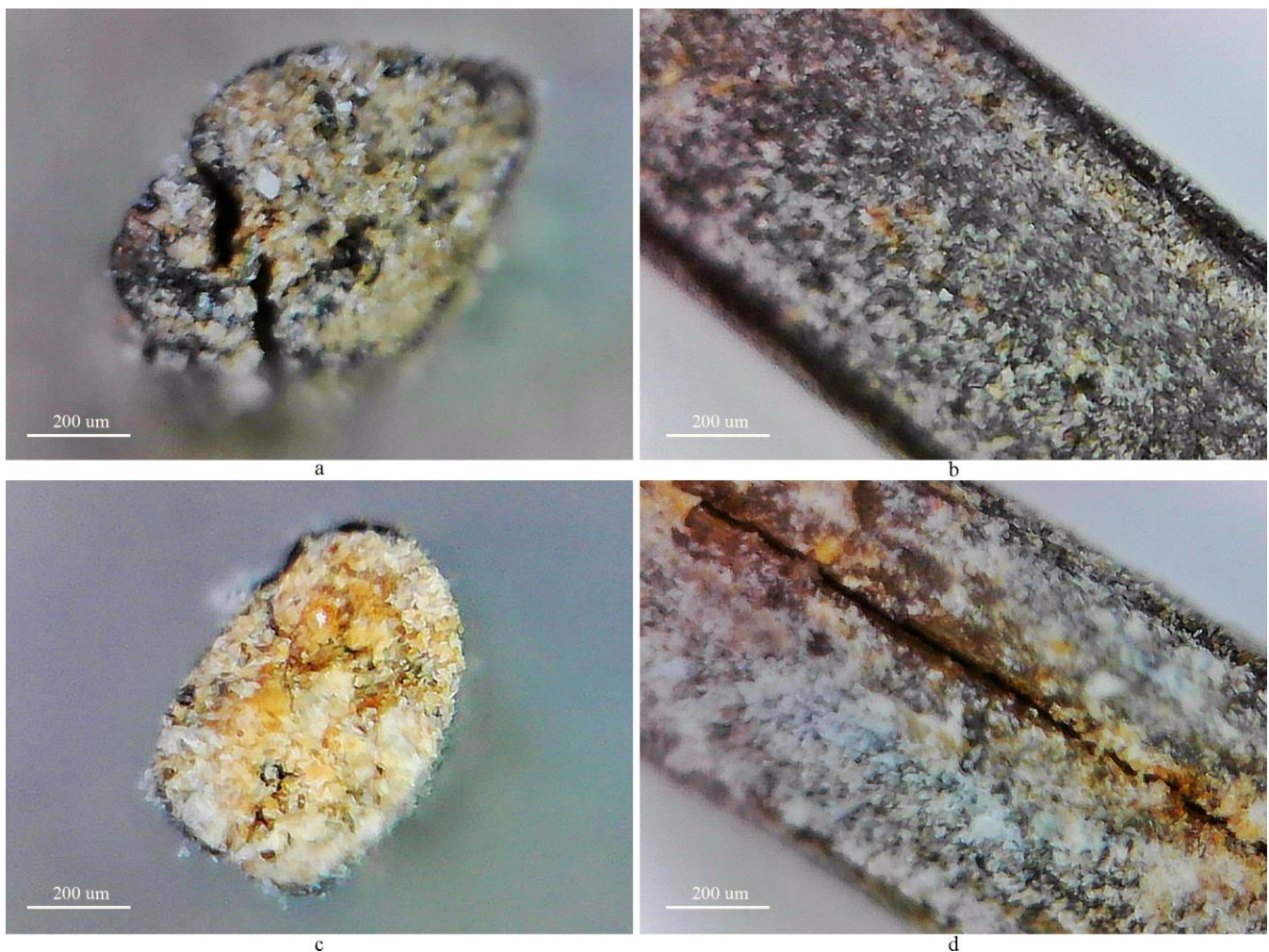


Fig.13. Microscopy of the extracted fibers from the hardened mortar after 28 days of curing: (a) Cross section of T00, (b) Fiber surface of T00, (c) Cross section of T01, and (d) Fiber surface of T01.

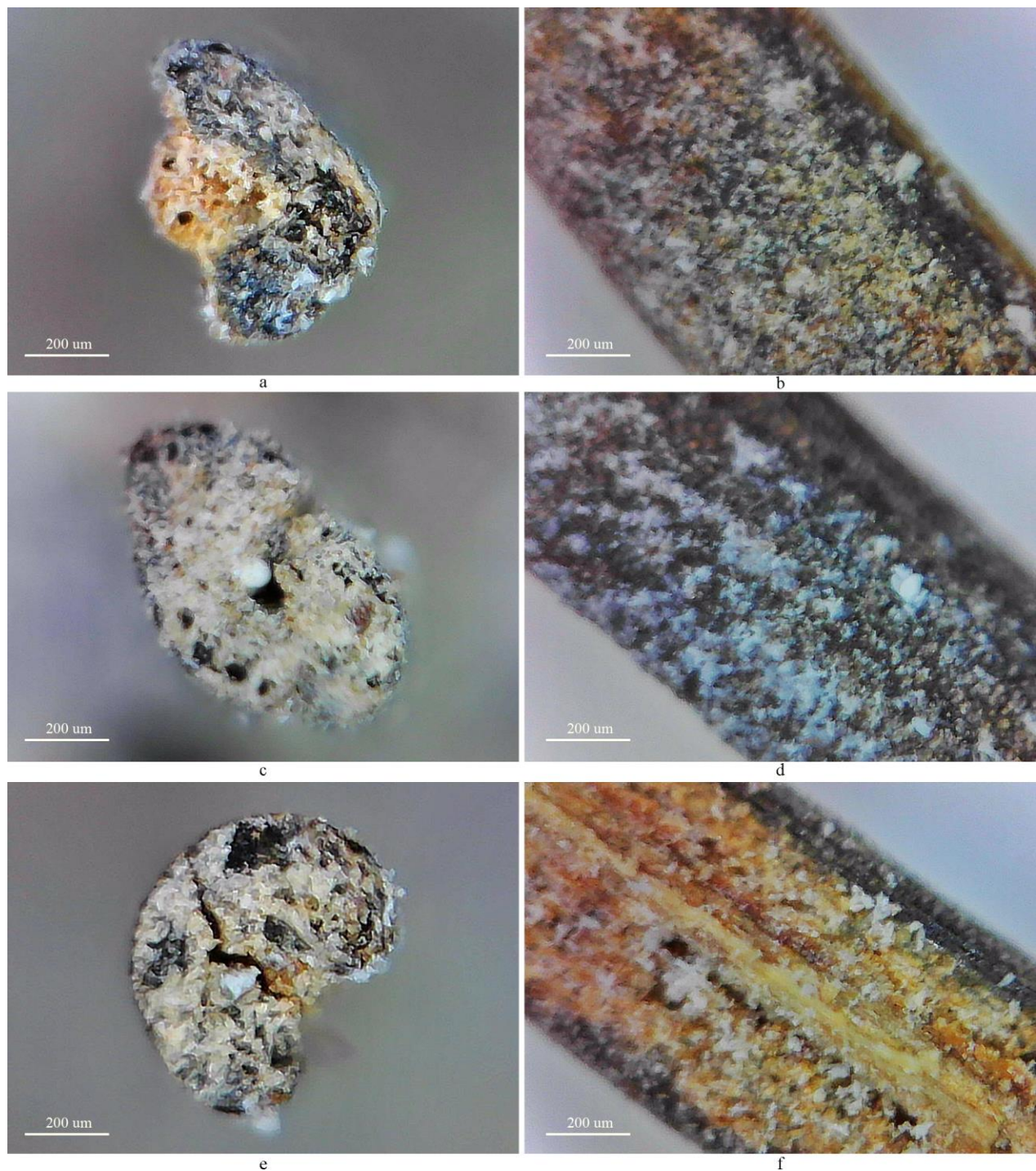


Fig. 14. Microscopy of the extracted fibers hardened mortar to 28 days of curing: (a) Cross section of T02, (b) Fiber surface of T02, (c) Cross section of T03, (d) Fiber surface of T03, (e) Cross section of T04, and (f) Fiber surface of T04.

bending, but with a decrease in its durability compared to the one of the conventional concrete (Behera, Bhattacharyya, Minocha, Deoliya, & Maiti, 2014b).

3) Treatment with flaxseed oil- SCM

Fig.12a and Fig.12b show the mass losses of the combined treatments of flaxseed oil- SCM at 28 days and 98 days of testing.

A 28 days: T05 and T06 exhibit similar degradation degrees, less than T00 and T01, and higher than T07. At 98 days, T05 and T06 have a lower degradation degree than T00, T01 and T07. For T05 and T06 there is a correspondence between the fibers mass loss and the mortars flexural strength. This results in a fibrous concrete with RHA and SF additions of better initial flexural strength and greater durability than the reinforced with untreated or alkaline treated fibers concrete.

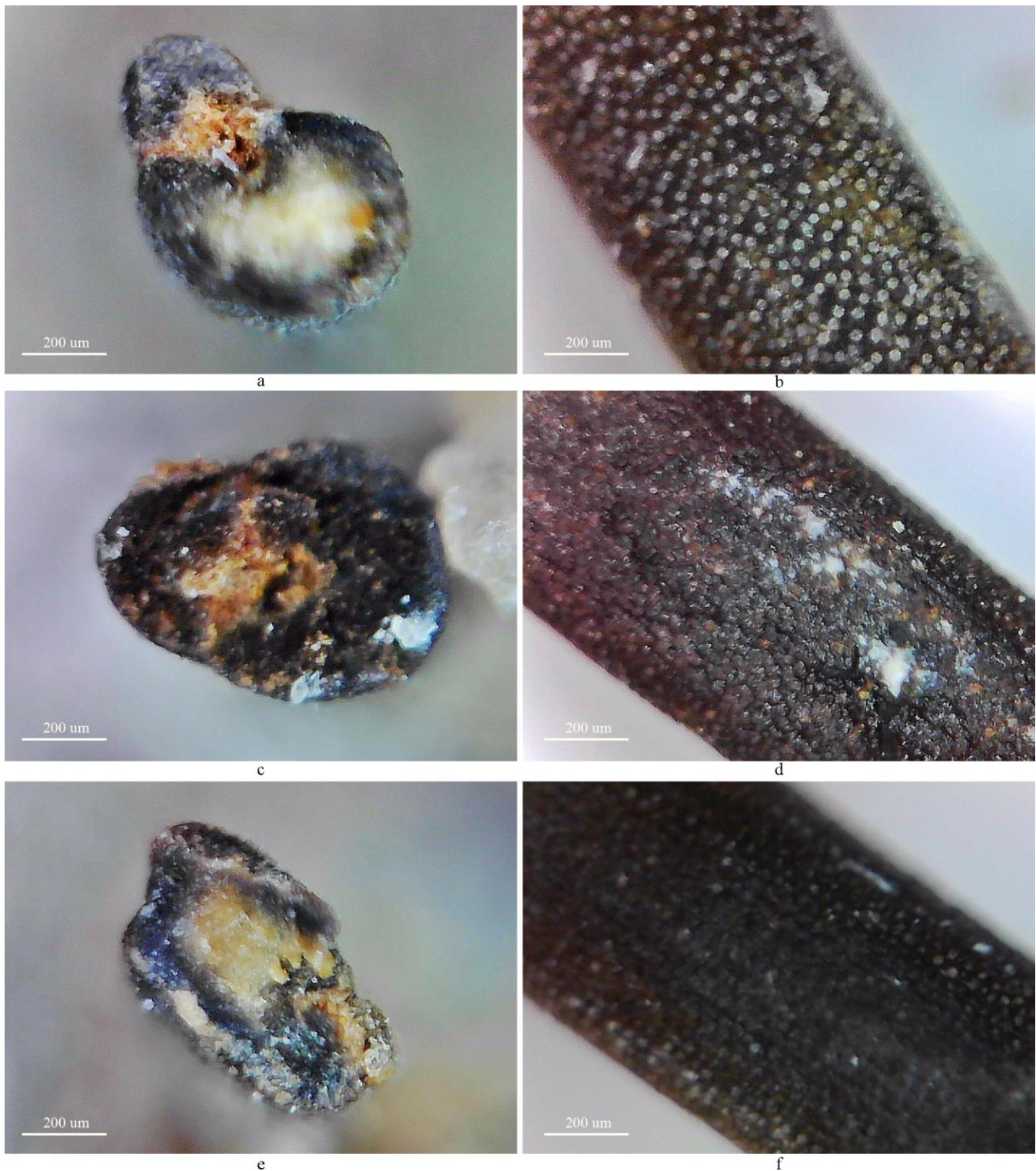


Fig. 15. Microscopy of the fibers extracted from the hardened mortar to 28 days of curing: (a) Cross section of T05, (b) Fiber surface of T05, (c) Cross section of T06, (d) Fiber surface of T06, (e) Cross section of T07, and (f) Fiber surface of T07.

To T07, it was not found the correlation between the fibers mass loss and the mortars flexural strength. This by the greater time exposure to air and at the fibers room temperature with the treatment tested in concrete, compared to the fibers with the treatment tested in the alkaline solution. I.e., the chemical

interaction between flaxseed oil and BRMF advances as the polymerization of the oil. This interaction accentuated with BRMF by the porous structure and the amount of cement paste bonded to the particles surface.

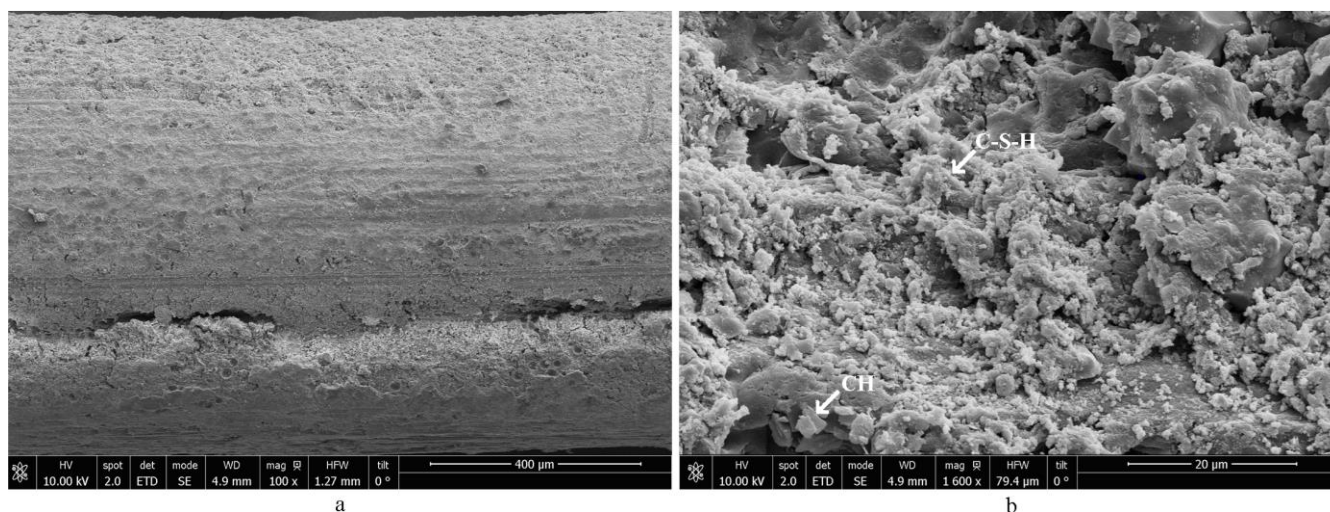


Fig. 16. Scanning electron microscopy (SEM) of a piassaba fiber previously treated with $\text{Ca}(\text{OH})_2$ obtained from the cement matrix at 28 days, which shows: (a) Fiber surface in longitudinal section, and (b) fiber surface with deposits of CH and C-S-H, seen in longitudinal section and in greater increase than a.

F. Optical microscopy

1) Calcium hydroxide treatment

The Figures 13a and 13b shows the surface of a non-treated fiber, removed from the matrix after the flexural strength essay. The figures 13c and 13d show the surface of a fiber treated with calcium hydroxide, withdrawal of the matrix after the flexural strength test. The greater whitening of the treated with calcium hydroxide fibers is due to the removal of residual lignin and cell wall extracts (depolymerization). This process improve the bonding of fiber- matrix (the bonding is failure reduced by fracture failure extraction, reducing the composite material ductility), but brings mineralization and fragileness of the fibers (Onuaguluchi & Banthia, 2016).

Figures 16a and 16b show the surface of a treated fiber with calcium hydroxide, removed from the matrix after the flexural strength test. The calcium silicate hydrate gel (C-S-H) and the portlandite (CH) on the fibers surface improves the fiber-matrix bond (Anggraini, Asadi, Syamsir, & Huat, 2017).

2) Treatment with natural rubber latex- SCM

Regarding to T02, the Fig.14a and Fig.14 b show along the fiber surface, the CH crystals formation and a C-S-H white gel, in greater intensity than T0 3 and T04. With respect to T03, Fig.14 c and Fig.14d show in parts of the fiber surface, the formation of CH crystals and a white C-S-H gel, at a lower intensity than T02. With respect to T04, Fig.14e and Fig.14f show in some parts of the fiber surface, the formation of CH crystals and a white C-S-H gel, at a lower intensity than T02 and T03. The highest speed of the BRMFs pozzolan reaction is the cause of the least amount of calcium compounds on the fiber surface.

3) Treatment with flaxseed oil- SCM

Regarding to T05, the Fig.15b show on the surface of the light brown fiber to a white gel formed by CH and C-S-H. With respect to T06, Fig.15d shows on the surface of the dark brown

fiber (healthy fibers characteristic) a greater amount of white gel formed by CH and C-S-H than T05 and T07. Regarding T07, Fig. 15f shows on the surface of the light brown fiber a (more intense than T05), a white gel formed by CH and C-S-H. That is, T05 and T07 have more damage than T06, and T07 has more damage than T05. This is due to the RHA porous structure and the BRMF, responsible for the greater chemical interaction with flaxseed oil and fiber mineralization.

IV. CONCLUSIONS

In the present research are studied the effects of various treatments on the piassaba fibers, through the physical and mechanical properties of the hardened mortars, the fibers optical microscopy that are separate from the mortars at 28 days, and the fibers mass loss in an alkaline solution. The following is concluded:

- The flexural strength results of the 28 days curing hardened mortars and the fibers optical microscopy show that treatments with natural rubber latex - SCM improve the mortar performance and decrease the fibers degradation.
- The size, shape, porous structure, and chemical composition of SCMs determine the compatibility with the polymer and oil. The results of the physical and mechanical properties of the hardened mortars, as well as the tests of fibers's mass loss in alkaline media, show that the RHA have greater compatibility with the polymer and the oil.
- Combination treatment of natural rubber latex - BRMF, shows the best mechanical performance at 28 days, however, the high capillary high values and the fibers accelerated degradation (even higher than the alkaline treatment), compromise the mortar durability to more than the 28 days of alkaline exposure.

ACKNOWLEDGEMENTS

The authors thank the technical support to María del Cisne Guamán, Diego Mata and Juan Carlos Quintuña.

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