Materials Research. 2021; 24(suppl. 2): e20210328
DOI: https://doi.org/10.1590/1980-5373-MR-2021-0328

Moringa *Oleifera* Seed Peel Structure and Its Performance in Cementitious Composite

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Received: June 30, 2021; Revised: February 10, 2022; Accepted: February 13, 2022

This article seeks to characterize the seed husk fiber of Moringa *oleifera* and understand its influence when added to a cementitious composite, in terms of mechanical performance. Moringa fibers were chemically and physically tested and were added to a cementitious composite. Specimens were molded for Ultrasonic Speed Pulse test and Uniaxial Compression Strength test, and subsequent observation in SEM. The results show a fiber with high lignin content and high absorption of water. Adding fiber to the composite, the water in the mixture is absorbed, which reduces the formation of hydrated cement compounds over time. Consequently, it results in a composite with low mechanical strength. The fiber/matrix interface analyzed in the micrographs is porous, has microcracks and a high concentration of calcium hydroxide. Despite this, the same lignin content that impairs mechanical strength in this composite is what makes the fiber resistant to weathering. More studies regarding the effectiveness of this quality should be carried out.

Keywords: *Natural fiber composites, Moringa oleifera, fiber structure, mortar, cement composites.*

1. Introduction

The civil construction industry has presented numerous technological advances, aiming to reduce costs and increase the efficiency of materials, as is the case of materials with cement matrix. One of the improvements made was the incorporation of natural fibers in the cement matrix. The natural fiber used in this kind of composites presents a solution of low cost, good availability and low environmental impact[1].

1.1. Moringa oleifera

The fiber chosen for use in this work is the seed peel of Moringa *oleifera*. Moringa has the ability to grow in places with nutrient-poor soils and a hot, dry climate (as is the case in the arid and semi-arid regions of Brazil). Regarding its cultivation, the moringa has a high growth speed, ease of propagation and the ability to accept large pruning[2].

The roots, leaves, fruits and seed have several applications, such as industrial (oil extracted from the seed in the cosmetic industry), medicinal (all parts of the plant), flavoring (roots), and fuel (wood and oil)[3]. The potential for exploration and use of moringa has attracted the scientific community and producers around the world. When working with this type of material, it is necessary, in addition to analyzing the behavior of the fibers reinforcing the matrix, to also analyze the behavior and properties of the pure fiber[4].

Moringa *oleifera* has already been used as reinforcement in polymer composites. Studies have successfully developed the composite of polyethylene terephthalate (PET) with mercerized fibers from the rind of moringa fruit. In addition to improving the mechanical properties of the PET matrix (reaching 65.92 MPa tensile strength and 98.49 MPa flexural strength), it also confirmed greater thermal stability of the composites[5]. Other studies indicated that moringa fiber can be considered as an ecological fiber and with potential for use in bio composites with application in engineering products[6]. Successful application of this fiber is also found in polypropylene (PP) matrices[7] and in high density polyethylene (HDPE) matrices[8].

However, the use of moringa fiber in non-polymeric matrices, such as the cement matrix, is still an open field of studies. Works using moringa seed powder with and without shell as a natural polymer in modified mortars for greater resistance to aggressive environments proved to be very promising, as the moringa powder acted as an additive that coagulates metallic ions in water[9,10].

1.2. Plant histology

Plant histology is the specific study of plant tissues. Tissues are groups of cells that generally perform the same

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functions[11]. Vegetable fibers basically consist of: cellulose, hemicellulose and lignin, which are polar molecules. Coexisting with lower percentages of pectin, wax and water-soluble substances. The individual percentage of these components varies with different types of fibers[12]. The variation can also be affected by growing and harvesting conditions.

Cellulose is a semi-crystalline polysaccharide and is responsible for the hydrophilic nature of fibers. Hemicellulose is a completely amorphous polysaccharide with lower molecular weight compared to cellulose. The amorphous nature of hemicelluloses results in them being partially soluble in water and alkaline solution[13]. Cellulose and hemicellulose are linked by hydrogen bonds and together are known as holocellulose, which is considered a structural component of fiber. It is the major constituent of fibers, usually present around 65-70% of plants dry mass[11,13]

Lignin is an amorphous polymer. It is a hydrophobic component of the fiber and acts as a cementation material, which gathers the various elementary fibers, forming the so-called technical fiber. The main function of the lignin is to make the cell wall rigid.

1.2.1. Epidermis

The epidermis is the outermost part of the plant organs and is in the direct contact with the external environment, its main function being the coating. It prevents the invasion of pathogens, restricts water loss and, at the same time, allows its passage with mineral salts, in addition to carrying out gas exchange with the environment[11].

2. Methodology

In this work, it was sought to analyze the structure of the Moringa *oleifera* husk added in five different mortar mixes in order to evaluate the possibility of its use in cementitious composites. The following topics describe the materials used and the methods to develop it.

2.1. Materials

The research was divided into stages, starting with the choice of the fiber source plant. Moringa *oleifera* was chosen, focusing on seed husk fibers. The material was provided by the Laboratory for Synthesis and Development of Polymers and Composites (LAPCOM) at UFOP.

As a matrix, the cement chosen was a national brand Portland cement with High Initial Strength coded CP V-ARI. It was chosen because it contains fewer additions and therefore fewer analysis variables (Table 1). The sand used is the common type, from the river. The fraction used is that which passed through the #16 Tyler sieve. For this experiment, it was used oven dried. The water used in the mixture was taken directly from the supply network managed by the Ouro Preto Municipal Water and Sewage Service (SEMAE).

Reference dosages were made in accordance with standard[14]. The mixtures containing the addition of moringa fiber were made in three stages:

1) Cement and half of the water were added I the mechanical mixer, which was mixed for one minute at low speed;
2) Then, the rest of the water was added and the mixture was submitted to high speed for thirty seconds;

Table 1. Composition of High Initial Strength Portland cement.

Type of cement portland	Abbreviation	Composition (mass percentage)	
		Clínquer + plaster	Carbonate material
High Initial Strength	CP V-ARI	100-95	0-5

3) After that, the fiber, *in natura*, was added and the mixture was homogenized at low speed for one minute and thirty seconds.

Dosages containing 2.5%, 5.0%, 7.5% and 10.0% w/w of moringa fibers with respect to the cement mass of each reference dosage were prepared. High-strength concretes need a greater volume of fibers to change the ascending branch of compressive strength *versus* specific deformation[15].

2.2. Methods

2.2.1 Fiber

To a better understanding of the properties of this fiber, in addition to the existing literature[16], the fiber was prepared for tests according to TAPPI standards[17,18]. The lignocellulosic materials were reduced to less than 1.0 mm in an industrial blender and then sieved to 40/60 mesh fraction.

The chemical characterization of total extractives content, ash content and lignin content were carried out, according to TAPPI standards[19-21].

Physical characterization was performed in the laboratory. The natural moisture, the water absorption content and the specific mass of the fiber was determined. The moringa seed peel morphology was determined by SEM operating with Secondary Electrons (SE).

2.2.2. Cementitious composite

The dosage choice was made through the consistency test (flow-test), which gave the water/cement ratio of the reference mixtures, shown in Table 2. The dosages are shown following the order: cement:sand:water:fiber and was chosen to represent the dosage in volume, which is most commonly used in fiber addition studies.

For each mixture four cylindrical specimens (5x10 cm) were manufactured and subjected to curing in a humid chamber at 26±1°C. At seven days old, those specimens were tested to determine the ultrasonic pulse speed that travels through them and evaluate the quality of the composite produced[22]. After that, they were tested for uniaxial compression strength[23].

To obtain the speed of the ultrasonic pulse, the TICO equipment model from PROCEQ Testing Instruments was used, with 54 Hz flat transducers. The mechanical tests were performed on the EMIC DL 20000 universal mechanical testing equipment. Those tests were performed at the Materials Laboratory of Civil Construction at UFOP.

The performance of the composite is related to the volume of fibers added and how it interacts with the matrix[24]. In order to investigate how the fiber is dispersed and acts in the matrix, after the destructive tests, samples of the mortar were put in isopropyl alcohol to stop the cement hydration. These samples were coated with epoxy resin to

Table 2. Used dosages.

Type	Identification	Natural Fiber %	Dosage	Materials Consumption (g)			
				Cement	Sand	Water	Fiber
1:2	REF-01	0	1:2:0.72:0	391,4	1143,8	281,8	0,00
	AF25-01	2,5	1:2:0.72:0.025	391,4	1143,8	281,8	9,79
	AF50-01	5,0	1:2:0.72:0.05	391,4	1143,8	281,8	19,57
	AF75-01	7,5	1:2:0.72:0.075	391,4	1143,8	281,8	29,36
	AF100-01	10,0	1:2:0.72:0.1	391,4	1143,8	281,8	39,14
1:3	REF-02	0	1:3:1.02:0	290,0	1272,0	295,8	0,00
	AF25-02	2,5	1:3:1.02:0.025	290,0	1272,0	295,8	7,25
	AF50-02	5,0	1:3:1.02:0.05	290,0	1272,0	295,8	14,50
	AF75-02	7,5	1:3:1.02:0.075	290,0	1272,0	295,8	21,75
	AF100-02	10,0	1:3:1.02:0.1	290,0	1272,0	295,8	29,00

further analysis in the Scanning Electron Microscope (SEM). Sample preparation was done at Thermal Treatment and Optical Microscopy Laboratory (LTM) and SEM analyzes were performed at NanoLab, both at UFOP.

3. Results and Discussion

3.1. Chemical characterization of the fiber

The results for the determination of total extractives, the lignin and ash content are shown in Table 3. The lignin content presents in the moringa seed husk is considerably higher when compared to other fibers[12]. This fiber has a low percentage of holocellulose, a consequence of the difference in extractive content[8]. As stated in the item 1.1, the fiber properties of the same plant can vary a lot from each other, as they depend on the degree of crystallinity, planting conditions and the part of the plant that it comes from. The major constituent of holocellulose is cellulose. A low amount of holocellulose represents a mechanically weak fiber.

A variation of up to 100% of the properties for the same fiber can be found[25]. So, this difference found for moringa fiber when compared to literature is not unusual (around 45.7%)[16].

The chemical composition of Moringa *oleifera* fiber reveals desirable characteristics for addition in cementitious composites. The high content of lignin present ensures that the fiber is weather resistant, fungus and bacteria resistant. Lignified tissues resist attack by microorganisms, preventing the penetration of cell wall-destroying enzymes[26]. But the amount of holocellulose is responsible for the hydrophilic character of the fiber.

The analyzed and used fibers have a lower holocellulose content and, consequently, a lower cellulose content. The lack of this structural component in the fiber makes its mechanical strength relatively low.

3.2. Physical characterization of the fiber

Table 4 shows the results of the tests for natural moisture, water absorption content and apparent and real specific mass. The raw fiber does not have a high percentage of natural moisture, with a found value of 8.79%. However, the fiber has the capacity to absorb almost four times its weight in water. Values for real specific mass and apparent specific mass are consistent with the literature[16]. Vegetable fibers are very porous and, due to their high water absorption content, it is to be expected that the difference between real and apparent density is relatively large.

Table 3. Moringa *oleifera* seed husk composition.

Moringa *oleifera* seed husk composition			
Lignin content (%)	Extractive content (%)	Ash content (%)	Holocellulose (%)
34,782	31,596	2,6	31,0

Table 4. Physical characterization of Moringa *oleifera* seed husk.

Physical characterization of Moringa *oleifera* seed husk	
Natural moisture (%)	8,79
Water absorption content (%)	390,16
Apparent specific mass (g/cm^3)	0,975
Real specific mass (g/cm^3)	1,291

Fiber micrographs taken on the SEM reveal a heterogeneous fiber anatomy. In Figure 1 it can be seen, at the same time, the frontal view of the epidermis and its cross section. The observed part is the dark region of the seed peel. In this region, the epidermis has beehive-shaped cuticle ornamentation. The cross section is parenchymal and contains sclereids and parenchymal cells with lignified thickening bars.

In Figure 2, where both dark and the light part of the husk can be seen, it is possible to see that the epidermis does not have a continuous ornamentation. In the light region of the peel the ornamentation is striated, almost smooth.

3.3. Cementitious composite

The consistency test indicated that the addition of fiber results in a less fluid mortar or a mixture with less workability. When incorporating the fiber into the cement mixture, there is a change in its consistency. The presence of fiber in the mixture causes an increase in the surface area, which demands wetting water[27].

The difference in mortar consistency is evident when compared in Figure 3. In addition to the increase in surface area, as stated in item 1.2, the cellulose from vegetable fibers is responsible for the hydrophilic nature of the fibers. The

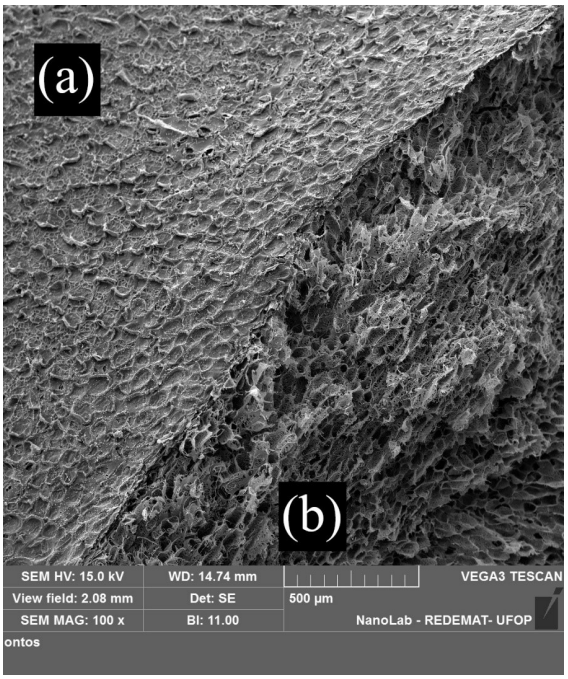

Figure 1. Fiber micrograph showing (a) frontal view of the epidermis of the fiber and (b) parenchymal region containing sclereids and parenchymal cells with lignified thickening bars.

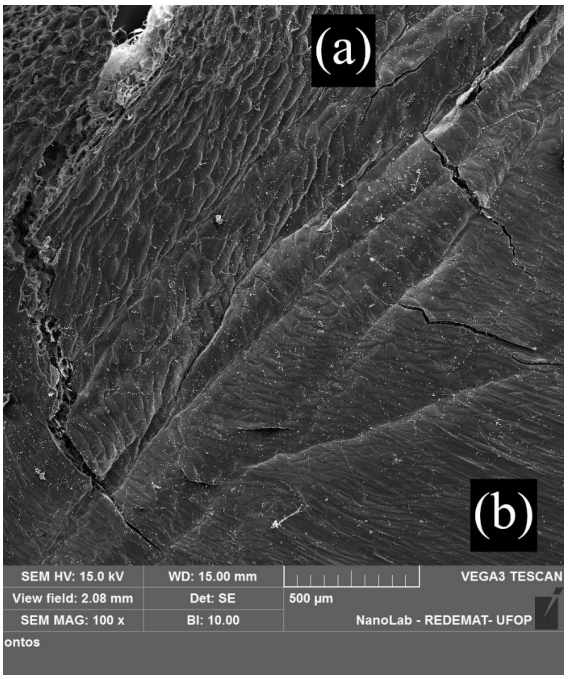

Figure 2. Non-uniform epidermis detail. The cuticle is (a) beehive-shaped and (b) striated.

fiber's high water absorption content (390.16%) confirms that the water present in the mortar mixture was absorbed by the fibers, thus decreasing the workability of the mixture.

Still in Table 5, it can be observed that, despite the tendency to decrease workability, the average spread of the

Table 5. Dosages and their respective consistency.

Type	Identification	Flow-test (average in cm)
1:2	REF-01	26,2
	AF25-01	19,7
	AF50-01	17,9
	AF75-01	24,5
	AF100-01	23,7
1:3	REF-02	25,8
	AF25-02	23,2
	AF50-02	17,4
	AF75-02	17,8
	AF100-02	19,5

Table 6. Criterion used to evaluate the quality of the concrete[28].

Linear propagation speed (m/s)	Concrete quality
V > 4500	Excelent
3500 < V 4500	Great
3000 < V <3500	Good
2000 < V < 3000	Regular
V < 2000	Bad

mixtures AF75-01 and AF100-01 was greater than that of the other mixtures, with a lower content of fiber addition. This variation may be due to the great variability of natural fiber properties. Just as the chemical properties are influenced by planting, physical properties, such as water absorption content, can also vary.

The main factors that influence the speed at which the ultrasonic pulse passes through the specimen are the distance between the contact surfaces of the transducers, presence of reinforcement, density of the cementitious composite (depends on the dosage), type and characteristics of the aggregate, type of cement and degree of hydration[22].

A previous evaluation of the quality of the mortar can be obtained, according to Table 6. This parameter must be evaluated together with the results of the mechanical tests. Higher pulse speed may indicate a more homogeneous composite[28]. When relating Table 6 to Figure 4, it is noted that the composite produced can be classified as good quality, since the average pulse speeds are around 3000- 3500 m/s. Despite this, when analyzing the results of the compression strength test, composite with lower-than-expected strength was obtained (Figure 5).

The dosages that have lower workability are: AF25-01, AF50-01, AF50-02, AF75-02 and AF100-02 (Table 5). From the results of the mechanical compression strength test (Figure 5), it is noted that the mixtures that had lower compression strength results are: AF75-01 and AF50-02 (7 days); AF100-01 and AF100-02 (28 days). The dosages that are more heterogeneous, according to the ultrasonic pulse speed test (Figure 4) are AF100-01 and AF100-02 at 28 days.

The compressive strength of the sample AF75-01 had an increase of 11.22% when comparing it in 7 and 28 days (Figure 5). Making the same comparison, AF25-02, AF50-02

Figure 3. Comparison between the consistency of REF-02 and AF100-02 mortars.

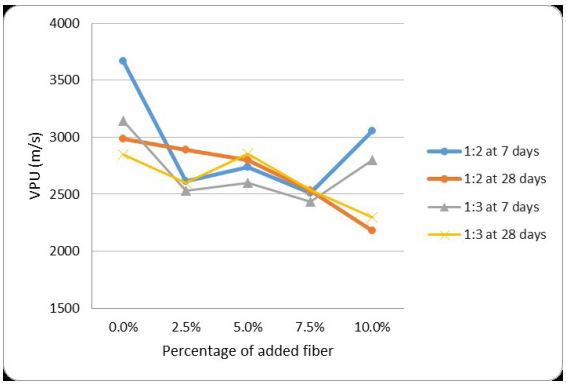

Figure 4. Average of the ultrasonic pulse propagation speed.

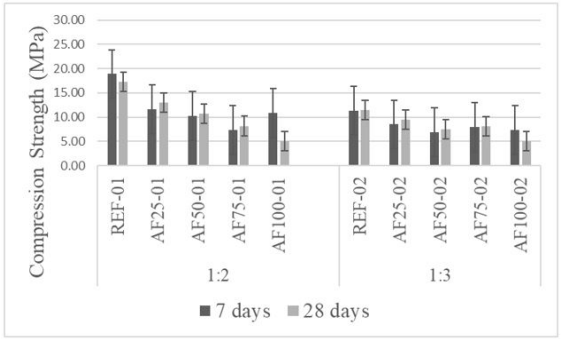

Figure 5. Compression Strength test results for 7 and 28 days.

and AF75-02 had an increase of 10.88%, 8.73% and 2.20%, respectively. This demonstrates that the addition of fiber can increase the compressive strength of the mortar. For younger ages, this difference is still small. When using CP-V (High Initial Strength), it can be inferred that the addition of fiber slows down the compression resistance strength curve of this type of cement. The presence of a high content of natural fibers reduces the kinetics of formation of hydrated cement compounds[29,30].

On the other hand, AF100-01 sample had 53.35% drop in resistance and sample AF100-02 had 31.19%. Although the sample AF100-01 presented a relatively good workability, its drop in compressive strength was the most expressive. It is important to note that its addition of fiber was greater and its percentage in the mixture is greater than in AF100-02 sample, when compared to the total mix. The fiber, being hydrophilic, absorbs water from the mixture. The higher its content, the greater the water absorption.

More advanced ages of these mixtures should be analyzed in order to determine the influence of fiber addition over time. The water absorbed by the fibers can be released at longer aging times, allowing the formation of late hydrated cement compounds.

The water absorbed by the fibers also forms occlusions. Where the water should be, a pore is formed. The porosity of the sample is confirmed with lack of homogeneity of the specimens by the ultrasonic pulse results and can be seen in the micrographs produced in the SEM analysis (Figures 6 to 9).

It is important to highlight at this point the hydration effects of Portland Cement, with consequent release of calcium hydroxide ($Ca(OH)_2$) on natural fibers. These types of composites tend to undergo aging, which reduces their strength. What occurs is the migration of $Ca(OH)_2$, which by mineralization, changes the structure of the natural fiber[31].

$Ca(OH)_2$ impregnates the lumen and the fiber wall, thus causing an early weakening of the plant fiber. In addition, there is a variation in the volume of fibers with the absorption of water, which also causes an early loss of mechanical strength[32].

Figure 6 shows the presence of partially hydrated cement grains (light gray) amid the fiber sclereids and Figure 7 shows

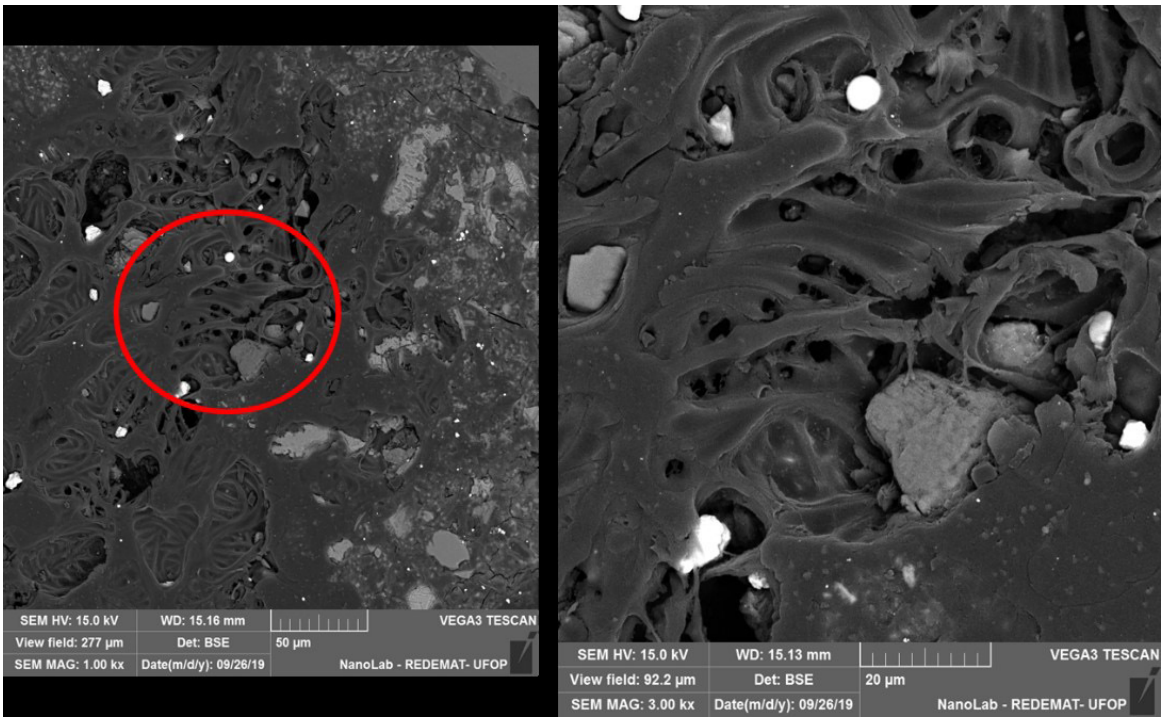

Figure 6. Micrograph of sample AF100-02 tested at 7 days with magnification of 1000x and 3000x.

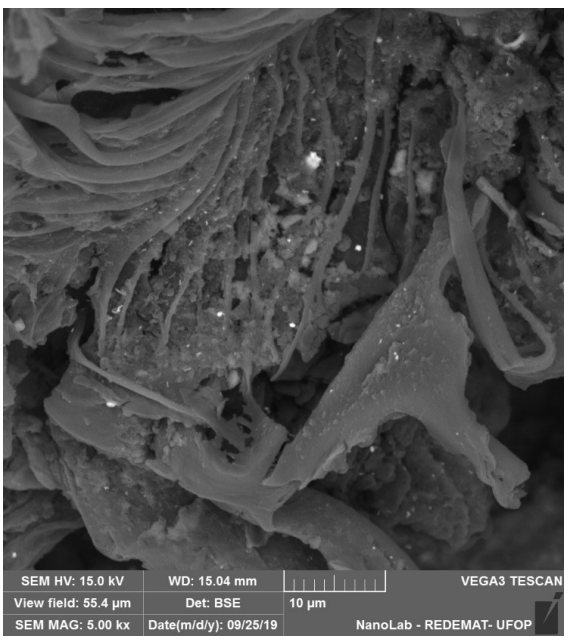

Figure 7. Micrograph of sample AF100-01 tested at 7 days with 5000x magnification.

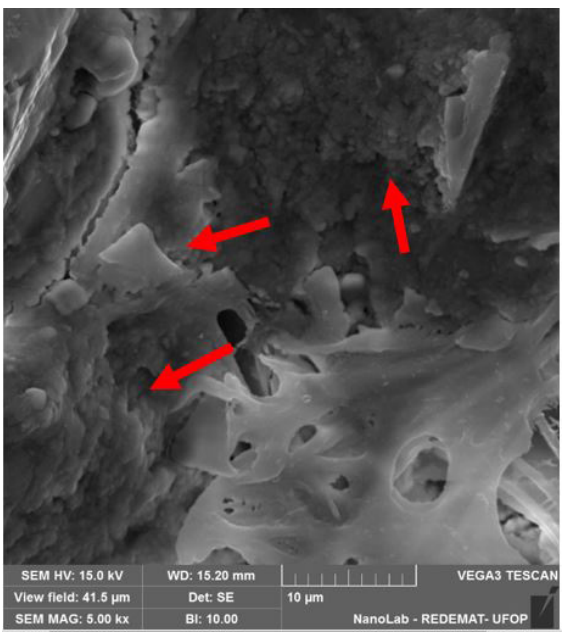

Figure 8. Micrograph of sample AF100-02 tested at 28 days with 5000x magnification.

the presence of calcium hydroxide crystals. The high porosity of the vegetable fiber induces the formation of portlandite crystals, which are not formed at the interface, but inside the transition zone[32]. With availability of space and water, there is the formation of nuclei of portlandite.

Figure 8 shows hydrated calcium silicate crystals (C-S-H) next to a sclereid. The morphology of these crystals depends on the curing conditions. As there was not enough water in the mixture to hydrate the cement, the C-S-H present could not fully develop its structure in sample AF100-02, which had a higher content of fiber addition (10%).

Figure 9 shows the concentration of calcium hydroxide in the fiber epidermis (presence of small portlandite crystals). At 28 days there is a greater accumulation of portlandite

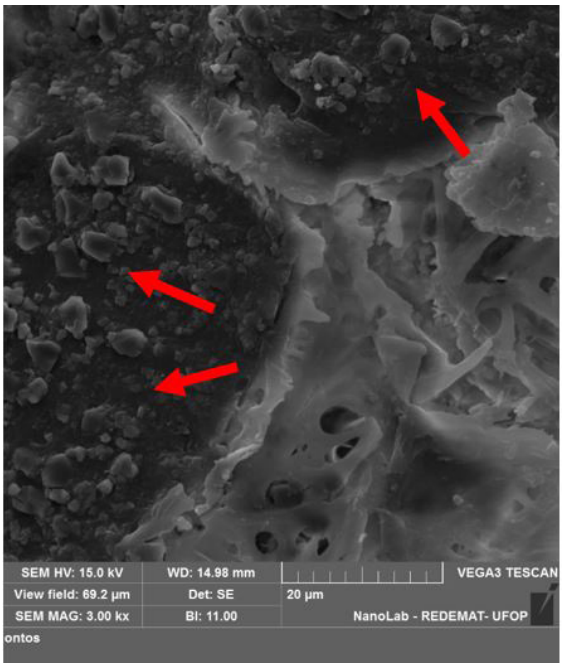

Figure 9. Micrograph of sampleAF75-01 tested at 28 days with 3000x magnification.

near the fiber. Portlandite is a key component for good mechanical performance. However, its presence along the vegetable fiber makes this type of composite weak because calcium hydroxide causes the petrification of the fibers with the filling of the lumen.

The information obtained from the micrographs is in agreement with the results of the mechanical tests. Hydrated cement composites were not sufficient to guarantee the mechanical strength of the composite produced. The fiber's high-water absorption delayed cement hydration. At 28 days, the compressive strengths were much lower than the standard foresees[33].

Insufficient water for cement hydration can also be observed by the amount of microcracks present at the composite interface. These microcracks are stress concentrators. The portlandite present next to the fibers also contributed to the low mechanical strength of the composite.

4. Conclusions

This work aimed to study the characteristics of the Moringa *oleifera*'s seed peel and its influence when added to a cementitious composite. From the tests performed, we can conclude:

1. Moringa fiber has a high content of holocellulose, which contributes to its hydrophilic character. It has a high-water absorption content (390.16%), which makes its structure weak due to volume variation;
2. The fiber presents incompatibility with the cement matrix, since the fiber undergoes alkaline attack, absorbs a lot of water from the mixture and does not allow the complete formation of hydrated cement compounds (at younger ages), which confer resistance to the composite;
3. Micrographs allow a better understanding of the fiber/matrix interface:
   a) Crystals of hydrated calcium silicate and calcium hydroxide are present at the interface and even inside the fiber. There is a high concentration mainly of calcium hydroxide in this region, a component that does not effectively contribute to the final strength of the composite;
   b) There are microcracks at the fiber/matrix interface. These are stress concentrators and can cause the composite to collapse as they are the most fragile part of the system;

The use of Moringa *oleifera*'s seed peel, *in natura*, did not bring benefits to the composite when studying its mechanical performance. Therefore, its use as an addition in cementitious composites is not recommended when there is a need for mechanical strength. However, its use in powder has been studied, with the potential to act in composites exposed to hostile environments, such as seawater [09]. Also, satisfactory results were obtained by emphasizing the durability of the composite produced with wood waste with a smaller particle size then the moringa fiber used in this work[34].

Other ways of using this fiber must be studied. Fiber pretreatment must be considered to improve its mechanical performance in the cementitious composite. Furthermore, the same high content of lignin that increases the fiber's hydrophilic content also makes it antifungal and weather resistant. More studies regarding the effectiveness of this quality should be carried out.

5. Acknowledgments

The authors would like to thank UFOP, REDEMAT, NanoLab, LTM and the Building Materials Laboratory for the infrastructure and support, and CAPES for promoting research.

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