

STUDY ON THE POLYMERIC TREATMENT WITH RICE HUSK SILICA ON SISAL FIBER IN CEMENTICIOUS COMPOSITES

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Highlights:

- novel application of EPS and RHS polymeric treatment for sisal fibers in cementitious composites;
- significant reduction (approx. 70%) in water absorption post-treatment, enhancing durability;
- microscopic analysis reveals discontinuous layer on fiber surface, impacting fiber-matrix interaction;
- direct traction tests highlight treatment's effect on fiber behavior, improving uniformity;
- enhanced fiber-matrix interactions observed despite no substantial increase in traction force;
- treatment compromises fiber-matrix adhesion, leading to lower breaking strengths and increased variability;
- pullout tests suggest the formation of a sealing layer by the hydrophobic polymer, limiting paste penetration.

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Abstract. This research evaluates how treating sisal fibers with expanded polystyrene (EPS) and rice husk silica (RHS) affects their absorption capacity, tensile strength, and adhesion when used in Portland cement matrices. The study on sisal fibers treated with EPS and RHS polymers found that the treatment significantly reduced water absorption by 70%, from 84.67% for untreated fibers to 15.18% for treated ones, due to the hydrophobic nature of EPS. Optical microscopy revealed an irregular polymer layer on the fibers, which, while improving dimensional stability, could impair fiber-matrix interaction. Despite these improvements, the treatment did not notably enhance the mechanical properties of the fibers, as the breaking strength remained similar to untreated fibers, and the rupture displacement slightly decreased.

Keywords: sisal fibers, cementitious composites, polymeric treatment, rice husk silica, expanded polystyrene (EPS).

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1. Introduction

The continuous development of cementitious materials emphasizes the importance of integrating innovative technologies into construction practices. While cement-based materials offer various advantages, they often exhibit limitations in tensile strength, leading to brittle behavior and low tenacity. To mitigate these issues, incorporating vegetable fibers as reinforcement has been recognized as beneficial due to their lower extraction energy requirements and potential to enhance mechanical properties in composites.

However, the inherent characteristics of natural fibers can be further enhanced through treatment methods, offering potential increases in mechanical resistance and fiber-matrix anchoring capacity. By leveraging natural and renewable materials, there is an opportunity to significantly

improve the mechanical properties of cementitious composites (Bentur & Mindess, 2007).

In this context, our research focuses on applying treatments using expanded polystyrene (EPS) and rice husk silica (RHS) to sisal fibers. Our objectives include evaluating their impact on water absorption capacity and analyzing the microstructure of sisal fibers to assess longitudinal uniformity. Additionally, the effects of polymeric treatment with Rice Hull Silica on the direct tensile strength of both natural and treated sisal fibers, as well as its influence on fiber-matrix adhesion capacity through pullout testing are investigated (Gram, 1983; Melo Filho et al., 2013; Bentur & Mindess, 2007).

Previous research has underscored the potential of surface treatments to enhance the performance of natural fibers in cementitious composites. Specifically, studies have demonstrated that treatment methods can reduce

water absorption rates, improve mechanical properties, and enhance fiber-matrix interaction. However, there remains a need to further explore the effectiveness of these treatments in enhancing the overall performance of cementitious composites (Lima & Toledo Filho, 2008; Wei & Meyer, 2003).

Various methods have been explored to mitigate degradation, including fiber protection treatments, cement matrix modification, and reduction of free calcium hydroxide content. Surface treatments, like the hornification process, show promise in enhancing mechanical performance and durability by reducing embrittlement and improving adhesion (Yan et al., 2016; Ferreira et al., 2017; Gopalakrishnan et al., 2023).

In this context, the present work aims to study the behavior of sisal fibers treated superficially using polymeric treatment based on EPS dissolved in toluene, with the addition of RHS, regarding the mechanical stresses of direct traction and its adhesion performance when used as reinforcement of cementitious composites in a matrix free of calcium hydroxide (Mohammed et al., 2022; Tonietto et al., 2019, Fadele et al., 2019).

Yimer and Gebre (2023) examined methods to enhance sisal fibers for concrete reinforcement. They varied NaOH concentrations and treatment durations to assess effects on fiber properties like water absorption, tensile strength, and surface morphology. Concrete characteristics such as slump, compressive strength, flexural strength, and toughness were also evaluated. Results indicated alkali treatment reduced water absorption and improved fiber morphology and mechanical properties, especially flexural strength and toughness in concrete, though compressive strength showed minimal improvement.

Prakash et al. (2022) explored eco-friendly fiber-reinforced concrete, using fly ash as a partial cement substitute and coconut shell as coarse aggregates. They incorporated steel fiber, sisal, and roselle fibers, studying their behavior and mechanical properties. The study showcased promising outcomes, especially with coconut shell aggregates.

Prakash et al. (2021) investigated lightweight concrete using coconut shells as substitutes for traditional aggregates. Various sisal fiber percentages were added to enhance mechanical properties. Results highlighted significant improvements in compressive strength (up to 6%), split tensile strength, flexural strength, and impact resistance, particularly with 3% sisal fiber incorporation.

Lilargem Rocha et al. (2022) explored lignocellulosic fibers in cementitious composites, emphasizing advancements in countries like Brazil. They compared natural and synthetic fiber properties, noting the lightweight advantage of vegetable-based composites. Fiber-matrix interaction and surface treatments' influence on composite characteristics were discussed, aiming to provide insights into lignocellulosic fiber applications in cementitious composites.

Ahmad et al. (2022) reviewed SSF-reinforced concrete advancements, focusing on its qualities, interaction with concrete, and resulting properties. SSF was found to

enhance concrete strength and durability, albeit reducing flowability. Future research directions were suggested.

De Klerk et al. (2020) aimed to counter sisal fiber degradation in concrete through alkaline treatment and acetylation. Various tests evaluated treatment effects on fiber strength, interaction with concrete, and durability. While effective in enhancing fiber durability, some degradation was observed despite chemical treatment, cautioning about potential strength reductions.

While the studies mentioned offer valuable insights into sisal fiber and natural materials in concrete reinforcement, they have drawbacks compared to a study on polymeric treatment with RHS. Firstly, there's a limited focus on specific treatments and their long-term effects on sisal fiber durability. Some studies explore treatments like alkaline treatment and acetylation but may not fully address potential drawbacks or long-term stability. Additionally, their effectiveness in different environmental conditions isn't thoroughly investigated. Furthermore, these studies may not comprehensively optimize sisal fiber properties for enhanced performance in cementitious composites. While they touch upon factors like fiber concentration and treatments, incorporating RHS and polymer treatments could further boost mechanical properties and durability. Moreover, there's limited discussion on the environmental impact and sustainability of sisal fibers. While they're eco-friendly, their cultivation and processing methods may have environmental implications needing further exploration. In summary, while these studies contribute significantly, there's room to optimize sisal fiber-reinforced concrete by thoroughly investigating treatment methods, optimizing fiber properties, and considering sustainability factors.

Recent advancements in composite materials made from natural fibers, particularly in the context of durability, highlight the role of treatments in enhancing the longevity and performance of these materials. Natural fiber composites, such as those with sisal fibers, benefit from treatments like RHS and polymer impregnation, which can reduce water absorption and improve mechanical properties. However, while these treatments help decrease the natural fibers' vulnerability to moisture and environmental degradation, the effectiveness of fiber-matrix adhesion often requires further optimization, as thick polymer layers may impede strong bonding (Singh et al., 2020; Bouafif et al., 2020).

The nano-fillers enhance the properties of polymer composites in ways that are not achievable with conventional micro-fillers. Their ability to improve mechanical, thermal, electrical, and barrier properties while being lightweight and cost-effective makes them invaluable in advanced technological fields (Zhang et al., 2022; Kim et al., 2022).

The paper highlights the synergistic effects of combining organic and inorganic materials, improving durability, thermal stability, and shielding effectiveness against electromagnetic radiation. Additionally, the paper underscores the importance of optimizing filler content and alignment

to enhance composite properties, making them suitable for applications in aerospace, automotive, and electronic industries (Hemath et al., 2020).

The research demonstrates that incorporating Al_2O_3 nanofillers significantly improves the tensile, flexural, and impact strength of the composite, with optimal performance observed at a specific filler concentration. Furthermore, the study explores the tribological behavior, showing reduced wear and friction due to the self-lubricating nature of the nanofillers. The findings highlight the potential of Al_2O_3 -reinforced basalt/epoxy composites for applications requiring superior wear resistance and mechanical performance, such as in construction, automotive, and wind turbine components (Vinay et al., 2020).

The scope of the problem investigates the impact of treating sisal fibers with EPS and RHS polymers on their properties in cement composites, particularly focusing on water absorption, stability, and mechanical performance. It aims to understand how these treatments improve fiber interaction with the cement matrix and enhance material durability.

2. Methodology

This section outlines the materials used and the experimental procedures employed for the polymeric treatment with RHS on sisal fibers, followed by the examination of superficial modifications through optical microscopy. The methods for sample preparation to assess absorption index and conduct direct tensile strength analysis of the treated fibers are discussed, along with the molding of

test specimens to evaluate pullout resistance, i.e., adhesion ratio between treated fiber and cementitious matrix.

The treatment of sisal fibers with a combination of EPS and RHS offers several benefits. This method reduces the water absorption capacity of the fibers, enhancing their durability in cementitious composites. It also improves tensile strength consistency by sealing the fiber surfaces, though it doesn't significantly increase breaking strength. However, challenges in fiber-matrix adhesion were noted, as the polymeric layer can hinder fiber anchoring. The study underscores the potential of EPS and RHS treatments to improve fiber properties, although further refinements may be needed for optimal adhesion (Fernandes et al., 2019; Gao et al., 2020; Bispo et al., 2022).

To enhance comprehension of the study's execution stages, Figure 1 presents a simplified flowchart of the experimental program depicting the sequential steps.

2.1. Materials

The materials utilized for composite production include Portland cement PC V-ARI (from Ultratech Cement), fly ash (from Dalmia Cement), metakaolin (Imerys), sand, expanded polystyrene (Sourced from Thermocool and Thermo-plast), toluene (Vishal Traders), rice husk silica (from Vijay Traders), sisal fiber (from Sisal Fiber Products, Nirma), and superplasticizer additive (BASF India).

Portland cement PC V-ARI (PC) was selected for this study, along with mineral additions such as fly ash (FA) and metakaolin (MC) (Figure 2). The fine aggregate, fine sand is shown in Figure 3 and exhibited specific properties as listed in Table 1.

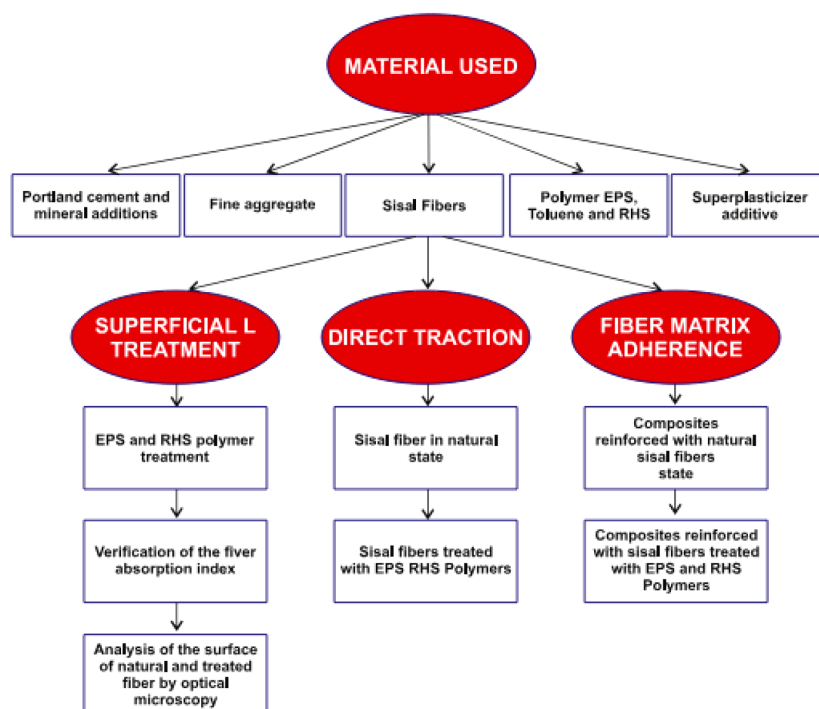


Figure 1. Simplified flowchart of the experimental program

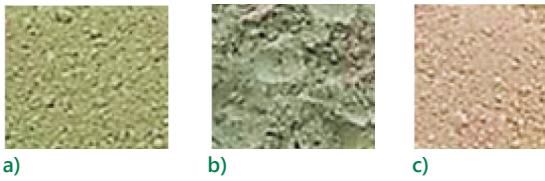


Figure 2. Binding material and mineral additions used:
a) Portland cement PC V-ARI; b) fly ash; c) metakaolin

Table 1. Characterization of fine aggregate (sand)

Fine sand		
Fineness modulus	Maximum characteristic dimension (mm)	Specific mass (kg/dm ³)
1.37	0.60	2.63



Figure 3. Fine aggregate used (sand)

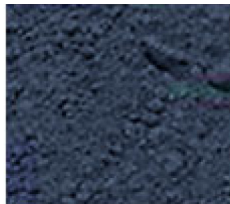


Figure 4. Material used to promote treatment in sisal fibers: RHS



Figure 5. Sisal fibers

RHS was employed for treating the sisal fibers (Figure 4). The sisal fibers, after being washed and dried, were not subjected to any prior treatment (Figure 5). The superplasticizer (SP) additive used was of the PA (Polyacrylate) Glenium 51 type.

The treatment process involved impregnating the thermoplastic polymer EPS with the addition of RHS in the fibers, facilitating a connection between the fiber and silica particles through an adhesion bridge formed from the polymer use. Toluene was employed as the solvent (Figure 6) due to its reactivity with EPS, ensuring complete dissolution at room temperature.

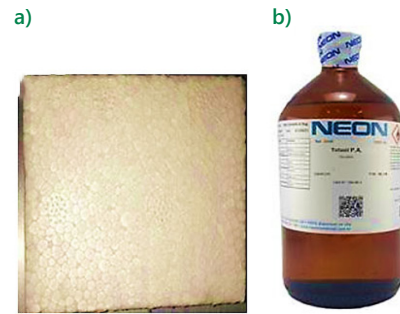


Figure 6. Materials used in the impregnation process:
a) EPS; b) Toluol P.A. (Toluene)

The overview of the materials used and their mechanical properties are provided below:

2.1.1. Portland cement (PC V-ARI)

Characterization: Portland cement serves as the primary binding agent in concrete.

Mechanical Properties: Compressive strength typically ranges from 20 MPa to 50 MPa.

2.1.2. Fly ash

Characterization: Fly ash acts as a supplementary cementitious material in concrete.

Mechanical Properties: Enhances compressive and flexural strength, contributing to improved durability.

2.1.3. Metakaolin

Characterization: Metakaolin is a pozzolanic additive used to boost strength and durability.

Mechanical Properties: Significantly improves compressive and flexural strength.

2.1.4. Sand

Characterization: Sand serves as the fine aggregate in concrete.

Mechanical Properties: Contributes to overall compressive and flexural strength.

2.1.5. Expanded polystyrene (EPS)

Characterization: EPS is a lightweight aggregate used to reduce density and improve insulation.

Mechanical Properties: Varies with density, typically has low compressive and tensile strength.

2.1.6. Toluene

Characterization: Toluene is a solvent used in industrial processes.

Mechanical Properties: Does not possess mechanical properties.

2.1.7. Rice husk silica

Characterization: Rice husk silica is a pozzolanic material.

Mechanical Properties: Enhances compressive and flexural strength, reduces cracking.

2.1.8. Sisal fiber

Characterization: Sisal fiber is a natural reinforcement material.

Mechanical Properties: Improves tensile and flexural strength, enhances toughness and impact resistance.

2.1.9. Superplasticizer additive

Characterization: Superplasticizers improve workability and flowability of concrete.

Mechanical Properties: Does not directly influence mechanical properties.

The quantities of materials used for sisal fiber treatment were determined to achieve a homogeneous mixture, as indicated in Table 2.

Table 2. Quantity of materials to promote treatment in sisal fibers

Expanded polystyrene	17 g
Toluol P.A.	100 ml
Rice Husk Silica	0.5 g

The EPS was solubilized in Toluene solvent through agitation in an ultrasonic bath equipment for 25 minutes, followed by the addition of RHS and stirring for 10 minutes until its particles were solubilized in the polymeric base (Figure 7). Subsequently, the sisal fibers were immersed in the solution and dried for 24 hours at a temperature of $25\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.

The treated fibers are depicted in Figure 8.

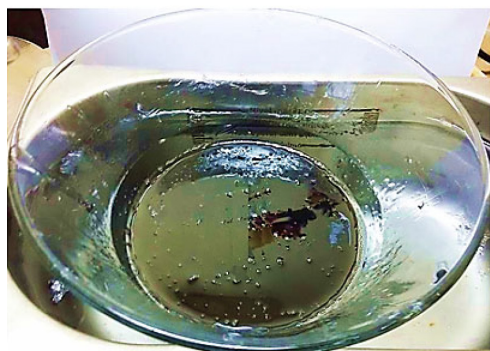


Figure 7. Polymeric solution using EPS with addition of RHS

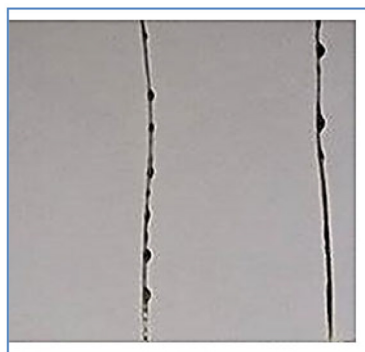


Figure 8. Sisal fibers after treatment

The novelty of this research lies in the innovative approach of using a combined treatment of EPS and RHS on sisal fibers for reinforcing Portland cement matrices. While the use of natural fibers in cementitious materials has been explored, this study is among the first to assess the effects of EPS and RHS treatments on sisal fibers, specifically focusing on their absorption capacity, tensile strength, surface modification, and fiber-matrix adhesion.

Key novel contributions of the research include:

- The simultaneous use of EPS and RHS is unique. While both EPS (for hydrophobicity and void reduction) and RHS (for silica content) have been studied individually, combining them to treat natural fibers is novel and may open new possibilities for fiber reinforcement in cement composites.
- The study demonstrates that this specific treatment significantly reduces the water absorption capacity of sisal fibers, an important factor for improving the durability and performance of fiber-reinforced composites. The reduction in capillary voids due to polymer treatment is a unique insight into enhancing natural fiber performance in construction materials.
- The treatment improves tensile strength uniformity, highlighting a novel method to standardize natural fiber mechanical properties, which can be inconsistent due to natural variability. This treatment method addresses one of the primary challenges with using natural fibers—mechanical property variability—by sealing fiber surfaces, though without improving breaking strength.
- The study provides a critical insight into the limitations of EPS and RHS treatment in enhancing fiber-matrix adhesion, particularly due to the formation of a thick polymeric layer that impedes fiber anchoring within the matrix. This points to the need for further refinement, suggesting that while the treatment improves certain properties, it may introduce new challenges for fiber adhesion.
- The observation of sealed fibers with occasional agglutination points adds a unique understanding of how polymer treatments interact with fiber microstructures. This microscopic perspective helps to explain why the treatment did not improve fiber-matrix bonding despite its positive effect on water absorption.

3. Experimentation

3.1. Evaluation of fiber absorption capacity

The methodology proposed by (Toledo Filho et al., 2009) was employed to determine the water absorption index (AI) of both treated and untreated sisal fibers. Two samples of natural and treated fibers were prepared, each comprising filaments with the same initial oven-dried weight.

The fiber samples were initially weighed while dry, and then immersed in water for 3 hours to achieve total saturation. Subsequently, the samples were re-weighed after each immersion to determine their wet weight. This

process was repeated three times for both untreated and treated fibers to obtain an average between the dry and wet weights.

The final result for the AI was obtained through Equation (1):

$$AI = \frac{P_{ww} - P_{od}}{P_{est}}, \quad (1)$$

where P_{od} represents the weight of oven-dried fibers and P_{ww} the wet weight, after saturation, thus being able to verify how much the treatment used influenced the absorption capacity of water by the sisal fibers.

3.2. Analysis of the microstructure

Surface evaluations were carried out for samples of untreated and superficially treated fibers, using an optical metallographic microscope.

3.3. Test of direct traction on the fibers

The direct traction test on the fibers was performed on a Shimadzu mechanical testing machine, model AGS-X, using a 500 N load cell.

The sisal fibers had a length of 50 mm and were tested at a speed of 0.1 mm/min. These were fixed in a paper mold, providing their alignment in relation to the machine and improving adherence between the sample and the grips that lock it, thus preventing possible sample slippage.

To enable the verification and statistical analysis of the load supported by the fibers submitted to direct traction, 20 samples were tested, this total being divided equally between: samples for direct traction test, untreated sisal fiber (DTT-UTSF) and samples for direct traction, sisal fiber subjected to polymeric treatment with RHS (DTT-PRHS).

The direct traction samples were prepared by adapting the procedure described in the ASTM C1557 standard, using 90 g/m² paper and masking tape, where first the fiber is aligned in the mold and then a strip of paper is glued on their ends to improve grip adhesion. After positioning the specimen in the machine, a transverse cut will be made on the sides of the mold to allow only the fiber to be pulled.

Figure 9 shows the sample prepared for carrying out the direct traction test, while Figure 10 shows the test configuration.

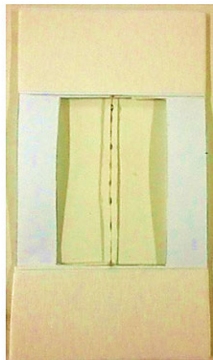


Figure 9. Mold used to perform the direct traction test

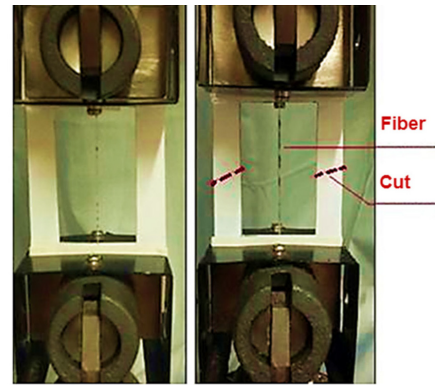


Figure 10. Configuration of the direct traction test on the treated sisal fiber

3.4. Cement matrix

The matrix used to produce the cementitious composite is an adaptation of a matrix already used by (Ferreira et al., 2020). This adequacy can be justified by the change in the type of cement, choosing to use PC V-ARI cement instead of PC II F-32, in order to coincide with local availability, facilitating the acquisition of the material.

In this way, the mix of materials for the production of the consonant matrix presented in Table 3 was established, maintaining for this study the proportion used by (Ferreira et al., 2020).

Table 3. Consumption of materials used in dosing the cement matrix

Name	Matrix (kg/m ³)							Spread (mm)
	PC	Sand	MC	FA	Water	w/c** Ratio	SP*	
M1	362	542	289	434	434	0.4	25	≥450

Note: * Solids from SP/MC; ** w/c: water/cementitious material.

3.4.1. Matrix production

The mortars were prepared following the methodology described by ASTM C1609, using a planetary-type mechanical mixer (MIXER HSD Series), with two speeds and an approximate capacity of 5 liters, as shown in Figure 11.



Figure 11. Planetary type mechanical mixer

The mixing process was carried out according to the steps listed below:

- Mixture of water + superplasticizer for 30 seconds in the mechanical mixer;
- Premix (in a separate container) of the fine materials for 1 minute;
- Adding the mixture of fine materials to the mixer over 2 minutes;
- Mixing all materials placed in the mixer for 2 minutes;
- 30-second stop to remove material adhering to the mixer walls;
- Mix all the material contained in the mixer for 2 minutes.

Also, according to ASTM C1609, after mixing to obtain the mortar, a consistency table test was carried out, verifying the measurement of the spreading diameter with the aid of a caliper, in order to assess whether it was above the limit shown in Table 3. Figure 12 shows the steps of the test performed in a simplified way.



Figure 12. Consistency test steps: a) filling of the conical trunk; b) removal of the formwork vertically; c) verification of the spreading measure

3.5. Molding of composites

The specimens for the pullout test were molded using Medium density fiberboard (MDF), as shown in Figure 13, based on the method developed by (Li et al., 2000).

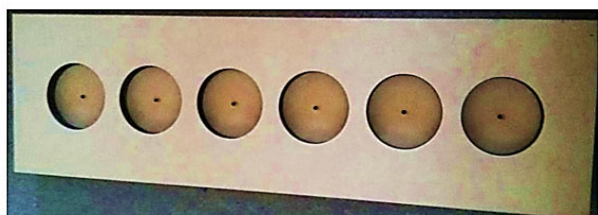


Figure 13. MDF plate for fitting the PVC pipes

These plates were manufactured with a 32 mm circular recess to allow the fitting of the PVC mold, with a central hole that aims to ensure fiber alignment (Silva et al., 2011) as shown in Figure 14.

First, the fibers were introduced and then the PVC molds were fitted onto the MDF board. The casting of the mortar for molding the specimens consisted of filling the molds manually with the aid of a pastry bag (Ferreira et al., 2020), a procedure similar to what can be seen in Figure 15.

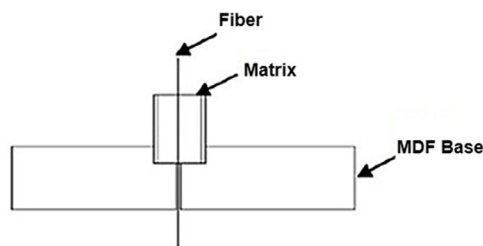


Figure 14. Schematic drawing showing fiber alignment



Figure 15. Procedure for molding the specimens for the pullout test

After filling the PVC molds, the MDF board was superimposed on the molds, acting as a top cover. Afterwards, the fiber was stretched in order to provide greater rigidity to the set and allow better alignment of the fiber in the middle of the matrix.

3.6. Pulling test

The pullout test was carried out on a Shimadzu AGS-X mechanical testing machine with a 500N load cell, in which the specimens were tested at a beam displacement speed of 0.1 mm/min.

The specimens had a length of 50 mm of sisal fiber embedded inside the composite. After 24 hours, the composites were demoulded and taken to a humid chamber ($T \pm 23^\circ\text{C}$ and $RH \pm 43\%$) for curing, remaining in this chamber for up to 24 hours before the control age stipulated for the test. After removing the specimens from the humid chamber, they were kept for 24 hours in an oven at an ambient temperature of $T = 23^\circ\text{C} \pm 1^\circ\text{C}$, in order to provide adequate drying.

Figure 16 shows some of the specimens used to perform the pullout test.

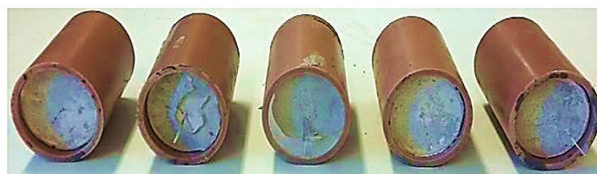


Figure 16. Test specimens with 50 mm fiber embedded

Figure 17 shows the configuration of the pullout test, where it is possible to visualize the aligned positioning of the fiber in relation to the center of the PVC mold.

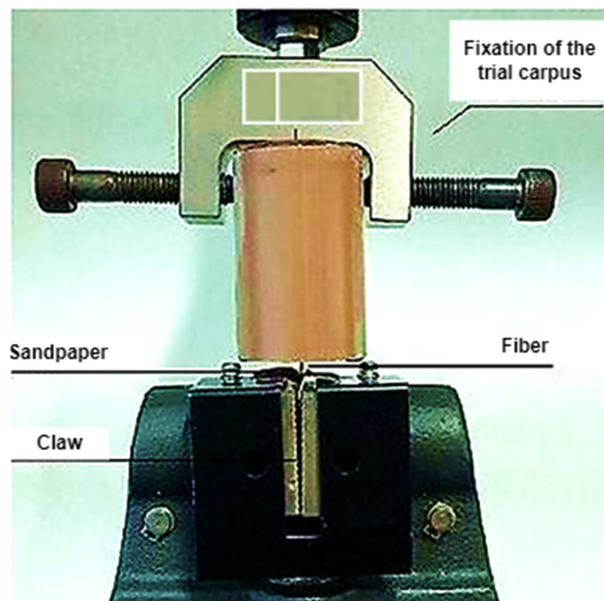


Figure 17. Configuration of the pullout test for a specimen with 50 mm of embedding

To enable the subsequent statistical analysis of the results obtained for the pullout test, a total of 40 samples were molded, which were equally divided into: reference specimens for the pullout test, composite reinforced with untreated sisal fiber (CRSF-UTSF) and specimens for pullout testing, composite reinforced with sisal fiber subjected to polymeric treatment with RHS (CRSF-PRHS). The aforementioned samples were tested at the control ages of 07 and 28 days.

4. Results analysis

4.1. Evaluation of fiber absorption capacity

For the evaluation of the water absorption index of the fibers, an AI of 84.67% was obtained through Equation (1) for the natural sisal fibers and 15.18% for the fibers submitted to the polymeric treatment based on EPS and RHS. It is possible to notice that, after the treatment application, the decrease in the index of absorption of the fibers is around 70%.

Similarly, for sisal fibers treated by the hornification process (Ferreira et al., 2020), a 30% decrease in water absorption by the fibers was obtained. Literature (Brancato, 2008) observed decreases of up to 50% in the water retention capacity of cellulose fibers after applying cycling treatments. Ferreira et al. (2020) observed decreases of 15%, 17.50%, 25%, and 50% for sisal fibers treated, respectively, through hornification, alkaline treatment with calcium hydroxide, polymeric impregnation with styrene butadiene, and hybrid treatment—a combination of hornification treatments and polymeric impregnation. Furthermore, for eucalyptus fibers treated with tetraethyl ortho-silicate (TEOS 98%), Defoirdt et al. (2010) found a 32% reduction in water absorption.

Based on the literature, for other types of treatment in natural fibers, this behavior can be explained due to the stiffening of the structure of the sisal fibrocells resulting from the treatment process used, allowing a greater packing of the internal structure of the fiber.

However, the behavior of a large drop in water absorption for the bundle of fibers that received treatment can also be attributed to the choice of polymer used for its application, since EPS is a nonpolar compound of hydrophobic nature. This polymer is characterized by having little or no interaction with water. Additionally, hydrophobic materials have the ability to form a film on the surface in contact, which may have caused a lower absorption rate for treated fibers, as it acted by repelling water molecules.

4.2. Microstructural analysis

Figures 18 and 19 show the surface characteristics of sisal fibers before and after treatment with EPS and RHS polymer.

From the images obtained for the surface of sisal fibers with the aid of an optical microscope, it becomes noticeable that the treatment created a discontinuous layer in the longitudinal wrapping of the fiber, causing agglutination points of the polymeric solution of EPS and RHS. Solvent evaporation due to the high viscosity increase of the mixture when exposed to the environment can be indicated as one of the main factors for this event.

Another relevant aspect is the possible lack of interaction between the natural fiber and the solvent used (toluene) to solubilize the mixture, or even with the polymer since this would act as the binding element in the

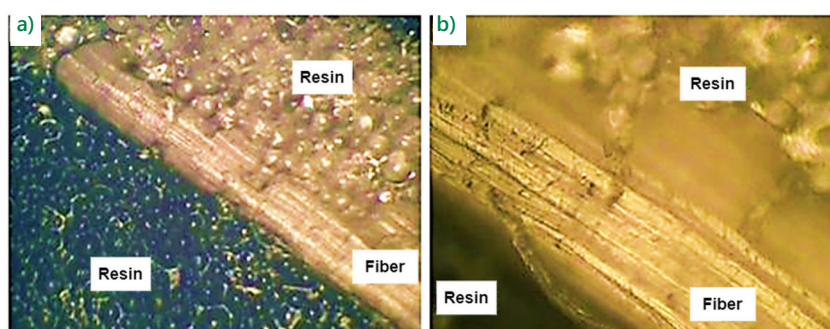


Figure 18. Optical microscopy images of the untreated sisal fiber surface: a) 100x magnification; b) 200x magnification

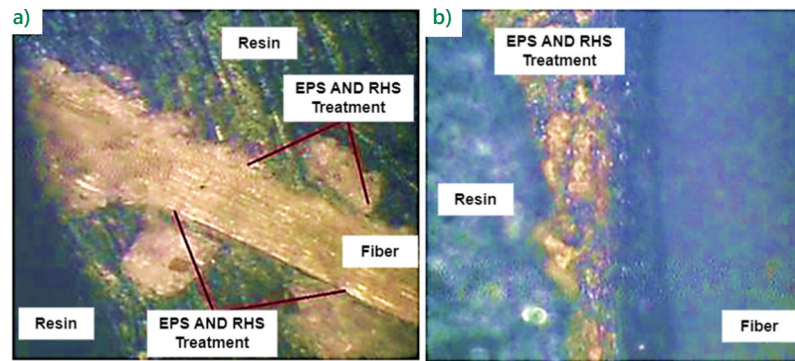


Figure 19. Optical microscopy images of the surface of sisal fiber chemically treated with EPS and RHS: a) 100x magnification; b) 200x magnification

polymer-silica-fiber aspect. Thus, as a treatment used which consisted of dissolving the polymer and silica in the aforementioned solvent, one of the probable causes of the non-uniformity of the treatment is that it was not absorbed homogeneously by the pores of the fiber, causing points of excess.

It is identified that the treatment may have been effective in terms of fiber sealing because, as can be seen in Figure 19b, there is a modification of the surface of the fibers caused by the treatment, where fiber encapsulation is observed by the polymeric solution with RHS. However, when checking the microscopy images obtained, it is noted that the use of the treatment seems to configure a very thick layer, which may negatively influence the interaction of the fiber with the matrix. However, it may be effective when analyzing the fiber as a single element, in the case of the direct tensile test (DTT).

4.3. Direct fiber traction

Table 4 shows the data obtained for average, maximum and minimum displacement and force, standard deviation and coefficient of variation comparing 10 samples of sisal fibers without treatment and 10 samples of sisal fibers treated with EPS and RHS.

Table 4. Direct traction test for DTT-UTSF and DTT-PRHS

DTT-UTSF			DTT-PRHS	
Rupture displacement (mm)		Rupture strength (N)	Rupture displacement (mm)	Rupture strength (N)
Average	2.800	10.109	2.198	10.705
Minimum	0.973	5.151	1.199	7.515
Maximum	5.877	15.730	6.119	17.805
Standard deviation	1.929	4.149	1.484	3.032
Coef. variation	68.89%	41.04%	67.53%	28.32%

Since the results obtained from direct traction for all untreated and treated samples showed great variability,

it was decided to carry out a selection of the force and displacement data, doing this visually through the correlation between those that presented rupture forces with lower variation. In this way, the average displacement and rupture force were verified, which are presented in Table 5.

Table 5. Average rupture displacements and forces for DTT-UTSF and DTT-PRHS

DTT-UTSF			DTT-PRHS	
Rupture displacement (mm)		Rupture strength (N)	Rupture displacement (mm)	Rupture strength (N)
Average	2.745	12.566	1.775	10.216
Standard deviation	1.677	3.395	0.675	1.836
Coef. variation	61.11%	27.02%	38.02%	17.98%

Analyzing the averages obtained, it is noted that the surface treatment was not able to increase the mechanical properties, maintaining only approximate normal levels when compared to the breaking force supported by the sisal fiber only in its natural state. Still, it appears that the values obtained for those without treatment are within the range found by other authors, such as (Ferreira et al., 2020; Muthukumaran et al., 2017; Pichaipillai et al., 2023), who, in their studies, obtained an average breaking strength of 10.28 N for natural sisal fibers under direct traction. Figures 20 and 21 show the curves for sisal fibers in their natural state and for those subjected to polymeric treatment with EPS and RHS, respectively, for the results shown in Table 5.

Based on the curves presented, it can be seen that the levels reached by the breaking strength are variable, which can be explained by the irregularity of the fiber diameter. According to Silva et al. (2011), sisal fibers have a hierarchical structure with variable morphology, including an irregular cross-section. Therefore, to obtain accurate results regarding resistance to direct traction and deformation, the irregular areas of the fibers must be considered, and tensile stress calculated accordingly.

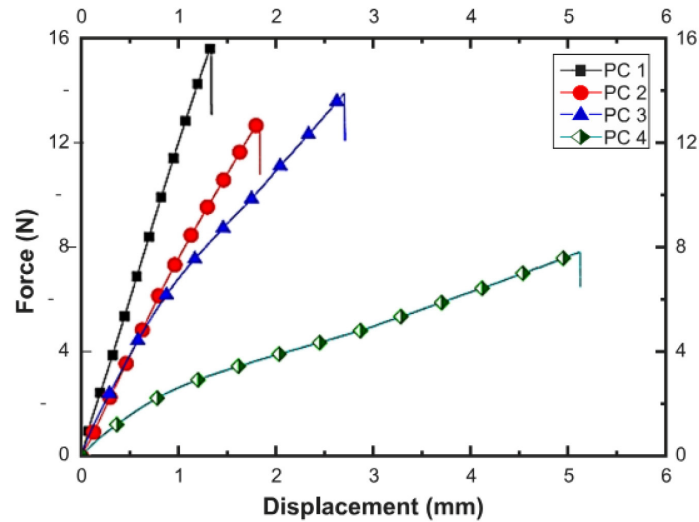


Figure 20. Force x Displacement Curves for DTT-UTSF

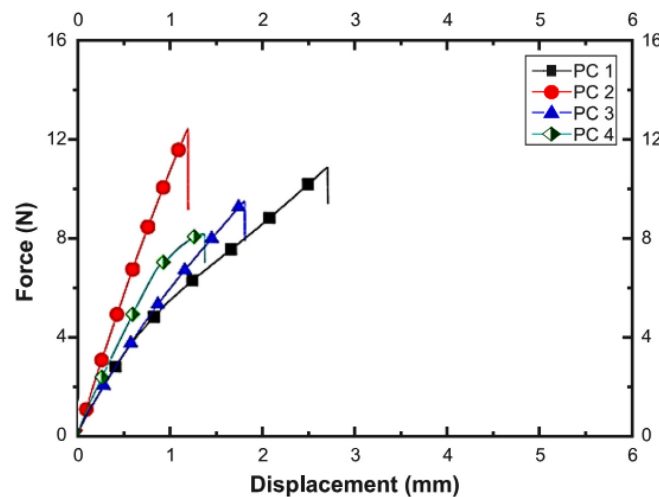


Figure 21. Force x Displacement Curves for DTT-PRHS

It is also noteworthy that the treatment was not able to significantly change the mechanical properties of the sisal fiber, as the breaking strength did not increase, remaining stable. This fact can be explained due to the variation in the cross-sectional shape of sisal fibers. However, the polymeric treatment combined with the addition of RHS may have been sufficient to perform the dimensional stabilization of the fiber, as can be seen through the analysis of images presented, where the encapsulation of the fiber by the polymeric EPS and RHS solution is observed.

Thus, even seeking to relate only the less discrepant data for rupture force to direct traction, it is possible to notice that there is a smaller variation in the average displacement and rupture force for those that were submitted to the treatment. This indicates that even though the treatment did not cause an increase in traction force, it provided better homogeneity in the Force x Displacement relation. This becomes noticeable when carrying out the graphical analysis of the breaking strength, as peaks in the

breaking strength for direct traction supported by the natural fibers are less pronounced when graphically observing the behavior of the treated fibers. They show greater homogeneity in the data, with closer results of force and displacement of rupture.

4.4. Pulling test

For cementitious composites reinforced with untreated sisal fibers and treated with EPS polymer with RHS, the mean force and displacement, minimum and maximum rupture strength, standard deviation and coefficient of variation were verified for all specimens at the control ages of 7 and 28 days.

Table 6 presents the results obtained for CRSF-ST and CRSF-PRHS at the control age of 7 days, considering that 10 specimens were analyzed for those using natural fibers and 10 specimens for those using fibers subjected to treatment.

Table 6. Mean, minimum and maximum value, standard deviation and coefficient of variation of the breaking load, displacement for CRSF-UTSF and CRSF-PRHS with 50 mm of fiber embedding at the age of 7 days

CRSF-UTSF			CRSF-PRHS	
Rupture displacement (mm)		Rupture strength (N)	Rupture displacement (mm)	Rupture strength (N)
Average	1.533	6.402	1.247	5.244
Minimum	1.044	2.113	0.329	1.583
Maximum	2.890	10.158	2.685	14.632
Standard deviation	0.534	2.636	0.731	3.910
Coef. variation	34.84%	41.17%	58.61%	74.56%

Table 7 shows the data acquired through the pulling test for CRSF-UTSF and CRSF-PRHS at the 28-day control age, in which the number of specimens used was the same as that mentioned for those broken at the 28-day control age. 7 days.

Table 7. Mean, minimum and maximum value, standard deviation and coefficient of variation of the breaking load, displacement for CRSF-UTSF and CRSF-PRHS with 50 mm of fiber embedding at the age of 28 days

CRSF-UTSF			CRSF-PRHS	
Rupture displacement (mm)		Rupture strength (N)	Rupture displacement (mm)	Rupture strength (N)
Average	1.330	7.729	0.962	5.731
Minimum	0.445	2.183	0.179	2.080
Maximum	2.275	15.120	1.827	13.318
Standard deviation	0.693	4.514	0.623	3.388
Coef. variation	52.10%	58.41%	64.81%	59.11%

Comparing the results obtained for composites reinforced with sisal fibers chemically treated by the fiber immersion process in EPS polymeric solution with RHS with those in which natural fibers with the same embedded length were used, it can be observed that the use of treatment impaired the adhesion performance of the fibers with the matrix. The average breaking strength supported by the composites reinforced using sisal fibers in a natural state was 18.09% and 25.85% higher at 7 and 28 days, respectively, compared to that obtained for those using treated fibers.

The deviations obtained also indicate that the treatment resulted in greater variability in the results for both strength and rupture displacement, which can be explained by the non-uniformity of the process used to treat the fibers. This statement is supported by the analysis of microscopic images of the surface of the fibers, as explained earlier, in which the formation of an enveloping film with

an irregular layer can be visualized due to the increase in the viscosity of the polymeric solution when exposed to room temperature.

Another aspect to be considered is the possible sealing of the fiber due to the treatment used, creating a very thick layer of EPS combined with RHS, as shown in the images. Such an occurrence may have promoted a slippery layer, originating points susceptible to loss of adherence of the fiber with the matrix when absorbing the pullout load. In this way, the dimensional variation caused by the coating created on the sisal fiber reduced the transmission capacity of efforts, weakening the interfacial connection, and thus forming a fiber-polymer-matrix interaction.

Data that presented less variability in relation to the pullout load supported by the composites were selected in order to enable the performance of graphic analysis with a smaller standard deviation and coefficient of variation. Thus, Tables 8 and 9 show data on force and mean rupture displacement, standard deviation, and coefficient of variation for 4 of the specimens in which the closest results were obtained when relating force and rupture displacement to CRSF-UTSF and CRSF-PRHS submitted to the test of pullout at the control age of 7 and 28 days, respectively.

Table 8. Mean value, standard deviation and coefficient of variation of the breaking load and displacement for CRSF-UTSF (PC 2, PC 4, PC 8 and PC 10) and CRSF-PRHS (PC 2, PC 5, PC 6 and PC 10) with 50 mm of fiber soaking at 7 days

CRSF-UTSF			CRSF-PRHS	
Rupture displacement (mm)		Rupture strength (N)	Rupture displacement (mm)	Rupture strength (N)
Average	1.302	8.784	1.113	6.008
Standard deviation	0.193	1.180	0.353	2.082
Coef. variation	14.85%	13.44%	31.71%	34.65%

Table 9. Mean value, standard deviation and coefficient of variation of the breaking load and displacement for CRSF-UTSF (PC 2, PC 4, PC 8 and PC 10) and CRSF-PRHS (PC 2, PC 5, PC 8 and PC 9) with 50 mm of fiber soaking at 28 days

CRSF-UTSF			CRSF-PRHS	
Rupture displacement (mm)		Rupture strength (N)	Rupture displacement (mm)	Rupture strength (N)
Average	1.147	12.205	1.343	8.798
Standard deviation	0.509	3.357	0.486	3.087
Coef. variation	44.37%	27.50%	36.20%	35.09%

It is noted that even when attempting to use only the composites that obtained similar behavior in terms of breaking strength, there is a decrease in the variation of the data both at 7 and 28 days. This fact may be explained

by the inconsistency of the cross-section of the sisal fibers, not only because of the irregularity of the proposed treatment.

The relation between the rupture forces obtained for the specimens tested at the age of 7 days did not show significant variation, so they maintained a similar average. However, at the age of 28 days, even when observing related data according to their proximity, there is still a decrease in terms of the breaking load supported by the composites, with untreated fibers presenting an average 27.91% greater adhesion performance compared to the composites that used treated fibers.

Figures 22 and 23 show the Force x Displacement curves obtained through the pullout tests at 7 days for composites reinforced with untreated and treated sisal fibers, using data previously selected based on proximity to the results.

Thus, when observing Figures 22 and 23 for the results of the pullout test, it can be noted that despite having a lower breaking strength, the composites in which the treated fibers were used show strength peaks during displacement, indicating points where there was a better adhesion interaction between fiber-polymer-matrix. Additionally, it is noticeable that before reaching breakage, the levels of resistance strength oscillate and do not undergo brittle breakage as observed in composites using natural fibers.

The load x displacement curves for the treated and untreated fiber-reinforced composites tested at 28 days of age for the selected data are shown in Figures 24 and 25.

Thus, when analyzing the strength levels obtained for the treated fibers, it can be seen that this treatment impaired the interaction of the fiber with the cementitious matrix at both rupture ages, causing an interaction

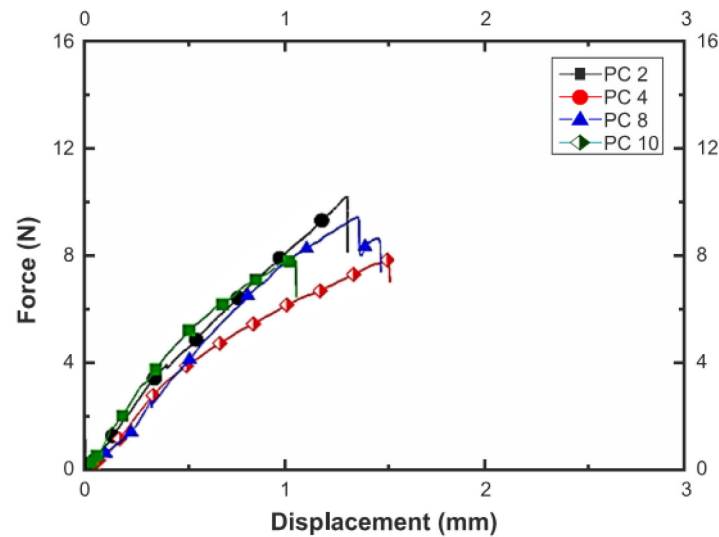


Figure 22. Force x Displacement curves for CRSF-UTSF pullout test at 7 days

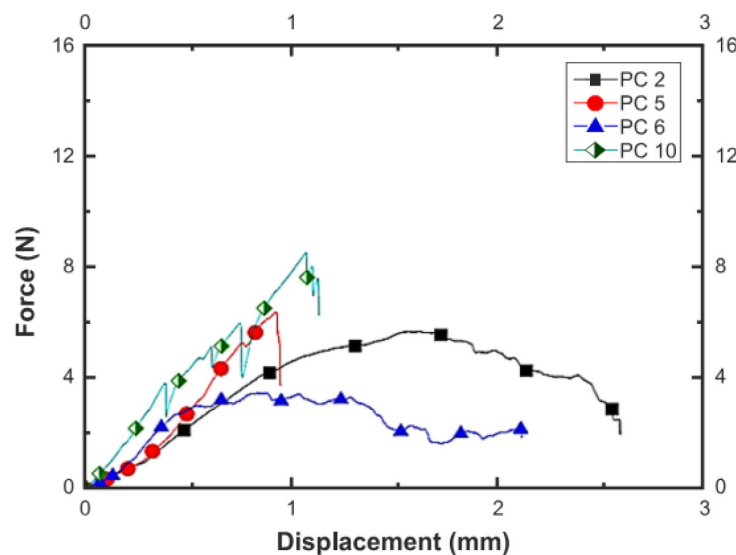


Figure 23. Force x Displacement curves for pullout test in CRSF-PRHS at 7 days

between fiber-polymer-matrix. As a justification for such an occurrence, the possibility is pointed out that, as it is a polymeric treatment with a hydrophobic polymer that creates a sealing layer to the fiber by repelling water molecules, the proposed treatment may not have allowed the paste to penetrate into the fiber pores, and as a consequence caused a decrease in adherence.

The conceptual discussion surrounding the novel results of the research on cementitious composites reinforced with sisal fibers treated with EPS polymer and RHS encompasses several key aspects. Firstly, the significant reduction in water absorption of sisal fibers after treatment with polymeric EPS and RHS is noteworthy. This reduction, approximately 70%, suggests improved durability and performance of the treated fibers in cementitious composites, which is crucial for enhancing the longevity and structural integrity of these materials.

Secondly, the microscopic analysis revealing a discontinuous layer on the fiber surface post-treatment indicates a potential trade-off between fiber sealing and matrix interaction. While the treatment effectively seals the fibers, creating encapsulation by the polymeric solution with RHS, it may also hinder fiber-matrix interaction in composite materials. This finding highlights the need for further optimization to balance the benefits of fiber sealing with the potential drawbacks of reduced interaction with the matrix, thereby advancing the understanding of fiber treatment effects on composite performance.

Furthermore, the detailed investigation of direct traction tests comparing untreated and treated specimens provides valuable insights into the treatment's effect on fiber behavior. Although the treatment didn't substantially increase traction force, it notably enhanced the uniformity of force-displacement behavior, indicating improved

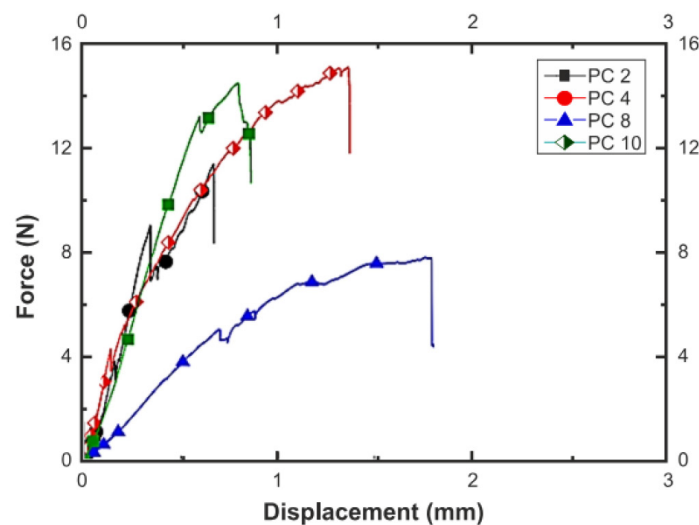


Figure 24. Force x Displacement curves for CRSF-UTSF pullout test at 28 days

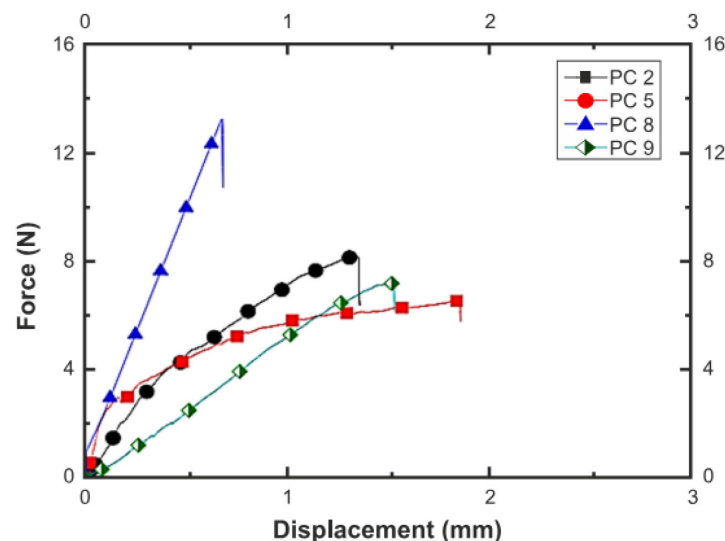


Figure 25. Force x Displacement curves for pullout test in CRSF-PRHS at 28 days

fiber-matrix interactions. This underscores the treatment's potential to optimize composite material performance by enhancing homogeneity in mechanical properties, despite not significantly altering individual fiber strength.

Lastly, the study's findings regarding the compromise in fiber-matrix adhesion due to the treatment, leading to lower breaking strengths and increased result variability, shed light on the complex interplay between fiber treatment and composite performance. The formation of a sealing layer by the hydrophobic polymer, limiting paste penetration into fiber pores and reducing adherence, underscores the need for a nuanced approach to fiber treatment to optimize composite properties effectively.

In summary, the research provides valuable insights into the multifaceted relationship between fiber treatment, fiber-matrix interaction, and composite performance, highlighting the importance of continued research and optimization efforts in this field to advance the development of sustainable and high-performance cementitious composites.

5. Explanation of results

5.1. Evaluation of fiber absorption capacity

The results indicate a substantial reduction (approximately 70%) in the water absorption index for sisal fibers treated with EPS and RHS. This reduction is significantly higher compared to other treatments reported in the literature, such as hornification (30%) and calcium hydroxide-based alkaline treatments (17.5%). The reduced absorption is attributed to the hydrophobic nature of EPS, which repels water molecules and forms a protective film around the fiber. This encapsulation stiffens the fibrous structure, leading to a lower water absorption rate.

5.2. Microstructural analysis

Optical microscopy images of treated and untreated sisal fibers reveal that the polymeric treatment modifies the fiber surface by creating a discontinuous layer of EPS and RHS. While this layer effectively seals the fiber, improving its water resistance, it also appears thick and non-uniform. This irregularity arises from solvent evaporation and the potential lack of interaction between the fiber and the toluene-based solution, leading to points of polymer agglomeration. Despite this, the treatment seems effective for encapsulating the fiber, though the thick layer might negatively affect fiber-matrix interaction in composites.

5.3. Direct fiber traction

The direct traction test results for untreated (DTT-UTSF) and treated (DTT-PRHS) fibers highlight key insights:

- The treated fibers exhibit reduced rupture displacement (average: 1.775 mm vs. 2.745 mm for untreated fibers) but slightly lower rupture strength (average: 10.216 N vs. 12.566 N).

- While treated fibers did not show a significant increase in breaking strength, the polymeric treatment reduced variability in the results, as seen from the lower coefficient of variation for treated fibers (17.98% for strength) compared to untreated fibers (27.02% for strength).
- The graphical force-displacement analysis shows greater homogeneity in treated fibers, likely due to dimensional stabilization from the polymeric encapsulation. However, the hierarchical and irregular structure of sisal fibers still contributes to variability in results.
- The findings suggest that the treatment enhances the uniformity and stability of fiber behavior but does not significantly improve tensile strength. This stability is beneficial for applications where consistency in performance is crucial.

5.4. Pulling test

For cementitious composites reinforced with untreated (CRSF-ST) and treated (CRSF-PRHS) sisal fibers, the results from specimens tested at 7 and 28 days reveal:

- Improved Consistency: Treated fibers show reduced variability in rupture strength and displacement compared to untreated fibers. This aligns with findings from the direct traction test, where treated fibers demonstrated more homogeneous behavior.
- Limited Strength Improvement: The treated fibers do not significantly enhance the mechanical performance of the composite. Instead, the primary benefit is dimensional stabilization and improved interaction consistency within the matrix.

5.5. Breaking strength and displacement

At 7 days:

- Untreated Sisal Fibers (CRSF-UTSF): The average breaking strength (6.402 N) and displacement (1.533 mm) were higher than those of treated fibers.
- Treated Sisal Fibers (CRSF-PRHS): The treatment resulted in lower average breaking strength (5.244 N) and displacement (1.247 mm).
- Observation: Untreated fibers displayed better adhesion with the matrix, allowing for higher force transfer before rupture. Treated fibers showed more variability (higher coefficients of variation) due to irregularities in the treatment process, which compromised adhesion.

At 28 days:

- Untreated Fibers: The average breaking strength (7.729 N) and displacement (1.330 mm) were again higher than treated fibers.
- Treated Fibers: The average breaking strength (5.731 N) and displacement (0.962 mm) were lower, reinforcing the adverse impact of the treatment on adhesion performance.

- Observation: As composites aged, hydration of the matrix typically improves fiber-matrix interaction. However, the sealing effect of the polymeric layer on treated fibers hindered this improvement, causing a decrease in adhesion and breaking strength compared to untreated fibers.

5.6. Effect of fiber treatment on fiber-matrix interaction

Reduced Adhesion:

- Microscopic analysis revealed that the treatment created a non-uniform, hydrophobic polymer layer that encapsulated the fibers. This layer reduced the matrix's ability to penetrate the fiber pores, leading to weaker adhesion.
- The sealing layer also acted as a slippery interface during pullout, diminishing the transmission of loads.

Improved Uniformity:

- While the treatment reduced overall breaking strength, it led to more consistent force-displacement behavior in some cases. This indicates that while adhesion was impaired, the treated fibers' behavior under load became less brittle and more predictable.

5.7. Variability in results

Higher Coefficient of Variation:

- Treated fibers showed significantly higher coefficients of variation in both displacement and strength compared to untreated fibers. This suggests that the treatment process lacked uniformity, leading to inconsistent fiber surface properties and performance.
- Possible reasons include variations in polymer viscosity and fiber coating thickness during treatment.

5.8. Force-displacement behavior

Untreated Fibers:

- The curves exhibited sharp peaks and a brittle failure pattern, reflecting strong but localized fiber-matrix adhesion.

Treated Fibers:

- The force-displacement curves displayed oscillations before rupture, indicating intermittent adhesion points where the fiber-matrix interaction momentarily improved. This pattern reflects the formation of localized adhesion zones due to the polymer layer, despite overall weaker bonding.

5.9. Microscopic and mechanical evidence

The polymeric treatment reduced water absorption by ~70%, enhancing fiber durability and resistance to degradation. However, this hydrophobic property also impaired bonding with the cementitious matrix, a critical trade-off.

The presence of a sealing layer disrupted the uniform transmission of stress across the fiber-matrix interface, further reducing mechanical performance.

5.10. Comparison across ages

7 days vs. 28 days:

- At both ages, untreated fibers consistently outperformed treated fibers in terms of breaking strength and displacement.
- The relative decrease in treated fiber performance at 28 days underscores the sustained adverse impact of the polymer layer on long-term fiber-matrix bonding.

6. Conclusions

The present study aimed to investigate the influence of polymeric treatment with EPS and RHS on sisal fibers to evaluate their absorption capacity, surface modifications after treatment, behavior under direct traction, and comparison with untreated sisal fibers. Additionally, the study aimed to analyze the adhesion behavior of sisal fibers when used to reinforce Portland cement matrices and compare composites using natural and treated fibers.

Regarding water absorption capacity, it was observed that fibers treated with EPS and RHS polymer obtained a lower index than natural fibers. This decrease may be attributed to a reduction in the rate of fiber capillary voids, possibly due to the closure of these voids after treatment application or the hydrophobic nature of the polymer used, which repels water molecules.

Surface microstructure analysis revealed that the fiber was sealed through treatment, but agglutination points were also observed. The thick protective layer formed by the treatment with EPS and RHS may have negatively affected fiber-matrix interaction but provided better homogeneity when analyzing treated fibers individually.

Results of the direct traction test showed significant differences in resistance capacity between natural and treated fibers. However, the lack of diameter verifications hindered the evaluation of how fiber diameter inconsistency influenced results variation.

Concerning sisal fiber's adhesion to the cementitious matrix, the treatment applied was insufficient to enhance adherence at the fiber-matrix interface, resulting in a distinct interface of fiber-polymer-matrix interaction. Modification of sisal fiber morphology did not occur through treatment, potentially reducing variability in load supported by fibers during pullout tests.

A significant factor contributing to the decrease in fiber anchoring within the cement matrix could be attributed to potential inconsistencies arising from the method and materials used for treating the sisal fibers. The rapid evaporation of the solvent likely led to an increase in EPS viscosity, forming a thick treatment layer on the fiber's surface, impeding adhesion at the fiber-matrix interface.

The proposed treatment to standardize the fiber structure and improve its mechanical capacity of traction and adherence was not effective. Future work suggestions include tests considering fiber diameter variation, analyzing polymeric treatments alongside fiber bleaching by alkaline

attack, exploring other types of binder and solvent polymers, and evaluating the use of different additions to promote better fiber-matrix adherence, such as Silica 325 or fine sand.

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Appendix

Graphical abstract

