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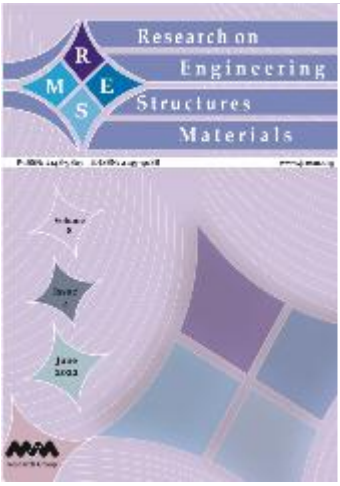

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Research Article

Effect of calcined clay on the properties of cementitious mortar reinforced by Posidonia Fiber

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Abstract

Posidonia fiber has a negative visual impact on tourism and produces bad odors due to the decomposition of microorganisms. Thus, its recovery for waste recycling and other sustainable uses is encouraged. In this context, the objective of this work was to study the influence of calcined clay as a mineral coating for fibers and the surface treatments (hydro-thermal by water boiling and chemical treatment by NaOH) on the durability of Posidonia fiber and to evaluate their effect on the physical and hygroscopic properties, rheological, mechanical and micro-structural performances, and chemical attack of cementitious mortars with supplementary materials. An experimental study was carried out by varying volume ratios (5%, 12.5%, and 20%). The effects of these treatments on the physical, thermal and morphology properties of the fibers were determined. The water absorption of mortar reinforced by fibers coated with calcined clay decreased compared to composite reinforced by fibers treated without coating. The calcined clay makes the fibers relatively less hydrophilic. According to the various mechanical tests, adding treated Posidonia fibers in a pozzolanic matrix improves the composite properties.

1. Introduction

Because of the various ecological and environmental problems, extensive research has been conducted over the recent decades on the use of vegetable fibers, e.g., flax, hemp, date palm, and jute, as reinforcing materials. These fibers are biodegradable and renewable [1, 2, 3]; however, they are hydrophilic because they are removed from cellulose. Generally, plant fibers are physically characterized by their density, length, and diameter [4]. The low density of plant fibers gives these materials specific mechanical properties [5]. Plant fibers are distinguished by their chemical composition, i.e., cellulose, hemicelluloses, lignin, pectin, waxes, and water [6], which determines the physical properties of fibers [7]. Plant fibers trap water between micro-fibrils due to the presence of hemicelluloses. This hydrophilic character is an essential feature to consider in the case of vegetable fibers. Indeed, water will influence the behavior of cementitious composites in the hardened state (mechanical properties, porosity, etc.) and the fresh state (workability and flow time) ([5, 8]. Furthermore, fluid movement is a central feature of the durability of cement materials [9, 10, 11].

Cement composites reinforced with natural fibers have recently been considered a viable alternative to traditional composites (steel and synthetic fibers). However, the main hurdle in their application remains their lack of long-term durability in the highly

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alkaline environment of the Portland cement matrix. Several studies have revealed that plant fibers exhibit limited resistance to alkalis [7, 12, 13, 14]. Alkaline environments, especially in lime released by cement during hydration, limit the use of fibers in cement, as lime causes fiber degradation or tensile strength decrease. Fiber degradation in the cement matrix occurs because of the dissolution of lignin and hemicelluloses due to the adsorption of calcium and hydroxyl ions [12, 13, 15]. In addition, a change in the method of fibers' fracture was noticed after long-term aging. Fiber mineralization is caused by the migration of hydration products (calcium hydroxide) on the fiber's surface and into the fibrous cell (lumen) simultaneously after prolonged contact with the cement matrix, causing deterioration and hardening of its structure. As a result, the fibers become brittle, and the transfer of strength between them and the matrix will be limited [7, 12, 13, 14, 15, 16]. Therefore, two solutions have been proposed to protect natural fibers from degradation in cementitious composites. The first solution is the modification of the matrix by decreasing its alkalinity, and the second entails treating the fibers' surface before adding them to the matrix [12, 13,14].

The use of low-alkali cementious the most widely used method of reducing matrix alkalinity. Some researchers suggest replacing part of the cement with slag, fly ash, or pozzolanic materials. For example, materials based on natural volcanic ash, metakaolin, and calcined clay can improve the durability of composites [7, 17, 18]. Calcined clays are recognized as pozzolanic substances because of their composition, silica or silica-alumina compounds [19, 20]. Using natural calcined clay as a supplementary cementitious material that substitute's part of cement in concrete is gaining significant attention[21]. Its application not only helps decrease the cement content in concrete production but also enhances the strength and durability of structural concrete [21, 22, 23]. Incorporating calcined clay is crucial in influencing the mechanical properties of mortars and concrete [17, 21, 24]. Calcium Aluminates hydrates (CAH) and calcium silicate hydrate (CSH) intensify as the addition percentage rises. These compounds play a significant role in enhancing the strength of the material, likely influenced by the mineral content. Besides, their chemical reaction with the calcium hydrate (CH) released during cement hydration further enhances this strength [21, 25]. Additions reduce pores sizes and change their distribution [18, 21, 24. The introduction of pozzolans results in closed porosity, leading to enhanced mechanical strength and durability. This improvement is attributed to the increased formation of CSH and the lack of interconnected pores. Consequently, integrating pozzolans into the cement promotes better hydration, reduces the number of pores, and decreases the volume of mixing water.

Thus, the coating of lignocellulose fibers can be achieved with a pozzolanic material, such as metakaolin [26] and silica fume [14], to reduce water absorption of fibers and thus increase the mechanical properties and fiber/matrix adhesion. Fiber absorption has been reduced, and the probability of a cavity between the fiber and the matrix is lower [8]. The replacement of cement with metakaolin and bentonite improves the sisal fibers' durability in cement-based materials as they increase their flexural strength [15]. Wei et al. [27] reported that adding supplementary cement materials (metakaolin, fly ash, etc.) could significantly slow down the degradation of natural fibers.

Fiber surface treatment is needed to improve the fiber/matrix interface, moisture resistance, and fluid absorption [10,11]. Alkaline treatment partially removes hydrophilic hydroxyl groups of natural fibers. It makes the fiber surface rougher. Naiiri et al. [28] studied the surface modification of palm fibers, and they showed that treatment with soda at a concentration of 1% for one hour improves tensile strength, which may be attributed to an increase in the arrangement of the cellulose and a decrease in the lignin content in the fiber. After alkaline treatment, the removal of amorphous parts increases the crystallization rate of the fibers [29]. They can also be easily treated with other chemicals, such as silane or acetic acid [28,30, 31,32].

Research on composite materials incorporating vegetable fibers has shown that the mechanical properties of composites improve. Ajouguim et al. [33, 34, 35] highlight the shift from a fragile composite to a ductile composite that exhibits controlled post-peak behavior. However, this change in behavior does not always lead to an improvement in bending resistance [36]. Generally, the compressive strength of composites is not significantly affected by the incorporation of vegetable fibers [7]. Asma et al. [4] show that compressive strength decreases with fiber volume. Chafei [8] asserts that incorporating flax fibers reduces compressive strength significantly. This decrease is due to poor fiber/matrix adhesion and the increased size and number of occluded pores in cement [31]. However, other researchers attributed this reduction to an increase in the number of defects and a non-homogeneous distribution of fibers. Composite porosity increases with the volume fractions of fibers, leading to a decreased density of composites and decreased mechanical properties [7].

The Posidonia fiber represents natural resources found on the beaches of the Mediterranean in the form of balls carried by the waves. Despite its crucial ecological role in protecting against erosion, it has a negative visual impact on tourism and produces bad odors due to the decomposition of microorganisms [37]. Thus, the recovery of Posidonia for waste recycling and other sustainable uses is encouraged. Several researchers have studied the effect of adding Posidonia fiber to a cement matrix on the physical, mechanical, thermal, and morphological properties. The results show that the bio-composite gives better bending resistance, higher ductility, lower density, and a better matrix/fiber interface than conventional cement materials [32, 38, 39, 40].

The present work aimed to study the effect of calcined clay as a mineral coating for fibers and surface treatments (hydro-thermal by water boiling and chemical treatment by NaOH) of Posidonia fiber on the physical and hygroscopic properties, rheological, mechanical, and micro-structural performances, and chemical attack of cementitious mortars with supplementary materials.

2. Experimental Program

2.1. Materials and Characterization

2.1.1 Sand Cement and Calcined Clay

A Portland cement type CEM I 42.5 MPa and standard sand of EN 196-1 were used. Moreover, calcined clay at 600°C for one hour of calcination [17] was used as a mineral coating. The physical and mechanical properties, such as pycnometer density, Blaine surface area, average particle diameter, and pozzolanic activity index, were measured (Table1). The chemical composition and X-ray diffraction curve (mineralogical composition) of calcined clay were determined (Table 2 and Fig 1).

Table1. Properties of calcined clay

Properties	Calcinedclay (600°C/1h)
Density	2.48
The Blaine area(m2/Kg)	457
Average Particle diameter (μm)	0.96
Pozzolanic activity index	1.19

The chemical composition was determined by X-ray fluorescence. The results of the chemical analysis of calcined clay at 600°C show that their main constituents were silica (45.54%), alumina (16.71%), and iron oxide (11.11%). The calcined clay is a good pozzolanic addition in that the total percentage of SiO_2, Al_2O_3, and FeO are greater than 70% (73.26%)

Table2. Chemical composition of calcined clay

% by mass	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	k_2O	Na_2O	TiO_2	$CaCO_3$	LOT
600°C/1h	45.54	16.71	11.11	8.96	1.81	1.84	1.05	1.24	0.03	13.7	13.8

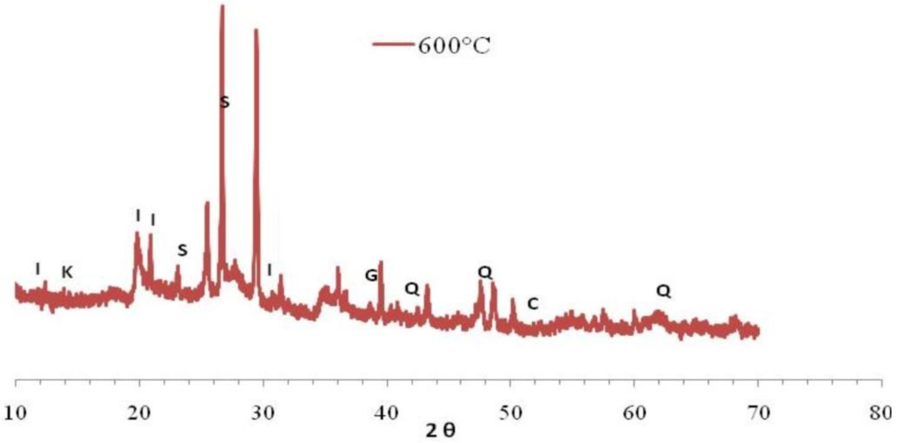

Fig. 1 Mineralogical composition of calcined clay (S: Smectite, I: Illite, K: Kaolinite, C: Calcite, Q: Quartz, G: Geothite)

2.1.2 Posidonia Oceanica Fibers

a. Extraction of Raw Fibers

The physical and mechanical properties of Posidonia fibers are comparable to other natural fibers commonly used in composites. Posidonia fiber is a natural fiber composed of celluloses (48.4%), lignin (23.12%), and hemicelluloses (18.9%) with a tensile strength equal to 5MPa [40].

The fiber of Posidonia oceanica, in the form of balls, was collected directly on the beach in Gabes-Tunisia. They are made of naturally agglomerated fibers. First, a manual extraction was necessary to separate the fibers and then rinsing in water several times to facilitate the removal of impurities, followed by drying in the air for days and in an oven at 65°C. After drying, crushing was carried out using a crusher (Retsch SM 100) to refine the size of the fibers and ensure the distribution and homogenization between the fibers and the matrix (Fig 2).

b. Surface Treatment of Raw Fibers

In this study, raw and crushed fibers are referred to as (T0). Two treatments were planned: a first combined treatment (T1): hydro thermal and chemical by NaOH for different fractions (0%, 5%, 12.5%, and 20%), and a second treatment (T2) combined and coated by calcined clay (12.5% E). Given the hydrophilic nature, the properties of the fibers had tobe modified by surface treatments to make them hydrophobic.

Fig. 2 Fibers from Posidonia Oceanica after crushing and sieving

The crushed fibers were boiled in water in a container for 30 minutes. Then, they were rinsed with water to remove impurities and organic matter, especially sugars [1]. Hydrothermal treatment alone does not profoundly affect the structure of the fibers. It is used only for the extraction of water-soluble materials [41]. Hence, additional treatment was proposed. In most cases, the chemical treatment of fibers with NaOH can improve their physical, mechanical, and insulating properties and promote their implementation, whether molded in the form of wool or mixed with materials (gypsum, cement matrix, etc.) [3].

Fig. 3 Fibers treated and coated with calcined clay

Mercerization of fibers promotes fibrillation, eliminates hemicelluloses, and makes the surface rougher [41]. In the present study, boiled fibers were immersed in NaOH solution with a molar concentration of 0.4 M for 2 hours. Subsequently, after chemical treatment, the fibers were rinsed with water to remove traces of NaOH and impurities until neutralization by checking the pH. Given that the interstitial alkaline solution of the cement matrix decreases the fibers’ durability and causes their degradation and microstructure modification [33, 34, 35], the mineral coating with a pozzolanic material was considered. This treatment consists of mixing treated fibers thermally and chemically with calcined clay to reduce the strong water absorption and make it waterproof and hydrophobic (Fig 3) [7].

2.1.3. Mixture Proportion

The composite consisted of a binder (90% cement + 10% calcined clay), water, and sand to form the matrix and the treated Posidonia fibers as reinforcement. The fibers were previously dried in an oven until a constant mass. The cement was initially mixed with 10% of calcined clay and water for a Water-Binder (W/B) ratio equal to 0.5(by mass). Afterward, the fibers and standard sand were added, in the wet state, for a Sand-Binder (S/B) ratio equal to 3 (by mass) to have better mixture homogeneity. The volume fractions of the fibers of the entire mix (sand: cement: water) were 0%, 5%, 12.5%, and 20%. Vegetable fibers have a significant impact on the handling and homogeneity of composites. For this reason, the mixing protocol is crucial as it affects the properties in the hardened and fresh state composite. Moreover, some protocols apply only to standardized mortars; thus, the presence of fibers is not taken into account [7], which is why a mixing plan that is more suited to the presence of fibers was developed. Some measures may be taken during the mixing process to reduce the effects of agglomeration. The fibers are usually added gradually at the end of the mixing process once the other ingredients are mixed [7]. Another critical aspect of the handling is the agglomeration of the fibers and the formation of pellets during mixing. The degree of agglomeration depends on the fraction volume, the length (crushing of fibers) and type of fibers used, and the maximum aggregate size in the composite. Fiber agglomeration should be avoided as it would affect resistance negatively. The mortar was put in cubic molds ($4*4*4cm^3$) to determine physical properties, in prismatic molds ($4*4*16$ cm^3) for flexural and compressive strengths, and in cylindrical ones ($11*22$ cm^2) for Brazilian tensile test (Fig 4). The molds were removed after 24h, and the samples were kept in water for 90 days.

Fig. 4 Cast samples to physical and mechanical properties

2.2. Test Methods

2.2.1. Rheological Properties

The workability of the pozzolanic mortar was determined to study its rheological behavior. The test was performed in accordance with NF P18-452.

2.2.2. Physical Properties

To determine the water-accessible porosity and bulk density of mortars, the gravimetric method was used in accordance with NF P18-459.

2.2.3. Hygroscopic Properties

a. Absorption-Sorption

To follow the water imbibitions kinetics of the mortars and in accordance with ASTM C1585-20, the samples were placed in an oven, with a gradual temperature rise of up to 80°C and obtaining a constant dry mass obtention. Then, a protective coating with an epoxy resin was applied to the circumference to avoid the accessibility of water from the sides. The samples were weighed, and then one side was put in contact with water up to 2 mm in height. The samples were taken after 15 min, 1h, 2h, 3h, 4h, 1d, 2d, 3d, 4d, and 7d. The mass gain due to water absorption was measured, and the capillary absorption coefficient (C) was calculated.

b. *Moisture Diffusion Under Controlled Relative Humidity*

The performance of mortar subjected to aggressive environments is a function of the penetrability of the pore system. The material is partially charged with moisture when exposed to a humid or aggressive atmosphere. The specimen is conditioned in an environment with controlled relative humidity to induce a consistent moisture condition in the capillary pore system. The moisture absorption or moisture diffusion kinetics of fiber-reinforced pozzolanic cementitious composites was measured (NF EN ISO 12571). These composites were previously dried in the oven at 80°C until a stabilized mass. The experimental device was composed of a sealed container to place the composites above a saline solution of a mass concentration of 35.9g NaCl/100g of water (HR= 79%). The samples were taken after 15min, 1h, 2h, 3h, 4h, 1d, 3d, 4d, and 7 days. Also, the moisture content of mortar in a controlled relative humidity environment (W) and the diffusion coefficient (D) were calculated.

2.2.4. Mechanical Properties

The mechanical tests for mortars were carried out in accordance with NF EN 196 and NF P18-400 standards.

2.2.5. Chemical Attack

To assess the samples' durability and chemical resistance to chemical attack (ASTM C267-20), we examined the change in specimens' weight and measured their compressive strength at 90 days of samples immersed in sodium chloride (NaCl). All specimens were compared before and after exposure.

2.2.6. Morphological Study

Scanning electron microscope (SEM-EDX) observations were used to compare the effect of fiber treatment on morphology, the interface matrix/fiber, and the pores size of the mortars.

3. Results and Discussions

3.1. Effect of Surface Treatment of Fibers

3.1.1. Physical Characterization

Fig 5 presents the particle size distribution of raw Posidonia fiber determined after sieving by laser diffraction apparatus. The raw and treated fiber exhibited a modal distribution. Table 3 shows that treated fibers' specific surface area (SSA) is higher compared to untreated fibers. It is a modification of the structure and the fibers. The mean diameter of treated fibers is higher, which could be attributed to an agglomeration effect [31].

Table3. Particle size distribution of treated and untreated Posidonia fibers

	SSA (m^2/Kg)	D_{50} (um)
Raw fiber T0	143.3	75
Treated fiber T1	153.1	85

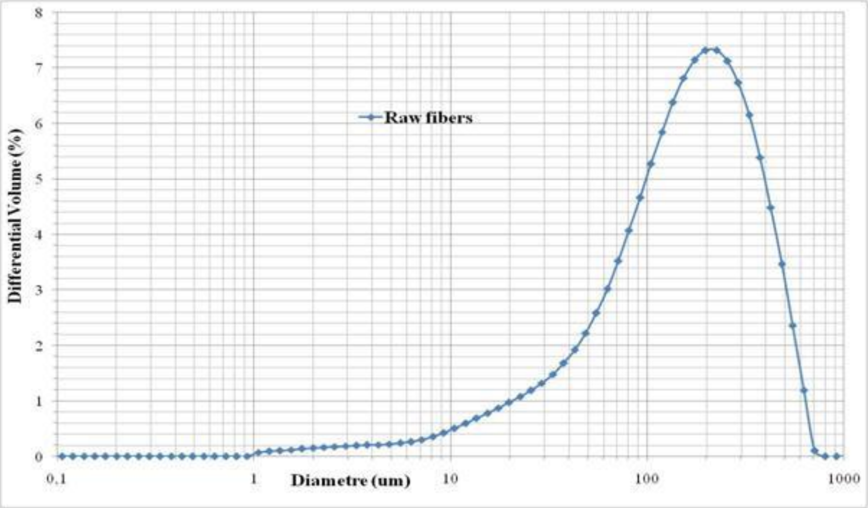

Fig. 5 The particle size distribution of raw Posidonia Fibers after sieving

To measure the fibers' water absorption (Fig 6), the natural water content, and the densities (Table4), the tests carried out were based on an experimental protocol developed by the RILEM TC 236-BBM group [7]**.** Fig 7 presents the percentage of mineral (C %) and organic (O %) composition of raw and treated Posidonia fibers determined by calcinations at 550°C.

Fibers treated with sodium hydroxide are lighter than raw fibers due to the removal of extractable and non-cellulosic materials on the fiber wall (lignin, sugar, etc.) [3]. The natural water content of treated fibers (T1) is lower than that of raw fibers, which explains the presence of organic matter in the raw fibers. The organic matter contents of the raw and treated Posidonia fibers are respectively 93.54% and 86.5%. Posidonia fibers have high organic matter content than the treated fibers. This finding shows that alkaline treatment appears to be effective for the removal of impurities and non-organic matter (sugars).

Table4. Physical properties of raw and treated fiber

Properties	Raw fiber	Treated fiber by NaOH
Bulk density (g/cm^3)	0.18	0.15
Density (g/cm^3)	1.49	1.34
Natural water content (%)	12.66	11.85

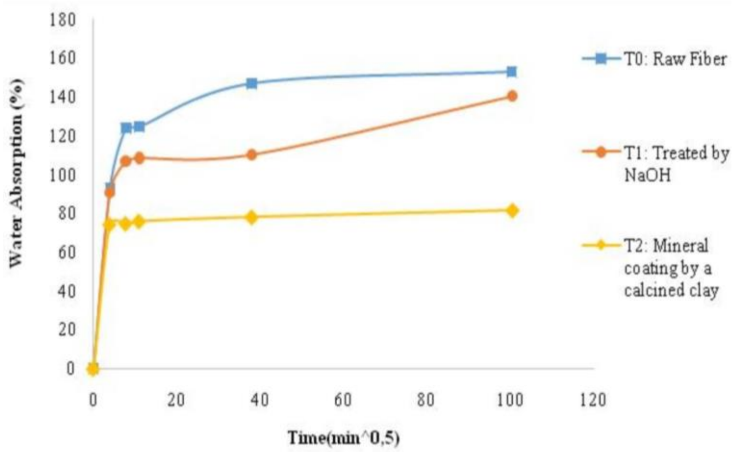

Fig. 6 Water absorption of raw and treated Posidonia fibers

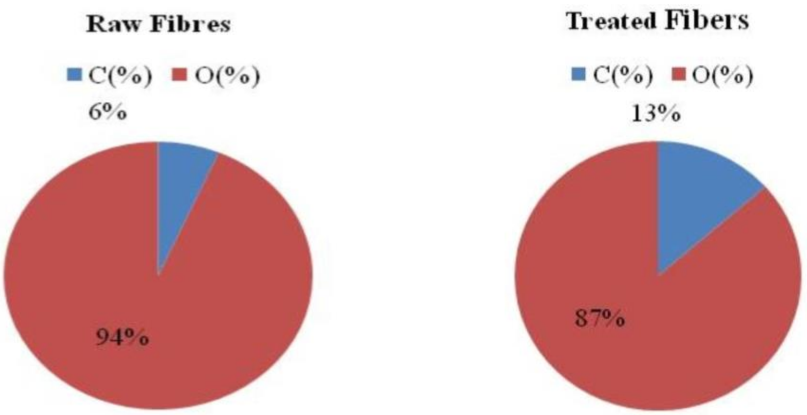

Fig. 7 The percentage of mineral (C %) and organic (O %) composition of raw and treated fibers

The general shape of the water absorption curves of raw and treated Posidonia plant fibers is similar to that of other plant fibers found in the literature [5, 7]. For Posidonia fibers, two stages of absorption can be distinguished: First, the mass of the samples increased rapidly during the first 15 minutes of immersion. In less than 60 minutes, the raw Posidonia fiber had absorbed almost 123%. Second, a continuous slow absorption occurred after two hours of immersion. Many properties of plant fibers, including physical and mechanical, are affected by water absorption [42]. Raw Posidonia fibers can absorb up to 146% of their mass after 24 hours of immersion. This high absorption capacity is mainly due to the capillary action produced by the high porosity of these fibrous structures and the presence of hydroxyl (-OH) and amorphous groups in the cellulosic structure of plant fibers. In the long term, this will result in significant dimensional changes in the fiber within the matrix [42]. Regardless of the type of fiber used, the literature on using plant fiber in the cement matrix indicates that a plant fiber absorbs much water and, in some cases, more than its weight. Fibers' water absorption kinetics allows knowing their intrinsic properties [43]. Indeed, the soda treatment has improved the Posidonia fiber absorption performance. On the contrary, another previous study also noted that the hydrophilic content of NaOH-treated fibers increased significantly [5].

3.1.2. Structural characterization

The treatment of Posidonia fibers with NaOH has an apparent effect on the infrared spectra (Fig 8). The Fourier transform infrared (FTIR) obtained using a spectrometry infrared UV allows checking the removal of lignin, pectin, and hemicelluloses. The alkaline treatment modifies the structure of the Posidonia fibers and reduces the intermolecular and intra-molecular hydrogen bond between the hydroxyl groups of cellulose and hemicelluloses of the vegetable fiber. After 2 hours of immersion in NaOH, the alkaline treatment causes damage to some components of Posidonia fiber, including lignin and hemicelluloses, which will be almost dissolved and extracted from the fiber. Indeed, the comparison of the two spectra shows the disappearance in the spectra of treated fibers of peaks at 1591 cm^{-1} and 1417 cm^{-1} attributed to lignin, pectin, and hemicelluloses [44, 45]. The decrease in band intensity at 2911 cm^{-1} presents the valence vibration of the C-H group of the CH_2 bond. In addition, the intensity of the absorption peak between 3100 and 3800 cm^{-1} is attributed to the hydroxyl groups of cellulose increases after treatment, confirming the increase in the cellulose content in the treated fibers [1, 46].

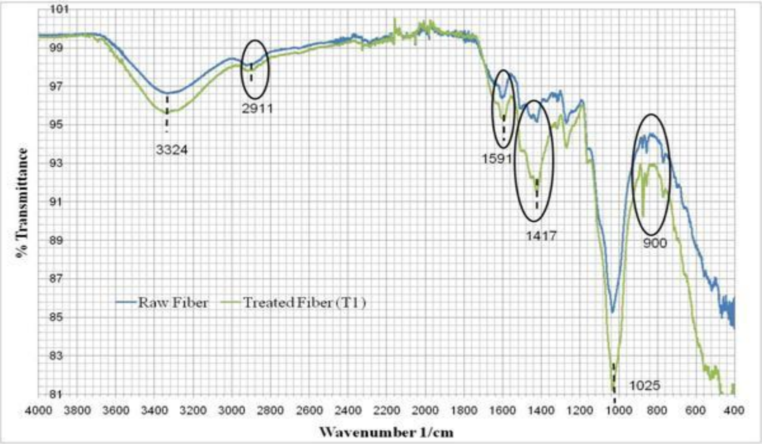

Fig. 8 FTIR spectra of raw and treated fibers

3.1.3. Thermal characterization

Differential thermal analysis (DTA) and thermo gravimetric analysis (TGA) were determined simultaneously in a Setaram differential calorimeter device (SETSYS evolution) with argon shielding gas and Helium, an inert gas. A finely ground sample was introduced into an oven where the temperature was raised from room temperature to 600°C at a speed of 10°C/min. The DTG curves shown in Fig 9 depict two endothermic peaks. Between 50°C and 100°C, the Posidonia fibers samples lost a small amount of mass due to evaporation and moisture removal from the fibers. It was thermally stable up to 220°C [47]. For raw fibers, the first peak at low temperatures (290°C) is the temperature of hemicelluloses decomposition. This peak does not exist for treated fibers, proving that hemicelluloses have been removed. The second peak is associated with the decomposition of cellulose and lignin (315°C for treated fibers and 330°C for raw ones) [1, 11, 46]. The treatment with NaOH is effective since the thermal stability is related to the level of cellulose decomposed during processing. The de-polymerization of hemicelluloses and cellulose is due to improved thermal stability. Hemicelluloses and lignin are amorphous and begin to degrade before cellulose, which is very crystalline [10, 46].

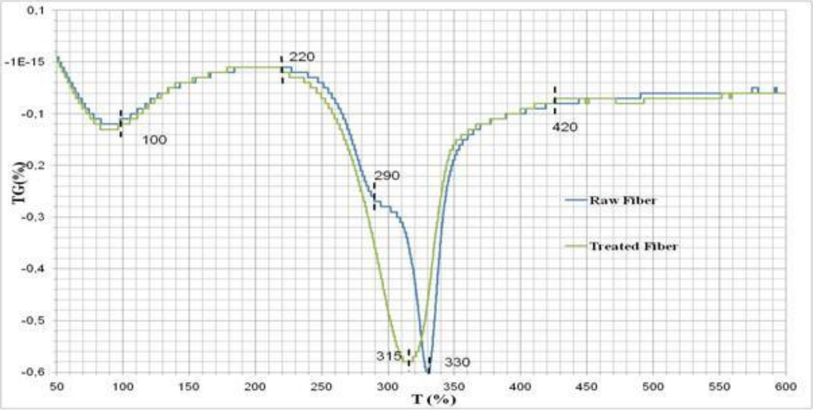

Fig. 9 The DTG of raw and treated fibers

3.2. Workability

Fig 10 shows the flow time of mortar for different volumetric fractions measured using the workability meter. The reference mortar (non-fibrous) has a soft consistency (fluid) with a flow time equal to 10 seconds. The flow time measured on the mortar (T1 (5%)) is in the order of 20 seconds.

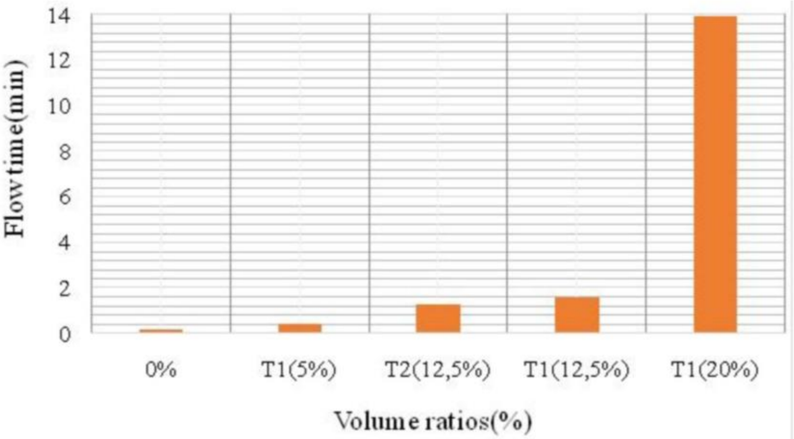

Fig. 10 Mortars flow time of raw and treated fibers

There is an increase in flow time with the fraction of fibers (Chafei et al., cited by [7]). However, mortar (T1 (20%)) has poor workability (stiff consistency) with a flow time of about 14 minutes. Finally, mortar (T2 (12.5%)) flow time is equal to 1.23 minutes, which is lower than mortar (T1 (12.5%)) (1.57 minutes). For the same Water/Binder (W/B) ratio and considering the high absorption capacity of fiber during the first 15 minutes (Fig 6), the flow time increased (Fig 11). The addition of fiber to a pozzolanic mineral matrix leads to decreasing its workability, which can be corrected by an adjustment of the W/B ratio. There is a loss of workability due to the high water-absorption power of the fibers [4, 14].

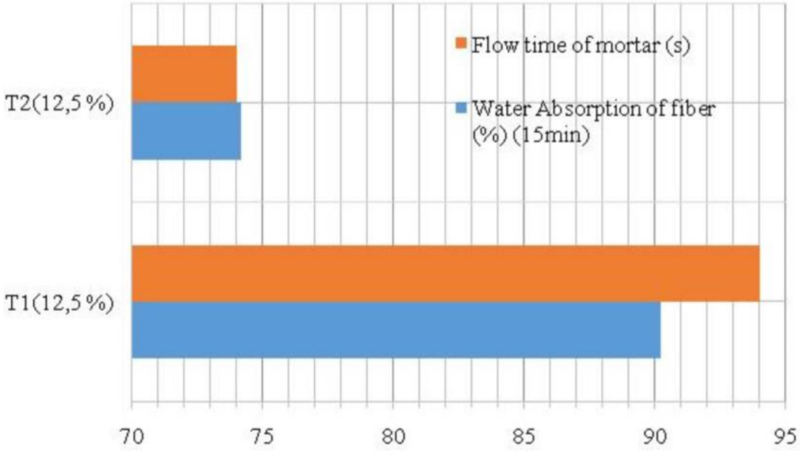

Fig.11 Effect of treatment on flow time of mortars and water absorption of fibers

3.3. Bulk Density and Water-Accessible Porosity

Fig 12 shows a significant decrease in density when Posidonia fibers are incorporated. The embedded mortar of 20% fiber has the lowest density, 6% less than the reference mortar, and has a greater porosity (14.27%). The porosity of the composite increases with the volume fractions, which can be explained by the fact that using fibers can create more voids and promote the accessibility of water. This increase can be caused by the swelling of fibers in the fresh mortar. These release the water absorbed, which creates voids and increases the porosity in the matrix [33, 34, 35, 48].

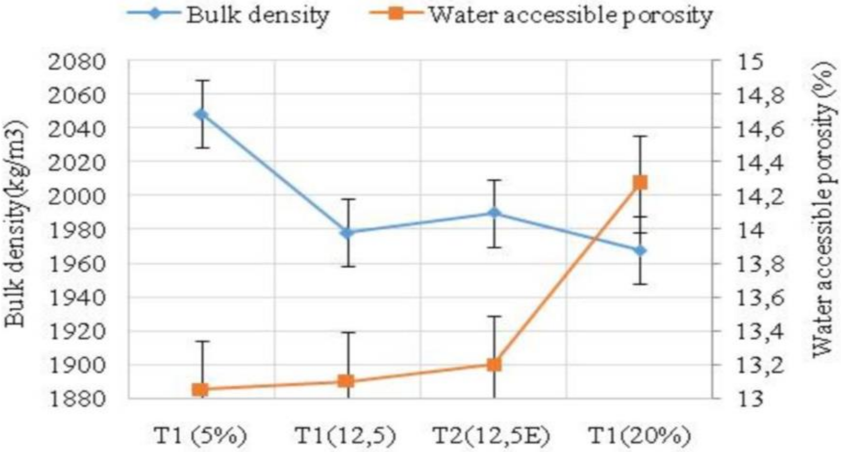

Fig. 12 Effect of treatment on bulk density and water accessible porosity of the mortars

3.4. Imbibition Kinetics

Fluid movement is a crucial feature of cement materials' durability[49]. The capillary absorption coefficients(C (Kg/m^2)) of the composites (12.5%) for different treatments are shown in Fig 13. The embedded mortar of 20% fiber has the highest capillary absorption. The incorporation of vegetable fibers in composites is known to increase their porosity [35, 50]. The absorption of composite water reinforced by fibers coated by calcined clay is lower than that of composite reinforced by NaOH-treated fibers without coating. This decrease is due to the reduced number of pores through the mineral coating treatment. Water diffusion is related to several factors, such as porosity, moisture, volume fraction of fibers, temperature, and matrix viscosity [51, 52]. Water absorbed by fibered composites represents the mobility and movement of liquid in the capillary pores due to capillary absorption [53]. The Posidonia fibers absorb water, causing hygroscopic swelling and changing their mechanical and physico-chemical properties [31]. The water absorption ensures the durability of the composite. The water diffusion kinetics in the composite is based on Fick models. The water absorption behavior is initially linear (Fickian diffusion); slows down until the moisture level approaches saturation level. Quasi-linear variation provides information on the diffusivity of water molecules and indicates the speed of water penetration into the material [54]. The water intake increases with the fiber fractions. This increase is related to the hydrophilic character of fibers and the formation of hydrogen bonds between water molecules and the hydroxyl and carboxyl groups of fibers present in pectin, hemicelluloses, and cellulose [54,55, 56, 57]. Increasing the fractions of the fibers improves their separation and dispersion in the composite, thus promoting water absorption in these composites because of a specific surface that is more important for the best separated fibers. The calcined clay decreases the fibers' hydrophilic nature due to a better adhesion to the fiber/matrix interface. The volume and porosity at the interface decrease, hence a reduction in the free space for water circulation and storage [55]. The coating agent decreases the number of short chains at the interface and makes OH clusters of fibers less accessible to water molecules [58, 59].

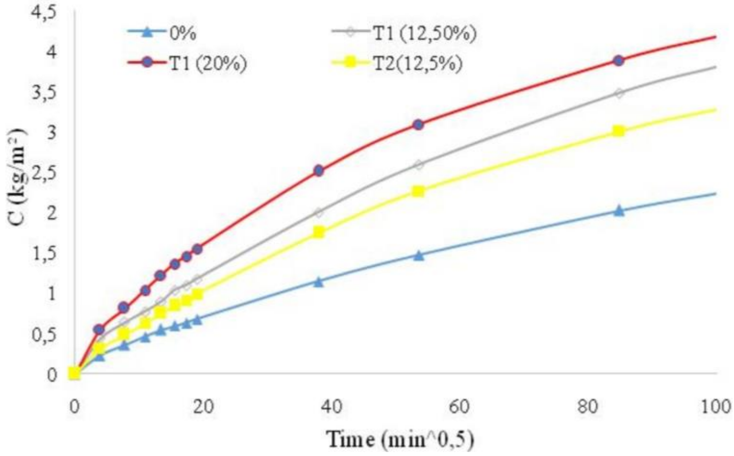

Fig. 13 Effect of treatment and volume ratios on water absorption coefficient of the mortars

3.5. Moisture Diffusion Under Controlled Humidity

The moisture content under controlled humidity (W) (79%) of the composites as a function of time is illustrated in Figs 13 and 14. The water content increases over time;

This finding is expected because mortars are packed in an environment with high relative humidity. As expected, the water intake was very fast from the first hours and then slowed down to stabilize. The water content (Fig14) and the diffusion coefficient (Table 5) increase with fiber content. There is a 27% increase for the 20% volume fraction. Composites reinforced by coated fibers were found to absorb less moisture, proving that the treatment of mineral coating by pozzolanic materials makes it relatively less hydrophilic (Fig 15). Water absorption capacity and humidity are directly related to the presence of voids and the fiber/matrix interface [53]. High moisture absorption results in a low fiber/matrix interface and poor stress transfer. Alkaline treatment reduces moisture absorption, decreases the hydrogen bonding capacity of cellulose, and eliminates open hydroxyl groups that tend to bind to water molecules. It dissolves hemicelluloses and disrupts the hydrogen bond in the network structure, thus increasing surface roughness [31, 58, 59].

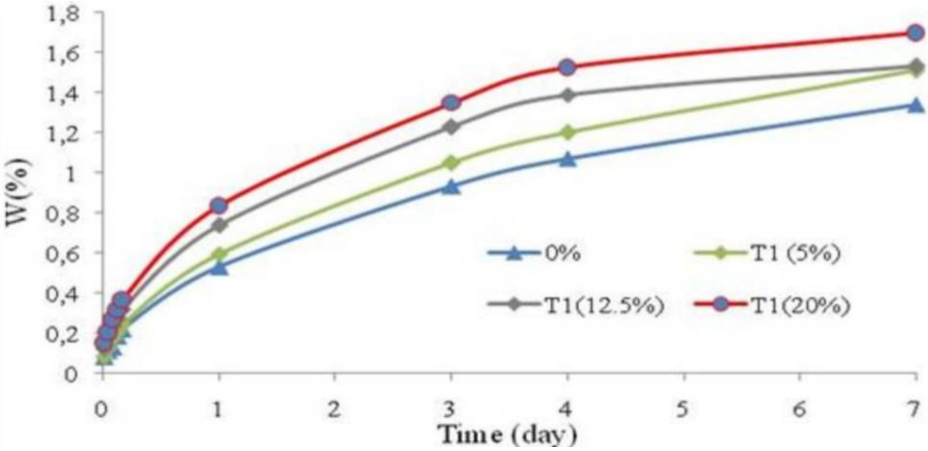

Fig. 14 Effect of volume ratios on water content under controlled humidity (79%) of the mortars

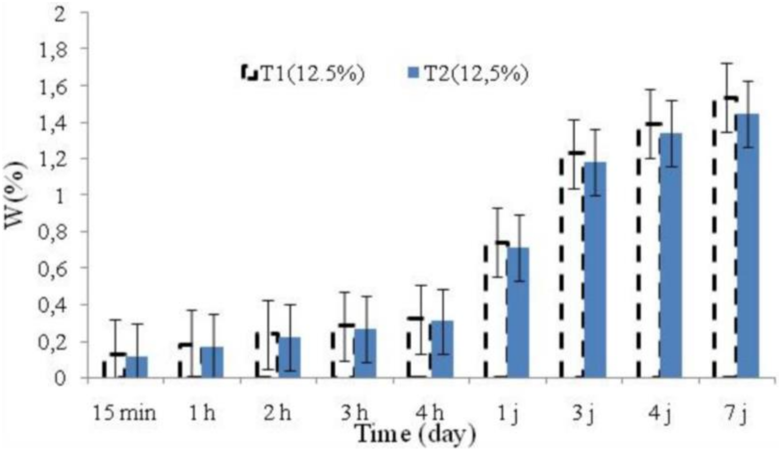

Fig. 15 Effect of treatment on water content under controlled humidity (79%) of the mortars

Table5. Effect of fiber fraction on the diffusion coefficient

		D (m2/s)	n<0.5 (Fick models)
T1	0%	5.7E-10	0.463
	5%	5.7E-10	0.463
	12.50%	8.46E-10	0.417
	20%	8.83E-10	0.421
12,5%	T1 (12.5%)	8.46E-10	0.417
	T2 (12.5%)	8.7E-10	0.404

3.6. Mechanical Properties

The results of the splitting tensile test in long-term (90 days) for different volume fractions and surface treatments are shown in Fig 16. There is a loss of ductility and an increase in resistance to the appearance of the first crack. The same results were found by Sedan et al. using hemp fibers and Canovas et al. using sisal fibers, cited by [7]. In mechanical studies on fiber-cement composites, the authors report that, in most cases, the ductility decreases over time, and the mechanical properties change considerably due to the reduced ductility of the material [4]. The factors contributing to the loss of ductility are the degradation of fibers in the alkaline matrix by partial dissolution of cellulose, hemicelluloses, and lignin and the decomposition according to two phenomena. The first was peeling off by the formation of iso-saccharine acids (CH_2OH) that would break away from the chain. The hemicelluloses are more sensitive to peeling off. The second was the alkaline hydrolysis that consists of dividing the molecular chain and reducing the degree of fibers' polymerization [7,14].

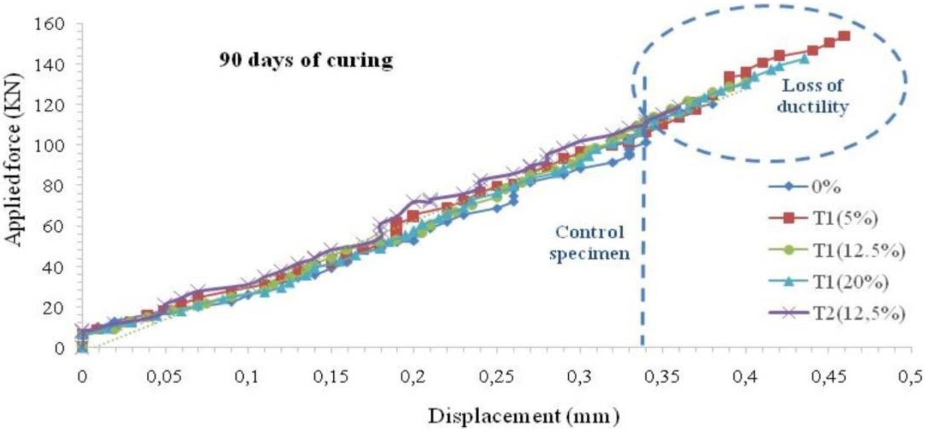

Fig. 16 Effect of treatment on splitting tensile strength of the mortars

The experimental results of the flexural strength (σ_f) at 28-day and 90-day for different volume fractions and surface treatment are shown in Fig 17. As can be seen in the figure, the tensile strength of the composite reinforced by fiber (T1 (5%)) improves compared to the reference composite, with a 1.5% increase. This finding can be attributed to the improvement of the fiber/matrix interface [38] and the pozzolanic reaction of calcined clay [18]. It eliminates the phenomenon of fiber mineralization and improves mechanical resistance. The reduction in the long-term bending strength of 12.5% and 20% volume fractions may be due to surface defects increase and higher porosity [33]. There is also a low variation (Fig17) in the bending resistance of composite reinforced by treated and coated fibers (T2), with an increase of 2.8% compared to (T1). The mineral coating seems to improve the fiber/matrix adhesion.

Fig18 shows that the compressive strength (σ_c) decreases with the addition of fibers. Several authors explain this decrease by the increase in pores in the mixture via oculus air and, thus, the reduction in the cohesion of the fibrous matrix [7, 31]. The increase in the percentage of fiber leads to a decrease in the amount of heat released following the hydration reaction, which explains the decrease in strength of 12.5% and 20%. This finding is probably due to the residual sugars present in the structure of fibers; accordingly, the hydration reaction can be inhibited [34].

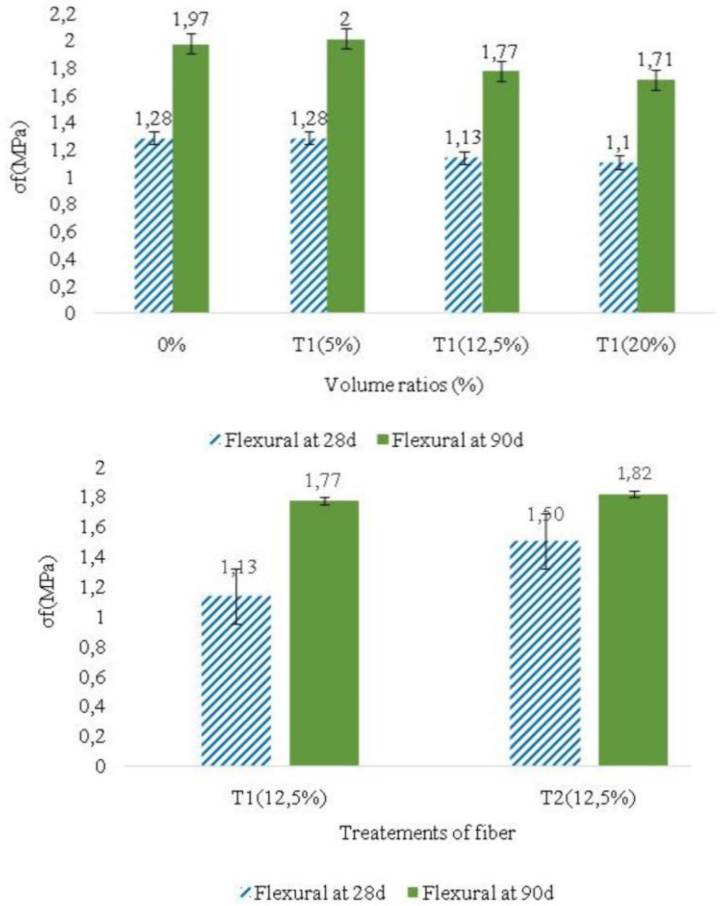

Fig. 17 Effect of volume ratios and treatment on flexural strength of the mortars

Adding fiber helps trap air inside the matrix and reduce its compactness. Abida [53] claims that this finding may be due to the orientation of the fibers during the preparation phase, which allows the agglomeration in the matrix and the non-homogeneous distribution of fibers. However, the best compressive strength is obtained for 5% fibers, with an 11% increase over the reference composite. This finding is consistent with that of Allegue et al. [38]. The alkaline treatment decreases the absorption rate of the fibers, explaining the resistance gain [53]. Also, fig 18 depicts a low variation in the compressive strength of the composites reinforced by the fibers treated and coated by the calcined clay (T2) compared to (T1). Mineral coating for fibers seems to improve the mechanical properties of composites. The larger the volume, the more likely it is to find notable defects that can lead to the breakdown of the fibers. In addition, it has been shown that fiber ruptures are due to a larger size but not to the number of defects [60]. Composite characteristics are determined by fiber, matrix composition, and fiber-matrix interface [61].

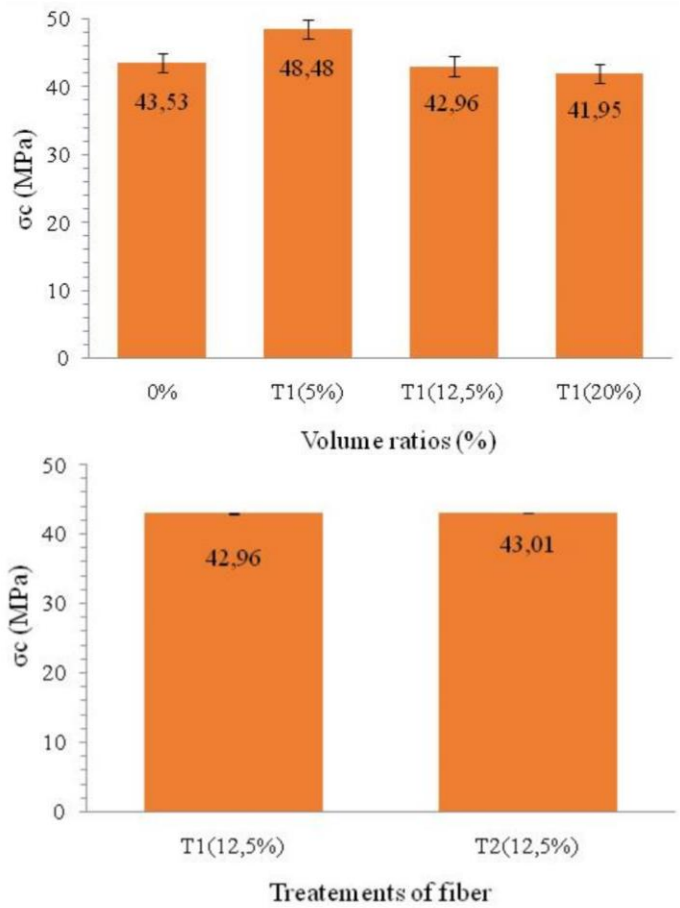

Fig. 18 Effect of volume ratios and treatment on compressive strength of the mortar (90days of curing)

3.7. Alkaline Degradation by Sodium Chloride (NaCl)

Fig 19 shows that the density of the mortars increases after 90 days of immersion in an alkaline solution. This increase is due to a swelling of the composites and the formation of calcium chloride. In the same vein, Asma [4] showed swelling of composites reinforced by Diss fibers after immersion in seawater. Fibers cause volume variation in all composite directions [33]. It is also noted (Fig 20) that the coating treatment by the calcined clay reduces the loss of strength, which can be explained by the rapid reduction of the calcium hydroxide in the cement paste caused by the pozzolanic reaction of the calcined clay [18].

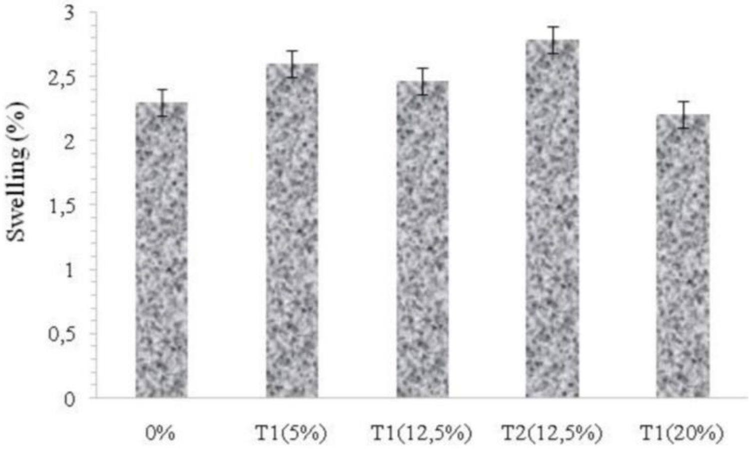

Fig. 19 Swelling of mortars after 90 days of immersion in ~~chloride~~sodium chloride(NaCl)

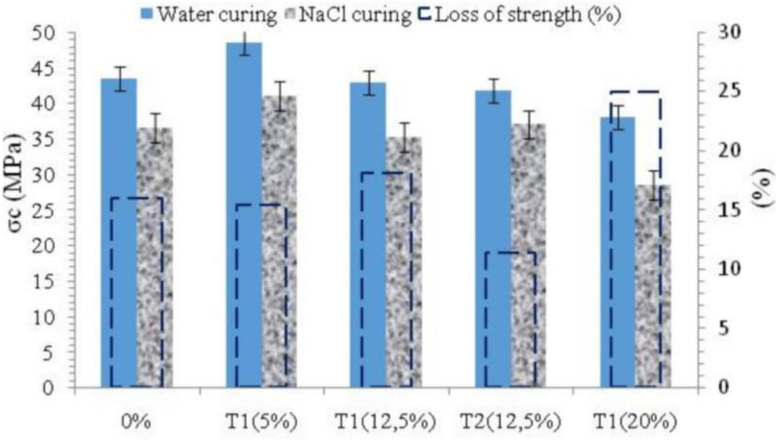

Fig. 20 Reduction in compressive strength of mortars after 90 days of immersion in chloride sodium chloride(NaCl)

3.8. SEM Microstructure Study

The shape of the fibers influences the interface and the anchoring with the matrix. The tubular form has a high adhesion as the fibrillar fibers form massively. This form absorbs more water, which can cause a durability problem. The nature of the fibers directly affects water absorption and the formation of barriers. There is an increase in the roughness of fiber surfaces after alkaline treatment. This regularity improves the adhesion between the fibers and the matrix. It can be suggested that there is also a reduction in impurities, lignin, and hemicelluloses [13, 16, 33]. Fig 21, 22and 23 shows the SEM-EDX micrographs of Posidonia fibers before and after treatment. To determine the efficacy of the treatment with NaOH (Na^+), the amount of Na+ in the treated fibers is evaluated. An additional peak shows the presence of the Na^+ molecule resulting from the performed chemical treatment. The results confirm the presence of a chemical reaction between Posidonia fibers and NaOH. Table 6 presents the major compositional elements (oxygen (O) and carbon ©) of raw and treated fiber and traces for silica (Si), alumina (Al), and iron (Fe), indicating the typical residual elements for plant biomass fibers and calcined clay (siliceous aluminous nature).

Untreated fibers have a thin layer of grass, wax, and impurities deposited on the surface. The hydrothermal and alkali treatment removes the weak external layer of the fibers, such as wax, lignin, and hemicelluloses. It creates a rough surface morphology resulting in better adhesion with the mortar matrix. A small canal with little pores confirms the porosity structure of the fiber. Mineral coating over the Posidonia surface provides a waterproof film that reduces water absorption from the matrix. A combined treatment would create both surface roughening and waterproofing effects [62]. The molecules of the matrix can be fixed to the surface of the fiber by a chemical reaction or adsorption, which determines the adhesion strength of the interface. In some cases, the interface may consist of additional components, such as an adhesive or an intermediate layer between the two components of the composite material. Due to the presence of cellulose and lignin hydroxyl groups, natural fibers can be modified. Surface properties, such as wettability, adhesion, surface tension, fiber surface roughness, and porosity, can be improved by treatment. These surface irregularities play a crucial role in the mechanical connection of the interface with the matrix [61]. The addition of Posidonia fibers allows the creation of voids inside the matrix leading to poor adhesion between fibers and the matrix. The mineral coating treatment by calcined clay minimizes voids (Fig 24 and 25).

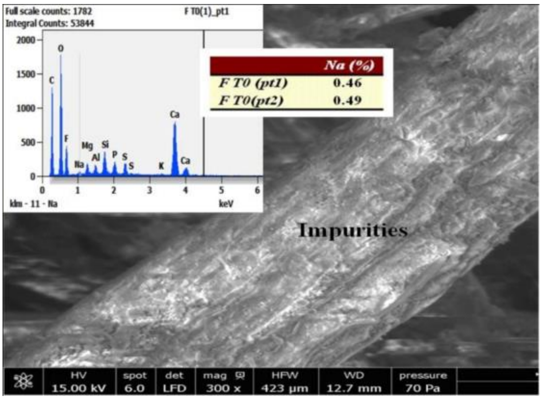

Fig. 21 SEM–EDX micrographs of Posidonia fibers before treatment

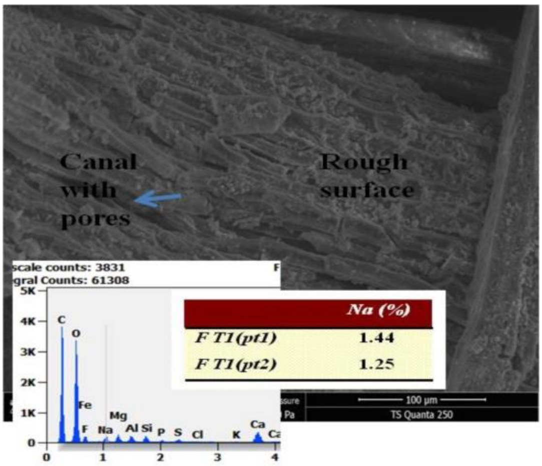

Fig. 22 SEM–EDX micrographs of Posidonia fibers after treatment (alkali and hydrothermal treatment)

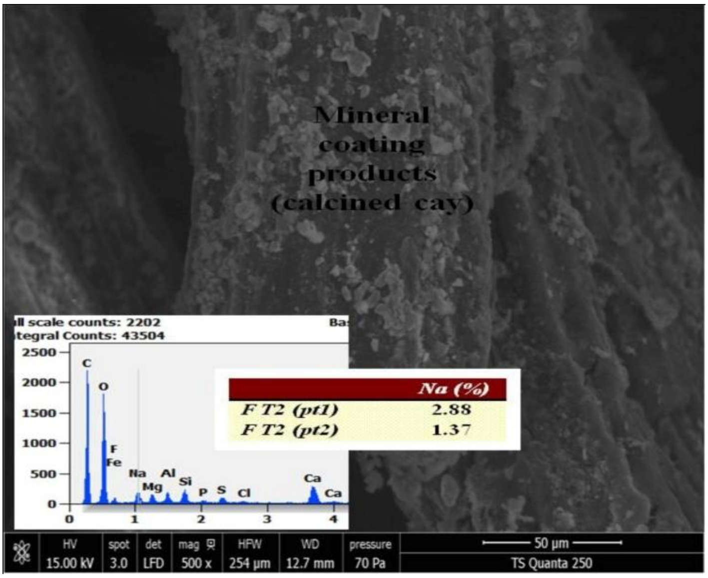

Fig. 23 SEM–EDX micrographs of Posidonia fibers with mineral coating (x100)

Table6. Compositional elements of raw and treated fibers

	Atom %	C	O	Si	Al	Fe
F T0	Point(1)	15.95	38.47	2.02	0.71	0.57
	Point(2)	14.30	34.33	0.98	0.60	-
F T1	Point(1)	40.29	51.01	0.65	0.80	0.25
	Point(2)	37.71	50.77	0.73	0.76	0.21
F T2	Point(1)	39.65	47.18	1.67	1.19	0.40
	Point(2)	23.46	59.05	3.42	2.03	0.62

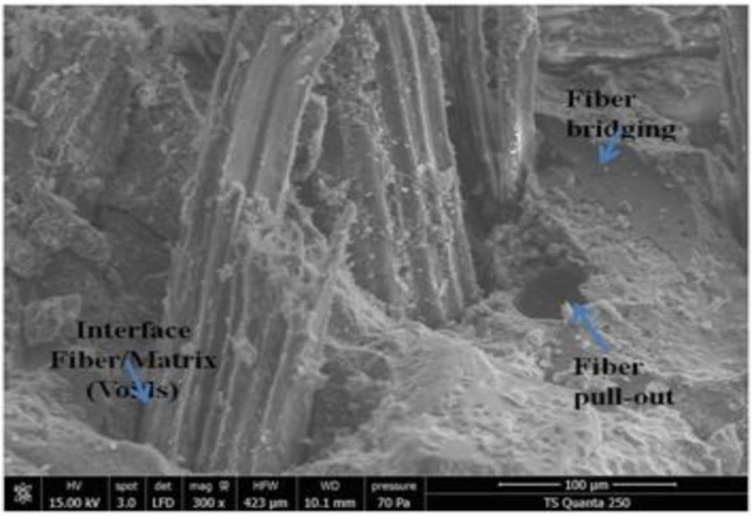

Fig. 24 SEM micrographs of mortars (12.5%) alkali and hydrothermal treatment

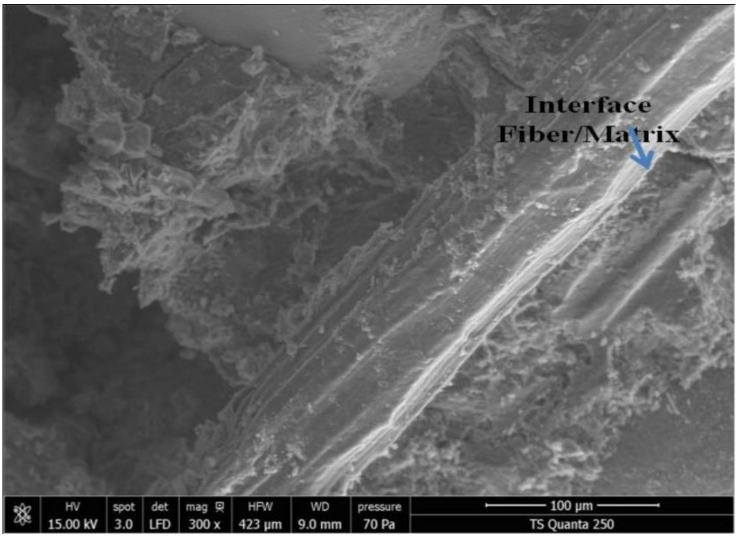

Fig. 25 SEM micrographs of mortars with mineral coating (x100)

5. Conclusions

Based on the analysis of the physical and hygroscopic properties, rheological, mechanical, and micro-structural performances, and chemical attack of cementitious mortars with supplementary materials (calcined clay) incorporating Posidonia fibers, the following conclusions can be drawn:

- Fiber characterization showed that alkaline treatment was effective in removing non-cellulosic materials and reducing water absorption from fibers.
- The incorporation of Posidonia fibers (volume fraction 5%, 12.5%, and 20%) in composite seems to reduce the usability in the fresh state significantly.
- A decrease in the water absorption of composite reinforced by fibers coated with calcined clay compared to composite reinforced by fibers treated with NaOH without coating. This decrease is due to the reduced number of pores through the mineral coating treatment.
- The maximum moisture content increases with the fiber content.
- The mineral coating treatment by the pozzolanic material makes the fibers relatively less hydrophilic.
- The tensile test shows a long-term loss of ductility and an increase in resistance to the appearance of the first crack.
- The flexural test shows an improvement in strength with the addition of treated Posidonia fibers up to an optimum of 5% by volume of fibers. This finding may be explained by the improvement of the fiber/ matrix interface and the pozzolanic reaction of calcined clay, which eliminates the phenomenon of fiber mineralization. However, beyond that percentage, resistance decreases, which may be due to the increase in surface defects.
- The highest compressive resistance was obtained at 5% volume fraction. The alkaline treatment decreases the absorption rate of the fibers, explaining the gain in resistance, and then decreases with the increase in the volume fraction of the fibers.

- The density of the mortars increases after 90 days of immersion in an alkaline solution. This increase is due to a swelling of the composites and the formation of calcium chloride. Fibers cause volume variation in all composite directions.
- The coating treatment by the calcined clay reduces the loss of strength after 90 days of immersion in an alkaline solution, which can be explained by the rapid reduction of the calcium hydroxide in the cement paste caused by the pozzolanic reaction of the calcined clay.

Future Scope and Limitations of The Study

Following the results of this research, other methods, such as lime or plasma treatment, can be proposed for the surface treatment of fibers. It is necessary to study the durability of composite reinforced by Posidonia fibers, such as the determination of migration coefficient and chloride diffusion, freeze-thaw test, and hygro-thermal behavior. It is advisable to study the behavior in adverse environments such as acid environments and elevated temperatures.

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