

(54) Title of the invention : **THE INFLUENCE OF DIFFERENT TREATMENTS APPLIED TO FLAX FIBERS ON DIFFERENT PROPERTIES OF MORTAR REINFORCED BY THESE FIBERS**

<p>(51) International classification :C04B0028020000, C04B0111000000, D06M0010020000, A61K0036550000, C04B0018240000</p> <p>(31) Priority Document No :NA</p> <p>(32) Priority Date :NA</p> <p>(33) Name of priority country :NA</p> <p>(86) International Application No :NA</p> <p>Filing Date :NA</p> <p>(87) International Publication No : NA</p> <p>(61) Patent of Addition to Application Number :NA</p> <p>Filing Date :NA</p> <p>(62) Divisional to Application Number :NA</p> <p>Filing Date :NA</p>	<p>(71)Name of Applicant :</p> <p>1)Dr.A.N.SWAMINATHEN Address of Applicant :ASSOCIATE PROFESSOR, DEPARTMENT OF CIVIL ENGINEERING, SREE DATTHA INSTITUTE OF ENGINEERING AND SCIENCE, SHERIGHUDA, IPRAHIMPATNAM, TELANGANA -501510 Telangana India</p> <p>2)Dr.R.SUBASHCHANDRABOSE</p> <p>3)Dr.J.REX</p> <p>4)Dr. S. ROBERT RAVI</p> <p>5)Dr.M.KARTHIKEYAN</p> <p>6)Dr. D. MARUTHACHALAM</p> <p>7)Dr.A.KUMAR</p> <p>8)Ms. ANCE MATHEW</p> <p>9)Mr. R. SRINIVAS PRABHU</p> <p>10)Dr. SHEELA. V</p> <p>(72)Name of Inventor :</p> <p>1)Dr.A.N.SWAMINATHEN</p> <p>2)Dr.R.SUBASHCHANDRABOSE</p> <p>3)Dr.J.REX</p> <p>4)Dr. S. ROBERT RAVI</p> <p>5)Dr.M.KARTHIKEYAN</p> <p>6)Dr. D. MARUTHACHALAM</p> <p>7)Dr.A.KUMAR</p> <p>8)Ms. ANCE MATHEW</p> <p>9)Mr. R. SRINIVAS PRABHU</p> <p>10)Dr. SHEELA. V</p>
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(57) Abstract :

THE INFLUENCE OF DIFFERENT TREATMENTS APPLIED TO FLAX FIBERS ON THE DIFFERENT PROPERTIES OF MORTAR REINFORCED BY THESE FIBERS Several solutions for treating flax fibers have been studied in this work. This need for treatment is explained by the high water absorption capacity of flax fibers which disrupts the rheological behavior of cementitious composites in the fresh state but also affects the physical and mechanical properties in the hardened state. Three treatment solutions were explored: atmospheric plasma, mineral cement/slag coating and linseed oil coating. These treatments were optimized before the incorporation of fibers into the mortars. The hygroscopic and hydroscopic behaviors of flax fibers have been studied. It has been found that flax fibers do absorb large amounts of water very quickly (up to 140% by mass). Atmospheric plasma treatment made it possible to modify the kinetics of water absorption but not the retention capacity. Conversely, linseed oil is the only treatment that actually reduces the water absorption capacity while the cement/slag coating treatment does not modify it. It appeared necessary to take into account the presence of the treatment in the interpretation of the results and therefore to relate the measurements to the mass of raw fibers only. Failure to take into account the treatment products could lead to erroneous interpretations.

No. of Pages : 30 No. of Claims : 5

FORM2
THE PATENT ACT 1970
(39 OF 1970) &
The Patents Rules, 2003
COMPLETE SPECIFICATION
(See section 10 and rule 13)

TITLE OF THE INVENTION:

**THE INFLUENCE OF DIFFERENT TREATMENTS APPLIED TO FLAX FIBERS ON
DIFFERENT PROPERTIES OF MORTAR REINFORCED BY THESE FIBERS**

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2.PREAMBLE TO THE DESCRIPTION :

Separate Sheet is attached

3.COMPLETE SPECIFICATION

Separate Sheet is attached

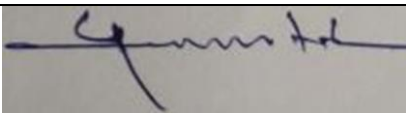
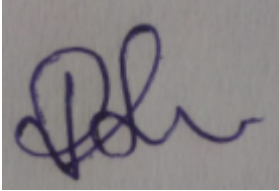
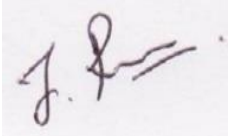

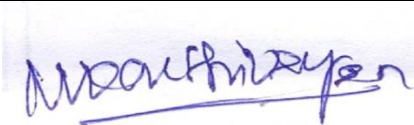
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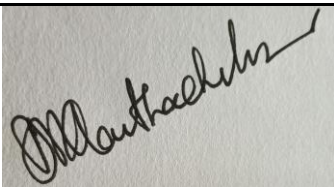
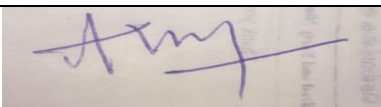
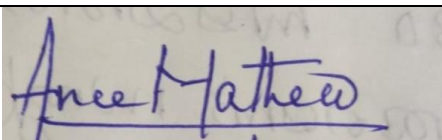
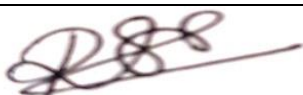
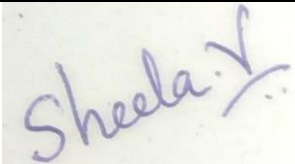
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5.CLAIMS

Separate Sheet is attached

6.

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7. ABSTRACT OF THE INVENTION		
_____Separate Sheet is attached_____		

PREAMBLE

THE INFLUENCE OF DIFFERENT TREATMENTS APPLIED TO FLAX FIBERS ON THE DIFFERENT PROPERTIES OF MORTAR REINFORCED BY THESE FIBERS

Several solutions for treating flax fibers have been studied in this work. This need for treatment is explained by the high water absorption capacity of flax fibers which disrupts the rheological behavior of cementitious composites in the fresh state but also affects the physical and mechanical properties in the hardened state. Three treatment solutions were explored: atmospheric plasma, mineral cement/slag coating and linseed oil coating. These treatments were optimized before the incorporation of fibers into the mortars. The hygroscopic and hydroscopic behaviors of flax fibers have been studied. It has been found that flax fibers do absorb large amounts of water very quickly (up to 140% by mass). Atmospheric plasma treatment made it possible to modify the kinetics of water absorption but not the retention capacity. Conversely, linseed oil is the only treatment that actually reduces the water absorption capacity while the cement/slag coating treatment does not modify it. It appeared necessary to take into account the presence of the treatment in the interpretation of the results and therefore to relate the measurements to the mass of raw fibers only. Failure to take into account the treatment products could lead to erroneous interpretations.

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ABSTRACT

THE INFLUENCE OF DIFFERENT TREATMENTS APPLIED TO FLAX FIBERS ON THE DIFFERENT PROPERTIES OF MORTAR REINFORCED BY THESE FIBERS

Several solutions for treating flax fibers have been studied in this work. This need for treatment is explained by the high water absorption capacity of flax fibers which disrupts the rheological behavior of cementitious composites in the fresh state but also affects the physical and mechanical properties in the hardened state. Three treatment solutions were explored: atmospheric plasma, mineral cement/slag coating and linseed oil coating. These treatments were optimized before the incorporation of fibers into the mortars. The hygroscopic and hydroscopic behaviors of flax fibers have been studied. It has been found that flax fibers do absorb large amounts of water very quickly (up to 140% by mass). Atmospheric plasma treatment made it possible to modify the kinetics of water absorption but not the retention capacity. Conversely, linseed oil is the only treatment that actually reduces the water absorption capacity while the cement/slag coating treatment does not modify it. It appeared necessary to take into account the presence of the treatment in the interpretation of the results and therefore to relate the measurements to the mass of raw fibers only. Failure to take into account the treatment products could lead to erroneous interpretations.

Atmospheric plasma treatment has shown that absorption is not the major cause of the rheological behavior encountered in fresh composites. It is the morphology and in particular the large specific surface area of the fibers that mainly explains the losses in workability. In the hardened state, raw or treated flax fibers have made it possible to more or less significantly increase the flexural strengths of the composites. The presence of flax fibers enhances the tenacity of the mortar, which becomes comparable to that of the material incorporating the glass fibers at the start of the cure. However, this action of flax fibers is weakened over time until it disappears in the medium term (90 days). No treatment has improved the durability of flax fibers in contact with the highly alkaline environment of the cement matrix. It therefore appears necessary to use alternative binders, with smaller amounts of Portlandite. Finally, we also noted an influence of flax fibers on the setting and hydration of mortars. The polysaccharides present in the fibers can go into solution and cause delays in setting and act as a hydration inhibitor of the cement.

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DESCRIPTION

THE INFLUENCE OF DIFFERENT TREATMENTS APPLIED TO FLAX FIBERS ON THE DIFFERENT PROPERTIES OF MORTAR REINFORCED BY THESE FIBERS

Plant fibers have a significant impact on the properties in the fresh state of cementitious composites, but also in the hardened state. When fresh, the rheology of composites is strongly affected by a considerable decrease in workability, even at low fiber incorporation rates. Furthermore, many authors have noted a poorly bonded state of the fiber-matrix interface, or even the absence of contact (free space) between the fiber and the cementitious matrix. This space around the fiber is probably generated by the shrinkage during desorption which follows the absorption of water during mixing. It therefore appears necessary to reduce the quantity of water absorbed by the flax fibers. Pretreatment of these fibers could limit or even eliminate water uptake. This work therefore examines the influence of different treatments applied to flax fibers on the rheological, physical and mechanical properties of mortars reinforced by these fibers. The effects of these treatments on the properties of flax fibers will first be studied. Then, a multiphysical characterization of the mortars will be carried out before proceeding to the study of the hydration of the composites.

INFLUENCE OF TREATMENTS ON THE PROPERTIES OF FLAX FIBERS

Description of processing

Surface treatment with atmospheric plasma

The first treatment to be tested is a physical surface treatment with atmospheric plasma. This treatment is minimally invasive because the depth of penetration into the material is only a few micrometers at most. It is used in various industrial fields to modify the surface properties of many materials such as textiles, plant fibers, metals, polymers or even composites. A surface modification technique comparable to that of atmospheric plasma is physical thin-film deposition. The operating mode of plasma spraying is as follows: the source used for the coating, here in liquid form, is blown into the plasma flame by means of a carrier gas. The source compound is then melted and projected at very high speed and with very high energy on the surface to be treated. The deposited thickness generally varies from a few nanometers to a few micrometers. The dissociation of the starting molecule in the plasma into numerous highly energetic fragments, reacting with each other on the surface of the part, ends up forming a more or less crosslinked layer. The adhesion of the coating results essentially from two adhesion processes: physical bonds and electrical interactions (van der Waals forces).

In our case the plasma is ionized nitrogen. The precursor used for the treatment is HMDSO (Hexamethyldisiloxane) is given in figure 1.

Parameters used for processing:

- Flow rate of the precursor = 150 g/h;
- N₂ plasma gas flow rate = 1300 L/h;

- Evaporator N₂ gas flow rate = 300 L/h;
- Evaporator temperature = 125° C;
- Displacement speed of the plasma nozzle = 100 m/min;
- No advance of the nozzle = 2mm;
- Distance nozzle - flax fibers = 30mm;
- Number of treatments per side = 5.

Coating treatment with cement/slag grout

The second treatment is a coating of the flax fibers with a grout of hydraulic binders. Its objective is the chemical insulation of fibers by minimizing exchanges with the matrix and reducing the rate of water absorption. The mineral substances were chosen for their low hydrophilicity and their resistance in an alkaline environment, which ensures that the treatment is maintained at the surface of the fiber. Thus, the mineral coating is made from white Portland cement CEM I 52.5N and blast furnace slag (BFS). The final hydraulic binder used for the treatment is thus composed of 50% by volume of BFS and 50% of CEM I cement. For this treatment, the fibers are coated with a grout of this binder prepared with an equal water/binder mass ratio at 1 and a fiber / binder ratio equal to 2/3. The fibers thus obtained are referenced FCL for: Fibers treated with Cement. The binders and water are first mixed together for 2 minutes at 140 rpm in a standardized mortar mixer (NF EN 196-1). Once the homogeneous grout is obtained, the fibers are added to the mixer, still for 2 additional minutes. The coated fibers are then stored in a chamber at controlled temperature and humidity (20 ± 1 ° C, 65 ± 5% RH) for 28 days before the preparation of the cementitious composites.

Coating treatment with linseed oil

Description of processing

Linseed oil (LO) is a viscous liquid that hardens by oxidation in air to form a solid film, so it is referred to as a drying oil. This strong siccativity explains its current use as a surface coating to waterproof wood. Linseed oil is composed of three fatty acids with long unsaturated aliphatic chains (oleic acid, linoleic acid and linolenic acid) and saturated (stearic acid and palmitic acid). Due to the siccative properties of linseed oil, great care must be taken as its exothermic oxidation can, at high temperature, lead to spontaneous combustion. This is a complex phenomenon whereby a combustible material can catch fire under the effect of its own heat of reaction, without external heat or another source of ignition being involved. The oxidation of a double bond leads to the oxidation of neighboring bonds and so on; since double bonds contain more energy, the temperature rises may be greater after oxidation. It is the linoleic and linolenic acids which will determine the temperature rise capacity of the oil: the proportion between these two acids can vary from one sample to another and this influences the quality and therefore the price of this oil. Likewise, oxidation is faster at elevated temperatures. This is why it is recommended not to use this oil at a temperature above 60° C. The linseed oil used in this work is a boiled linseed oil allowing accelerated oxidation. It is oil extracted from flax grown, harvested and produced in Normandy. It was obtained by

cold pressing without solvent, filtered at 1 μm , and then baked at 150° C without adding a drying agent. This linseed oil has a density of 0.93. To carry out the treatment, the fibers dried at $50 \pm 0.1^\circ \text{C}$ are introduced into a planetary mixer. The mixer is then started at a speed of 30 rpm-1 and after 30 seconds the linseed oil is introduced for a period of 2 minutes 30 seconds. For the study, the oil/fiber mass ratio was varied from 0.25 to 2. Mixing is maintained for an additional 2 minutes 30 seconds to ensure uniform coverage of the fibers. The fibers thus treated are placed in a ventilated oven at $50 \pm 0.1^\circ \text{C}$ for 2 weeks.

Optimization of LO/fiber ratio

In Figure 2, the mass gains of flax fibers relative to the initial mass of oil are presented as a function of the time elapsed after treatment, for each LO/fiber ratio. We can thus observe for all samples an increase in mass of linseed oil of between 8 and 12% approximately. The lower the LO/fiber ratio, the higher and faster the oil mass increase. This increase in mass is the result of the oxidation reaction of flaxseed oil. The polymerization of cooked linseed oil therefore seems to take place between 2 and 3 days after application of the treatment and setting in an oven.

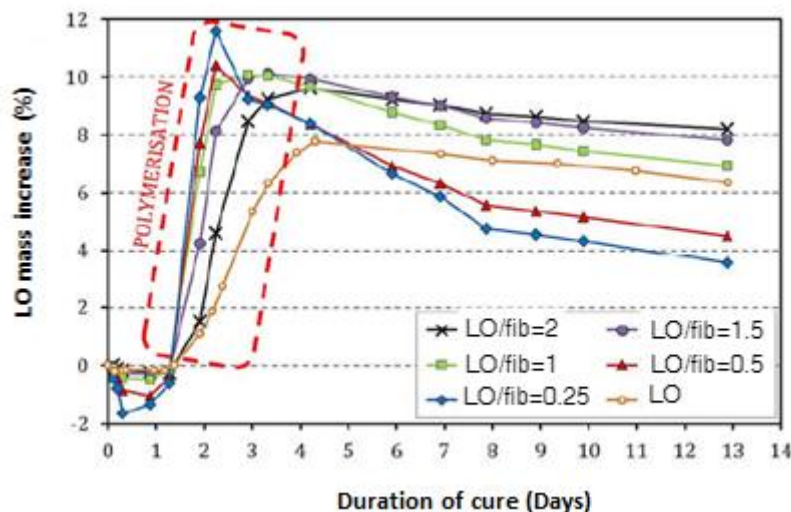


Figure 2: Mass monitoring of fibers treated with linseed oil during the cure at 50°C

The linseed oil coating treatment of the fibers has the effect of decreasing the density of the fiber + LO coating entity (Figure 3). The specific density of crude flax fibers is equal to $1.521 \pm 0.001 \text{ g.cm}^{-3}$. Following the treatment, the density of the fiber + LO coating assembly is equal to $1.205 \pm 0.008 \text{ g.cm}^{-3}$ for a ratio of 2. This is explained by the low density of linseed oil, equal to 0.93 g.cm^{-3} . Thus, as the LO/fiber ratio increases, the proportion of oil is higher, which leads to a decrease in the density of the fibers treated. Note that the absolute density of these fibers was measured using a helium pycnometer, on 6 different samples for each LO value. Note also the very small standard deviations,

indicated by the error bars in Figure 4-5. This indicates good homogeneity in the application of the polymeric coating.

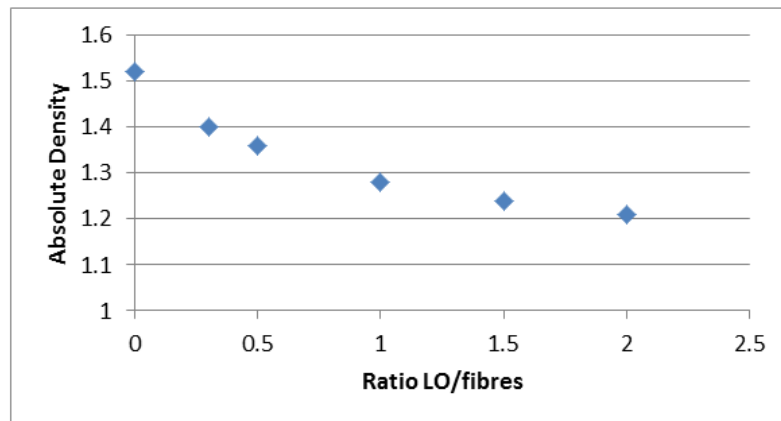


Figure 3: Absolute density of flax fibers treated with linseed oil

Remember that the goal of treatment with linseed oil is first to decrease the water absorption of the fibers. It is therefore mainly on this criterion that the LO/fiber ratio should be optimized.

Thus, water absorption measurements were carried out on these treated fibers, after 5 and 60 minutes of immersion. The results of these water absorption tests are presented in terms of mass ratios, relative to the mass of fibers treated. This type of representation was chosen to highlight the absorption results compared to the linseed oil present in the treated fibers. Indeed, this linseed oil does not absorb water. It is therefore normal that the absorption of treated flax fibers is lower than that of raw fibers. It is then necessary to calculate the mass of water absorbed compared to the mass of crude fibers, without the mass of linseed oil. This curve (in brown on the graph) is used to determine the optimum ratio allowing the absorption of crude fibers to be reduced as much as possible, while using as little linseed oil as possible. It appears that the raw fibers absorb the most liquid water: $90.3 \pm 9.6\%$ after 5 minutes of immersion and $114.5 \pm 3.8\%$ after 60 minutes. We also note that the higher the LO/fiber ratio increases, the lower the quantity of water absorbed.

However, it is important to compare this absorbed water to the amount of raw fibers actually present in the sample to find out which treatment is really the most effective.

The LO/fiber ratio of 0.25 turns out to be the most effective in reducing water absorption, with mass water, abs/mass fiber ratios, gross of 0.59 after 5 minutes of immersion and 0.83 after 60 minutes. The mass water absorption curves of flax fibers (treated or not) as a function of time: was observed. Thus, without correction, the LO/fiber ratio of 2 might seem the most effective in reducing water absorption because it is with these fibers that the amount of water absorbed is the least. However, for this ratio, a large part of the fiber mass is polymerized linseed oil. So it does not represent the absorption of flax fiber. After correction, we see that the LO/fiber ratio does not

ultimately have a great influence on water absorption. Even with a small amount of oil, absorption is drastically reduced. After correction, for the LO/fiber ratio of 0.25, water absorption is reduced by approximately 30% compared to raw fibers. Without making the correction on the flaxseed oil, absorption was reduced "apparently" by about 45%.

Thus, the optimal LO/fiber ratio was determined for the flaxseed oil treatment of the fibers, namely the LO/fiber ratio of 0.25. It is the most effective in decreasing the water absorption of flax fibers and also the most economical since it requires less oil for processing. Subsequently, the fibers will therefore be treated with linseed oil with this ratio.

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DESCRIPTION WITH COMPLETE SPECIFICATION

THE INFLUENCE OF DIFFERENT TREATMENTS APPLIED TO FLAX FIBERS ON THE DIFFERENT PROPERTIES OF MORTAR REINFORCED BY THESE FIBERS

CHARACTERIZATION OF MORTARS INCORPORATING TREATED FLAX FIBERS

The state of the art of this document has made it possible to highlight the problems encountered with biofiber cementitious composites, both in the fresh state and in the hardened state. It turns out that a pretreatment of plant fibers is necessary. The approach taken for this study was therefore to develop test pieces to assess the influence of treatments applied to plant fibers on the rheological, physical and mechanical properties of mortars reinforced with flax fibers.

Formulation and preparation of mortars

Formulation

The flax fibers having different densities and absorptions depending on the treatments, we have chosen to keep effective water/cement ratio constant, equal to 0.51. An additional amount was added to the various mortars to take into account the absorption of aggregates and fibers. The length of the flax fibers, in this case, was set at 12 mm. The dosage of flax fibers (raw or treated) was set at 1.0% by absolute volume relative to the total volume of the batch. In order not to modify the theoretical volume of the batch, when adding fibers, a volume of saturated sand equal to the volume of saturated fibers was removed from the formulation. The theoretical final compositions of the various mortars produced are given in Table 1. In addition, the names will be CM, GFRM, MRFF, MFFAP, MFFC/S, MLFLO represents Control Mortar (non-fiber), Glass fiber reinforced mortar, Mortar reinforced with raw flax fibers, Mortar reinforced with flax fibers treated with atmospheric Plasma, Mortar reinforced with Flax fibers treated with Cement/slag, and Mortar reinforced with linseed fibers treated with linseed oil respectively

Table 1: Composition of mortars reinforced with 1% flax fibers (kg.m⁻³)

Details	CM	GFRM	MRFF	MFFAP	MFFC/S	MLFLO
Cement	507.2	507.2	507.2	507.2	507.2	507.2
Superplasticizer	5.0	5.0	5.0	5.0	5.0	5.0
Viscosity agent	2.51	2.51	2.51	2.51	2.51	2.51
Water	258.3	258.5	278.7	272.3	276.4	270.1
Sand0/4	1525.2	1499.1	1446.8	1462.4	1452.7	1468.1
Fiber	0	26.1	15.5	15.6	18.4	14.1
Volumic mass theoretical (kg.m ⁻³)	2300.5	2300.5	2256.2	2266.1	2263.2	2268.5
E _{eff} /L _{eq}	0.50	0.50	0.50	0.50	0.50	0.50
E _{tot} /L _{eq}	0.520	0.520	0.560	0.550	0.554	0.545

Mixing, making test specimens and curing conditions

The mortars were manufactured using a mortar mixer, conforming to Standard 196-1. The fibers are introduced last in this process to prevent clumping. The fresh mortar was poured into 4 x 4 x 16 cm test tubes. The physical and mechanical tests were then carried out on these same specimens, at the appropriate time. The test pieces were removed from the mold 24 hours after manufacture and then placed in a storage chamber at 20 ± 1 ° C and $> 90\%$ RH, until the end of the tests.

Fresh properties

Maneuverability

Figure 4 shows the spreads of the different mortars. In this case, these are relative spreads with respect to the initial diameter of the test cone of 10 cm. For the reference mortar (non-fiber), noted CM, the average relative spread is $175 \pm 8\%$. This value is very high due to the use of a superplasticizer for the preparation of the mortars. The incorporation of 1.0% by volume of glass fibers in the mortar (GFRM) results in a significant reduction in the spread of the material, with a spread reduced to $92 \pm 17\%$. However, despite this significant reduction in spread, the mortar still retains a fluid consistency. On the other hand, the addition of 1.0% by volume of crude flax fibers has a considerable impact on the flow of the mortar (MRFF); the spread after vibration is only $14 \pm 1\%$, which corresponds to almost zero workability of the mortar. It should also be noted that the treatment with atmospheric plasma did not make it possible to improve the workability of the mortar (MFFAP), since the measured relative spread is $13 \pm 1\%$, ie a value almost identical to MRFF. However, we have seen in Figure 4-13 that this treatment significantly reduces the water absorption of flax fibers, at least in the short term (< 60 minutes), as is the case during the d test. 'spread at the shock table. We would therefore have expected absorption to be greatly reduced, which is not the case here. This therefore means that the water absorption of the fibers does not play a major role in the rheology of the composites. Cement/slag (MFFC/S) and linseed oil (MLFLO) coating treatments have resulted in significant improvements in the workability of the mixes. Indeed, the relative spreads obtained for these two mortars are respectively $69 \pm 14\%$ and $61 \pm 13\%$. These improvements in the workability of mortars are due to the increase in the diameter of the fibers and to the coating, which tends to decrease their specific surface. As we have seen, the very large specific surface area of flax fibers results in a considerable need for cement pulp volume to meet correct workability criteria. These two treatments therefore make it possible to improve the consistency of cementitious composites by coating the fibers.

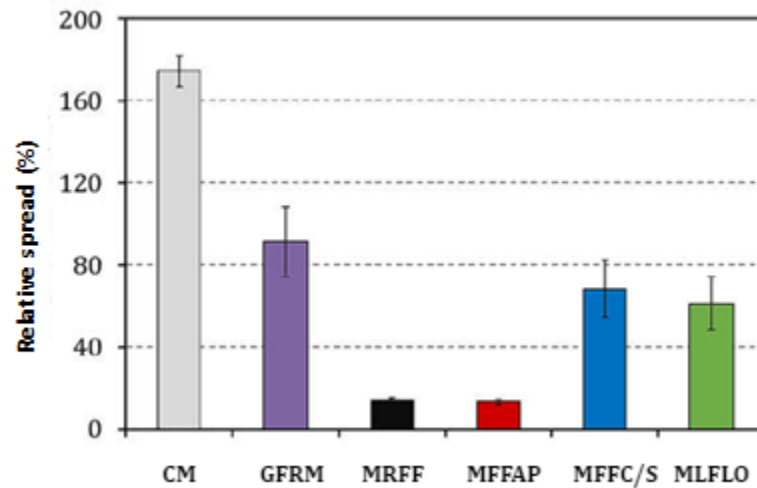


Figure 4: Relative spreads of mortars at the impact table

The same trends are seen with the maneuverability meter flow test (Figure 5).

CM mortar has a very fluid consistency, with a flow time of less than 2 seconds. MRFF mortar has poor workability, with a flow time of over 20 seconds, which corresponds to a very firm consistency. A flow time of about 17 seconds was measured on the MFFAP mortar, which is lower than MRFF, but still very high. Finally, mortars with coated fibers (MFFC/S and MLFLO) again obtained similar flow times (around 5 seconds), which translates to a notable improvement in workability.

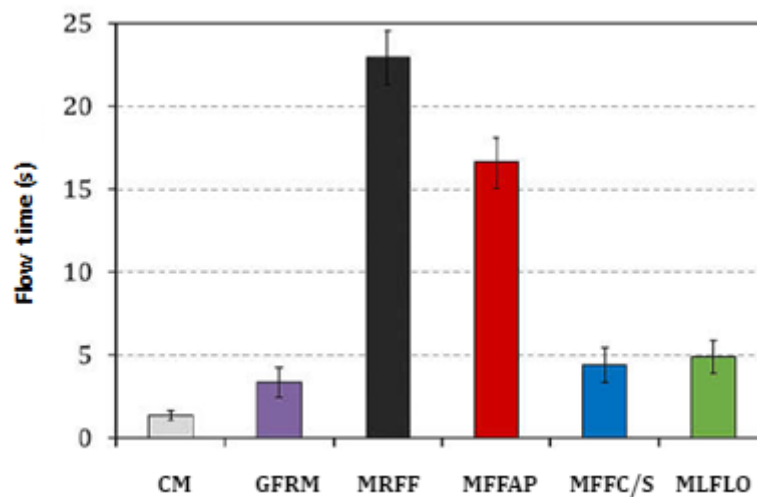


Figure 5: Flow time of mortars with the maneuverability meter

Air Content

The values for the entrained air, determined from experimental measurements of fresh densities, are shown in Figure 6. The air content of the reference mortar (CM) is equal to $1.81 \pm 0.63\%$. This entrained air value is relatively low for a mortar, the usual values being between 3 and 6%. This low value is due to the use of superplasticizer which, by

improving the fluidity of the material, also increased its compactness, which reduces the air content. The addition of fiberglass does not seem to have

a notable influence on the air content since the GFRM mortar has an air content very close to the CM mortar. However, it is observed that the incorporation of flax fibers in the mortars has the effect of significantly increasing the air content of the mixtures. The air content of MRFF is equal to $5.95 \pm 0.81\%$, which is approximately three times higher than the reference mortar (CM). Again, atmospheric plasma treatment does not appear to have a significant influence on the occluded air. Indeed, the MRFF mortar has an air content of $5.54 \pm 0.80\%$, ie a value very close to MRFF. The treatment with hydraulic binders reduced the air content of the composite with a value of $4.92 \pm 0.54\%$, which reflects an increase in the compactness of the corresponding mortar (MFFC/S). The mortar incorporating fibers coated with linseed oil (MLFLO) has the highest air content, equal to $6.99 \pm 0.82\%$.

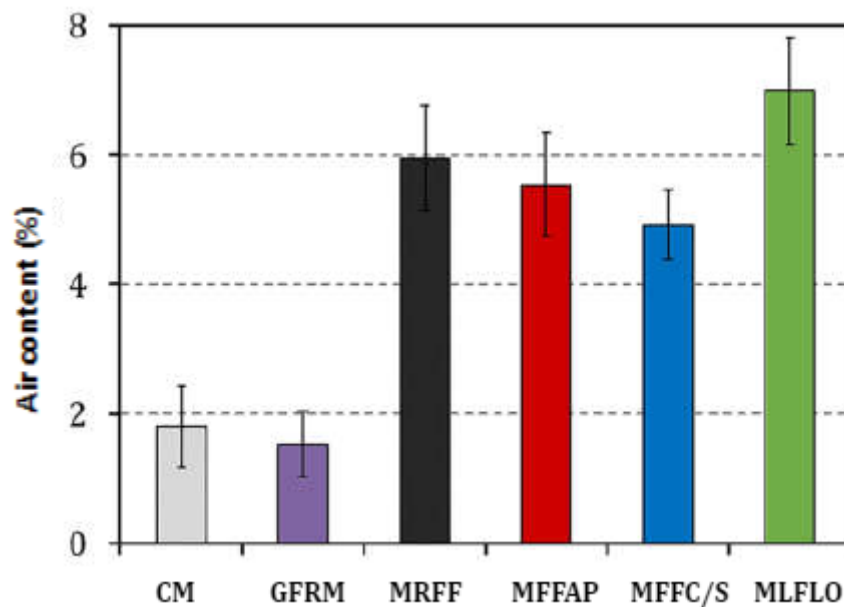


Figure 6: Air content of mortars

Furthermore, the increase in the occluded air also leads to a decrease in the theoretical proportion of cement, a constituent which has a strong influence on the resistance of cementitious materials. It is therefore important to recalculate the volume proportions of the constituents of the mortars, taking into account the air content.

Properties in the hardened state

Porosity accessible to water

The addition of flax fibers significantly increases the porosity of the mortars. We can also clearly distinguish the two mortars without flax fibers (CM and GFRM), which respectively have porosities equal to 13.8 ± 0.3 and $14.8 \pm 0.5\%$, from the mortars incorporating fibers of lin, with porosities between 18.5 ± 0.4 (MFFC/S) and 20.8 ± 0.2 (MLFLO). It would appear that the increase in porosity in mortars is directly related to

the increase in air content since a very clear linear relationship can be observed between these two structural parameters. On the other hand, a decrease in porosity was noted for the mortar incorporating the fibers coated with cement/slag (MFFC/S) in comparison with MRFF, of about 10%. Hydraulic binders (cement and slag) have therefore made it possible to reduce the porosity of the structure of the flax fibers and consequently reduce the porosity of the composites incorporating this reinforcement.

3-point bending behavior

The mechanical properties in a fiber composite mainly depend on the fiber content, their orientation, but also on the quality of the charge transfer at the fiber-matrix interface. This is particularly true for composites reinforced with short fibers due to the multiplication of interfaces. The transfer of forces between the fiber and the matrix takes place at their interface and plays an essential role in the behavior of the composite. Interfacial adhesion is a combination of different phenomena acting simultaneously to different degrees, such as physical and chemical adhesions between fibers and the matrix at this interface.

Concerning the bending behavior of mortars, the evolution of the applied stress as a function of the displacement of the fulcrum measured at the center of the test piece for a sample of non-fiber mortar (CM) and for Mortar specimens incorporating a volume fraction of flax fibers (raw or treated) is equal to 1.0%. Two types of behavior can be observed. The first one corresponding to the sample of the reference mortar (CM) is characterized by a linear behavior until rupture occurs suddenly (brittle character). The second relates to biofiber mortars. The behavior is linear until the first stall which reflects the first cracking of the matrix. The neighboring fibers of the crack tip are then stressed and this results in a rise in the load. The resulting nonlinear behavior persists up to the maximum load. The post-peak behavior is of the softening type with a gradual decrease in load and persistent elongation. . The addition of flax fibers therefore avoids brittle fracture of the material in bending and even induces behavior. We note that at 28 days, the first incident on the stress-displacement curves of the biofiber mortars occurs at a deformation almost identical to the reference mortar (CM). This deformation value characterizes the microstructure of the cement matrix in use. It is also observed that only MRFF and MFFAP mortars manage to display a higher stress after the first cracking of the matrix, thanks to the absorption of forces by the fibers. For mortars incorporating coated fibers (MFFC/S and MLFLO), the stress does not exceed the flexural strength of the matrix. A ductile behavior is nevertheless observed with these two mortars. It would therefore appear that the coating treatments (cement/slag and linseed oil) limit adhesion at the fiber/matrix interface, preventing a consequent transfer of tensile forces from the cement matrix to the flax fibers.

The mechanical behaviors of these fiber composites (MRFF, MFFAP, MFFC/S and MLFLO) are considerably modified after 90 days of age without accelerated aging. On the stress-displacement curves, we observe only a linear slope up to the maximum

stress, followed by brittle failure, such as non-fiber mortar (CM). The postpic zone is no longer present, or very little. The modification of the mechanical behavior of these composites could be explained by the evolution of the nature of the interphase and of the fibers themselves. Indeed, some authors have reported mineralization of fibers by a calcification process sometimes observed from 28 days of age. This mineralization process could continue over time and thus create an increasingly strong bond between the fibers and the cement matrix. The modification of the fiber/matrix interface from a weak state to a strong state is reflected in the mechanical behavior of composites which changes from ductile to brittle. Indeed, at 90 days of age, the stress values at the end of linear behavior are different, unlike the mortars at 28 days. After 90 days of curing, the constraints of the biofiber mortars are all greater than the control mortar (CM), which clearly indicates a contribution of fibers that is still present. However, these no longer make it possible to maintain a ductile behavior in bending but only to slightly increase the maximum bending stress.

In Figure 8, the maximum flexural strengths of the different mortars are shown after 7, 28, 90 and 320 days of curing. First of all, there is a consistent development of the resistances of the control mortar (CM) during the curing time. Glass fiber reinforced mortar (GFRM) is the one that shows the highest strengths, between 10.6 and 12.5 MPa, which is explained by the higher breaking stress of this reinforcement. However, there are very large variations in resistance for the same expiry date. Despite these strong variations, it is the GFRM mortar that has by far the highest mechanical resistance. The four biofiber mortars (MRFF, MFFAP, MFFC/S and MLFLO) have higher flexural strengths than the control mortar, with these strengths increasing with the maturities of 7, 28 and 90 days. The variations in resistance between the four biofiber mortars remain relatively low. The strengths of MRFF and MFFAP mortars are almost similar at all maturities. Atmospheric plasma treatment does not therefore seem to have no influence on the strength of composites. This result is consistent with observations, namely that this treatment does not reduce the water absorption of flax fibers in the long term, but only in the short term (less than 60 minutes). The strengths of MFFC/S mortar are lower than MRFF at 7 and 28 days but higher after 90 days of cure. This could be due to the presence of blast furnace slag in the fibers. This binder, with a pozzolanic reaction, is known to have lower strengths than Portland cement for short maturities (less than 28 days), but higher after 90 days. The mortar incorporating the fibers treated with linseed oil has overall lower strengths than MRFF. These lower strengths may be due primarily to a lower amount of cement present, given the very high air content and porosity of this mortar. Linseed oil could also affect the hydration of the nearby cement matrix to varying degrees. Finally, the interface between the cementitious matrix and the fibers treated with linseed oil is likely to be of lower quality than with raw flax fibers. In fact, the very low apparent roughness of linseed oil could cause greater slippage of the treated flax fibers within the matrix and therefore reduce the adhesion between these two elements at the interface. Other authors have also observed increased porosity at the interface between hemp aggregates coated with

linseed oil and a pumice-based mineral matrix. Furthermore, in the case of the four biofiber mortars, a decrease in flexural strength is observed at 320 days compared to 90 days. It is probable that at this time, the prolonged contact of the flax fibers with a cement matrix led to too much alkaline hydrolysis, which would then have damaged the very structure of the flax fibers, making them ineffective for resuming forces. tensile strength in the composite. The treatments do not seem, in view of these results, to improve the durability of flax fibers in contact with a cement matrix.

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Diagram

THE INFLUENCE OF DIFFERENT TREATMENTS APPLIED TO FLAX FIBERS ON THE DIFFERENT PROPERTIES OF MORTAR REINFORCED BY THESE FIBERS

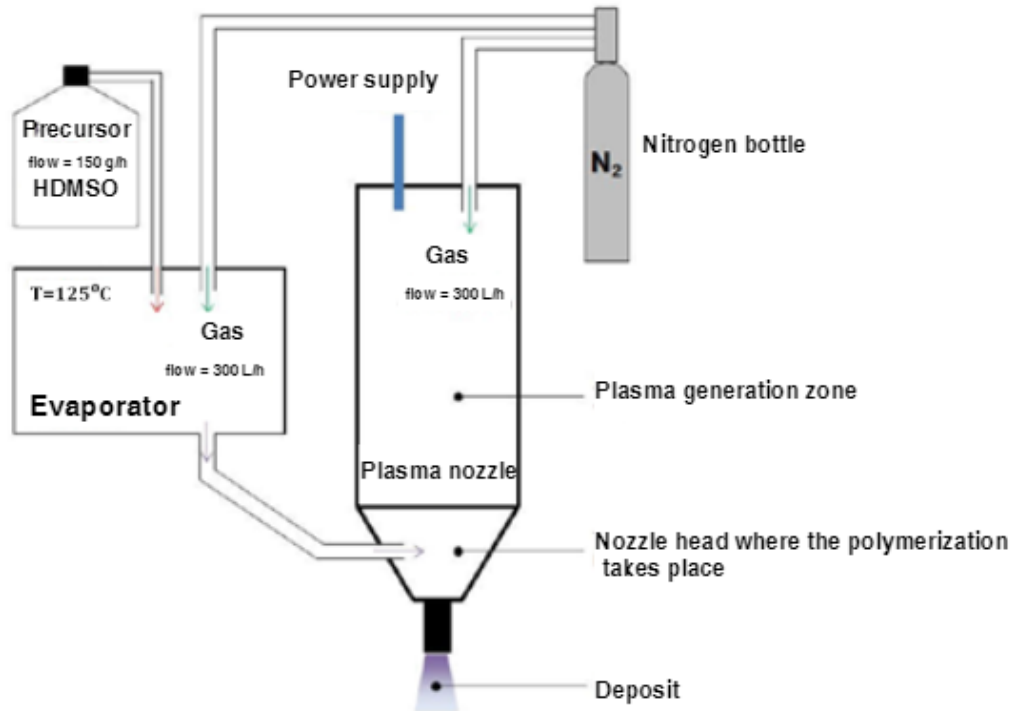


Figure 1: Principle of HDMSO deposition by plasma nozzle under atmosphere, $T=125^{\circ}\text{C}$

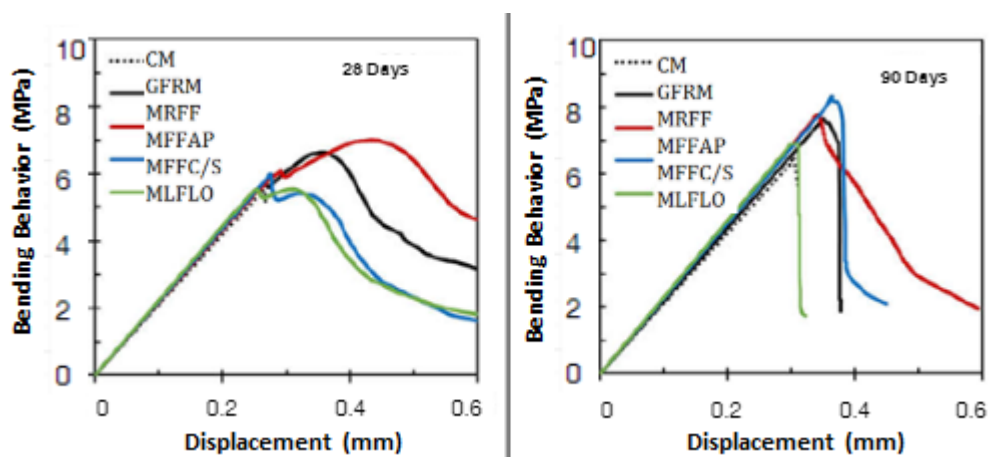


Figure 7: 3-point bending behavior curves of the different mortars after 28 and 90 days of curing

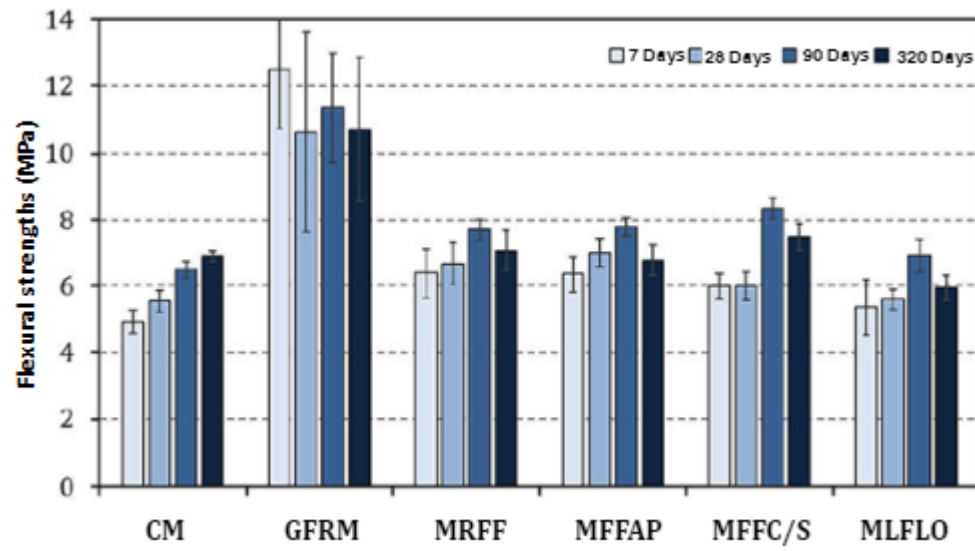


Figure 8: Flexural strengths of mortars after 7, 28, 90 and 320 days of curing

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Claims

THE INFLUENCE OF DIFFERENT TREATMENTS APPLIED TO FLAX FIBERS ON THE DIFFERENT PROPERTIES OF MORTAR REINFORCED BY THESE FIBERS

1. The need of treating flax fibers is explored by the high water absorption capacity of flax fibers which disrupts the rheological behavior of cementitious composites in the fresh state but also affects the physical and mechanical properties in the hardened state.
2. Atmospheric plasma treatment solution is explored
3. Mineral cement/slag coating treatment solution is explored and
4. Linseed oil coating treatment solution is explored.
5. The hygroscopic and hydroscopic behaviors of flax fibers have been studied.

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