OPTIMIZATION OF THE LINSEED OIL TREATMENT OF FLAX FIBRES: INFLUENCE ON FRESH PROPERTIES OF FIBRE-REINFORCED MORTARS

J. Page, S Amziane, M. Gomina

3rd International Conference on Bio-Based Building Materials

June 26th - 28th 2019
Belfast, UK

OPTIMIZATION OF THE LINSEED OIL TREATMENT OF FLAX FIBRES: INFLUENCE ON FRESH PROPERTIES OF FIBRE-REINFORCED MORTARS

J. Page[1]*, S. Amziane[2], M. Gomina[3]

[1] Univ. Artois, EA 4515, Laboratoire de Génie Civil et géo-Environnement (LGCgE), 62408 Béthune, France

[2] Univ. Clermont Auvergne, UMR 6602 CNRS, Institut Pascal, 63178 Aubière, France

[3] Univ. Caen Normandie, UMR 6508 CNRS, CRISMAT/ENSICAEN, 14050 Caen, France

*Corresponding author; e-mail: jonathan.page@univ-artois.fr

Abstract

For several years, many studies have focused on the mix-design and characterization of cementitious composites reinforced with plant fibres. It appears that in the fresh state, the morphology and the hydrophilic nature of these fibres significantly affect the rheology of the material. In the hardened state, the mechanical performance of the composites is lower than that of the composites including synthetic fibres. These low mechanical performances are mainly due to the weak fibre/matrix interface. Indeed, the high-water absorption capacity causes swelling/shrinkage of the fibre within the matrix, which subsequently leads to a lack of fibre/matrix bond. This work proposes to study the influence of a coating treatment with linseed oil applied to flax fibres on the properties of cementitious composites. The first part of this study deals with the optimisation of this treatment. The second part focuses on the effect of this treatment on the fresh state properties of the composite. The results showed a strong improvement in the workability of the treated fibre composites. A pronounced effect of the linseed oil treatment on air content was noted for high fibre content.

Keywords:

Plant fibre; Composite; Surface treatment; Water absorption, Workability.

1 INTRODUCTION

For several decades, fibres have been used in cementitious materials to improve the post-cracking and toughness behaviour. The most commonly used in cementitious composites are steel fibres, glass fibres and polypropylene fibres. But all these reinforcement materials have the disadvantage of being derived from non-renewable resources. Economic issues related to the rising costs of fossil resources, their increasing scarcity, and environmental impacts inherent to their production therefore lead to explore other material sources. Based on this observation, plant fibres could be a solution for the future of the construction industry. Among the plant fibres, flax stands out because of its high mechanical properties and its low density.

Much researches dealt with the incorporation of bio-based fibres in cementitious materials. It was highlighted that in fresh state, flax fibres affect the rheology of the material [Chafei 2014, Page 2017, Page 2019]. These rheological disturbances are mainly attributed to the high demand for water due to the high-water absorption capacity of flax fibres (up to 150 % of dry fibre mass) and their high specific surface area [Chafei 2015]. In hardened state, plant fibre-reinforced composites present an improvement of the flexural toughness or strength and an increased ductility [Merta 2013, Page 2019, Sedan 2008]. The transition from a brittle matrix to a ductile fibre-reinforced composite having a controlled post-peak behaviour is noted by several authors. However, this change in the stress-strain behaviour is not always accompanied by an improvement in the flexural strength [Kriker 2005].

Despite the interesting physical properties obtained for plant fibre-reinforced cementitious composites with, many studies highlight problems occurring at the fibre-matrix interface, sometimes manifested by the presence of a gap between these two components, without being able to quantify it precisely enough [Sedan 2008, Tonoli 2012]. This gap is often attributed to the swelling of the fibres during the concrete mixing process (in fresh state) followed by their shrinkage during the curing of the material (in hardened state). Moreover, these studies have shown that the hydrophilicity of plant fibres is responsible for these dimensional variations, which could lead to poor fibre/matrix interface.

Water absorption by plant fibres is therefore an important issue limiting their use in composite materials. Surface treatment may be a solution to limit or eliminate the water absorption by these fibres. Given the problems encountered when using plant particles with a cementitious matrix, many studies have been conducted to modify some of their characteristics. Most of the treatments experimented were aimed at limiting the hydrophilic behaviour of the fibre while others aimed at modifying the surface of the fibres. Chemical treatments are the most commonly experimented. They consist of a prolonged immersion of plant fibres in water-based solution [Chafei 2015, Olorunnisola 2008, Sellami 2013, Le Troedec 2008] or in an alkaline solution (sodium or calcium hydroxide, etc.) [Le Troedec 2008, Nozahic 2012, Sedan 2007, Toledo Filho 2000] or by esterification [Hill 1998, Khazma 2014]. The chemical treatments generally allow solubilization of plant extractables potentially troublesome and surface modification. However, they may cause a partial destruction of the fibre structure. In addition, these treatments have significant environmental impacts. Thermal treatments are regularly applied on wood; they have therefore been experimented on plant fibres [Bilba 2008]. Like chemical treatments, thermal treatments allow elimination of hemicelluloses and other extractables, and limition of the volume variations of the fibre. However, these treatments remain difficult to control and require heating for several hours. A mineral coating of the lignocellulosic fibres or aggregates can be carried out using a hydraulic binder (cement, lime, etc.) [Chafei 2017, Khazma 2008, Monreal 2011]. Mineral coatings seem to reduce the hydrophilicity of the fibres, chemically isolate the fibre from the matrix and limit the volume variations. However, it causes a considerable mass increase of the composite. Moreover, the long-term mechanical performance is degraded by the mineralisation of the fibres in contact with the binder used for the coating. Still with the aim to isolate and limit the plant fibre or aggregate water absorption, another solution is to apply an organic coating (polymer) such as paraffin wax [Juarez 2007], acrylic fluoropolymer [Chafei 2015, Chamoin 2013], linseed oil [Khazma 2014, Monreal 2011, Nozahic 2012]. These treatments make it possible to significantly reduce the water absorption of the aggregates and to improve the composite properties. Nevertheless, the surface created could modify the adhesion with the matrix. Depending on the nature of the polymer, the environmental impact can also be significant.

From the literature review, linseed oil stands out because of its bio-based origin and its efficiency to reduce the water absorption of plant aggregates. Thus, this work proposes to investigate a way to improve the fresh properties of flax-fibre-reinforced mortar using linseed oil as flax fibre coating.

2 EXPERIMENTAL PROGRAM

2.1 Materials

Binder

The hydraulic binder used was developed from two commercial powders: CEM I cement (67%), metakaolin (33%). The cement used is a CEM I 52.5 R ordinary Portland cement in accordance with EN 197-1 standard. The compressive strength on standardized mortar at 28 days is 72.0 MPa, the Blaine fineness is 4200 cm^2.g^{-1}, and the density about 3.13 g.cm^{-3}.

A Metakaolin was also used as an additive of Portland cement to develop the binder, in accordance with NF P18-513 standard. This is a commercial product named Argicem, supplied by Argeco Développement. This powder has a Blaine fineness of 4270 cm^2.g^{-1}, and the density is about 2.57 g.cm^{-3}.

In addition to cement and metakaolin, a high purity limestone was used (total carbonates equal to 98.7%), in accordance with NF P 18-508 standard. The Blaine fineness of the limestone filler is 5440 cm^2.g^{-1} and the density is 2.70 g.cm^{-3}.

Admixtures

In the literature, many studies have shown that plant fibres have a considerable influence on the rheological behaviour of mortars and concretes [Chafei 2015, Page 2019]. The use of a superplasticizer and a viscosity modifying admixture significantly reduces these disturbances [Le Hoang 2012, Page 2016]. Therefore, these additives were used for mortars reinforced with flax fibres. A high range water reducer superplasticizer based on polycarboxylates was added with a mass ratio of 0.30% relative to the binder (dry extract) to provide a better workability in the fresh state.

In addition, a viscosity-modifying agent (VMA) based on a high-molecular weight bio-polymer was added to the mixture with a mass ratio of 0,02% relative to the binder (dry extract) to avoid segregation between the paste and the aggregates.

Aggregates and fibres

An alluvial sand with grain size 0/2 mm was used. This sand presents a specific gravity of 2.66 g.cm^{-3}, an absorption coefficient of 0.15%.

Flax fibres used were harvested in Normandy (France) in 2014, cut at a length of 12 mm and provided by Vandecandelaere Company (Depestele Group), specialized in flax farming and scutching. These raw flax fibres present a real density of 1.52 g.cm^{-3} [Barbulée, 2015], and a mean diameter of 14.66±2.95 µm. The water absorption rate of the flax fibres after 24 hours of immersion is 132.4±4.2%. This value was used to correct the effective water content for mortars mix design.

2.2 Linseed oil treatment

Linseed oil (LO) is a drying oil susceptible to auto-oxidation and polymerization resulting in a tough elastic film upon air exposure [Sharma 2006]. Linseed oil is composed of three fatty acids (oleic, linoleic and linolenic) with long unsaturated aliphatic chains and saturated acids (stearic and palmitic). Both linoleate and linolenate acids rapidly oxidize, since their unsaturated bonds are close the one to the other. The reactivity strongly depends on temperature and linoleic fatty acid over linolenic fatty acid ratio [Stenberg 2005]. Moreover, the heat released during the oxidation-polymerization of linseed oil increases the kinetics of reaction and may induce its spontaneous combustion [Abraham 1996]. For this reason, it is recommended not to use linseed oil at a temperature above 60°C.

The linseed oil used in this work is a boiled linseed oil with an accelerated oxidation, supplied by Natura® laboratories. It is an oil extracted from farmed flax, harvested and produced in Normandy. It was obtained by cold extraction without any solvent, filtered at 1 µm, and then heated at 150°C. This linseed oil has a density of 0.93 g.cm^{-3}.

For carrying out the treatment, the flax fibres were previously dried at 50±1°C and then introduced into a planetary mixer. The mixer is then started at a speed of 30 rpm and after 30 seconds the linseed oil is introduced for a period of 2 minutes 30 seconds. The oil / fibre mass ratio was varied from 0.25 to 2. Mixing was maintained for 2 minutes 30 seconds more to ensure a uniform coating of the fibre. The fibres thus treated are placed in a ventilated oven at 50±0.1°C for 14 days.

Tab. 1: Composition of mortars.

Mortar	Ref. Mortar	Raw Fibres Mortars			Linseed Oil treated Fibres (ratio = 0.25) Mortars			Linseed Oil treated Fibres (ratio = 0.5) Mortars		
Fibre content	**0 %**	**1 %**	**2 %**	**3 %**	**1 %**	**2 %**	**3 %**	**1 %**	**2 %**	**3 %**
Sand 0/2 (g)	800.0	800.0	800.0	800.0	800.0	800.0	800.0	800.0	800.0	800.0
Binder (g)	600.0	600.0	600.0	600.0	600.0	600.0	600.0	600.0	600.0	600.0
Limestone (g)	200.0	200.0	200.0	200.0	200.0	200.0	200.0	200.0	200.0	200.0
Eff. Water (g)	300.0	300.0	300.0	300.0	300.0	300.0	300.0	300.0	300.0	300.0
Flax fibres (g)	0.0	13.4	26.8	40.2	12.3	24.6	36.9	12.0	24.0	36.0
Add. water (g)	0.0	17.4	34.8	52.3	10.3	20.7	31.0	8.8	17.6	26.4
Density ($g.cm^{-3}$)	2.158	2.130	2.103	2.077	2.137	2.117	2.098	2.139	2.120	2.103

2.3 Mortars mix design

The respective proportions of the mortar's components are detailed in Table 1. For all compositions, the effective water on binder (CEM I cement + Metakaolin) ratio was fixed to 0.50 and the sand on powders (binder + limestone) ratio was fixed to 1.0.

Flax fibres were added with different proportions: 1.0, 2.0 and 3.0 vol.% to the total batch volume. An additional amount of water was added to take into account the water absorption by flax fibres. These quantities are given in Table 1.

After mixing, the mortars were cast in two layers into prismatic moulds (40x40x160 mm). To expel the air from the mixture, the moulds were subjected to 60 hits on a vibrating table. The test pieces were demoulded 24 hours after manufacturing, and then placed in a climate chamber (20±1°C and 65±5%RH).

3 TESTING METHODS

3.1 Flax fibre characteristics

Water absorption

Due to their nature and chemical composition, flax fibres are very sensitive to moisture. The flax fibres were dried by heating at 50±0.1 °C in a ventilated oven for 48 hours prior to testing. The water absorption kinetic was obtained by placing 3.00 grams of flax fibres in a microperforated bag, then dipping this bag in water for different times (from 1 minute to 48 hours). The experiment has been repeated six times for each soaking time. After immersion, the bags were placed in a centrifuge operating at 500 rpm for 30 seconds to remove the excess water on the surface of the fibres.

Real density

The real density measurements of raw and treated flax fibres was conducted using a helium pycnometer, on oven-dried fibre samples of about 1.0 gram; six samples were tested for each coating treatment.

3.2 Mortar's fresh properties

Air content

The fresh state density has been determined by measuring the mass and volume of samples filled in vibrated metallic moulds. The air content of fresh mortar was estimated from the relative difference of the mortar theoretical density and the measured fresh density.

Workability

The workability of the different mortars was assessed using the flow table method, in accordance with EN 1015-3. In this procedure a sample of the fresh mortar is placed on the flow table tamper by means of a truncated conical mould, and given 15 vertical hits by raising the flow table and allowing it to fall freely from a height of 10 mm. The flow value of the mixture is estimated by the average of the slump diameters measured in two orthogonal directions.

4 OPTIMIZATION OF THE LINSEED OIL TREATMENT

Previous studies using linseed oil for the treatment of plant particles had used variable oil/particle ratios. For example, Monreal had chosen ratios of 0.33, 0.5 and 1 [Monreal 2011]. Nozahic et al. have adopted an oil / sunflower aggregates ratio of 0.5 [Nozahic 2012] while Khazma had retained oil / flax shives ratios of 1, 2 and 3 [Khazma, 2008]. Finally; Sellami had used an oil / fibre ratio of 0.78 [Sellami 2013]. A wide range of ratios has therefore been experimented, depending on the type of the plant particles. Thus, the literature review does not allow making a reasoned choice of the linseed oil ratio for the treatment of flax fibres. Therefore, in order to optimize the mass quantity of linseed oil relative to the flax fibres, five LO/fibre ratios were investigated: 0.25, 0.5, 1, 1.5 and 2. Microscopic observations of raw and LO-treated flax fibres are given in Fig. 2.

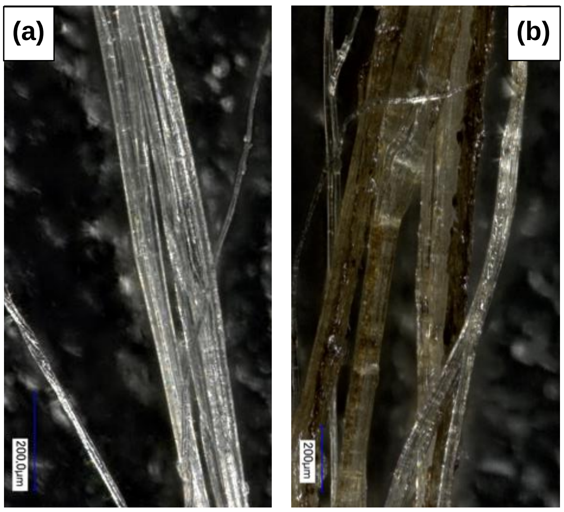

Fig. 2: Microscopic observations of raw (a) and linseed oil treated flax fibres (b).

4.1 Mass monitoring

As a reminder, after treatment, flax fibres were placed in an oven at 50°C for 2 weeks. A portion of the treated fibres was placed into metal cups in order to follow the evolution of the mass of the samples over time. On Fig. 3 are presented the mass gains of the treated flax fibres relative to the initial mass of oil, as a function of the elapsed time after treatment, for each LO/fibre ratio.

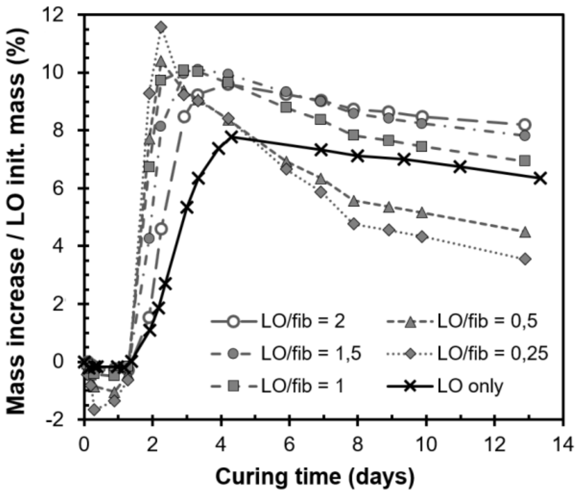

Fig. 3: Mass monitoring of fibres treated with linseed oil during the treatment cure.

For all samples, there is a mass gain of linseed oil in the first stages of cure (up to 4 days). Low LO/fibre ratios (0.25 and 0.5) led to more important (up to 12%) and faster (up to 2 days) oil mass increase. This mass increase is associated with oxidation of linseed oil. The polymerization of the boiled linseed oil seems to set between 2 and 4 days after application of the treatment. This is in agreement with the results obtained by Lazko et al. who observed a mass increase of linseed oil of between 10 and 15% [Lazko 2011].

4.2 Density

The linseed oil coating treatment of fibres has the effect of reducing the overall density of the treated fibres (Fig. 4). The specific density of the raw flax fibres is 1.521±0.001 g.cm^{-3} and the one of the linseed oil is 0.93 g.cm^{-3}. As a result of the coating treatment, the density of flax fibre coated with LO is 1.205±0.008 g.cm^{-3} for LO/fibres ratio of 2.

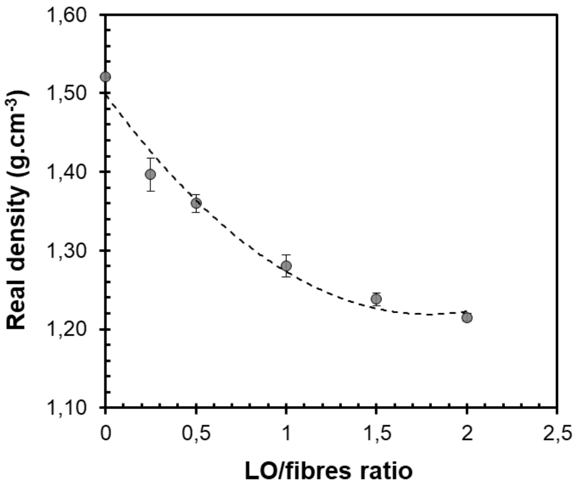

Fig. 4: Density of the coated fibres versus LO/fibre ratio.

Due to the low density of linseed oil when the LO/fibre ratio increases, the treated fibre density raises as the proportion of oil is greater. We can note the very small standard deviations in Fig. 4. That suggests a good homogeneity of the coating on the fibres.

4.3 Water absorption

The main purpose of the linseed oil treatment is to reduce water absorption by the flax fibre. The linseed oil (LO) / fibre ratio will therefore be optimized especially according to this criterion. Water absorption measurements were therefore carried out on these treated fibres immersed for 60 minutes in water. The results are presented in terms of mass ratios, based on the mass of coated flax fibres (Fig. 5).

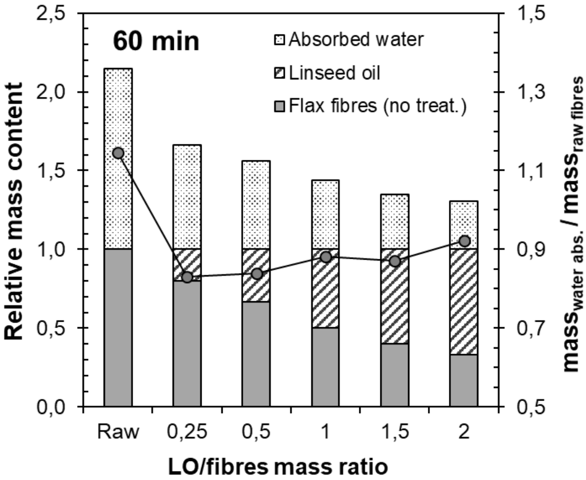

Fig. 5: Absorbed water mass relative to the mass of fibres treated, according to the LO/fibre ratio after immersion for 60 minutes.

This type of representation was chosen to highlight the absorption results with respect to the linseed oil present on the treated fibres. Indeed, this linseed oil does not absorb water. It is therefore obvious that the absorption of the treated flax fibres is reduced compared to raw fibres.

The correct representation would be to compare the mass of absorbed water to that of uncoated fiber ($mass_{water,abs.}/mass_{raw,fibres}$). The calculated values are reported in Fig. 5 and the associated curve (with black points in the figure) can be used to determine the optimal ratio for a given linseed oil quantity. It appears logically that raw fibres are those that absorb the higher water ratio: 115.0±4.2% after 60 minutes of immersion. It is also noted that the higher the LO/fibre ratio is, the lower is the amount of absorbed water.

However, it is important to express this rate of absorbed water as a function of the amount of raw fibres present in the sample to identify the most effective LO/fibre ratio. Therefore, the curve (relative to the right y-axis in Fig. 4) shows the mass of absorbed water relative to the mass of raw fibres. Thus, the LO/fibre ratios of 0.25 and 0.5 seem to be the most effective for decreasing the water absorption. These two ratios will therefore be retained for the continuation of this study.

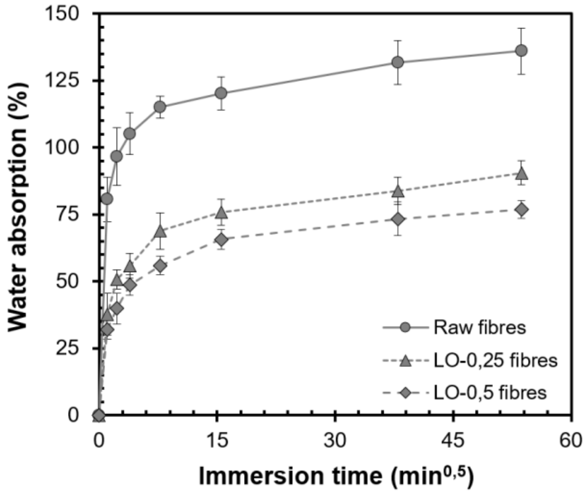

Fig. 6: Water absorption of raw and treated flax fibres.

The water absorption kinetics of raw and treated flax fibres are presented in Fig. 6. Two distinct stages are evidenced for all fibres. First, a quick water absorption is observed in short time. in less than 6 minutes the raw fibers absorb more than 50% of the estimated water mass for their saturation. A second absorption stage shows a less steep raise toward the saturation level. The highly hydrophilicity of plant fibres by the nature of their constituents explains the strong dependency of their morphology and their physical and mechanical properties on the water content [Stamboulis 2001, Thuault 2014]. This essential characteristic also induces a stiff competition between flax fibres and binder on the water demand, which starts with concrete mixing. As it can be seen in Fig. 6, the linseed oil treatments (0,25 and 0,5 LO/fibre ratios) strongly reduce the water absorption of the fibre. The water absorption rate after immersion for 24 hours (WA_{24}) is only 83.8±5.2% and 73.4±6.2% for LO/fibre ratios of 0.25 and 0.5, respectively. That means relative reductions of 36% and 44%, respectively when compared to the raw fibres.

5 PROPERTIES OF MORTARS IN FRESH STATE

Linseed oil treatment showed a significant decrease of water absorption by the fibre, especially for 0.25 and 0.5 ratios. Mortars incorporating raw and treated (with these two ratios) fibres were manufactured and characterized in both fresh and hardened states. Three volume proportion of flax fibres were tested: 1.0, 2.0 and 3.0 vol.%.

5.1 Air content of fresh mortars

The incorporation of flax fibres into mortar mixes increases the air content due to entrainment of additional air (Fig. 7). The reference mortar has the lowest air content of 1.7±0.1%. As expected, increasing the fibre content raises the rate of entrapped air, raw or treated fibres. This influence of the fibres on the occluded air content was also underlined by other authors [Pickering 2016 and Page 2019]. It is mainly attributed to the low ability of fibres to compact. Indeed, the fibres have a significant impact on the granular compactness of concrete mixtures [Page 2017].

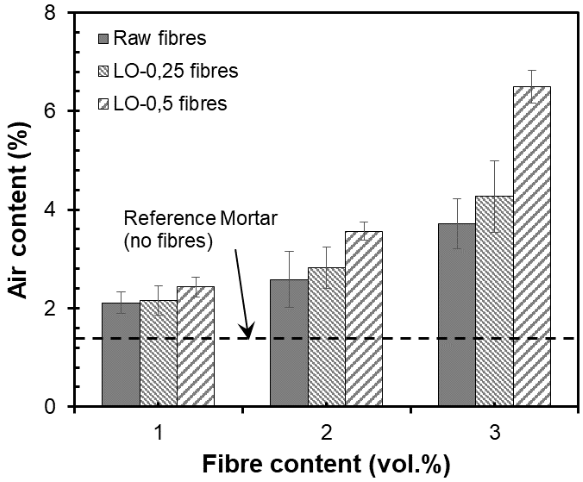

Fig. 7: Estimated air content of fresh mortars.

5.2 Consistence of fresh mortars

Fig. 8 reports the diameter measurements obtained by the method of the flow table. Firstly, the reference mortar shows a plastic consistency, with a flow of 18,0±0,4 cm. Note that this mortar does not contain a superplasticizer. In contrast, mortars with flax fibres all contain a superplasticizer and a viscosity-modifying admixture to overcome the problems encountered with the use of flax fibres in cementitious composites [Page 2017]. Moreover, despite the use of admixtures, the raw fibre mortars collapse less than the reference mortar: 17.8±0.3 cm with 1.0 vol.% of raw flax fibres, 13.7±0.6 cm with 2.0 vol.% and 11.4±0.6 cm with 3.0 vol.%. Thus, as the fibre content increases, the flow decreases, reflecting a reduction of workability. This trend was also highlighted by other studies [Tung 2013, Page 2017].

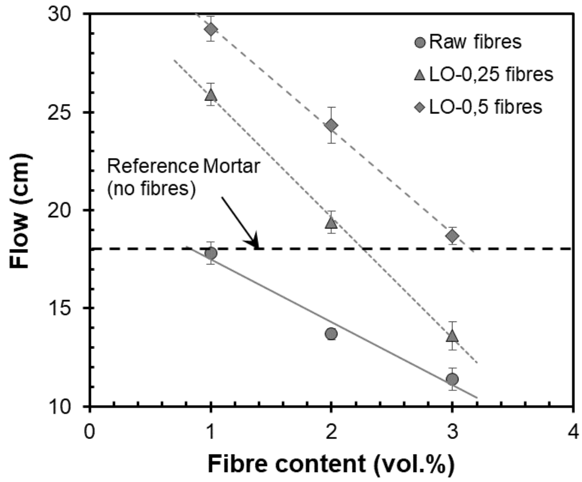

Fig. 8: Slump diameter measured with flow table for the different mortars.

The linseed oil treatment of flax fibres significantly enhances the workability of fresh fibre-reinforced mortars. Indeed, with the LO-0.5 treated fibres, the measured diameters are on average 69% higher than raw fibre-reinforced mortars. With these treated fibres, the diameter is always higher than 18 cm, even with 3.0 vol.% of fibres in the mixture. With 1.0 vol.%, the measured flow is 29 cm, that means a very fluid consistency. With the LO-0.25 treated fibres, the workability of the mortars is also improved compared to raw fibre-mortars, but to a lesser extent. For 1.0 vol.% fibres, the diameter is greatly increased, up to about 26 cm, i.e. 45% higher than raw fibre-mortar. On the other hand, the improvement is less significant with 3.0 vol.% of fibres, with a flow diameter of about 17 cm, i.e. an improvement of 19%.

The significant reduction of the water absorption of flax fibres due the linseed oil treatment can explain these workability enhancements. However, the coating effect of this treatment can also explain this improvement. Indeed, the linseed oil treatment have the effect of increasing the apparent cross-section area (due to coating) of the fibrous reinforcement, and therefore their specific surface area is reduced. In doing so, the demand for cement paste to meet the workability criteria is greatly reduced.

For mortars reinforced with 1.0 vol.% of fibres, the linseed oil treatment has no significant impact on the air content, which is between 2.1±0.2% and 2.4±0.2%. For mixtures with 2 vol.% of fibres, the linseed oil treatment (especially with the 0.5-LO/fibre ratio) results in a noticeable increase in air content compared to raw fibres. The effect of the linseed oil treatment on air content is even more pronounced for mixtures containing 3 vol.% fibres. For raw fibres, the air proportion represents 3.7±0.5% against 4.3±0.7% and 6.5±0.3% for 0.25 and 0.5 LO/fibre ratio, respectively. This effect of fibre treatment on air content could be explained by the coating characteristics specific to linseed oil. Indeed, linseed oil seems to coat sometimes unit flax fibres and sometimes bundles of fibres. In the latter case, air could be entrapped in the coated fibre bundles. Once these fibres are mixed with the mortar, the air remain entrapped, which would have the effect of increasing the air content of the material.

6 CONCLUSIONS

This works has investigated a way to improve the physical properties of flax-fibre-reinforced mortars using linseed oil coating treatment. The linseed oil treatment has been optimized by using a smaller content of linseed oil while minimizing water absorption. The following main conclusions can be pointed out:

- The water absorption of flax fibres disturbs the rheological behaviour of the cementitious composites in the fresh state but also affects the mechanical properties in the hardened state. The hydroscopic behaviour of raw and linseed oil coated flax fibres was first studied. It appeared that the flax fibres absorb very rapidly a huge quantity of water (up to 130% by mass). The linseed oil treatment decreases the water absorption capacity. Thus, the LO/fibre ratios of 0.25 and 0.5 seem to be the most effective for depressing the water intake.
- Linseed oil treatments have reduced the water absorption of fibres, resulting in a significative improvement of workability for composites made with these treated fibres. Furthermore, these coating thus increased the fibre diameter, which lead to a decrease of the specific surface area and therefore of the water demand of flax fibres.
- The incorporation of flax fibres into mortar mixes increases the air content due to entrainment of additional air. Moreover, a pronounced effect of the linseed oil treatment on air content was noted for high fibre content.

However, important issues remain to be investigated, as the effect of this treatment on hardened mortar properties. This point is the subject of work in progress.

7 REFERENCES

[Abraham 1996] Abraham, C.J.; A solution to spontaneous combustion in linseed oil formulations. Polymer Degradation and Stability, November 1996, Vol. 54, n°2-3, p. 157-166.

[Barbulée 2015] Barbulée, A.; Compréhension des effets du défibrage sur la morphologie, les propriétés et le comportement mécanique des faisceaux de fibres de lin. Etude d'un composite dérivé lin/époxyde. Université de Caen Basse-Normandie, 2015.

[Bilba 2008] Bilba, K.; Arsene, M.A.; Silane treatment of bagasse fiber for reinforcement of cementitious composites. Composites Part A: Applied Science and Manufacturing, September 2008, Vol. 39, n°9, p. 1488-1495.

[Chafei 2014] Chafei, S.; Khadraoui, F.; Boutouil, M.; Gomina M.; Optimizing the formulation of flax fiberreinforced cement composites. Construction and Building Materials, March 2014, Vol. 54, p. 659-664.

[Chafei 2015] Chafei, S.; Khadraoui, F.; Boutouil, M.; Gomina, M.; Effect of flax fibers treatments on the rheological and the mechanical behavior of a cement composite. Construction and Building Materials, March 2015, Vol. 79, p. 229-235.

[Chafei 2017] Chafei, S.; Gomina, M.; Khadraoui, F.; Boutouil, M.; Dependence of the properties of cementitious composites on the nature of the hydraulic binder coating the reinforcing flax fibers. Journal of Mechanical and Civil Engineering, November 2017, Vol. 14, n°6, p. 27-33.

[Chamoin 2013] Chamoin, J.; Optimisation des propriétés (physiques, hydriques et mécaniques) de bétons de chanvre par la maîtrise de la formulation. INSA de Rennes, 2013.

[Hill 1998] Hill, C.A.S.; Khalil, H.P.S.A.; Hale, M.D.; A study of the potential of acetylation to improve the properties of plant fibres, Industrial Crops and Products, March 1998, Vol. 8, n°1, p. 53-63.

[Juarez 2007] Juarez, C.; Durán, A.; Valdez, P.; Fajardo, G.; Performance of "Agave lecheguilla" natural fiber in Portland cement composites exposed to severe environment conditions. Building and Environment, March 2007, Vol. 42, n°3, p. 1151-1157.

[Khazma 2014] Khazma, M.; Goullieux, A.; Dheilly, R.M.; Rougier, A. et al.; Optimization of flax shive-cementitious composites: Impact of different aggregate treatments using linseed oil. Industrial Crops and Products, November 2014, Vol. 61, p. 442-452.

[Kriker 2005] Kriker, A.; Debicki, G.; Bali, B.; Khenfer, M.M. et al.; Mechanical properties of date palm fibres and concrete reinforced with date palm fibres in hot-dry climate, Cement and Concrete Composites, May 2005, Vol. 27, n°5, p. 554-564.

[Lazko 2011] Lazko, J., Dupré, B., Dheilly, R.M., Quéneudec, M.; Biocomposites based on flax short fibres and linseed oil, Industrial Crops and Products, March 2011, Vol. 33, n°2, p. 317-324.

[Le Hoang 2012] Le Hoang, T.; Boutouil, M.; Khadraoui, F., Gomina, M.; Mechanical and microstructural characterization of flax fibre-reinforced cement composite. In: Proceedings of the 11th Japan-Korea-France-Canada Joint Seminar on Geoenvironmental Engineering, Caen, France, 2012, vol. 11, p. 131-136.

[Le Troedec 2008] Le Troedec, M.; Sedan, D.; Peyratout, C.; Bonnet, J.P. et al.; Influence of various chemical treatments on the composition and structure of hemp fibres, Composites Part A: Applied Science and Manufacturing, May 2005, Vol. 27, n°5, p. 514-522.

[Merta 2013] Merta, I.; Tschegg, E.K.; Fracture energy of natural fibre reinforced concrete. Construction and Building Materials, March 2013, Vol. 40, p. 991-997.

[Monreal 2011] Monreal, P.; Mboumba-Mamboundou, L.B.; Dheilly, R.M.; Quéneudec, M.; Effects of aggregate coating on the hygral properties of lignocellulosic composites. Cement and Concrete Composites, February 2011, Vol. 33, n°2, p. 301-308.

[Nozahic 2011] Nozahic, N.; Amziane S.; Influence of sunflower aggregates surface treatments on physical properties and adhesion with a mineral binder. Composites Part A: Applied Science and Manufacturing, November 2012, Vol. 43, n°11, p. 1837-1849.

[Olorunnisola 2008] Olorunnisola, A.O.; Effects of pre-treatment of rattan (*Laccosperma secundiflorum*) on the hydration of Portland cement and the development of a new compatibility index. Cement and Concrete Composites, January 2008, Vol. 30, n°1, p. 37-43.

[Page 2016] Page, J.; Sonebi, M.; Amziane, S.; Étude expérimentale des propriétés d'un béton de chanvre incorporant un adjuvant viscosant. Revue des Composites et des Matériaux Avancés, 2016, Vol. 26, n°3-4, p. 349-366.

[Page 2017] Page, J.; Khadraoui, F.; Boutouil, M.; Gomina, M.; Multi-physical properties of a structural concrete incorporating short flax fibers. Construction and Building Materials, June 2017, Vol. 140, p. 344-353.

[Page 2019] Page, J.; Khadraoui, F.; Gomina, M.; Boutouil, M.; Influence of different surface treatments on the water absorption capacity of flax fibres: Rheology of fresh reinforced-mortars and mechanical properties in the hardened state. Construction and Building Materials, February 2019, Vol. 199, p. 424-434.

[Pickering 2016] Pickering, K.L.; Efendy, M.G.A.; Le, T.M.; A review of recent developments in natural fibre composites and their mechanical performance. Composites Part A: Applied Science and Manufacturing, April 2016, Vol. 83, p. 98-102.

[Sedan 2008] Sedan, D.; Pagnoux, C.; Smith, A.; Chotard, T.; Mechanical properties of hemp fibre reinforced cement: Influence of the fibre/matrix interaction. Journal of the European Ceramic Society, January 2008, Vol. 28, n°1, p. 183-192.

[Sellami 2013] Sellami, A.; Merzoud, M.; Amziane, S.; Improvement of mechanical properties of green concrete by treatment of the vegetals fibers. Construction and Building Materials, October 2013, Vol. 47, p. 1117-1124.

[Sharma 2006] Sharma, V.; Kundu, P.P.; Addition polymers from natural oils – A review. Progress in Polymer Science, November 2006, Vol. 31, n°11, p. 983-1008.

[Stenberg 2005] Stenberg, C.; Svensson, M.; Johansson, M.; A study of the drying of linseed oils with different fatty acid patterns using RTIR-spectroscopy and chemiluminescence (CL). Industrial Crops and Products, March 2005, Vol. 21, n°2, p. 263-272.

[Stamboulis 2001] Stamboulis, A.; Baillie, C.A.; Peijs, T.; Effects of environmental conditions on mechanical and physical properties of flax fibers. Composites Part A: Applied Science and Manufacturing, August 2001, Vol. 32, n°8, p. 1105-1115.

[Thuault 2014] Thault, A.; Eve, S.; Blond, D.; Breard, J. et al.; Effects of the hygrothermal environment on the mechanical properties of flax fibres. Journal of Composite Materials, June 2014, Vol. 48, n°14, p. 1699-1707.

[Tonoli 2012] Tonoli, G.H.D.; Belgacem, M.N.; Smith, A.; Bras, J.; Pereira-da-Silva, M.A. et al.; Mechanical properties of hemp fibre reinforced cement: Influence of the fibre/matrix interaction. Journal of Materials Science, May 2012, Vol. 47, n°9, p. 4167-4177.