COMPARATIVE STUDY OF DIFFERENT LINSEED OIL TREATMENTS ON WOOD AND HEMP

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Abstract

Just as the majority of vegetable materials, wood presents an important absorption rate. This property limits its use in cementitious matrix where a part of water dedicated to hydration of binder, and even the totality, is consumed by the vegetable part, affecting in a negative way mechanical and physicochemical properties of the final product. To limit this water absorption, one of the avenues is to coat vegetable material by linseed oil.
The study conducted allowed to evaluate the impact of pure and emulsion linseed oil coatings on maximal absorption rate of wood shavings as a function of different conditions of curing. In the same way, a comparative work has been realized in order to appreciate the efficacy of such treatments on another type of vegetables: hemp.
Results show that it is possible to identify the better conditions to polymerize linseed oil coatings and significantly reduce the water uptake of green aggregates. Moreover, a comparison between wood and hemp reveals a correlation between efficacy of studied treatments and intrinsic properties of considered green materials.

Keywords:
Vegetables – Wood treatment – Linseed oil – Absorption – Hemp treatment

1 INTRODUCTION

Since the 2000's, the number of studies dealing with incorporation of bio-sourced materials in concrete construction in total or partial substitution of coarse aggregates has significantly increased. Works essentially focus on flax shives, wood or hemp, but other vegetable materials have already been tested as coconut shell, rice husk, beet pulp, bamboo or fibres of date palms, for examples [Amziane 2016]. In an environmental point of view, this approach allows to reduce the carbon footprint of the final product and to preserve non-renewable natural resources. However, introduction of these bio-sourced materials in a cementitious matrix usually causes a decrease of mechanical performances, which increases with the introduction rate. This can be explained by intrinsic properties of these aggregates (high absorption rate, leaching of water soluble extractives in the matrix,...).

In order to enhance compatibility of these bio-sourced aggregates with cementitious matrix, one of the potential solutions studied in literature is to coat surface of materials with linseed oil. For that, several parameters are determining, in which it can be cited type of oil (crude, boiled,…), ratio (oil / dry aggregates) and the choice of curing, essentially. Moreover, one of the obstacles is to limit the spontaneous combustion phenomenon particularly observed in confined spaces.

In their works, Ledhem and al. [Ledhem 2000] coat pine wood shavings with crude linseed oil, with a ratio oil / dry aggregates varying between 0.25 and 0.75, and a curing at 20°C / 50%RH during at least one month. The water uptake obtained is lower than 50% for a ratio equal to 0.75 and around 250% for untreated wood.

Khazma and al. [Khazma 2014] work as for them on flax shives treated with linseed oil (pure or in emulsion form). After several tests, authors have chosen a ratio oil (pure or emulsion) / dry flax shives equal to 2, a lower or higher ratio leading respectively to a discontinuous coating or an excess of oil.

Flax shives are dried at 50°C until constant mass is reached. Results of water uptake obtained are 202%, 75% and 50% for respectively untreated, treated by emulsion of linseed oil and treated by pure linseed oil flax shives.

Linseed oil treatments were also tested on other types of vegetable materials. For example, Monreal and al. have conducted a study on beet pulp [Monreal 2011]. Merzoud and al. have studied linseed oil based treatment on Diss fibres, and Nozahic worked on hemp and sunflower aggregates [Nozahic 2012].

In a general way, literature proves that linseed oil treatment is efficient in terms of the water uptake. Nevertheless, and after advanced researches, it appears that there is linseed oil in the water used for absorption test, a sign that a part of the oil added for the treatment has not polymerized. This fact has a slight impact on calculation of water uptake because mass of this oil should be taken into account. Moreover, in this case, it is clear that the compatibility with cementitious matrix is poor, with a weak adhesion between vegetable aggregates and cementitious binder, and so low mechanical performances.

A comparison of results given in literature allows to show that a same treatment can present different level of efficiency depending on type of bio-sourced materials considered. The aim of this study is to make a correlation between performance of linseed oil coating as a function of several parameters (oil diluted or not in water, mass ratio Pure Linseed Oil / Dry Aggregates (LO / A) or Linseed Oil Emulsion / Dry Aggregates (DLO / A), temperature and time of curing), and as a function of two types of materials: wood and hemp.

One of the principal issues of this work is to understand the relation between treatment efficiency of a linseed oil treatment and intrinsic properties of treated bio-sourced materials.

2 MATERIALS AND METHODS

2.1 Raw materials

Bio-sourced aggregates

Wood shavings used come from the grinding of resinous wood, essentially. They do not undergo any chemical treatment. Hemp wood is provided by Biofibat calibrated and dusted. It is obtained after a mechanical operation which allows to separate the heart of the stem (used in this study) and fibres.

These materials are dried in an oven at 60°C until constant mass is reached (i.e. mass variation lower than 0.1% between two consecutive weighings) before use.

Some porosity characteristics of these materials, obtained by Autopore Mercury Porosimeter are given in Tab.1.

Linseed oil

Linseed oil used is a crude oil obtained by cold-pressed of flax seeds.

Binder for compatibility test

The binder used is Portland cement CEM I 52.5 N.

2.2 Test methods

Water uptake of aggregates

To determine water uptake of aggregates, an exact mass of material is introduced in a given volume of water, and undergoes a forced immersion during 24h.

Water absorption is calculated according to equation (1).

$$w\ (\%) = \left(\frac{mh-ms}{ms}\right) * 100 \quad (1)$$

With:

w (%) : water uptake of sample after immersion

mh (g): moist weight of sample after immersion

ms (g): corrected dry weight (taking into account real moisture content of treated sample before immersion)

At the same time as this assay, a part of tested bio-sourced materials is dried in an oven at 105°C in order to determine moisture content of the samples and calculate the equivalent dry weight really introduced in water.

During this test, colour of water is also noted. Water is then stored for analysis.

Linseed oil treatments

Pure Linseed Oil

Linseed oil is weighted to obtain LO / A desired. Oil is then progressively introduced on a container including aggregates. They are mixed together during 2 min. No oil must stay in the bottom of the container.

Linseed Oil Emulsion

Linseed oil emulsion is an emulsion of water in oil with volumetric ratio oil / water equal to 4 (set on literature basis [Khazma 2014]).

Stabilisation of the emulsion is performed by addition of sugar ester emulsifier, with a recommended dosage included between 1 and 8%wt. This dosage was set to 5% in this study. Emulsifier is diluted in oil phase before addition of water.

Treatment is performed in the same way that pure linseed oil treatment.

Microscopic observations

Observations at microscopic scale were performed on a Carl Weiss optical microscope with an illuminator HXP 200 C and Delta Pix software.

FTIR Analysis

FTIR spectra of samples were obtained by measure of absorbance. This technique of analysis allows to determinate the level of polymerization of a linseed oil coating. According to Lazzari and al. [Lazzari 1999], some spectral changes (in relation with structural modifications) appear during the drying and are visible on FTIR spectra. It can be noted an almost complete disappearance of peaks at 3011 and 1654 cm^{-1} and a diminution of absorption at 723 cm^{-1}, attributed to isolated double bonds.

In addition, it appears a large strip around 3430 cm^{-1} linked to hydroxyl groups, a broadening of carbonyl absorption at 1800 cm^{-1}, a low absorption at 1633 cm^{-1} linked to formation of conjugated double bond, and other complex changes in regions of skeletal vibrations.

Tab.1: Porosity characteristics of hemp shives and wood shavings.

Materials	Total intrusion volume (cm^3/g)	Total pore area (m^2/g)	Median pore diameter (µm)	Porosity (accessible) (%)	Source
Hemp	2.415 ± 0.104	57.61 ± 7.44	0.510 ± 0.056	76.67 ± 2.03	[Jiang 2017]*
Wood	1.531 ± 0.170	9.57 ± 2.04	6.063 ± 2.370	67.54 ± 3.05	Author work**

*Average of 4 measures **Average of 7 measures

Dosage of total sugars

Dosage of total sugars is an important parameter for the evaluation of treatment efficiency in terms of extractives retention. Indeed, an efficacy coating must be able to retain a high quantity of sugars for limiting their diffusion in the matrix. For that, total sugars content in the samples is determined by the Dubois Method [Dubois 1956].

Compatibility with cementitious matrix

Studies show that a good quality of the interface between the vegetal part and the binder is required to obtain good mechanical performances of the composite. That's why it appears really important to make a compatibility test with cementitious matrix. Tests were made according to those of Diquélou and al. [Diquélou 2012] on hemp. Samples are partially sunk in a standardized cementitious paste.

When a vegetal aggregate does not present any problem of compatibility with cementitious matrix, a continuity appears between surface of the aggregate and paste. No ring is visible (Fig. 1 case (b)). Otherwise, i.e. when vegetal aggregate is treated by a coating with low efficiency in relation to retention of substances like sugars for example, an Interfacial Transition Zone (ITZ) appears, visible with a ring around aggregate (Fig. 1 case (a)). In a general way, mechanical performances of a composite are limited by mechanical strength of the ITZ, and fracture occurs at the interface aggregate / paste, and not in paste. This test of compatibility with a cementitious matrix is a good indicator to predict the behaviour of treated aggregates in concrete.

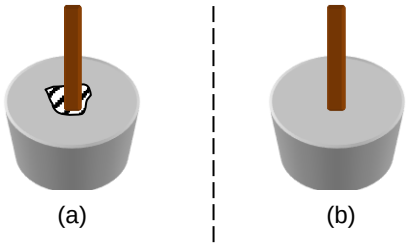


Fig. 1: Results of a compatibility test with a cementitious paste for a non-compatible (case a) and a compatible aggregate (case b).

3 PRELIMINARY STUDY

In order to set LO / A or DLO / A mass ratios, a preliminary study was conducted on wood. Several pure and emulsion linseed oil based treatments were tested with a ratio LO / A or DLO / A between 0.5 and 1.5, and a curing time under ambient laboratory conditions (± 23°C / 50%RH) between 7 and 28 days. Fig. 2 presents results obtained for the case with pure linseed oil. The increase of oil quantity used for the treatment leads to a decrease of the water uptake of wood shavings, and so to a better efficiency of the treatment. Nevertheless, a threshold value appears for a ratio near to 1.5, with a decrease of absorption rate near to 90%.

These results, alone, seem encouraging. However, a ratio equal to 1.5 leads experimentally to an excess of oil in recipients of treatment and to a slower polymerization of coatings. More precisely, it can be noted during tests that a ratio higher than 1 inevitably generates an excess of unpolymerized oil which stays later in water absorption, distorting the water uptake calculation. Similar results are obtained for emulsion based treatment.

Incorporating these treated wood shavings in a cementitious matrix, the unpolymerized oil provocates a delay of the concrete hardening, minimizing de facto mechanical properties of the final product. Tests have shown that the addition of 10 g of pure oil in a normalized mortar mix (so an addition of 0.49% wt) reduces compressive strength of prismatic samples 4x4x16 cm to 84%, and flexural strength to 16% after 7 days of curing under laboratory conditions (± 23°C / 50%RH).

This series of test runs allowed to show that samples treated with a ratio LO / A or DLO / A higher than 1 were still oily after 28 days of curing under ambient laboratory conditions. In all cases, water used for absorption test was yellow. It was very oily for ratios equal to 1.25 and 1.5. The colour of treated wood shavings was darker and darker with the increase of the ratio LO / A or DLO / A. Absorption results highlight the low difference of water uptake between samples dried during 15 and 28 days. It can be concluded that absorption at 15 days is almost the same that absorption after 28 days of curing.

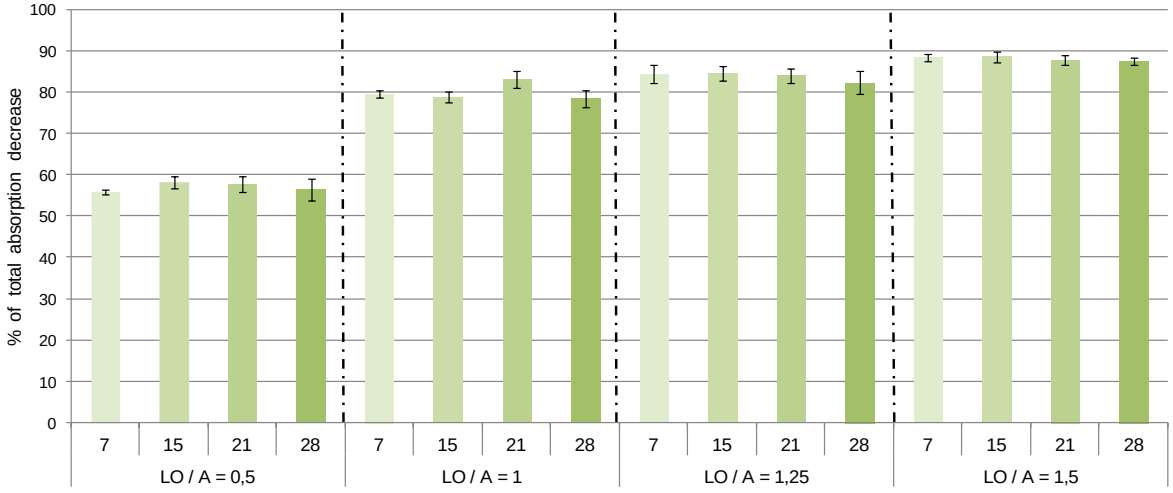


Fig. 2: % of total absorption decrease as a function of ratio LO / A (0.5, 1, 1.25 and 1.5) and time of curing (7, 15, 21 and 28 days) in comparison to untreated wood shavings (for pure linseed oil treatments).*

*Absorption taking into account recalculated dried weight of wood before immersion in water

Treatments tested in this work are presented in Fig. 3.

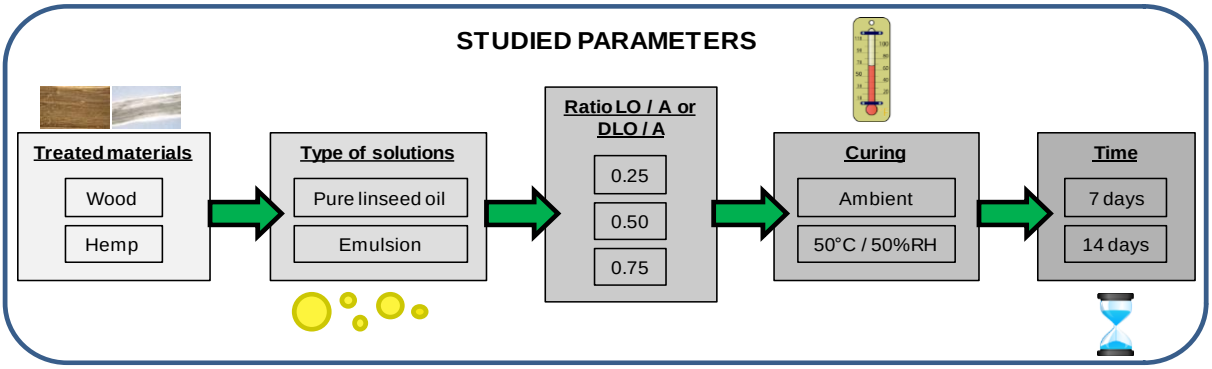


Fig. 3: Key parameters for this study.

According to results of this preliminary study, maximum ratio LO / A or DLO / A was set to 0.75, testing 0.25, 0.5 and 0.75. Moreover, time of curing was arbitrarily set to 15 days. It could be justified by the fact that coatings requiring times of curing longer are not really relevant.

In this work, the ambient curing corresponds to a dry environment away from rain and wind, with temperatures between 2 and 10°C. Hygrometry was not constant but equal to 50%RH or higher, depending on climatic conditions.

4 RESULTS AND DISCUSSION

Efficacy of treatments is evaluated in terms of:

- Water uptake of aggregates
- Mass evolution as a function of curing time
- Oil polymerisation
- Rate of total sugars leaching
- Presence or not of free oil in absorption water
- Compatibility with cementitious matrix

These criteria can help to predict the behaviour of treated bio-sourced aggregates in concrete. However, it appears clearly determining to include in the approach a part for studying durability of the treatment and compatibility between products used and vegetable materials (long-term degradation of shavings by products used, fungal resistance,...). These parameters are not mentioned in this study.

4.1 Water uptake of treated aggregates

Fig. 4 presents results of absorption tests on treated aggregates after 14 days of curing, knowing that the same trend is obtained at 7 days. In a general way, absorption decreases with increase of ratio LO / A or DLO / A. Moreover, samples treated by emulsion based coatings always present a higher absorption rate.

Results on hemp allow to conclude that linseed oil treatment (pure or emulsion) coupled with a curing at 50°C / 50%RH during at least 14 days leads to a lower absorption rate. In the case of pure linseed oil coating, with ratio LO / A equal to 0.75, the total absorption of samples are reduced by 53%wt for an ambient curing, against 62%wt for a curing at 50°C / 50%RH.

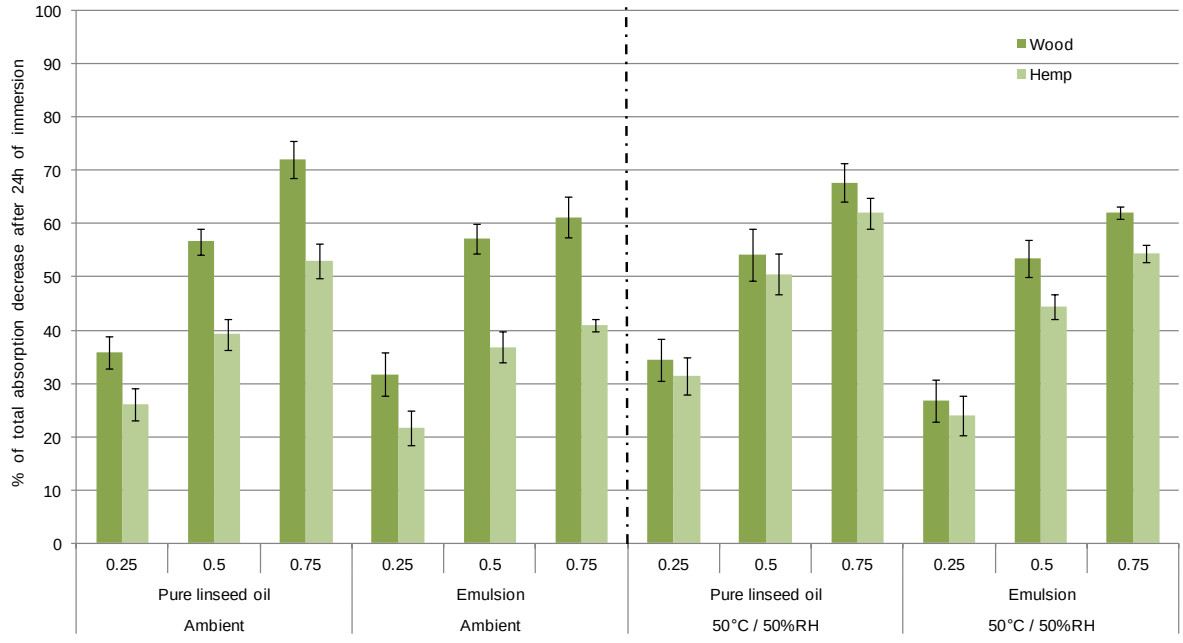


Fig. 4: % of total absorption decrease after immersion of 24h in water of wood and hemp shavings treated with linseed oil (pure or in emulsion) and after 14 days of curing, in comparison with untreated materials.

A gain of 10% of supplementary absorption reduction is obtained with treated hemp at 50°C / 50%RH. On wood, it can't be noticed any significant difference of absorption between samples dried under ambient conditions and those dried at 50°C / 50%RH after 14 days of curing.

Overall, treated hemp samples have a higher water absorption rate than wood shavings, but the difference of efficiency of water uptake between these two materials for a same treatment is more important under ambient curing that at 50°C / 50%RH.

4.2 Mass evolution

Fig. 5 and Fig. 6 present respectively the curves of mass evolution for pure linseed oil and emulsion treated hemp. Repeatability of results is also evaluated through the monitoring of mass evolution of 4 containers (for one same treatment). It must be noted that, for each type of treatments, sample in container 1 is partially used after 7 days of curing, and container 2 after 14 days of curing (for analysis). That's why the drawing of corresponding curves is stopped before the other curves.

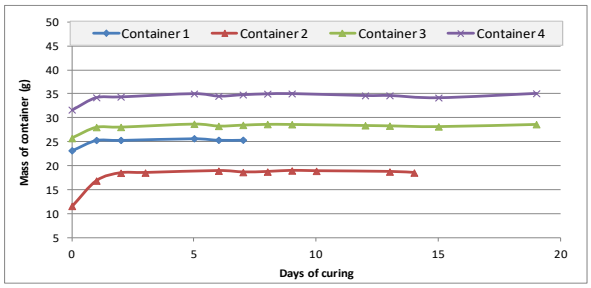


Fig. 5: Mass evolution of hemp treated with pure linseed oil (LO / A = 0.75) as a function of time of curing at 50°C / 50%RH.

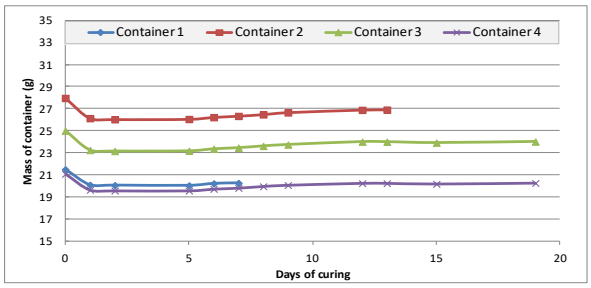


Fig. 6: Mass evolution of hemp treated with emulsion (DLO / A = 0.75) as a function of time of curing at 50°C / 50%RH.

Mass evolutions of treated samples are in agreement with literature. Indeed, Lazko and al. [Lazko 2011] explain that polymerization occurs in two steps, on condition of choosing the good curing. Firstly, treated material undergoes a step of maturation in which water evaporates. Then, a second step of maturation takes place and polymerization begins. Thus, for treated flax shives with a ratio LO / A equal to 0.07, 0.14 and 0.28, and dried at 50°C in an oven (so, RH < 10%), all of the process to obtain a complete polymerization lasts 20 days. This time is similar to the one found by Khazma [Khazma 2014] for 2 mm-thick layers of linseed oil spread on a non-absorbent smooth surface and placed in an oven at 50°C (21 days for pure linseed oil, 9 days for emulsion with volume ratio oil / water equal to 4).

On hemp, Fig. 5 shows that step 1 does not take place because there is no water to evaporate. However, in Fig. 6, the two steps are present: there is a decrease of mass of containers in the first days of curing, and then an increase of mass related to polymerization phenomenon, until a mass stabilization. The different curves obtained show that there is no evolution of mass beyond two weeks of curing.

This study, performed in the same way on wood, allows to notice the same trend in the two cases (pure linseed oil and emulsion). Concerning the impact of curing, it can be noted that this behaviour is different for emulsion when samples are dried under ambient conditions. Indeed, the second step of maturation is absent, which it can be explained by the fact that conditions are not enough favourable towards evaporation of water.

4.3 Optical microscope

Microscopic observations of untreated wood (Fig. 7 case (a)) show the presence of resinous canals. This structure is the same whatever the side of aggregates analyzed. Conversely, macroscopic observations of untreated hemp highlight a significant difference of structure between external and internal sides of hemp. This is confirmed at microscopic scale, with a honeycomb in the core hemp wood, and a plane and rough structure in the side of plant epidermis (Fig. 7 cases (c) and (e)). This difference has an high impact on water uptake, which is logically higher in alveolar part.

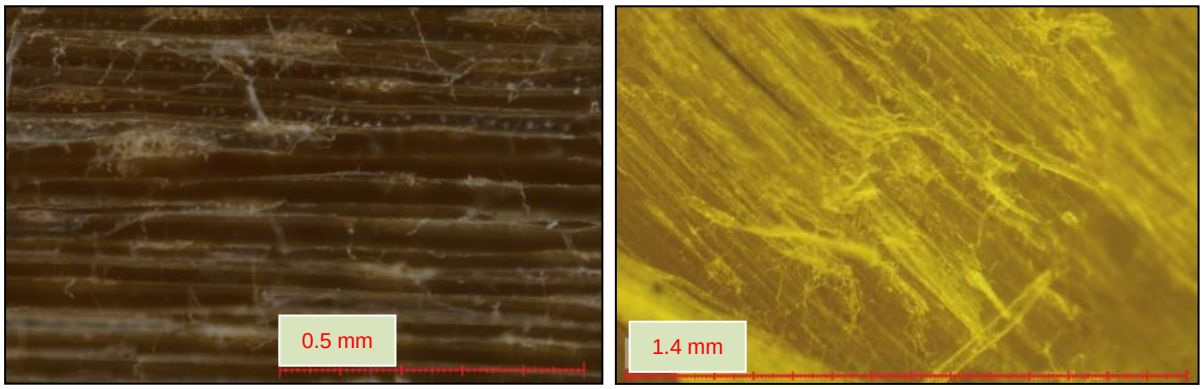
As it can be noted in observations (b) and (d), the same linseed oil treatment does not lead to the same result. Polymerization of oil on wood generates the formation of a polymeric network which looks like a spider's web. This phenomenon is observed on emulsion treated wood too (with curing at 50°C / 50%RH). Observations on treated hemp do not reveal any polymeric network but colour of samples shows that shives are well coated. Moreover, whether it be for pure linseed oil or emulsion, oil does not enter in the honeycomb structure.

4.4 FTIR analysis

Fig. 8 presents some results of FTIR analysis for wood and hemp treated by linseed oil (pure or in emulsion) for a ratio LO / A or DLO / A equal to 0.5 and after 14 days of curing. Thanks to this data, it's possible to compare IR spectra of the two sides of a same sample, and two different samples which have been coated by same treatment. Generally, it appears that aggregates are covered similarly in a same container.

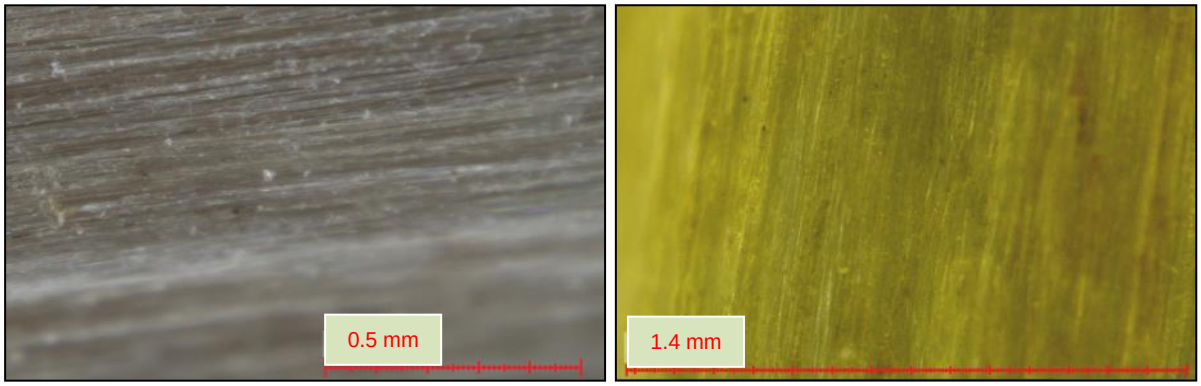
It can be observed that IR spectra of wood dried under ambient conditions presents several peaks characteristics of the presence of unpolymerized oil, at 1160 cm^{-1} a peak related to ester groups of linseed oil, cellulose and hemicelluloses, at 1746 cm^{-1} a peak corresponding to triglycerides of linseed oil and wax at surface of vegetable material, at 2850 and 2920 cm^{-1}, peaks related to elongation (C-H)(CH_2) of wax and natural fats, and at 3011 cm^{-1}, a peak linked to elongation (C-H) of the cis-double bond (=CH) of crude linseed oil. These peaks are also clearly visible on the first sample of treated hemp (on internal side), but not present on external side and on the second sample (two sides).

Partial or total disappearance of the peaks at 1746, 2850, 2920 and 3011 cm^{-1} on IR spectra of samples dried in an oven at 50°C shows that these conditions of curing are more in favour of oil polymerization. For identical parameters, there is no significant difference of IR spectra between wood and hemp, whatever ratio LO / A or DLO / A chosen.



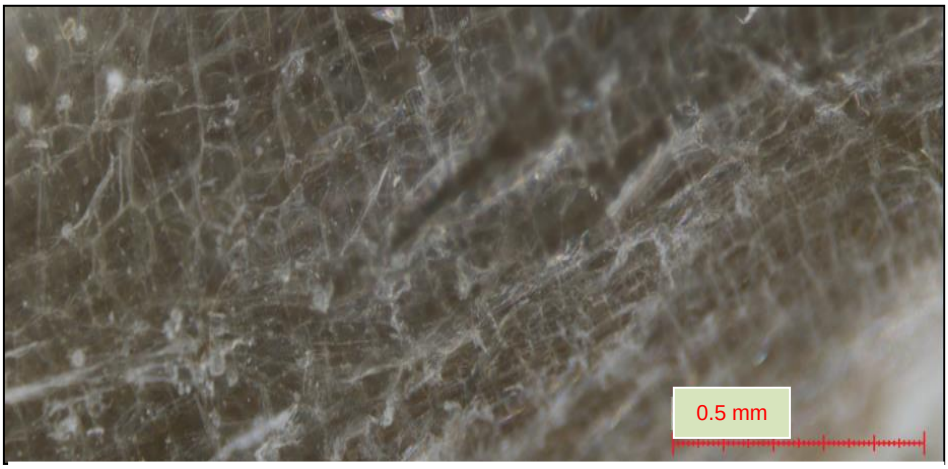
(a) Untreated wood – M x 80

(b) Treated wood with pure linseed oil – M x 80
LO / A = 0.50 – Curing at 50°C / 50%RH



(c) Untreated hemp (external side) – M x 50

(d) Treated hemp with pure linseed oil – M x 80
(external side)
LO / A = 0.50 – Curing at 50°C / 50%RH



(e) Untreated hemp (internal side) – M x 63

Fig. 7: Microscopic observations of untreated and treated materials.

M : magnification used to make observation

4.5 Dosage of total sugars

Leaching of sugars in cementitious paste is a phenomenon which considerably limits the use of bio-sourced materials as wood or hemp in construction sector. It represents a key parameter to validate or not a treatment. Two cases are taken into account: an ambient curing (case 1) and a 50°C / 50%RH curing (case 2). Interpretation of data is difficult to interpret due to a large variation of results. This can be explained by, on one hand, an incomplete polymerization of oil, and on the other, a lake of homogeneity of coatings leading to a discontinuous covering of aggregates. In a general way, total sugars for wood materials are at least twice as total sugars for hemp.

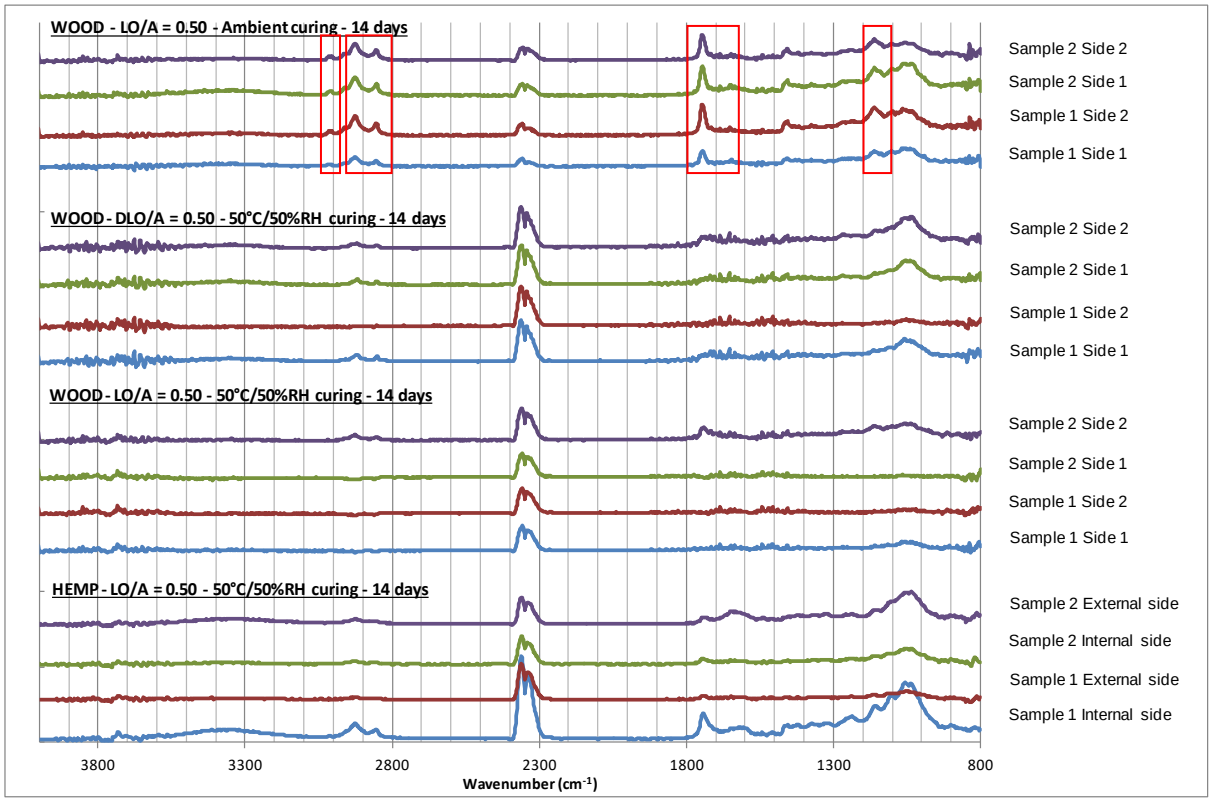


Fig. 8: FTIR spectra of linseed oil treated materials for a ratio LO / A or DLO / A = 0.5 and 14 days of curing.

The increase of ratio LO / A or DLO / A decreases the total sugars content in leachates, as well as the increase of time of curing. It can also be highlighted that total sugars content for emulsion is slightly higher than the equivalent for pure linseed oil.

4.6 Oil in water absorption

In this work, a colorimetric dosage by addition of lye (KOH) (and phenolphthalein as colour indicator) was performed on water used for absorption test. It results that there is not enough oil in samples to obtain a clear change of colour. Finally, the presence of oil in water absorption was visually evaluated. Only one sample seems to leach a large quantity of oil in water: wood treated with pure linseed oil with a ratio LO / A equal to 0.75 and a drying under ambient conditions (during 7 and 14 days). It would be false to conclude that other treated samples do not lose a part of oil during absorption test, but this quantity is lower.

4.7 Compatibility with cementitious matrix

Treated samples are partially sunk in a standardized cementitious paste. After 3 days of curing under laboratory conditions, the surface is observed at macroscopic scale. Two different behaviours can be highlighted. For wood, all of the samples (including untreated wood) dried under ambient conditions present a ring around them (Fig. 9). No ring is visible when coated wood shavings are dried at 50°C / 50%RH during 14 days (a ring appears when time of curing is reduces at 7 days). For hemp, no samples present rings (untreated hemp included).

Nevertheless, visually, a gap around particles is visible in all the cases, sign that it does not exist continuity between the vegetal part and the cementitious matrix. Results are confirmed by a simple test which consists in trying to remove the sample from the matrix, only pull it out with hand force. No sample has resisted.

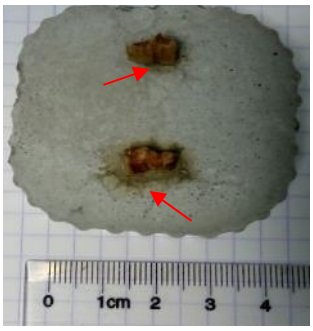


Fig. 9: Example of compatibility test result for untreated wood

5 CONCLUSION AND PERSPECTIVES

Absorption tests on treated aggregates show that the increase of ratio (LO or DLO / A) leads to a decrease of water uptake of wood and hemp. Nevertheless, an excess of oil is prejudicial because a part of oil goes to water absorption (and in cementitious paste if aggregates are put in this type of matrix). Results of absorption can be interesting but calculations would be wrong. The type of curing does not change the maximal absorption rate of treated aggregates. Better results are obtained for pure linseed oil treatments, that's can be explained by the fact that water can be degrade siccativity of oil, and coating could be discontinuous after drying.

Analyses performed highlight that it would be better to limit ratio LO / A to 0.50 for wood. However, behaviour of treated hemp in water does not show any clear limit between 0.25 and 0.75 (no excess of oil). A ratio higher than 0.75 or 1 could be tested. Furthermore, 50°C / 50%RH represents the better conditions tested here to obtain a polymerization of oil, but coatings could

be even more effective if hygrometry is reduced to 10% (in an oven for example).

In a general point of view, it would be useful to work on a method to well cover aggregates keeping in mind that ratio LO or DLO / A must be lower as possible to facilitate the drying and polymerization of the coating. Particles shapes of wood and hemp are important to well understand why a conventional mix with low quantity of oil does not totally perform well. It could be interesting to test the same procedure used in this article adding a second mix with oil after 14 days of curing to enhance homogeneity of aggregates.

Compatibility tests with a cementitious paste allow to conclude that linseed oil coating modifies surface roughness of aggregates, generally transforming a rough surface in a smooth surface. Consequently, treated particles are easy to remove from cementitious part.

Intrinsic properties have a significant effect on efficacy of oil treatments. Manufacturers of linseed oil recommend washing surface to treat with Marseille soap or hot water containing soda crystals to remove fatty substances, and to sand the surface to open the pores. Moreover, coating adheres better on smooth surface. Characteristics of materials treated in this study do not respect any of these recommendations. Nevertheless, opened porosity accessible to mercury (on Autopore Mercury Porosimeter) of hemp shives is higher than those for wood shavings (77 vs 67%) (See Tab.1) and median pore diameter is almost twelve times lower for hemp, in comparison to wood. In other terms, hemp presents more porosity but opened pores are generally smaller than those of wood. It's perhaps easier to seal in surface pores of hemp with oil coating than to seal pores with bigger diameter. Moreover, external surface of hemp shives is smooth, absorbs less than internal surface and is easier to cover.

If the environmental impact of linseed oil treatments is beneficial and some of results are interesting, this study shows that it is necessary to work on adhesion of vegetal part with mineral matrix.

6 ACKNOWLEDGMENT

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