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Effect of flax fibers treatments on the rheological and the mechanical behavior of a cement composite

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HIGHLIGHTS

- Effect of flax fiber treatments on its properties.
- Effect of flax fibers treatments on the rheological properties of cement mortar incorporating this reinforcement.
- Consequences of flax fibers treatments on the mechanical strengths of the cementitious composites studied.

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ABSTRACT

The association of plant fibers with cement paste raises a number of problems in both fresh and hardened states. Different treatments are applied to flax fibers with various methods within the aim to enhance the rheological properties of the cement mortar–fiber mixtures. Thus, the effects of these treatments on the properties of the fibers are evaluated. The rheological characteristics of the cement mixtures and the mechanical properties of the composites are assessed. Results show an improvement in the properties of the fibers, better rheological behavior of the cement mixtures and an increase of the mechanical strength of the composites.

1. Introduction

Research is increasingly interested in studying the possibility of using plant fibers as reinforcement for cement matrix, due to their high specific mechanical properties, their natural character and their low environmental impact in production. However, the incompatibility between plant fibers and cement matrix generates different problems. Indeed, on account of their hydrophilic character [1,2], plant fibers absorb large quantities of the mixing water and consequently the consistency of the cementitious mixture is greatly reduced [3]. Moreover, the dissolution of extractables from the fiber's surface in the cement mixture disturbs the hydration of the cement and delays the initial setting time of the composite [4,5]. Several studies have discussed the incompatibility issues between plant fibers and cement matrix and have suggested different treatments to overcome these problems. Khazma et al. [6] have evaluated the effect of coated flax shives with pectin/polyethylenimin mixtures on their properties and on the properties of the cement composite incorporating these particles. They have noted a decrease in their water absorption rate and in their initial setting time as the cement hydration enthalpy is increased. That correlates with an amelioration of their mechanical behavior and a great reduction in dimensional variations. In another study, Tonoli et al. [7] have assessed the effect of the surface modification of eucalyptus kraft pulp on the properties of cement composite reinforced with these fibers. The surface modification was made with different chemical substances (methacryloxypropyltri-methoxysilane (MPTS), aminoproplytri-ethoxysilane (APTS) and an aliphatic isocyanate (AI)). Results show that only MPTS-treated fibers decreased the water retention and improved the fiber-cement stability.

In this study, an attempt is made to improve rheological and mechanical properties of a cement mortar incorporating treated flax fibers. The treatments are intended to clean the fiber from the surface impurities and greases, to protect them against water and to minimize migration of extractables from fiber surface into the cementitious matrix. The efficiency of these treatments will be assessed through the measurement of the water absorption by the fibers, the identification of chemical groups existing on raw

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and treated fibers and through the measurement of the consistency and the initial setting time of the cementitious mixtures. The consequences of these treatments on the flexural and the compressive strengths of the cement composite will be also evaluated.

2. Materials and methods

2.1. Materials and fiber treatments

The cement used in this study is an ordinary Portland cement CEM I 52.5 N in accordance with European standard NF EN 196-1 [8]. The sand is a 0/4 dried and rolled one in accordance with the French standard XP P 18-545 [9]. Tap water is used.

Flax fibers (2.5 cm long) cultivated in Normandy are provided by the Vandecandelaere Company of the Depestele Group.

The first treatment is a water repellent chemical substance called Rheomac Deco Oleo which is dedicated to protect concretes from water, oil and other pollutions without modifying their surface appearance. This patented product is made from fluorinated copolymers in aqueous phase [10]. In this study, it is used to lower water absorption by the fibers and then ameliorate the consistency of the cement mixture. Fibers are immerged in the substance for two hours, and then dried at ambient temperature for four hours. Fibers obtained are called RHF.

The hydrothermal treatment consists in cleaning the fibers with boiling water for five minutes, then they are rinsed with water in order to remove the released extractives. The clean fibers are then dried at ambient temperature for two days. The obtained fibers are called BF.

In the last treatment the flax fibers are coated with CEM I 52.5 R cement grout prepared with mass ratio given by the formulation (1) [11]. The obtained fibers are called CF.

$$\begin{cases} \frac{\text{Water}}{\text{Cement}} = 1 \\ \frac{\text{Fibers}}{\text{Cement}} = \frac{2}{3} \end{cases} \tag{1}$$

The aim of this treatment is to limit water absorption by a shielding effect against water. This mineral substance is chosen due to its ability to resist in an alkaline environment.

Standard prismatic with size $4 \times 4 \times 16\ \text{cm}^3$ of control mortar (without fiber) and mortars incorporating raw or treated fibers are prepared. The formulation of the control mortar is that of standardized mortar, composites are made by using the same formulation but 2 vol.% of sand substituted fibers. They are demolded after two days to be sure the final setting time is reached, and cured in controlled condition at 20 ± 2 °C and 50% RH until mechanical tests after 7, 14 or 28 days. This RH value is a compromise because we cannot let the samples submerged in water due to the presence of flax fibers.

2.2. Water absorption test of flax fibers

The determination of the water saturation rate of the fibers is investigated in accordance with the method proposed by Magniont [12]. It consists in drying 5 specimens of approximately 1 g of flax fibers in an oven at 60 °C until constant weight, and then immersing for 5, 15, 30 min and 24 h in water. After each immersion time, the specimens are superficially dried with paper towels and their masses are measured. The water saturation rate of the fibers is then calculated with the formulation (2):

$$\%\ \text{Absorption} = \frac{\text{saturated mass} - \text{dried mass}}{\text{dried mass}} \times 100 \tag{2}$$

2.3. Real density of the fibers

The measurement of the real density of raw and treated fibers is assessed using a helium pycnometer (Micromeritics AccuPyc 1130).

2.4. Tensile testing single fibers

To evaluate the effect of the treatments (except the cement coating) on the mechanical properties of flax fibers, tensile tests are implemented with the method described by Charlet [13] using a universal mechanical testing apparatus (Instron 5566) equipped with a 10 N capacity load cell, at a crosshead displacement rate of 1 mm/min. 22 valid tests are considered for each type of fibers (RF, RHF, BF). The test was not conducted on CF because the presence of the cement layer modifies significantly the stiffness of the fiber and may affect the evaluation of the ultimate stress.

2.5. FTIR spectroscopy

The FTIR spectroscopy analysis is performed to investigate the changes in the chemical nature of the compounds on the surface of the fibers due to the applied treatments. The apparatus used is a Spectrum One FT-IR (Perkin Elmer) assisted by a computer. A total of 10 scans were acquired at a resolution of $4\ \text{cm}^{-1}$ with a scanning range between $4000\ \text{cm}^{-1}$ and $650\ \text{cm}^{-1}$.

2.6. Consistency of cement composites

The measurement of the consistency of the control mortar called CM, the reference composite (composite with raw fibers) called RFM, the composite with Rheomac-treated fibers RHFM, boiled-treated fibers mortar BFM and the cement coated fibers mortar CFM is assessed with a consistometer in accordance with NF EN 12350-3 standard [14]. This method is chosen because it is suitable for concrete and mortar of poor workability whose maximum aggregate size does not exceed 63 mm [14], which is the case of our fiber–cement mixtures. However, it was also conducted on the control mortar in order to compare it to other formulations. Consistency is defined as the time required for the underside of the transparent disc of the device to be completely covered with mortar (Vebe time). For a valid measurement, the time should be between 5 s and 30 s. Otherwise, another apparatus should be used.

2.7. Initial setting time

The initial setting time of the different formulations is determined with a Vicat apparatus in accordance with the French standard NF P 15-431 [15]. The method consists in the measurement of the time necessary for a plunger assembly with total weight of 1000 g to penetrate into the material. The initial setting time is defined as the time passed since the contact of the water with the cement to the time when the plunger in the mixture is at 2.5 mm from the base-plate.

2.8. Compression and three point bending tests

The mechanical properties of the different mortars are determined by implementing compression and three point bending tests in accordance with NF EN 196-1 standard [16] using a mechanical testing machine (Instron 5566) equipped with a 50 kN capacity load cell with a loading rate of 50 N/s.

3. Results and discussion

3.1. Real density of raw and treated fibers

The real density of raw and treated fibers is presented in Table 1. Compared to the raw fiber, the lower density (5% reduction) of the boiled fiber may be explained by the removal of the extractives and volume reduction after boiling (see Table 2). At the contrary, an increase in density is noted for the Rheomac treated fiber (4%) and the cement coated fiber (27%).

3.2. Tensile testing

Mechanical properties of RF, BF and RHF are gathered in Table 2. Both treatments result in a reduction of the scattering of the tensile strength values. The diameter reduction for BF can be explained by the removal of the extractables from the fiber surface.

3.3. Water absorption of raw and treated fibers

The water intake of the treated fibers must be investigated as this is an important issue for the consistency of the mixtures. Besides, the absorbed water is released by the fibers during the

Table 1
Real density of raw and treated fibers.

Fibers	Real density
RF	1.54
BF	1.46
RHF	1.6
CF	1.95