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Optimizing the formulation of flax fiber-reinforced cement composites

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HIGHLIGHTS

- Behavior of composites with fiber treatment and/or adhesion additive is analyzed.
- Treatments affecting rheology of fibered mortars are outlined and discussed.
- The efficiency of adhesion additive is observed.

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ABSTRACT

Because of their hydrophilic nature, flax fibers absorb water during the mixing stage and release it gradually during the curing step. Thus, the rheological properties and the setting of the cement paste are deeply disturbed. In addition, the diametrical shrinkage of the fiber in the desorption stage can lead to a weakening of the fiber–matrix bond. It is therefore necessary to carry out treatment of the fiber and/or cement mixture to prevent the fiber/matrix debonding. The results obtained from these formulations show an improvement in the rheology and the mechanical performances of the composites.

1. Introduction

The presence of flax fibers in a cement matrix disturbs the rheological behavior of the cement in the fresh state and its setting. Indeed, the non-cellulosic polysaccharides compounds (estimated at 5–15% of the dry mass of the fibers depending on the flax variety [1,2]), perturb the hydration of the cement and delay the initial setting time. Besides, fibers absorb an important quantity of the mixing water (up to 200% of their mass) [3], which makes the composite extremely dry and decreases its workability. In the hardened state, fibers improve the flexural resistance of the composite until maturity. Water absorption by the fibers and the alkaline attack by the cement matrix lead to the reduction of their performances over time [4,5]. Several other phenomena can take place within the composite in its hardened state, which could reduce the adhesion between the fibers and the matrix [6]. Indeed, the hydrophilic fibers absorb a large amount of water and start releasing it from the start of mixing, and even during the curing stage. It follows a diametrical shrinkage of the fiber, which results in fiber–matrix debonding. Surface treatments are then necessary to prevent the fibers from these dimensional changes. In this context, Sellami et al. [7] have evaluated the effect of two treatments of diss fibers on the mechanical properties of a mortar incorporating these fibers. The first treatment consists of extracting sugars by distillation in water; they noted an improvement in flexural strength compared to the composite with raw fibers. The second treatment is made by waterproofing the fibers with flax oil; this treatment reduced significantly the diss fiber absorption rate and improved the flexural strength of the cement composite. In another study, Claramunt et al. [8] have assessed the effect of the hornification of the vegetable fibers on the mechanical performance and durability of softwood kraft pulp and cotton linters cement mortar composites. The hornification of the fibers consists in four drying and rewetting cycles (mechanical treatment in water) and a filtration of the pulp suspension. Mortars reinforced with these fibers have shown better mechanical performances in flexural and compressive tests.

Chakraborty et al. [9] have modified the chemical composition and the surface properties of jute fibers for their homogeneous distribution into the cement matrix by the combination of NaOH and polymer emulsion treatment. They have then evaluated the effect of this fibers modification on their durability in alkaline cement environment and on the physical and mechanical properties of a

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cement mortar. They have noted the amelioration of the compressive strength, the modulus of rupture and the flexural toughness compared to the control mortar. They have also investigated the durability of treated and raw jute fibers by exposing them in alkaline cementitious environment for 7–360 days and then quantifying their tensile strength retention after 360 days. They have reported 93% tensile strength retention for the treated fibers against 74% for the raw fibers.

In this work the effects of NaOH fiber treatment and the addition of a latex adhesion promoter to the cement mixture are investigated in terms of rheological behavior and mechanical performances of flax fiber-reinforced cement composites. All formulations were characterized in the fresh state through workability and setting time tests. The mechanical properties were assessed in the hardened state by implementing three point bending tests. The physical and mechanical properties of flax fibers used in this study were also determined.

2. Materials

The flax plants used in this study were harvested in Normandy in 2011. The raw fibers were cut in the desired length by a professional cutter, and then were stored at ambient conditions. Table 1 presents their physical and mechanical properties measured from 22 samples.

The cement used was ordinary Portland cement CEM I 52.5 in accordance with European standard NF EN 196-1 [10]. In accordance with the French standard XP P 18-545 [11], dried 0/4 rolled sand was used in the mixture. Tap water was used for the preparation of the mixtures. Total mixing water had been adjusted for all composites to consider for the water absorption by the fibers. Standard prismatic specimens of 40 × 40 × 160 mm in size were used for the mechanical tests in the hardened state. They were demolded after 48 h and they were kept right after in wet cured condition at 20 ± 2 °C and 50% H_R until mechanical testing.

The sodium hydroxide solution of 6 wt.% was used for the fibers treatment. It is chosen in order to clean the fibers by removing the impurities from their surfaces (grease, wax, etc.), which makes them rougher [12] and able to better adhere to the matrix. Besides, it would eliminate the amorphous substances from their surfaces which would increase their degree of crystallinity [13,14].

The latex-based adhesion promoter provided by BASF "BARRALATEX 30T", known for its great adhesive power, is a chemical milky solution composed with highly active synthetic compounds designed for mortars, micro-concretes and coatings. In this study it is tentatively used to improve the workability of the mixture due to its plasticizing effect, but also with the aim to improve the adhesion between the fibers and the matrix due to its adhesive ability.

3. Set up and procedure

3.1. Tensile test

Tensile tests were conducted on 22 samples of ultimate fibers, using the protocol described by Tung et al. [15]. The test was performed with an Instron 5566 equipped with a 10 N capacity load, at a crosshead displacement rate of 1 mm/min (Fig. 1).

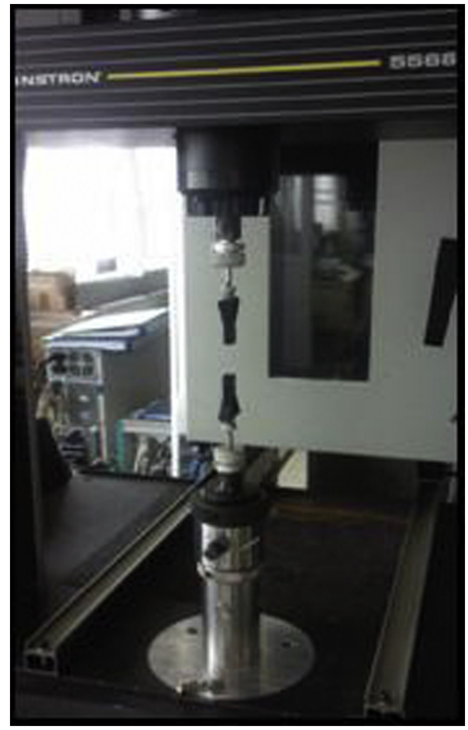

Fig. 1. Loading device for the tensile test.

3.2. Measurement of the density of the flax fibers

The measurement of flax fiber density was conducted using a helium pycnometer; three samples have been tested.

3.3. Treatments and mortar preparation

3.3.1. Treatment of flax fibers with the sodium hydroxide

2.5 cm long fibers were immerged for 48 h in 6% by weight NaOH solution, then rinsed with 1% concentrated acetic acid and with distilled water until chemical neutrality of the rinsing water, verified by successive measurements of the pH using a pH meter. The NaOH-treated fibers were dried for 48 h in an oven and an amount of 2% by volume was added to the mortar as partial replacement of the sand. The obtained composite material is referenced [SHF], while the mortar with raw fibers (the reference composite) is [RF] and the control mortar (without fiber) is [C].

3.3.2. Use of "BARRALATEX 30T" adhesion promoter

The BARRALATEX 30T was added for the preparation of the mortar with raw fibers 2.5 cm long by replacing 1/3 of the mixing water.

3.4. Setting time and workability of the cement composites

The workability of all the different formulations was assessed in accordance with the French Standard NF P 18-452 [16]. The recommended device allows measuring the time necessary for a defined quantity of mixture to cross a reference line while flowing under a specified vibration sequence.

The initial setting time was also measured for each composite. The principle of this test is 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 effective when the distance between the end of plunger and the base-plate is 2.5 mm in accordance with the French Standard NF P 15-431 [17].

3.5. Flexural tests

Three point bending tests were carried out on an Instron 5566 machine equipped with a 50 kN capacity load cell in accordance with Standard NF EN 196-1 [18]. The loading rate was 50 N/s.

Table 1
Physical and mechanical properties of the flax fibers.

Diameter (μm)	Density	Young's modulus (GPa)	Ultimate strain (%)	Tensile strength (MPa)
22.75 ± 6	1.54	44 ± 21	1.78 ± 0.6	849 ± 482