Study of thermal and mechanical properties of cement-based composites reinforced with vegetal sponge wastes and silica fume

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Abstract

This paper examines the thermal and mechanical properties of vegetal fiber cement based composites. These composites contain cement, sand, silica fume and a waste of industry composed of vegetal sponge containing cellulosic fibers. The objective was to obtain good mechanical properties combined with thermal insulation. The addition of vegetal sponge lowers the density (from 2092 to 1171 kg.m^{-3}), the thermal conductivity (from 1.78 to 0.47 W.m^{-1}.K^{-1}) and compressive strength (from 73 MPa to 4MPa). Scanning Electron Microscopy observations and density measurements were also carried out.

Keywords: Lightweight concrete, compressive and flexural strengths, thermal conductivity and diffusivity, silica fume, vegetal sponge.

1. Introduction

As 40% of the global energy consumption and 56.7% of carbon dioxide emissions are relative to the building sector, the development of new insulating concretes based on biomaterials remains an innovative subject [1]. Biomaterials like vegetal fibers are biosourced and biodegradable, they are renewable resources which are not participating to the emission of carbon dioxide [2; 3]. A wide range of vegetal fibers prepared by different pulping methods were studied in various matrix systems (cement, mortars, etc.) [2, 4]. In France, hemp concrete has been developed but the degradation of mechanical properties due to aging in humid conditions may limit its use [5]. In specific conditions, some micro-organisms alter the hemp shiv microstructure leading to a modification of the porosity of concrete. This induces a decrease of the toughness and post-cracking strength of the cement [2]. Moreover, some constituents of vegetal fibers are very sensitive to the alkaline environment of cement which causes the reduction of the composite durability [6].

In this context the addition of silica fume to any vegetable fiber based concrete appears as a good alternative. Indeed silica fume reduces the concentration of hydroxyl ion in the pore solution producing a less aggressive environment for the cellulose fibers [4]. Silica fume is composed of spherical particles of silicon dioxide (SiO_2) with an average diameter of 140 nm. These particles are 100 times smaller than the cement ones. In the presence of hydrating Portland cement, silica fume will react as any finely divided amorphous silica-rich constituent in the presence of CH- the calcium ion combines with the silica to form calcium-silicate hydrate through the pozzolanic reaction. A well-crystallized form of CSH-I can be formed [7]. Thus, a concrete with a compressive strength as high as 138 MPa has been obtained using the silica fume. Large pores are reduced and continuous pores are transformed to discontinuous pores which modify the permeability of silica-fume concretes. The increase of the bonds between particles and the modification of the porosity lead to the reinforcement of

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the concrete improving its compressive and flexural strength [8]. The incorporation of silica fume also leads to remarkable rheological characteristics of concrete and significantly reduces the permeability and chemical degradation reactions (carbonatation, alkali-reaction, sulfatic reactions). Consequently the incorporation of silica fume gets the triple advantage of a better processability, better mechanical properties and resistance to ageing.

2. Material and methods

The Vegetable Fiber High Performance Concrete (VFHPC) samples were obtained by mixing :

- **Cement :** CEM I 42.5 MPa R Portland cement
- **Silica fume :** Elkem Microsilica Grade 971-U Norway
- **Sand** : SIBELCO France CV32.
- **Vegetal sponge:** an industrial waste of vegetal sponge has been chosen. This industrial cellulosic paste has been obtained from a mixture of flax fibers, cotton fibers, wood pulp, … The material has been crushed in small particles of around 5 mm in diameter or less as visible in figure 1b.

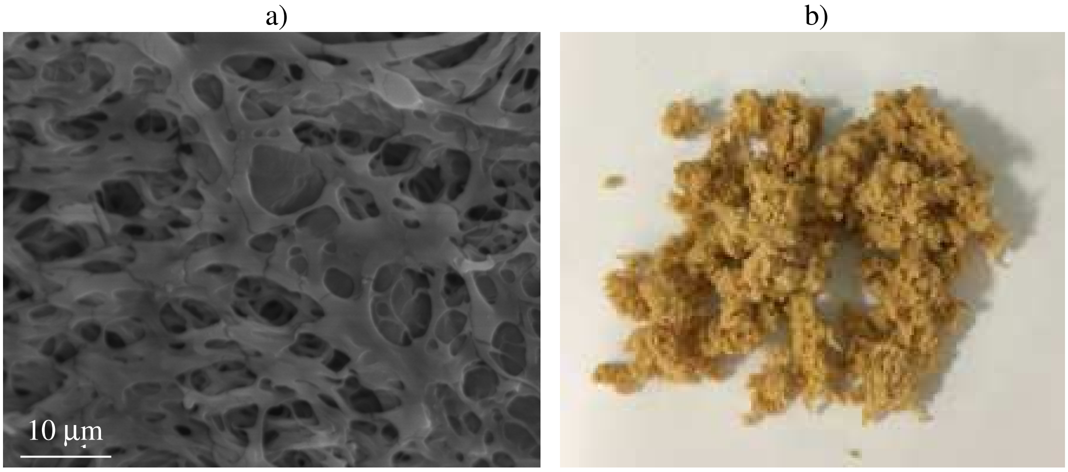

Figure 1 : *a) Observation by SEM of vegetal sponge*
b) Photography of the crushed vegetal sponge incorporated to cement

The concrete was formulated by choosing a cement content, a determined sand dosage and a constant W/C ratio. Vegetable fibers have a mass percentage instead of sand of 0%, 5%, 10%, 15% and 20%. The composition of the different samples is given in Table 1. The water / cement ratio (W/C) is 0.5, the Cement / Sand ratio (C/S) is fixed at 0.6 and the silica fume / cement ratio (SF/C) 0.2. The Vegetable Fiber High Performance Concrete (VFHPC) samples were named as a function of the sand substitution percentage, for example when 5% of sand mass is substituted by vegetal fibers the obtained concrete is named VFHPC5.

sample	Cement	Sand	Vegetal Fiber	Silica fume	Fiber weight fraction
	(g)	(g)	(g)	(g)	(%)
Concrete	500	1000	0	100	0
VFHPC5	500	950	50	100	3.1
VFHPC8	500	920	80	100	5
VFHPC10	500	900	100	100	6.3
VFHPC15	500	850	150	100	9.4
VFHPC20	500	800	200	100	12.5

Table 1 : *Composition of the Vegetable Fiber High Performance Concrete (VFHPC)*

Protocol:

- The fibers were dried 24 hours at 40 °C and then rehydrated with 1/3 of water before being incorporated into the mixture. They should not be incorporated dry as they would absorb the water necessary for the hydration reaction of the cement.
- The cement, the silica fume and the sand were mixed in order to well homogenize the mixture, then 2/3 of the water was added.
- The wet fibers are then gradually incorporated into the mixture while mixing.
- The concrete is then poured in stainless steel molds for 4*4*16 cm^3 test-pieces.
- The 3 samples were vibrated with a specific system coupled with the mold as visible in figure 2a and pressed under 3 bars as visible in figure 2b. The vibration was done thanks to compressed air inducing the rotation of a ball in a semi-circular cavity. A pseudo-circular wave in a parallel plan at the mold base (horizontal) allows vibrating the concrete in the horizontal sense. A vibration in the vertical plane would favorite the cement stratification. The vibration frequency is around 500 – 1000 Hz. This process of vibration and pressure is close to the production press industrial process. Vibrations are used in order to avoid air bubbles and the compression to favor the cohesion between the different constituents.
- Samples were kept one day in the mold covered with a plastic film, dried in air at ambient temperature and then placed in a storage room (T°=20°C, RH=50%) during 5 months.

Figure 2 : *a. Samples of concrete molded for mechanical tests in stainless steel molds with a vibration system, b. Samples in the mold on the hydraulic press.*

1.1. Methods

Samples were observed using a scanning electron microscope JEOL 6301 F. The density of the samples was obtained by weighing the samples and measuring their dimensions. Bending tests on 4x4x16 cm^3 molded test piece and compressive strength on half-prism after bending test were performed according to NF EN 196-1. Thermal properties were determined using the transient plane source method "Hot Disk" with the TPS 2500 apparatus, using the bulk (type I) standard isotropic measurement module. This method uses a plane probe sensor composed of a nickel foil (with a double spiral pattern) placed between two layers of Kapton sandwiched between two samples. The sensor is used as a heating source and a temperature sensor at the same time, as its electrical resistance is measured (by using a Wheatstone bridge) over the time. The thermal conductivity and diffusivity are then identified using a thermal model. The probe was chosen according to the thickness of the sample (6.403 mm and 3.189 mm radius probe on samples with an average thickness of 10 mm). The average values of thermal conductivity and diffusivity were calculated for six performed measurements on 2 samples turned up. The uncertainties of thermal conductivity and diffusivity where evaluated from standard deviations of the 6 experiments (using reproducibility conditions). Heat capacity C_p has been calculated using density ρ, thermal conductivity k and diffusivity a

($C_p = k / (\rho \times a)$). C_p combined standard measurement uncertainty is then computed by propagating standard measurement uncertainties on ρ, k and a according to the GUM method.

3. Results and Discussion

3.1. SEM observations

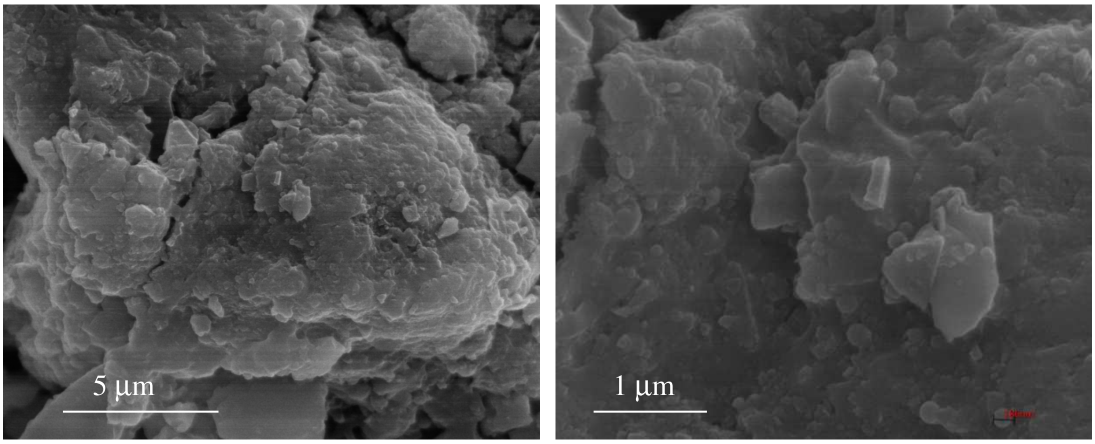

Figure 3 : *SEM micrographs of concrete containing sand and silica fume.*

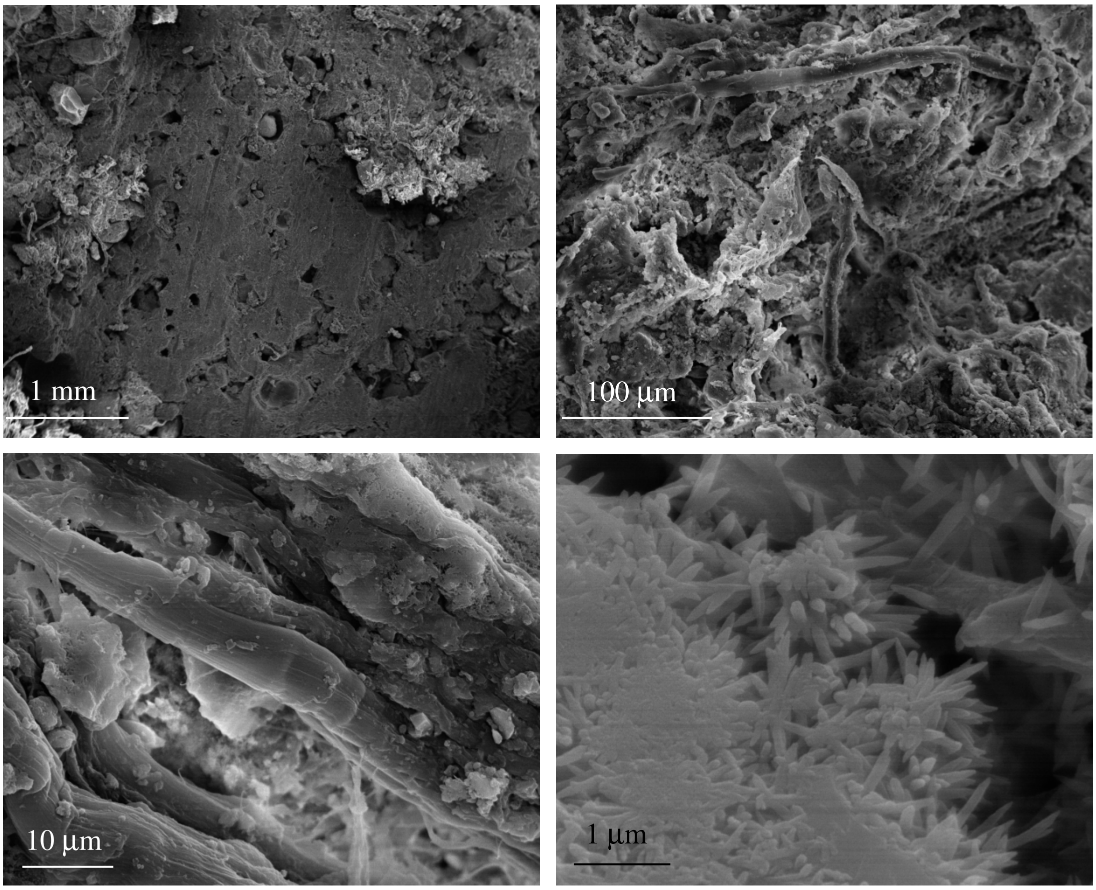

Figure 4 : *SEM micrographs of vegetal fibers concrete containing around 5 % of fibers and silica fume at different magnifications.*

Scanning Electron Microscopy (SEM) observations are presented in figures 3, 4 and 5. In figure 3 the micrographs of high performance cement containing silica fume show a very dense material. The addition of silica fume induces a very dense microstructure compared to sand cement without silica fume [9]. Silica fume increases the bond strength between cement paste and aggregate by making interfacial zone denser. The silica fume induces a less porous structure in the interfacial zone thus the cement is more resistant to the penetration of aggressive agents [9]. The Scanning Electronic Microscopy observation of vegetal fibers concrete containing around 5 % of vegetal sponge at different magnifications is presented in figure 4. The pieces of sponge of approximatively 1 mm in diameter are visible in the first image. They are surrounded by cement. On the adjacent image, at the scale of 100 µm, long vegetal fibers are visible after a zoom in the piece of sponge. Lowering the scale to 10 µm allows seeing the presence of crystals of CSH. These crystals are more visible at the scale of 1 µm on the last image of figure 4.

3.2. Density

The variation of density with the mass fraction of fibers is presented in figure 6. It is observed that the density decreases clearly when the mass fraction of fibers increases. The density is halved when the mass fraction of fibers is 12.5 %.

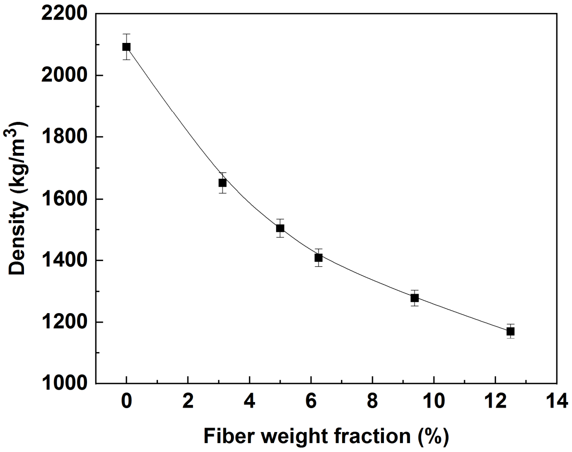

Figure 6 : *Evolution of density as a function of fiber weight fraction*

Here the density of the concrete containing silica fume is 2092 kg.m^{-3}. The addition of vegetal fibers decreases the density until 1171 kg.m^{-3} for a fiber mass fraction of 12.5%. Thus all the samples may be considered as lightweight concrete. Indeed, lightweight concrete has a density lower than 1840 kg/m^3 compared to normal weight concrete with a density in the range of 2240 et 2400 kg/m^3 [10]. The addition of vegetal fibers decreases the density of the vegetal fiber composite because the density of vegetal fibers is much lower than the density of cement. Moreover as vegetal fibers are forming a porous material in vegetal sponge their incorporation in cement leads to a lightweight concrete.

3.3. Thermal conductivity and diffusivity

The evolution of thermal conductivity of concrete as a function of the fibers mass fraction has been plotted in Figures 7. Thermal conductivity decreases with the fiber mass fraction. This decrease is expected because vegetal fibers have lower thermal conductivity than concrete. Such a decrease has ever been observed by different authors [12, 13]. The thermal conductivity goes from 1.785 ± 0.036 W.m^{-1}.K^{-1} for the cementitious matrix to 0.476 ± 0.010 W.m^{-1}.K^{-1} for the composite containing 12.5% by mass of fibers, which corresponds to a reduction to around ¼ of the initial value. Moreover, the addition of fibers in the matrix creates numerous interfaces which act thermally as successive contact resistances and lead to a decrease in thermal conductivity [13]. Part of these interfaces is filled by CSH formed by silica fume.

In most publications, the addition of silica fume decreases thermal conductivity and thermal diffusivity and increases the specific heat [14, 15]. For example the thermal conductivity of cements goes from 0.52 $W.m^{-1}.K^{-1}$ to 0.40 $W.m^{-1}.K^{-1}$ when 15% by weight of cement is replaced by silica fume [14]. Thermal conductivity of cement paste (Portland type I+ water) is 0.53 $W.m^{-1}.K^{-1}$ and thermal conductivity of mortar (cement + sand + water) is 0.58 $W.m^{-1}.K^{-1}$ [14]. A thermal conductivity of 0.46 $W.m^{-1}.K^{-1}$ was obtained for example for sugar cane bagasse fibers reinforced cement composites containing 3% of fibers with respect to Portland cement [1]. A thermal conductivity of 0.19 $W.m^{-1}.K^{-1}$ and 0.175 $W.m^{-1}.K^{-1}$ has been obtained for concrete obtained with cement, sand and coconut or durian fibers wastes (30% Fibers/cement), the corresponding compressive strength being respectively 1.81 MPa and 1.97 MPa [12].

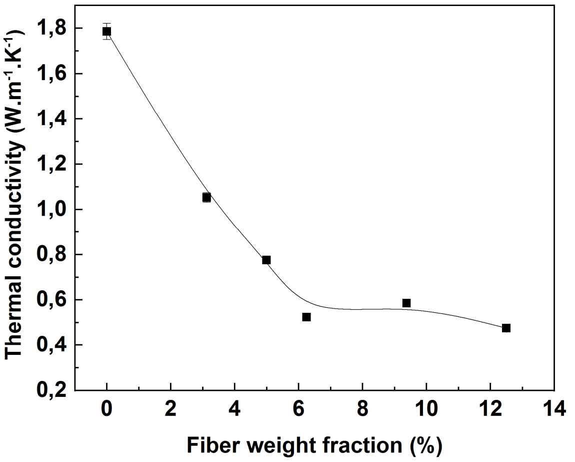

Figure 7 : *Thermal conductivity as a function of fiber weight fraction*

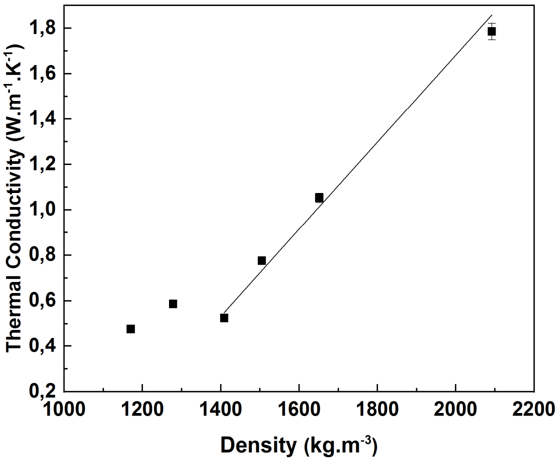

Figure 8: *Variation of the thermal conductivity as a function of density*

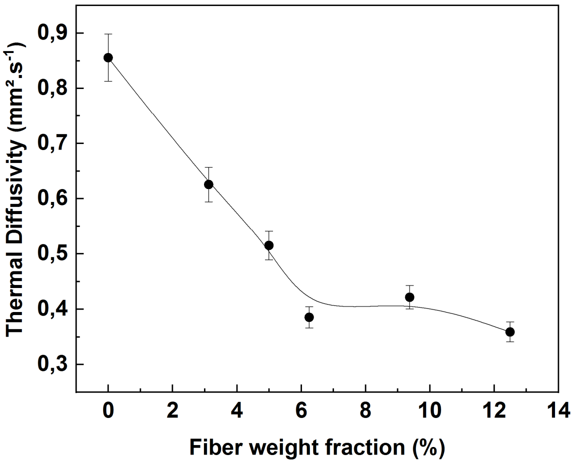

Figure 9 : *Thermal diffusivity as a function of fiber weight fraction*

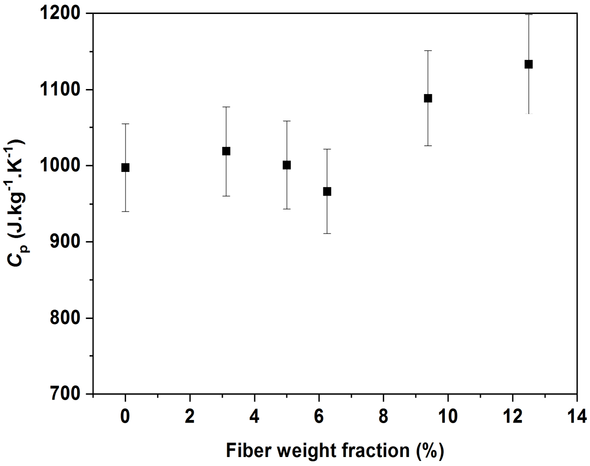

Figure 10: *Heat Capacity as a function of fiber weight fraction*

In other respects, the plotting of the relative variations in thermal conductivity as a function of density shows the existence of a correlation between these properties for fiber weight fractions lower than 6.3% (cf. figure 8). This result confirms those already observed in previous works for other types of natural fibers [1; 11]. Thermal conductivity of concrete is also related to its apparent density [15]. A linear decrease is also observed when replacing the cement with 10 to 30% silica fume, the thermal conductivity varying from 0.32 to 0.28 $W.m^{-1}.K^{-1}$ [15]. These properties are related to porosity, the addition of vegetal fibers can rise the number of voids particularly at the interface between vegetal fibers and cement [16]. At the same time, the addition of silica fume reinforces interfaces. In the case of sponge, and more particularly for high fiber weight fraction the penetration of cement paste inside the porous sponge may be difficult.

The evolutions of thermal diffusivity and heat capacity of concrete as a function of the fibers mass fraction has been plotted in Figures 9 and 10. Like thermal conductivity, thermal diffusivity decreases with the mass fraction of fibers (see figure 9). The diffusivity of the reference concrete is 0.855 ± 0.043 $mm^2.s^{-1}$, the minimum value is 0.359 ± 0.018 $mm^2.s^{-1}$. Heat capacity remains stable until 6.3% of fibers, then it increases slightly.

3.4. Mechanical properties

The mechanical properties in compression and in flexion are presented in figure 10. The mechanical resistance in compression is 72.8 MPa for the sample not containing fibers and drops to 32 MPa as soon as 3% by mass of fibers are incorporated. We conclude that adding fibers to concrete weakens our material, however the mechanical resistance in compression is still acceptable for samples not exceeding 6.3 % of fibers because it is greater than the limit value of 25 MPa necessary to make load-bearing materials (beams, walls, etc.). Above this percentage, the mechanical module in compression is close to the limit value of 4 MPa necessary to produce insulating materials for filling the wall of individual houses. A stability of the thermal and mechanical properties for higher fiber weight fractions than 6.3% is observed. SEM observations showed that this comes from the low quantity of cement which cannot recover all vegetal sponge pieces.

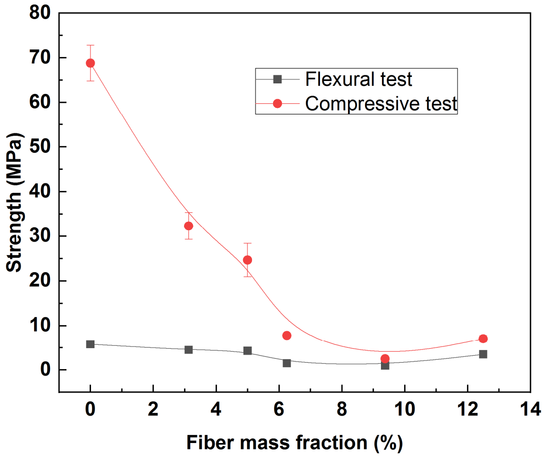

Figure 10 : *Evolution of flexural and compressive strengths as a function of fiber mass fraction.*

4. Conclusion

This study showed that vegetal sponge wastes can be reused in order to obtain insulating lightweight concrete. The thermal characterizations allowed verifying that the addition of porous vegetable fibers well reduces the thermal conductivity and diffusivity of the fiber-reinforced concretes. Thermal conductivity passes from 1.785 $W.m^{-1}.K^{-1}$ to 0.476 $Wm^{-1}.K^{-1}$. The addition of vegetal fibers decreases the density of the samples and therefore provides certain lightness to the material. The density drops from 2092 $kg.m^3$ to 1171 $kg.m^3$. Nevertheless, the integration of the fibers reduces the compressive strength, for samples containing no more than 6.25% of fibers; the compressive strength remains greater than 25 MPa which allows producing load-bearing materials (beams, walls, etc.). For samples containing up to 12.5% of fibers, the compressive strength is close to 4 MPa, so it may be used as an insulating material for filling walls. For a compressive strength of approximately 25 MPa, a thermal conductivity of 0.75 $W.m^{-1}.K^{-1}$ is obtained. The use of vegetal sponge wastes in concrete is therefore an attractive alternative in order to minimize the environmental impact by reducing energy consumption in buildings using biosourced industrial wastes.

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