EFFECTS OF ADDING WASTE CELLULOSIC FIBERS ON THE FROST RESISTANCE OF SCC

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Abstract
The aim of this study is to evaluate frost durability of self-compacting concrete (SCC) and cellulose based fibred self compacting concrete (FSCC). The two materials were prepared with cement content of 350 kg/m³, water/powder ratio of 0.35 and cellulose based vegetable fiber content of 21% by volume of cement for the fibred one. At this fiber content it was found that the mechanical properties enhancement of FSCC is accompanied by a decrease in the porosity and the intrinsic permeability compared to SCC.

The performance of two concretes exposed to freezing-thawing cycles was assessed from length change, mechanical properties, resonance frequency and permeability measurements of the test specimens before and after cycles. The results show that the two materials are not frost resistant and that FSCC is more sensitive to frost action than SCC in spite of its better mechanical properties. This low resistance is attributed to the low water transport properties which prevent water movement under driven hydraulic pressures and to the poor pore size distribution. Moreover the role of used aggregates in the frost resistance was also discussed.

Keywords: SCC, cellulose based fiber, Frost action, mechanical properties, permeability, and pore size distribution.

1. INTRODUCTION
The objective of this work is to present the influence of introducing vegetable based microfibers, resulting from paperboard recycling, on the frost durability of SCC. In previous papers [1,2], we have provided answers on its optimal dosage in a cementitious material and the methodology for the mix design of microfibers reinforced SCC. From theses previous studies an optimal microfibers content of 21% by binder volume was found.
This manuscript focuses on the frost durability of vegetable based microfibred self compacting concrete, FSCC, compared to non fibred one SCC. First, the physical and mechanical properties of two concretes are exposed and the difference between their characteristics is emphasized. In the second part, the influence of repeated freezing-thawing cycles on both materials is presented. Finally, the obtained results are interpreted in terms of microstructure properties and used aggregates properties.

2. MATERIALS AND EXPERIMENTAL PROGRAM

2.1 Raw materials

CEM I CALCIA cement 52.5 N CE CP2 NF, manufactured in France and certified in conformity with the recommendations of standard EN 197-2 was used with a natural river sand (0.125-4 mm) and crushed gravels (4-16 mm). The bulk densities of sand and gravel were equal to 2.55 and 2.51 respectively. Gravels are composed of 65% silico-calcareous aggregates and 35% siliceous aggregates. The chosen limestone fillers contain primarily carbonate of natural calcium manufactured by the French group OMYA SAS. Its bulk density is about 2.7 and the Blaine surface was equal to 500 m²/kg. Finally, the used superplasticizer was Cimfluid 2002 produced by Axim Italcementi group with solid contents of 35%. Micro-cellulose based fibers were provided by OMYA SAS. They were resulting from the recycling of resinous origin cardboard. The micro-cellulose based fibers contains about 80% of cellulose with a density $\rho = 30$ kg/m³, a mean length of about 1.1 mm, 0.045 mm mean diameter and a factor of form of 24.4 (factor of form = length/diameter).

2.2 Mix design of SCC and FSCC

Two self compacted concretes were formulated without air entrainment agents based on following requirements: class of environmental exposure XF2 according to NF EN 206-1 [3] and slump flow $D_{\text{mean}} = 68 \pm 2$ cm according to AFGC recommendations [4]. SCC formulation is based also on the maximum packing theory for the determination of solid particles content and the method of concrete equivalent mortar, CEM, for superplasticizer dosage [3]. Starting from the SCC of reference composition, vegetable based microfibers were introduced at a content of 21% by volume of cement. This content were found in a previous work to be the optimum [1,2]. Water absorbed by the microfibers is estimated about 670% by mass and has been taken into account in the total mix water. To compensate this surplus, an additional quantity of filler was added to maintain constant water/binder ratio. The mix proportion of both concretes is recapitulated in table 1.

<table>
<thead>
<tr>
<th>Constituent kg/m³</th>
<th>SCC</th>
<th>FSCC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement</td>
<td>350</td>
<td>350</td>
</tr>
<tr>
<td>Filler</td>
<td>116</td>
<td>168</td>
</tr>
<tr>
<td>Water</td>
<td>164</td>
<td>168</td>
</tr>
<tr>
<td>Sand 0/4</td>
<td>948</td>
<td>948</td>
</tr>
<tr>
<td>Gravel 4/10</td>
<td>758</td>
<td>758</td>
</tr>
<tr>
<td>microfibers</td>
<td>-</td>
<td>0.7</td>
</tr>
<tr>
<td>Superplasticizer (% of binder)</td>
<td>1,1</td>
<td>1,1</td>
</tr>
</tbody>
</table>
2.3 Experimental program

Cylindrical 16x32 cm$^2$ and prismatic 10x10x40 cm$^3$ specimens were prepared in order to follow, compressive and flexural strengths, elastic modulus and length change evolutions during freezing-thawing test. 24 hours after moulding, all specimens were preserved in water at room temperature for 28 days before submitting them to freezing-thawing test. Cycles of 12 hours duration were imposed according to the French standard NF 18-425 [3] and Rilem recommendations [5]. The freezing-thawing cycles were carried out without water contribution so each specimen was covered by a plastic film to avoid evaporation. Starting from +10 °C the temperature was lowered in 3 hours with a cooling rate of 10 °C/h down to -20 °C, and kept constant during 3 hours at this temperature. Then it was increased during 2 hours to +10 °C with a heating rate of 15 °C/h and kept constant during 4 hours. These steps were programmed in order to stabilize the temperatures and to allow the liquid transfers to occur. The basic loop includes then two cycles per day [6].

Uniaxial compression test was performed using a servo-hydraulic INSTRON machine with a capacity of 3500 KN by imposing a stress increment rate of 0.5 MPa/s. The main objective of the uniaxial compression test was to investigate the compressive strength, the elastic modulus, and the complete behaviour law of the studied concretes. 3 points bending test was performed using the same machine by imposing a displacement increment rate of 1 mm/min and dynamic modulus of elasticity is determined using E-Meter MK II device.

Intrinsic permeability was measured using a constant head permeametre CEMBUREAU with oxygen as the percolating gas and finally thermal characteristics were measured with a Hot Disk TPS 1500 thermal conductivity system.

3. PHYSICAL AND MECHANICAL PROPERTIES OF SCC AND FSCC 21%

Pore size distribution of both studied concretes is characterized by mercury intrusion porosimetry (MIP). The results are depicted in figure 1 where it can be shown that SCC and FSCC are characterized by a narrow distribution of an average pore diameter ranged between 40-45 nm. Mercury and water porosities, intrinsic permeability and thermal properties of two concretes are given in table 2 where it can be shown that FSCC porosity is lower than that of SCC resulting in a lower intrinsic permeability.

Table 2. Physical and thermal properties of SCC and FSCC.

<table>
<thead>
<tr>
<th></th>
<th>SCC</th>
<th>FSCC</th>
</tr>
</thead>
<tbody>
<tr>
<td>MIP porosity %</td>
<td>12.4</td>
<td>11.5</td>
</tr>
<tr>
<td>Water porosity %</td>
<td>13.9</td>
<td>12.6</td>
</tr>
<tr>
<td>Intrinsic permeability (m$^2$)</td>
<td>0.4x10$^{-16}$</td>
<td>0.28 x10$^{-16}$</td>
</tr>
<tr>
<td>Thermal conductivity (W/mK)</td>
<td>2.53</td>
<td>2.55</td>
</tr>
<tr>
<td>Specific heat (J/kgK)</td>
<td>670</td>
<td>685</td>
</tr>
</tbody>
</table>
The results of mechanical tests are given in table 3. It can be seen that the 21% microfibers reinforcement improves mechanical characteristics by 20-25%. This enhancement is explained by the porosity reduction related to the microfibers introduction in the cementitious matrix.

Table 3. Mechanical properties of SCC and FSCC.

<table>
<thead>
<tr>
<th></th>
<th>SCC</th>
<th>FSCC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Static elastic modulus (GPa)</td>
<td>41</td>
<td>49</td>
</tr>
<tr>
<td>Dynamic elastic modulus (GPa)</td>
<td>41.5</td>
<td>52</td>
</tr>
<tr>
<td>Compressive strength (MPa)</td>
<td>60</td>
<td>70</td>
</tr>
<tr>
<td>Flexural strength (MPa)</td>
<td>6.3</td>
<td>7.8</td>
</tr>
</tbody>
</table>

4. FROST DAMAGE EVOLUTION DURING FREEZING-THAWING CYCLES

4.1 Influence of microfibers on length change during freezing-thawing cycles

The effect of adding microfibers on the length change of SCC is reported on figure 2 where a swelling for both SCC which increases with cycle’s number is observed. This swelling is more important for FSCC up to 120th cycle then it tends towards a threshold of 1000 µm / m. If we refer to the damage criterion defined by the standard P18-424, being 500 µm/m, the frost resistance is 40 cycles and the test may be interrupted.
Swelling of SCC is progressive during the first 100 cycles and then increases sharply between 100 and 180th cycle and never change thereafter. This deformation reached the damage criterion, 500 µm/m, around the 110th cycle.

Figure 2. Length change of SCC and FSCC during freezing-thawing cycles.

4.2 Resonance frequencies evolution during freezing-thawing test

Damage factor related to change in frequency of resonance and consequently the dynamic modulus of elasticity can be assessed by the relationship:

\[ D^F_n = 1 - \frac{F_n^2}{F_{n0}^2} \]  

With \( F_n \) the resonance frequency measured after \( N \) cycles and \( F_{n0} \) the resonance frequency of concrete before frost test. According to standard NF P18-425, the material is qualified non frost resistant if the damage factor reaches a value of 0.4 (loss of 40% of its initial value).

Damage factor calculation from the measured frequencies using equation 1 gives the results presented on figure 3. Obviously, FSCC is more frost sensitive than SCC because damage factor reaches the value 0.4 at the 70th cycle while it reaches this value at 110th cycle for SCC. Damage factor increase, related to the decrease of the dynamic modulus of elasticity is due to microcracking then macrocracking for subsequent cycles. This is mainly explained by the fact that the waves propagate less quickly in damaged media than in undamaged one.
4.3 Influence of cycles on mechanical properties

Frost action induces often irreversible modifications in the microstructure of the cementitious matrix. So, for the studied materials, the effect of repeated freezing-thawing cycles on the mechanical resistance was investigated. To quantify this effect, the relative variation of the mechanical strengths during the test was calculated. This variation can be defined by the expression:

\[ D^c_F = \frac{\mathcal{F} - \mathcal{F}^c}{\mathcal{F}} \]  

(2)

With \( \mathcal{F} \): the property related to the non damaged material, \( \mathcal{F}^c \): the same property after \( N \) freezing-thawing cycles and \( D^c_F \): the damage factor.

Figure 4 shows the evolution of the damage factor related to the compressive strength with the number of cycles. It can be pointed out that damage factor, \( D^c_F \), reaches a value of 0.4 after only 30 cycles for FSCC while SCC reaches this value after 150 cycles.

The evolution of damage factors related to elastic modulus and flexural strength showed the same trend where FSCC was more vulnerable than SCC.

4.3 Porosity evolution during-freezing thawing cycles

The effect of freeze-thaw cycles on the porous texture of both concretes was assessed by changes in water porosity. To overcome the initial state and to study the cinematic of porosity’s change. We depicted in figure 5 the change in relative porosity with cycle’s number. As shown in this figure, the porosity of FSCC is more affected by frost action than that of SCC. The increase in the porosity is the result of cracking and indicates a progressive pore network connection or cracks opening.
Figure 4. Evolution of damage factor related to compressive strength.

![Graph showing the evolution of damage factor related to compressive strength for SCC and FSCC.](image)

Figure 5. Relative porosity evolution during freezing-thawing cycles.

![Graph showing the relative porosity evolution for SCC and FSCC.](image)
5. **INTERPRETATION OF OBTAINED RESULTS**

FSCC has mechanical properties which are more important than SCC prepared with the same cement content and the same W/C ratio. Moreover, it is characterized by a lower permeability. This permeability’s decrease leads to an increase in the magnitude of hydraulic pressure generated during ice formation [7]. When water cannot move to the empty void to freeze, the pressure generated may exceed the tensile strength of the material and causes cracking.

The accelerated degradation of FSCC is related also to the hydrophilic nature of the microfibers. These can be considered as cavities filled with free water inside the material. Water in these cavities, being independent of the effect of geometric restrictions, freezes before pore water nearby, and the amount of formed ice at a given temperature is larger than that formed in the non-fibered concrete and consequently the generated hydraulic pressure is more important.

A part of the frost sensibility of two concretes is attributed to the presence of silico-calcareous aggregates. Visual analysis of damage showed that these aggregates are completely destroyed when the number of cycles is important. The poor frost resistance of these aggregates is explained by their high porosity and their poor abrasion resistance.

6. **CONCLUDING REMARKS**

Freezing-thawing test has unveiled the frost sensibility of microfibred self compacted concrete, FSCC . The harmful effect of microfibers on the frost durability is due to the decrease in the permeability of reinforced materials despite their beneficial role on the mechanical properties. Also, their hydrophilic accelerates the degradation process because they act like cavities filled with water.

From the last point of view, the use of microfibers could improve the fire resistance of concrete. Their ability of water absorption would reduce the pressures generated at high temperatures.

**REFERENCES**