DURABILITY AND MECHANICAL PROPERTIES OF NANOCOPOSITE FIBER REINFORCED CONCRETE

Durability and mechanical properties of nanocomposite fiber reinforced concrete

Bartolomeo Coppola, Paola Scarfato, Loredana Incarnato, Luciano Di Maio
Department of Industrial Engineering, University of Salerno, Fisciano (SA), Italy
ldimaio@unisa.it

Abstract

In this study we investigated the influence of polypropylene/organoclay fibers on durability and mechanical behaviour of concrete. Pure polypropylene fibers and polypropylene nanocomposite fibers of two different lengths (20 and 60 mm) have been mixed in concrete at two volume fractions (0.1% and 0.3%). Nanoclay addition increases fibers elastic modulus (about 27%) reducing ductility. Workability of concrete is greatly influenced by fibers length and volume fraction: increasing these two values workability decreases. Fibers are not influent on compressive and flexural strength while post-cracking toughness is increased. Nanocomposite fibers have a better pull-out strength due to a better friction during slipping, but this doesn't ensure a better adhesion. Water absorption, freeze/thaw cycles and the sulfate attack test demonstrate that concrete durability increases with the volume of the fiber fraction.

1. Introduction

Durability of structures is an important issue for construction industry but also for maintenance costs. Shrinkage of concrete represents a serious concern for structures durability due to concrete low tensile strength and thus cementitious materials are subjected to cracking during their life. One crack control method widely accepted by researchers, is the use of randomly distributed fibers, particularly fine synthetic fibers with a volume fraction below 0.5%. The composite material made of concrete and short fibers is named fiber-reinforced concrete (FRC). Several authors investigated the influence of fibers, and particularly of PP fibers, on durability and transport properties of concrete structures, particularly on permeability (Kakooei et al., 2012). Polypropylene is one of the most widely used synthetic polymers as it is easy to process, cheaper, has excellent chemical resistance and is stronger than other synthetic polymers. The addition of nanoclay as reinforcement in the PP matrix can further enhance the physical properties of synthetic fibers. Nevertheless, studies on PP/clay nanocomposite fibers are so far limited. Richardson et al. (2012) reported that polypropylene fibers effect is to reduce water absorption and increase freeze/thaw resistance, having an increase of material durability. Their researches show that fibers inclusion could increase void system, offering an alternative way to improve void content. As in any fiber reinforced composite, fiber-matrix bond in FRC is extremely important. Mechanical behaviour of fiber reinforced composites is greatly governed by adhesion that allows stress transmission between matrix and fibers. To increase FRCs toughness or energy absorption, fiber slip has to be avoided. Fiber matrix bond is investigated by pull-out tests that are able to describe fiber/matrix interface, changing test parameters, i.e. fiber inclining angle, embedded length, specimen geometry etc. (Kim et al., 2008; Bartos, 1981; Singh et al., 2004). Deterioration processes of concrete structures are due to several causes: corrosion of the reinforcement, sulfate attack, frost action and alkali aggregate reactions. Sulfate attack depends on environmental and exposure conditions, representing an important issue affecting concrete durability. Neville's review (2004) about sulfate attack point out the differences between the mere occurrence of chemical reactions of sulfates with hydrated cement paste and the damage or deterioration of concrete. Jianming et al. (2013) investigated durability of concrete exposed to sulfate attack and wetting/drying cycles assessing that these could increase structures deterioration. Concrete transport parameters are influenced by water content of the test specimens. The measurement of transport properties is sensitive to the test procedure and attention to preconditioning of the test specimens is necessary (RILEM TC 116, 1999). The aim of this paper is to study the influence of different fibers on concrete behavior. Particularly the research interest lies in the lack of studies on the addition of polypropylene nanocomposite fibers in cementitious composites.

2. Materials and methods

Concrete was prepared using a CEM IV/A 32.5 R (ITALCEMENTI S.p.A.) and natural aggregates deriving from limestone crushing. Characteristics of aggregates are listed in Table 1.
Two different fibers were used: pure polypropylene and nanoclay reinforced polypropylene. Both types have been produced in the laboratory of Polymer Technology of the Department of Industrial Engineering of University of Salerno. Fibers have been produced by a twin screw extruder (COLLIN ZK 25, L/D = 32) using a feeder for polypropylene granules (MOPLEN V79S) and a feeder for the commercial layered organoclay powder (DELLITE 43B). Polypropylene and nanoclay blend comes out from the extruder at the molten state, is cooled into water and wound by a winder (COLLIN TECH-LINE BAW 130 T). This extrusion process produces a filament that after has been cut in short fibers at two different lengths: 20 and 60 mm. Fibers morphology is described in Table 2.
Mechanical properties of fibers have been determined by a tensile test, according to ASTM C 1557-03. The test was carried out at two cross-head speed (10 and 50 mm/min) using a universal testing machine with a load cell of 1 kN. Gauge length is 40 mm.
Concrete production was made by a concrete mixer: cement and aggregates are mixed for 10 minutes, than water is gradually introduced to have a homogeneous mixture (UNI EN 206-1, 2014). Concrete mix composition is indicated in Table 3.
Throughout the work will be used the FRC classification listed in Table 4.
Concrete workability has been evaluated by slump test. Workability is synonymous of consistency, i.e. the facility of concrete to flow. The test was carried out using a mold known as a slump cone or Abrams cone (UNI EN 12350-2, 2009).

Table 1 – Characteristics of aggregates.

Aggregate	Diameter [mm]	Water absorption [%]	Density [g/cm³]	Chloride content [%]
Sand	0 - 4	0.9 ± 0.1	2.68	0.01 ± 0.02
Fine	4 - 8	0.2 ± 0.1	2.70	0.01 ± 0.02
Coarse	8 – 20	0.5 ± 0.1	2.71	0.01 ± 0.02

Table 2 – Description of different fibers.

Fiber designation	Material	Cross section	Length [mm]	$\Phi_{average}$ [mm]
PP20	pure polypropylene	circular	20	1.05
PP60	pure polypropylene	circular	60	
PPNC20	polypropylene nanocomposite	circular	20	1.20
PPNC60	polypropylene nanocomposite	circular	60	

Table 3 – Concrete mix composition.

Component	Dosage [kg/m³]
Cement CEM IV/A 32.5 R	434
Sand (0-4 mm)	594
Fine aggregate (4-8 mm)	443
Coarse aggregate (8-20 mm)	333
Water	217
Water/cement ratio	50 %

Table 4 – FRC classification.

Concrete designation	Fibers length [mm]	Fibers material	Fibers volume fraction [%]
Reference	-	-	-
FRC_PP20A	20	PP	0.1
FRC_PPNC20A	20	PP NC	0.1
FRC_PP60A	60	PP	0.1
FRC_PPNC60A	60	PP NC	0.1
FRC_PP20B	20	PP	0.3
FRC_PPNC20B	20	PP NC	0.3
FRC_PP60B	60	PP	0.3
FRC_PPNC60B	60	PP NC	0.3

Mechanical properties of FRCs have been determined by flexion and compression tests at 28 days. For determining flexural strength two beams 15x15x60 cm for each mix were tested, according to UNI EN 12390-5. Notched specimens (45 mm on the lower edge) were used. For the four point bending test, flexural strength is given by the equation:

$$f_{cf}= \frac{F \cdot L}{d_1 \cdot d_2^2} \qquad \text{(eq. 1)}$$

where f_{cf} is the flexural strength, F is the maximum load, l is the distance between the supporting rollers, d_1 and d_2 are the lateral dimensions of the specimen (Figure 1).

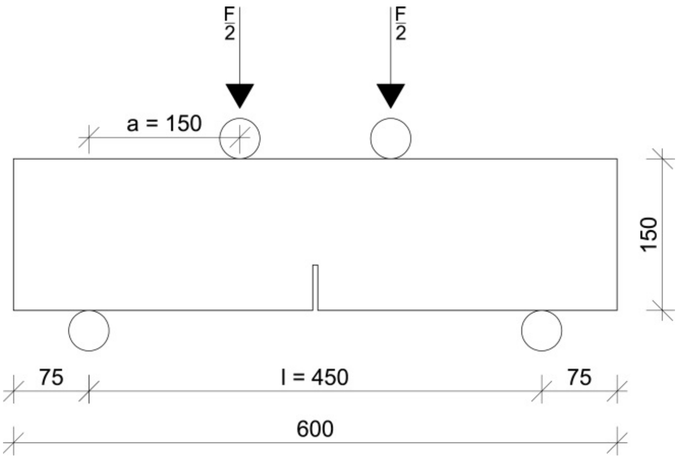


Figure 1 – Four-point bending test scheme.

Compressive test was carried out on three cubic specimens (15x15x15 cm) for each mix, according to UNI EN 12390-3. Load was applied by incremental increments, between 0.2 and 1.0 N/mm²s, until failure is reached. The maximum compressive load is registered and used for determining the compressive strength:

$$f_c= \frac{F}{A_c} \qquad \text{(eq. 2)}$$

where f_c is the compressive strength, F is the maximum load at failure and A_c is the cross-sectional area of the specimen on which the compressive force acts.

Durability of FRC was investigated by water absorption test, sulfate attack and freeze/thaw cycles, on prismatic specimens (7.50x7.50x15 cm) after 28 days curing. Rate of absorption, i.e. sorptivity, of water by FRC was evaluated according to UNI 9526. Specimens were dried inside an oven until constant mass is reached, after have been cooled at environmental temperature and then the weight was measured (M_0). Specimens were placed on supports at the bottom of a pan, filled of tap water so that the water level is 5 ± 1 mm. Water level was maintained constant during all the tests. Immediately after that the lower face on the specimen was immersed the timing started. The mass has been recorded after 1, 5, 10, 20, 60, 120, 240, 300 minutes and 24, 48 and 72 hours (M_i). The absorption I_i (eq. 3), at the time t_i, is the change in mass divided by the cross-sectional area of the test specimen (A):

$$I_i= \frac{(M_i-M_0)}{A} \qquad \text{(eq. 3)}$$

The sorptivity coefficient S_i is calculated as follows:

$$S_i= \frac{I_i}{\sqrt{t_i}} \qquad \text{(eq. 4)}$$

Water absorption for total immersion has been evaluated according to ASTM C 642-97. Specimens were totally immersed in water until the constant mass is achieved (mass variation less than 0.1% in 24 hours), M_1. Water absorption percentage (Ab) is evaluated as a percentage of the mass of the dry specimen M_0:

$$A_b= \frac{M_1-M_0}{M_0} \cdot 100 \qquad \text{(eq. 5)}$$

Standard ASTM C 88-05 defines a test method for testing aggregates, to estimate their soundness when subjected to weathering action in concrete or other application. FRC samples were cyclically immersed in a saturated solution of sodium sulfate (Sodium Sulfate Anhydrous, Na_2SO_4, provided by J.T. Baker) then dried in oven. Solution was prepared into a beaker on a hot plate magnetic stirrer device at a temperature between 100 and 110 °C. Before its use, the solution was maintained at room temperature for 48 hours, covering the beaker to avoid evaporation. Three samples for each mix were tested: specimens are immersed in the solution for 16 hours then are left on a desk for drain. After are dried in oven at 110 ± 5 °C for 8 hours, cured at room temperature and weighed, then immersed again in the solution. Nine cycles were performed.

Durability at freeze/thaw cycles was assessed according to UNI 7087. Three specimens for each mix were used: two are subjected to alternate freeze/thaw cycles and one is the reference sample. This specimen is immersed in tap water at 20 ± 2 °C during the testing period. Extent of deterioration is evaluated by the durability factor (eq. 7) that is the ratio between relative dynamic modulus of elasticity at the N cycle and the same property measured on the reference specimen. After 28 days curing the specimens are thermally stabilized in water at 5 ± 2 °C for 3 hours. Each cycle has two phases: cooling and maintaining at the lower limit of the cycle; warm-up and maintaining at the upper limit of the cycle. During the first phase specimens were kept in the freezer at -20 ± 2 °C for 2 h ± 10 min. After are placed in water at 5 ± 2 °C for at least 15 minutes then weighed. Generally a concrete is considered resistant at freeze/thaw cycles if after 300 cycles the relative dynamic modulus of elasticity is at least 80 % of the initial one. FRCs were submitted to 15 freezing/thawing cycles. The relative dynamic modulus of elasticity is measured by ultrasonic pulse method. Ultrasonic pulse velocity, V, and dynamic modulus of elasticity, E_d, are related by eq. 6:

$$E_d = \rho V^2 \frac{(1+\upsilon)(1-2\upsilon)}{(1-\upsilon)} \qquad \text{(eq. 6)}$$

where ρ is concrete density and υ dynamic Poisson's ratio (υ = 0.2).
Durability factor is given by:

$$DF = \frac{E_n}{E_i} \text{x100x} \frac{n}{300} \qquad \text{(eq. 7)}$$

where n are the cycles performed, E_n and E_i are dynamic modulus of elasticity of the samples undergone to freeze/thaw cycles and reference, respectively.

3. Results and discussion

3.1 Fibers tensile test

Elastic modulus, stress at yielding and tensile strength of fibers were determined by tensile tests and values are reported in Table 5.

Table 5 – Mechanical properties of fibers.

Fiber	E [MPa]	σ_y [MPa]	σ_b [MPa]	ε_b [%]
PP	785 ± 55	8 ± 2	22 ± 3	1500
PPNC	914 ± 9	11 ± 2	27 ± 2	1279 ± 83

Nanoclay addition leads to an increase of elastic modulus and tensile strength of 16% and 23% respectively. Nanocomposite fibers are slightly stiffer than pure polypropylene ones but are less ductile. Strain at failure of nanocomposite fibers is lower (-16%) than the maximum strain allowed at polypropylene fibers. Due to the limit of the testing machine, tensile tests for polypropylene fibers was arrested at a strain of 1500 % corresponding to an elongation of 600 mm.

3.2 Workability of fresh concrete

Workability of concrete was measured in terms of slump. Slump values of the different mixes are listed in Table 6. Increasing fibers volume fraction slump decreases, but a slightly higher viscosity is registered for the mixes with longer fibers. With same volume fraction, fiber having longer length reduces workability of concrete of a higher percentage. Fibers can form a network structure in concrete, which restrain mixture from segregation and flow. Slump classes are S3 and S4, so concrete is classified as fluid. Polypropylene and Nanocoomposite fibers have the same effect on concrete rheological behaviour.

Table 6 – Slump values of tested concretes.

Mix	Slump [mm]	Slump variation [%]	Slump class
Reference	200	-	S4
FRC_PPNC20A	160	-20	S4
FRC_PPNC20B	150	-25	S3
FRC_PPNC60A	150	-25	S3
FRC_PPNC60B	140	-30	S3
FRC_PP20A	165	-18	S4
FRC_PP20B	155	-23	S3
FRC_PP60A	145	-28	S3
FRC_PP60B	140	-30	S3

3.3 Test on hardened concrete

Compressive strength was obtained by eq. 2. As stated by several authors, fibers addition is not effective on compressive strength and variation of compressive strength is very negligible. Variation of compressive strength over unreinforced reference mix is very low, varying between -7.06 % and +11.42 % for FRC_PP20B and FRC_PPNC20A, respectively.

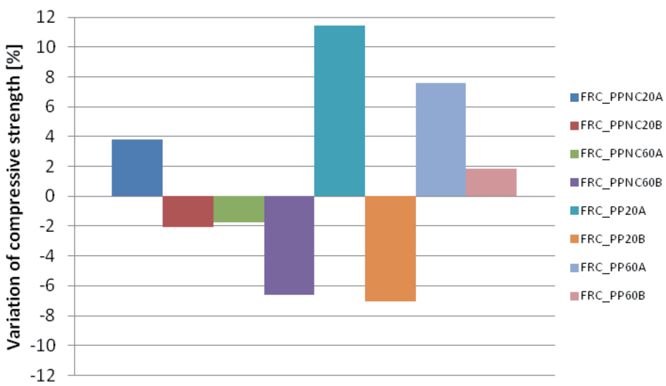


Figure 2 – Variation of compressive strength over reference mix.

Observing specimens after compressive test is clear the different failure mechanism: specimens without fibers show the so-called "hourglass" shape (Figure 3a) due to shear forces generated by friction at the interface with the plates of the press, which prevent the cross expansion of the specimen (Poisson's effect). Specimens containing fibers have a different rupture because fibers act as confinement (Figure 3b).

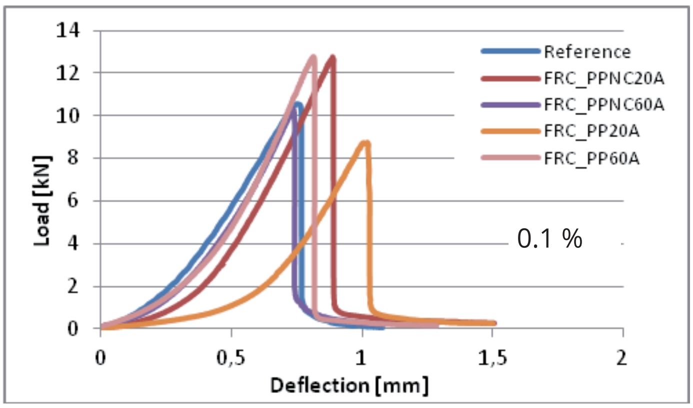
Values of flexural strength are reported in Table 7.

Four-point bending tests report a low variation of flexural strength. Fibers are effective in the post-cracking behaviour for the increase in toughness: load-deflection curves show an elastic behaviour until the peak is reached and cracking is achieved. Post-cracking behaviour is strain-softening and the residual stress increases increasing fibers volume fraction and fibers length (Figure 4 and Figure 5).

Figure 3 – a) Specimen without fibers; b) specimen with fibers.

Table 7 – Flexural strength of concrete at 28 days.

Mix	Flexural Strength [N/mm²]
Reference	1.54
FRC_PPNC20A	1.62
FRC_PPNC20B	1.67
FRC_PPNC60A	1.25
FRC_PPNC60B	1.45
FRC_PP20A	1.17
FRC_PP20B	1.48
FRC_PP60A	1.60
FRC_PP60B	1.47



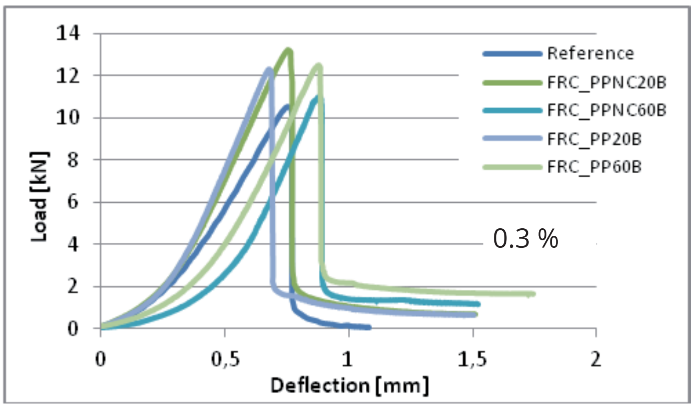


Figure 4 – Load-deflection curves, a) 0.1% fibers volume fraction; b) 0.3% fibers volume fraction.

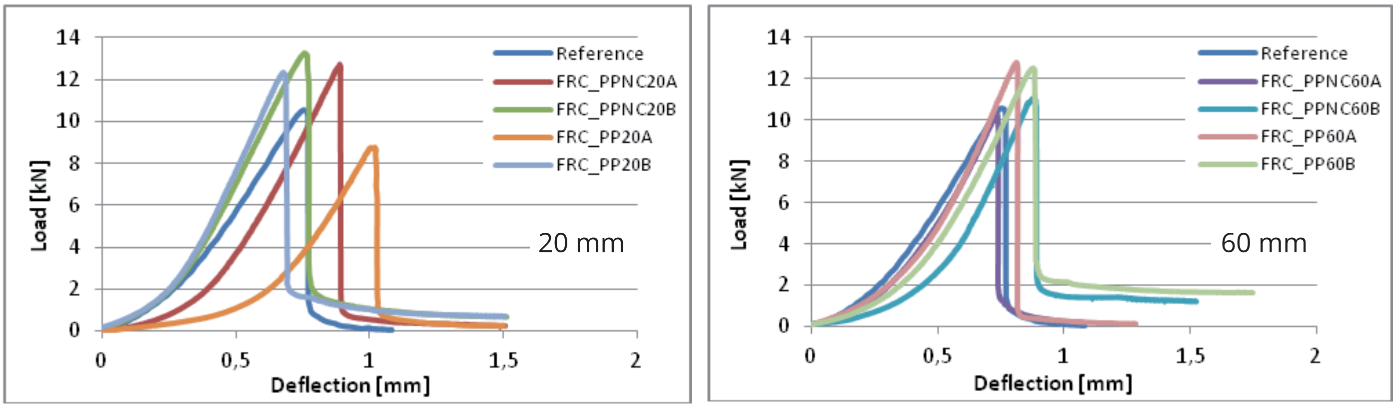


Figure 5 – Load-deflection curves, a) fibers 20 mm long; b) fibers 60 mm long.

Considering the same fibers volume fraction (Figure 4) and the same fibers length (Figure 5), we have the higher toughness increase for FRC_PP60B and FRC_PPNC60B, respectively. These mixes contain longer fibers (60 mm) and higher fibers volume fraction (0.3%). Thus, post-cracking behaviour is influenced by fibers volume fraction and length. Fibers could bridge fracture faces and delay specimens failure, showing residual stress after crack openings. An important parameter for toughness increase is the amount of fibers present in the notch (Figure 6).

Concrete water absorption was calculated by eq. 5 and the percentage of absorbed water versus time is reported in Figure 7. Reference sample presents the higher absorption rate, 6.64%. FRCs show a lower percentage of absorbed water rather than the reference mix, varying between -70% and -75% for FRC_

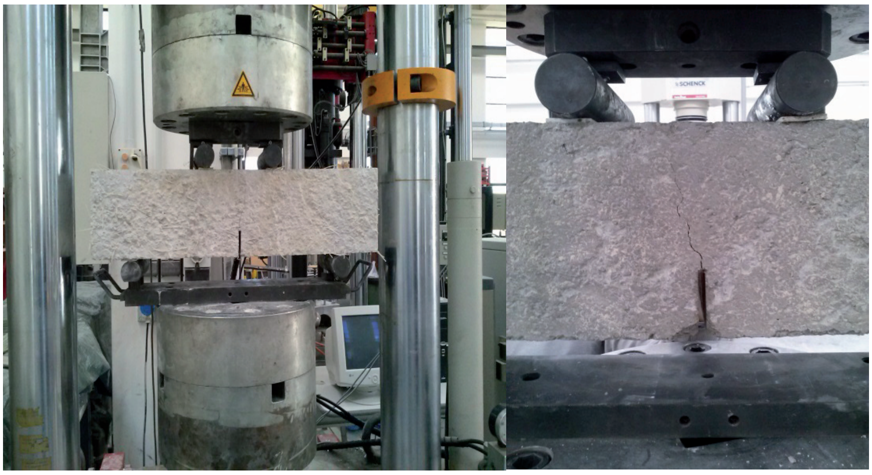


Figure 6 – Four-point bending test, notched specimen.

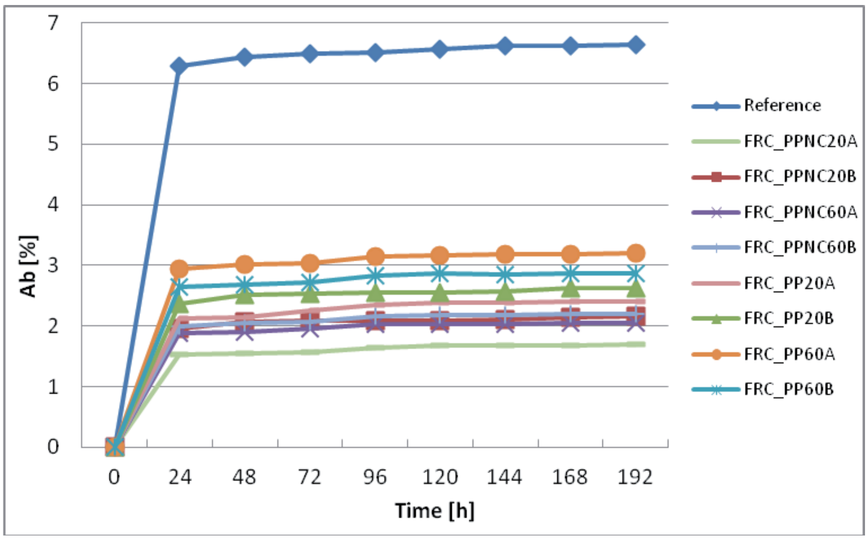


Figure 7 – Absorbed water percentage vs time

PP60A and FRC_PPNC20A respectively. Curves show a similar behaviour for the different mix: during the first 24 hours concrete absorbs the higher percentage of water and after 4 days tend to plateau. The presence of fibers leads to a lower water absorption rate and the best behaviour is registered for mix FRC_PPNC20A which contains nanocomposite fibers of 20 mm at the lower volume fraction (0.1%). Increasing fibers length and volume fraction a slightly greater percentage of water is absorbed (Figure 8 and Figure 9).

FRCs containing nanocomposite fibers absorb a slightly lower water amount compared to FRCs containing polypropylene fibers. Figure 9b shows that FRCs reinforced with longer fibers absorb more water than FRCs containing shorter fibers (Figure 9a). Thus, FRCs water absorption increases increasing fibers length and volume fraction.

Capillary water absortpion test is a further evidence of this phenomena. FRCs absorb less water, presenting clearly lower values of absorbed water per surface unit ratio, I_i (Figure 10).

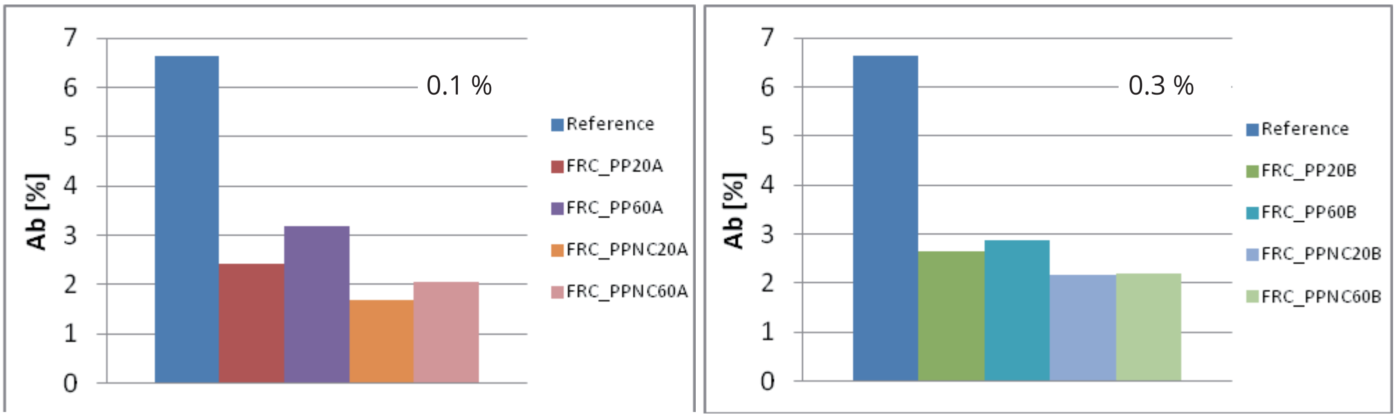


Figure 8 – Absorbed water percentage, a) 0.1% fibers volume fraction; b) 0.3% fibers volume fraction.

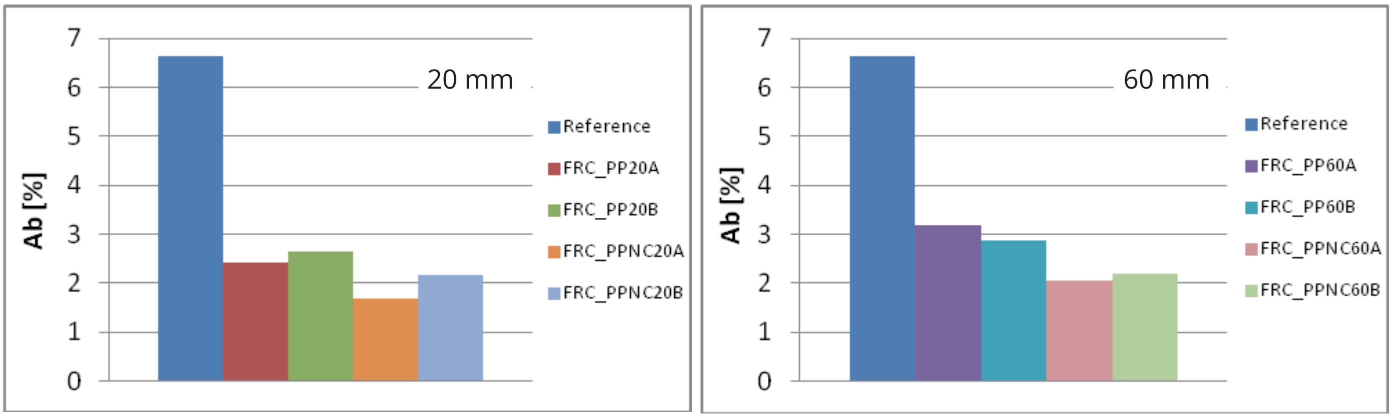


Figure 9 – Absorbed water percentage, a) fibers 20 mm long; b) fibers 60 mm long.

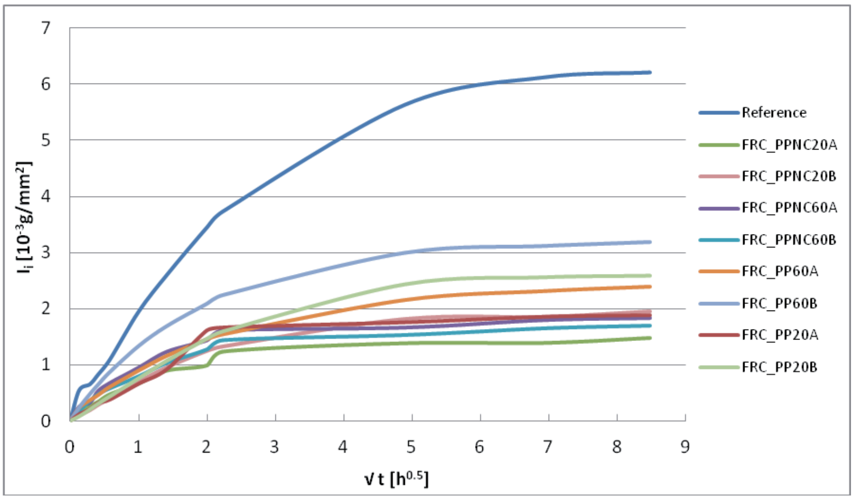


Figure 10 – Absorbed water per surface unit vs square root of time.

At the end of the test capillary rise has been measured (Figure 11). Fibers addition clearly leads to a decrease of absorbed water. Capillary rise of unreinforced reference mix is 50 mm while for FRCs it ranges between 31.50 mm and 15.10 mm for FRC_PP60B and FRC_PPNC20A, respectively. It is generally observed that FRCs reinforced with nanocomposite fibers have a lower capillary rise and increasing fiber length also capillary rise increases.

Sorptivity coefficient, S_i, has been calculated by eq. 4 and results are listed in Table 8. Sorptivity coefficient is expected to increase by increasing the capillary pores in concrete. The presence of fibers changes pores structure: fibers length, diameter and volume fraction are important parameters governing this aspect.

Sulfate attack has been evaluated by specimens weight loss over cycles number (Figure 12). For all the specimens was registered an increase of weight after the first cycle, due to the absorption of sulfate solution. Weight starts to decrease after the fourth cycle when expansive action of ettringite causes particles loss.

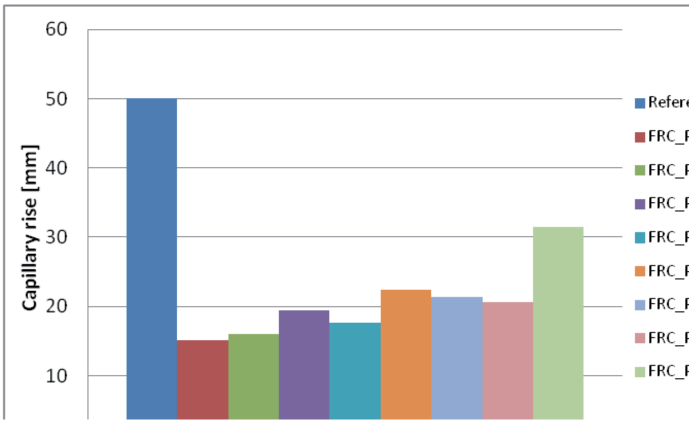


Figure 11 – Capillary rise at the end of the test.

Table 8 – Sorptivity coefficient [10^{-3} g/mm^{2}h$^{0.5}$].

Mix	S_1	S_2	S_3	S_4	S_5	S_6	S_7	S_8	S_9	S_{10}	S_{11}
Reference	4.19	2.25	2.01	1.88	1.94	1.83	1.72	1.66	1.15	0.88	0.73
FRC_PP20A	0.89	0.71	0.77	0.65	0.66	0.65	0.81	0.74	0.36	0.26	0.22
FRC_PP20B	0.60	0.69	0.74	0.74	0.75	0.75	0.73	0.71	0.49	0.36	0.30
FRC_PP60A	1.78	1.26	1.11	1.04	0.89	0.81	0.72	0.68	0.43	0.33	0.28
FRC_PP60B	1.81	1.65	1.57	1.49	1.33	1.19	1.04	1.00	0.61	0.44	0.37
FRC_PPNC20A	1.04	0.90	0.83	0.81	0.66	0.63	0.49	0.55	0.28	0.20	0.17
FRC_PPNC20B	0.69	0.659	0.69	0.69	0.69	0.67	0.62	0.59	0.37	0.26	0.22
FRC_PPNC60A	1.93	1.11	1.30	1.16	0.94	0.87	0.71	0.72	0.34	0.25	0.21
FRC_PPNC60B	1.36	1.24	1.16	1.00	0.79	0.73	0.63	0.64	0.31	0.23	0.20

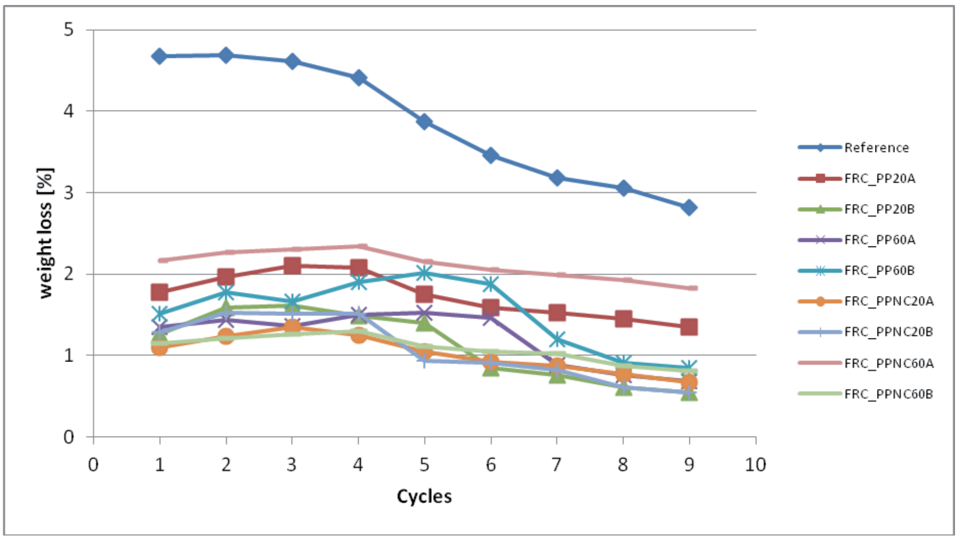


Figure 12 – Weight loss during sulfate attack cycles.

Samples are not completely destroyed after the last cycle. Reference samples show a higher mass variation after the third cycle while FRCs curves rise until the fourth cycle and then slightly decreasing. Again fibers volume fraction and fibers length are fundamental parameters: increasing fibers volume fraction weight loss decreases.

Durability factor is a parameter to assess freeze/thaw cycles resistance of concrete. Due to the low number of cycles performed in this study, is considered only the variation of durability factor over reference mix (Figure 13 and Figure 14).

The greater increase of durability factor is registered for FRCs containing the higher fibers volume fraction (Figure 13b and Figure 14). Fibers length is effectiveness at lower fibers volume fraction: FRCs with shorter fibers have higher durability factor (Figure 13a). Thus, for freeze/thaw resistance the most important parameter is the fibers volume fraction while fibers length influence plays a role at lower fibers volume fraction.

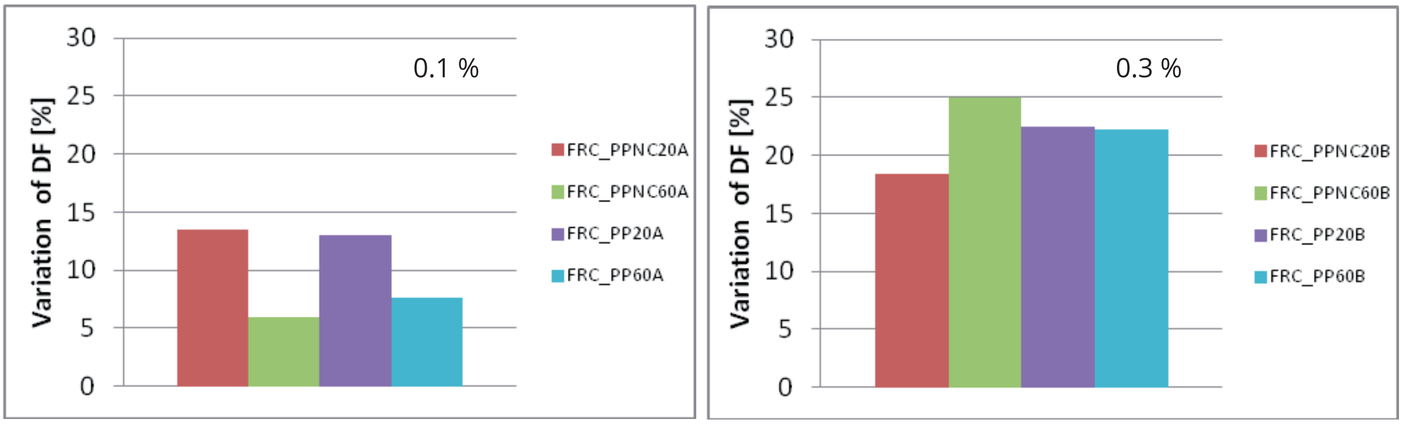


Figure 13 – Variation of durability factor over reference mix, a) 0.1% fibers volume fraction; b) 0.3% fibers volume fraction.

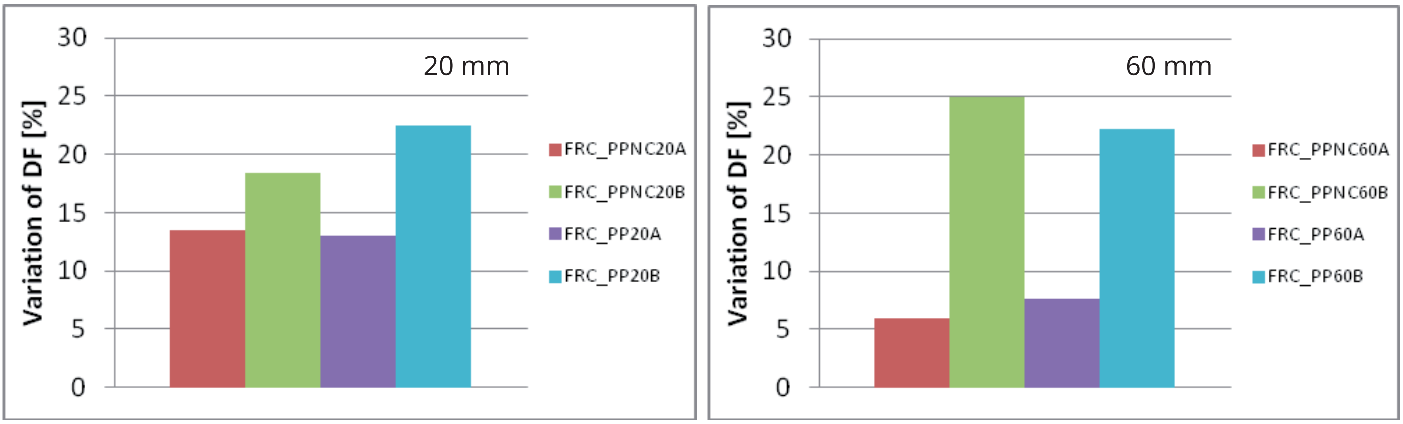


Figure 14 – Variation of durability factor over reference mix, a) 20 mm length fibers; b) 60 mm length fibers.

4. Conclusions

In the present work the influence of Nanocomposite fibers on FRCs was investigated. Nanoclay addition increases elastic modulus and tensile strength compared to pure PP fibers. Nanocomposite fibers are slightly stiffer than pure PP ones but are less ductile.

Increasing fibers volume fraction workability of concrete decreases and considering the same volume fraction, longer fibers reduces workability of concrete of a higher percentage.

Compressive and flexural strength of FRCs are not influenced by fibers addition while post-cracking behaviour is affected by fibers volume fraction and length. Fibers could bridge cracks and delay specimens failure, increasing toughness.

Fibers modify transport properties: the presence of fibers leads to a lower amount of absorbed water. The best behaviour is registered for FRCs containing few and shorter fibers. Increasing fibers length and volume fraction a slightly greater percentage of water is absorbed.

FRCs show a better durability compared to unreinforced concrete. FRCs exposed to sulfate attack show a lower weight variation during cycles, in this case the volume fraction represents an important parameter: increasing the volume fraction the weight loss decreases. Regard freeze/thaw resistance, the greater increase of durability factor is registered for FRCs containing the higher fibers volume fraction. Fibers length is effectiveness at lower fibers volume fraction.

Thus, nanoclay addition plays an important role on fibers properties but slightly differences are registered on FRCs containing nanocomposite fibers compared to pure PP fibers.

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