EVALUATION OF THE DURABILITY OF SYNTHETIC MACROFIBERS IN CEMENT MATRICES

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Abstract

The work presented in this paper focus on the evaluation of the durability of two synthetic macrofibers, one made from polypropylene (PP) and the other made from recycled polyethylene terephthalate (PET), in cement matrices. An accelerated test was performed by the immersion of samples of each fiber in an alkali solution for 30 days at 60°C. Besides, prismatic beams were molded with concrete reinforced with 0.50% of those fibers for bending tests for the determination of residual strength at the age of 28 and 168 days. It was observed that the PET fiber was severely attacked by the alkali solution and that the residual strength of the concrete reinforced with that macrofiber at the age of 168 days corresponded to 52% of the residual strength at the age of 28 days. However, the PP fiber did not show any indication of degradation on its surface. Moreover, residual strength of concrete reinforced with PP fibers at the age of 168 days presented a slight increase (7%) compared to the residual strength at the age of 28 days.

Keywords: synthetic macrofibers, durability, residual strength

1. Introduction

The use of synthetic macrofibers for concrete reinforcement has been growing continuously. Their main function is to provide toughness and ductility to the concrete matrix and to avoid the propagation of cracks [1]. The polymers most used for fiber manufacturing are polyethylene, polypropylene, polyamides and polyvinyl alcohol, because they present durability and mechanical properties suitable for that application [2, 3].

The source of the raw materials for fibers is not renewable. In Brazil, in an attempt to recycle some sorts of plastic, macrofibers produced from the recycling of polyethylene terephthalate (PET) bottles have been introduced in the market. That polymer corresponds to 20% of the solid residue generated in the country [4]. This fact generates a motivation for the development of some recycling processes in order to make the reuse of the material feasible, such as in fiber-reinforced concrete.

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In the recycling operations of PET, alkaline solutions may be used to accelerate some steps of the process. By using that, the depolymerization of PET may occur, with the cleavage of ester groups, originating smaller fragments and increasing the number of carboxyl end groups [5, 6]. The resulting polymer presents smaller molar mass and fewer numbers of cross-links when compared with the initial polymer, presenting smaller crystallinity and poor mechanical and chemical properties [7]. Hence, its durability should be taken into account when the application in fiber-reinforced concrete is the goal. In that sense, an experimental study was carried out in order to provide an analysis of the durability risks involved in concrete reinforced with PET macrofibers.

2. Objectives

The objective of this study is to make an experimental evaluation of the durability of synthetic macrofibers, produced from recycled PET, in a concrete matrix.

3. Materials and methods

To evaluate the durability of the fiber produced from recycled PET, an accelerated immersion test was performed and the residual strength of concrete reinforced with that fiber was measured. Concurrently, the same tests were performed with a polypropylene fiber made from virgin raw materials.

The properties of fibers used in this experimental campaign are described in table 1.

Tab.1: Properties of the macrofibers

Property	**PP fiber**	**Recycled PET fiber**
Raw material	Virgin copolymer / polypropylene	Recycled PET
Form	Monofilament / fibrillated	Monofilament
Fiber count (filaments/kg)	221,000	18,900
Length (mm)	54	53
Equivalent diameter (mm)	0.32 (for a single filament)	1.08
Density (g/cm^3)	0.91	1.18
Tensile strength (MPa)	570 – 660	-

Two different tests were performed. The first one was an accelerated degradation test, performed by the immersion of 10.0g of each fiber in 200.0g of 1M sodium hydroxide solution (40g/L, pH 14), during 30 days, at the temperature of 60^{o}C. The accelerated conditions are equivalent to the period of 13 years of exposure to the pore solution of a cement matrix (pH 13) at the temperature of 20^{o}C, approximately, considering the Arrhenius equation for the calculation of the reaction rate [8]. For each fiber, three samples were prepared and the results presented correspond to their average.

The analysis of this test is based on the mass loss of the fiber, the formation of residues and the modification of its visual characteristics, due to the degradation reaction. To determine the mass loss after the end of the test, the fiber was filtered from the solution, washed with tap water and dried at 60^{o}C until constant mass.

The second part consisted on the characterization of the mechanical behavior of concrete reinforced with those fibers. One concrete matrix, described on tables 2 and 3, reinforced with 0.50% of fibers was used. The flexural tests were performed at the ages of 28 and 168 days to check how residual strength changes with time.

Tab.2: Concrete matrix composition.

Material	**Dosage (kg/m^3)**
Type III cement	330.00
Fine aggregate	825.00
Coarse aggregate	1023.00
Water	198.00
Superplasticizer	1.32

Tab.3: Concrete matrix characteristics.

Characteristic	**Result**
Mix proportion	1.00 : 2.50 : 3.10 : 0.60
Unit mass (kg/m^3)	2287
Entrapped air (%)	3.2
Slump (mm)	120
Dry mortar content (%)	53.0
Compressive strength (28 days) (MPa)	35.2 ± 0.4
Flexural strength (28 days) (MPa)	4.53 ± 0.07

For each fiber and age, ten 150mm x 150mm x 500mm beams were molded for the flexural test according to ASTM C1609 (2010) [9] and six 150mm x 300mm cylindrical specimens for the compression test, according to ABNT NBR 5739 (2007) [10].

For the flexural test, a 120kN servo-actuator with closed-loop displacement control, two LVDTs with a sensitivity of 1.0 x 10^{-3}mm placed in a yoke and a data recording system at the frequency of 50Hz were used. The test was performed under 4-point-bending and the load application is controlled by the net vertical deflection of the specimen. The beam is loaded in a 450mm span up to a deflection of 3.0mm. The rate of increase of net deflection from 0 to 0.75mm was 0.12mm/min and from 0.75 to 3.0mm, the rate was 0.24mm/min.

The smaller rate at the beginning of the test is used to avoid instabilities when the rupture of the specimen occurs [9].

For each specimen, a load-deflection curve is plotted. For the specimens of the same series, an average curve is calculated. From each curve, the residual strength (*f*) at the deflection of 0.75 and 3.0mm are calculated using the equations 1 and 2, respectively.

$$f150{,}0.75 = P150{,}0.75.L/bd2 \tag{1}$$

$$f150{,}3.0 = P150{,}3.0.L/bd2 \tag{2}$$

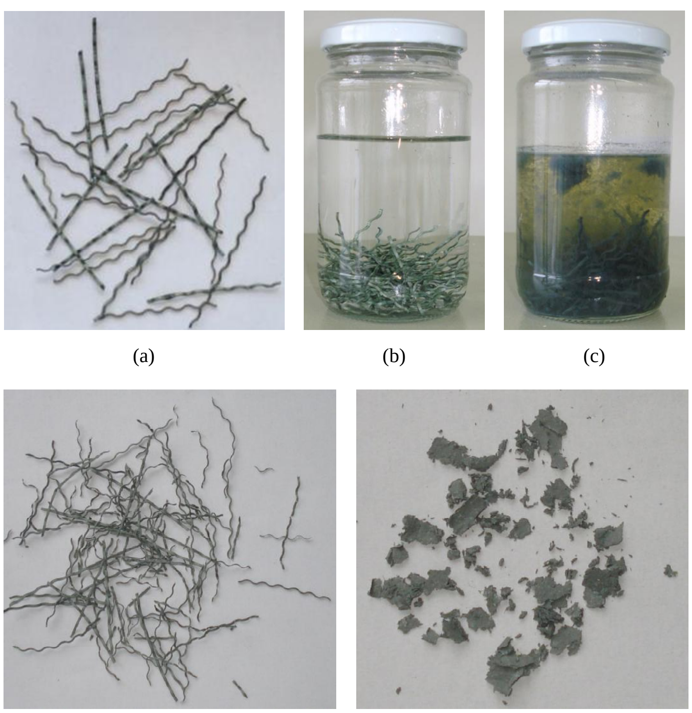
where P150,0.75 and P150,3.0 correspond to load values at 0.75 and 3.0mm, respectively, L is the span, b is the width of the beam and d is the depth of the beam.

For the compression test, an 1100kN equipment was used and the load was applied at a constant rate of 0.45MPa/s.

4. Results and discussion

4.1 Accelerated immersion test

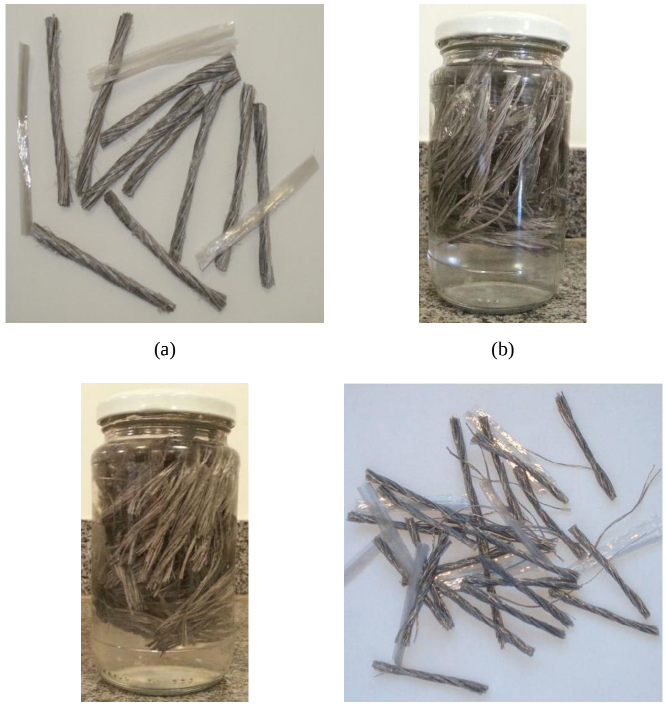
Figures 1 and 2 show some aspects of the test performed with the PET and the PP fibers, respectively. In table 4, the results of the mass loss of each fiber are presented.



(a) (b) (c)

(d) (e)

Fig. 1: Pictures of the PET fiber: as received (a); immersed in the solution at the beginning of the test (b); immersed in the solution at the end of the test (c); after the test (d); residue formed (e).



(a) (b) (c) (d)

Fig. 2: Pictures of the PP fiber: as received (a); immersed in the solution at the beginning of the test (b); immersed in the solution at the end of the test (c); after the test (d).

Tab.4: Mass loss after the test.

Fiber	**Mass loss / %**
PET	68.0
Polypropylene	0.73

As it can be observed on figure 1 (especially in pictures ‘a’ and ‘e’), the PET macrofiber did not present chemical resistance to the solution used. A reaction occurs on the macrofiber surface, degrading it and generating a solid residue, as shown in figure 1(e). After the test, the fiber surface becomes very irregular and the filaments are broken. The mechanical resistance of the fiber is damaged and the filaments can be easily broken by hands. Besides, 68.0% of the initial mass of the sample were degraded. This high mass loss is due to the solid residue formed and to the water-soluble hydrolysis products (removed when the fiber was washed).

The degradation was already expected, because PET – a polyester – is hydrolyzed in the presence of alkalis. The equation that represents the degradation reaction is shown in figure 3 [7].

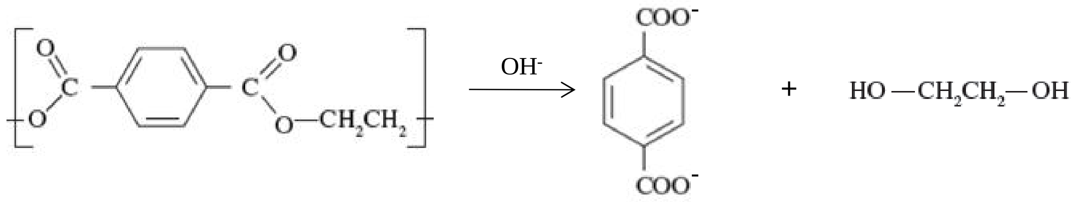


Fig. 3: Equation representing the degradation of the PET fiber in an alkali solution [7].

However, the polypropylene fiber did not have any significant mass loss and no modification of its initial characteristics was observed. PP is a polyolefin and does not have any reactive sites to nucleophilic attack by hydroxides. That is, PP macrofibers are inert to alkali solutions and do not undergo hydrolysis or depolymerization when kept in an alkali environment [7].

The results obtained in this test are a good sign of the durability of those fibers in cement matrices. As the PET fiber is continuously attacked by the alkalis contained in concrete, its degradation and damage in the fiber-matrix interface will occur in the majority of cases. Then, the residual strength provided by that PET macrofiber is expected to be reduced with time, until possibly zeroed out.

4.2 Compressive and flexural tests

The average compressive strength and the standard deviation of the compressive tests are shown in table 5. It can be observed that the presence of fibers does not alter that property, no matter what the age of the concrete is. The results presented very similar values, making the composites comparable. As the cement used a type III (high early strength), there was no expectative for any significant increase in the strength level from 28 to 168 days. So, all the results were very similar. The increase of the strength level of the PET macrofiber-reinforced concrete at the age of 168 days is not relevant due to the inherent variability of the test method itself.

Tab.5: Compressive strength results.

Fiber	**Age / days**	**f_{cm} / MPa**
PET	28	34.3 ± 1.1
	168	34.8 ± 0.6
PP	28	34.3 ± 0.2
	168	36.2 ± 1.1

The load-deflection curves obtained with the concretes reinforced with the PET and the PP macrofibers, at the ages of 28 and 168 days are shown in figure 4. The average curves from each set of specimens are shown in figure 5. From the analysis of the curves, the modulus of rupture and the residual strengths at the deflection of 0.75 and 3.0mm were calculated. These results are presented on table 6.

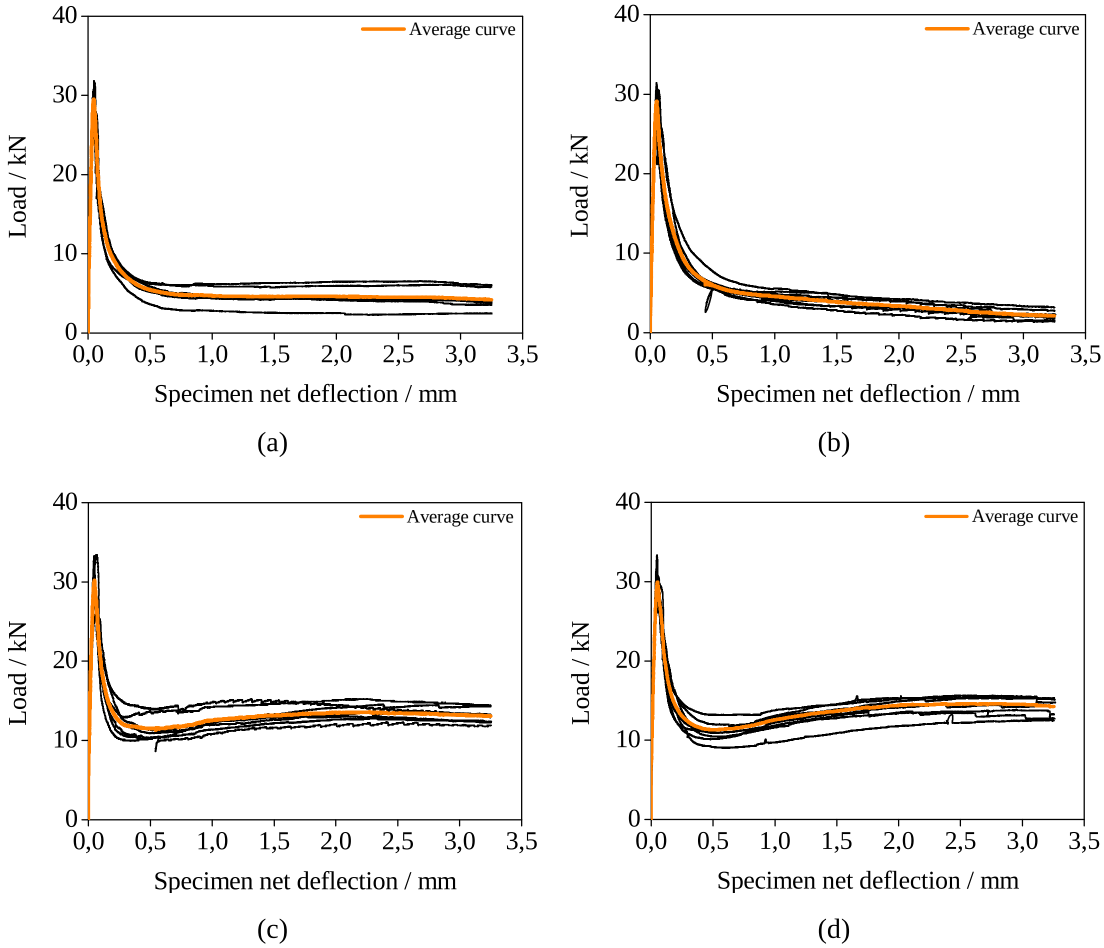


Fig. 4: Load-deflection curves of concrete reinforced with the PET fiber at 28 days (a); PET at 168 days (b); PP at 28 days (c); PP at 168 days (d).

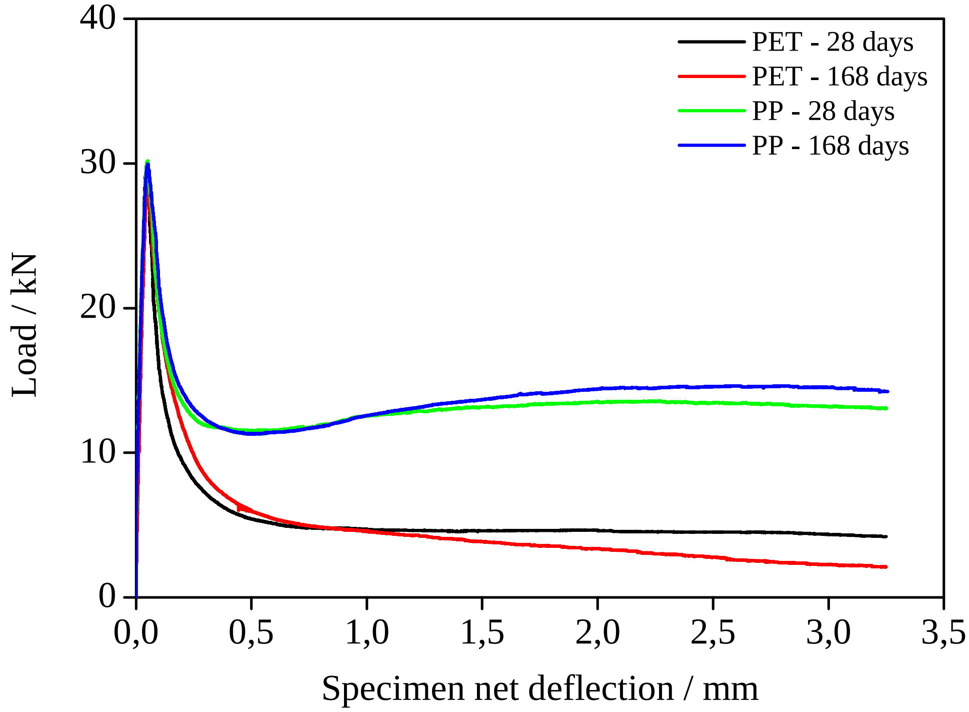


Fig. 5: Average load-deflection curves of concrete reinforced with PET and PP fibers at the age of 28 and 168 days.

Tab.6: Results calculated from the load-deflection curves from figure 4.

Fiber	**Age / days**	**MOR / MPa**	**$f_{cm150,0.75}$ / MPa**	**$f_{cm150,0.75}$ / MPa**
PET	28	3.90 ± 0.18	0.643 ± 0.141	0.586 ± 0.173
	168	3.91 ± 0.18	0.661 ± 0.072	0.302 ± 0.078
Polypropylene	28	4.00 ± 0.33	1.58 ± 0.19	1.77 ± 0.13
	168	4.06 ± 0.17	1.51 ± 0.17	1.90 ± 0.16

It can be observed that there is a low variability in the curves and that the post-crack behavior is very uniform, without post-peak instability. All the ruptures occurred by the formation of a single crack, which propagated in a stable manner. Due to the low macrofiber content, all the beams presented a deflection-softening behavior [11].

Regarding the PET fiber-reinforced concrete, it can be observed that the composite sustains a lower level of load when compared to the curve at 28 days and presents a slip-softening behavior. So, the residual strength presented a tendency of reduction, especially for higher levels of displacement and crack opening. This happens because the fiber surface is attacked by the alkalis from the concrete, degrading it and damaging the fiber-matrix interface.

On the other hand, the PP macrofiber-reinforced concrete presents similar behavior at 28 and 168 days, with a slight increase, around 7%, in the residual strength at 3.0mm at 168 days. Due to the densification of the matrix at higher ages, fiber anchorage is enhanced, causing an increase in toughness. Moreover, this composite presents a slip-hardening behavior.

Comparing both fibers, the residual strength provided by the PP macrofiber is around three times higher than that provided by the PET macrofiber at 28 days. At 168 days, that difference is even higher, around six times, mainly because the PET macrofiber tends to lose its capacity to sustain load as time goes by.

5. Conclusions

It was observed that PET fibers do not have good chemical resistance and that the concrete reinforced with that kind of macrofiber could present reduced residual strength at higher ages, due to the degradation of the fibers inside the concrete matrix. So, it can be concluded that the PET fiber used in this study is not suitable to be applied as a concrete reinforcement. The raw materials used in its manufacturing process are not alkali-resistant and, hence, the fiber does not provide good residual strength and tends to lose its capacity to sustain loads at high ages. Although there is a trend to study the recycling of PET for this purpose, the problem of durability in concrete cannot be ignored.

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References

[1] FIGUEIREDO, A. D. **Concreto reforçado com fibras.** 248p. Tese (Livre-Docência) - Escola Politécnica, Universidade de São Paulo, São Paulo, 2011.

[2] BENTUR, A.; MINDESS, S. **Fibre reinforced cementitious composites**. United Kingdom: Elsevier, 2007.

[3] ZHENG, Z.; FELDMAN, D. Synthetic fibre-reinforced concrete. **Prog. Polym. Sci.**, Canada, v. 20, p. 185-210, 1995.

[4] ROMAO, W., SPINACE, M. A. S., PAOLI, M. Poli(tereftalato de etileno), PET: uma revisão sobre os processos de síntese, mecanismos de degradação e sua reciclagem. **Polímeros**, São Carlos, v. 19, n. 2, Junho 2009.

[5] CAPARANGA, A. R. et al. Factors affecting degradation of polyethylene terephtalate (PET) during pre-flotation conditioning. **Waste Management**, v. 29, 2009.

[6] SANTOS, A. S. F., AGNELLI, J. A. M., MANRICH, S. Estudo da influência de resíduos catalíticos na degradação de plásticos reciclados (Blenda HDPE/PP e PET) provenientes de lixo urbano. **Polímeros**, São Carlos, v. 9, n. 4, Dezembro 1999.

[7] BRUICE, P.Y. **Organic Chemistry**. 6. ed. USA: Prentice Hall, 2010.

[8] ATKINS, P, de PAULA, J. Physical Chemistry. 7th edition. United Kingdom: Oxford University Press, 2001.

[9] AMERICAN SOCIETY FOR TESTING MATERIALS. **ASTM C1609**: Standard test method for flexural performance of fiber-reinforce concrete (using beam with third-point loading). Philadelphia, 2010a. 8 p.

[10] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. **NBR 5739**: Concreto: ensaio de compressão de corpos-de-prova cilíndricos. Rio de Janeiro, 2007a. 9p ABNT NBR 5739 (2007)

[11] NAAMAN, A. E. High performance fiber reinforced cement composites. In: **RILEM Proceedings**, **PRO 30**, RILEM Publications SARL, 2003.