CHARACTERIZATION OF SELF-FIBRILLATING SYNTHETIC MACROFIBERS FOR CONCRETE REINFORCEMENT

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Abstract

The work presented in this paper is about the chemical, physical and mechanical characterization of a self-fibrillating synthetic macrofiber for concrete reinforcement. For the analysis of the macrofiber chemical composition, infra-red (IR) and energy-dispersive x-ray (EDS) spectroscopies were performed. The tensile test indicated that the mechanism of fiber failure is through fiber fibrillation and not rupture by the formation of a constriction (necking). Analysis by scanning electron microscopy showed that fiber surface is rough and irregular and that the process of mixing it in the concrete promotes a high increase in its surface area. Moreover, fiber-matrix interface is very homogeneous, without the presence of pores or voids, and presents microstructure similar to that of the bulk. Bending tests revealed that concretes reinforced with fiber contents above 0.50% present slip-hardening behavior, which is associated with the chemical composition of the fiber, making it behave as self-fibrillating.

Keywords: synthetic macrofibers, self-fibrillating, slip-hardening composites

1. Introduction

Synthetic macrofibers for concrete reinforcement were developed in the 1990's. The first applications were in shotcrete, especially in Australia and in Canada [1]. Nowadays, these fibers are used in many segments, such as concrete floors, tunnel linings and precast concrete [2]. Their mechanical properties are dependent on their molecular structure, being the molecular mass of the polymer, the bond strength between chains and the intermolecular interactions the main factors that influence their strength [3].

Their main function is to provide toughness and ductility to the concrete matrix and to avoid the propagation of cracks [2]. The energy absorption mechanisms that control the residual strength and the ultimate deformations of the composite are related to the debonding and pull-out of the fibers bridging the cracks [4].

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Interfacial microstructure changes can occur during macrofiber pull-out when the composite is loaded, resulting in damage to the macrofiber or to the surrounding matrix, depending on their relative stiffness [4]. Some polymeric macrofibers, which are soft, tend to be abraded and fibrillated during pull-out, increasing their surface area and, hence, the fiber-matrix interaction and bonding, leading to a slip-hardening behavior [5].

The chemical composition of the macrofiber is one of the main factors that promote the slip-hardening behavior and its self-fibrillation [5, 6]. These properties tend to compensate the macrofiber's low elastic modulus and high Poisson ratio [4]. Hence, the macrofibers characterization is essential in order to explain the composite behavior.

2. Objectives

To present a study on the chemical, physical and mechanical characterization of a synthetic macrofiber for concrete reinforcement.

3. Materials and methods

A macrofiber composed of polypropylene in a monofilament / fibrillated system was used in this study to demonstrate the characterization process. The fiber length was 54mm and its equivalent diameter was 0.32mm (for a single filament). The density of the macrofiber was 0.91g/cm^3 and its tensile strength between 570 and 660MPa. These data were reported by the fiber manufacturer.

In order to obtain information about the chemical composition of the macrofiber, infrared (IR) and energy dispersive X-ray (EDS) spectroscopies were used. The first test was performed using a KBr pellet, in the ratio of 1:100, scanned from 4000 to 400cm^{-1}, in an equipment Nicolet 6700 from *Thermo Scientific*. The second test was performed in the microscope used for scanning electron microscopy, described forward.

The second step of the macrofiber characterization was to evaluate its mechanical behavior through a tensile test, according to ASTM D2256 (2010) [7]. Ten 500mm-long filaments (formed by 3 monofilaments glued together – equivalent diameter of 0.51mm) were tested in a 250mm gage length, at the rate of load application of 200N/min, in an equipment *5569* from *Instron*, using pneumatic flat-faced clamps and a 1000N load cell. First, it is obtained a load-elongation curve, from which a stress-strain curve is calculated using equations 1 and 2. The angular coefficient of the stress-strain curve corresponds to the fiber elastic modulus.

$$sT = F/A \tag{1}$$

where σT is the tensile strength, F is de breaking force and A is the section area of the fiber before the test.

$$ef = (Lf - Li)/Li \tag{2}$$

where ef is the deformation of the fiber, Lf and Li its final and initial length, respectivelly.

The third step of the experimental program was the characterization of the macrofiber using scanning electron microscopy (SEM). This analysis was performed in a *Stereoscan S440* microscope using four different samples: fiber as received, fiber washed out from a

fresh concrete, where it had been mixed for 20 minutes, and fibers that presented rupture by fibrillation and by pull-out, taken from a fractured prismatic specimen.

The last part consisted on the characterization of the mechanical behavior of concrete reinforced with this macrofiber. One concrete matrix (mix proportion of 1.00 : 2.65 : 3.24 : 0.66, using a type III cement and 0.4%bcw of superplasticizer), reinforced with fiber contents of 0.22, 0.33, 0.50, 0.66, 0.82 and 1.0% by volume was used. The flexural tests were performed when the concrete was at the age of 28 days. The characteristics of the matrix used are presented in table 1.

Tab.1: Concrete matrix characteristics.

Characteristic	**Result**
Specific weight	2251kg/m^3
Entrapped air	3.4%
Slump	120mm

Characteristic	**Result**
Dry mortar content	53.0%
Compressive strength (28 days)	(30.1±0.6)MPa
Flexural strength (28 days)	(4.26±0.02)MPa

For each fiber content, ten 150mm x 150mm x 500mm beams were molded for the flexural test according to ASTM C1609 (2010) [8] and six 150mm x 300mm cylindrical specimens for the compression test, according to ABNT NBR 5739 (2007) [9].

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 using a 4-point-bending apparatus and the load application was controlled by the net vertical deflection of the specimen. The beam was 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.

For each specimen, a load-deflection curve is obtained. From each curve, the residual strength (f) at the deflections of 0.75 and 3.0mm are calculated using the equations 3 and 4. For the group of specimens with the same fiber content, an average curve is calculated.

$$f150,0.75 = P150,0.75.L/bd2 \quad (3)$$

$$f150,3.0 = P150,3.0.L/bd2 \quad (4)$$

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. These determinations were made accordingly to the ASTM C1609 (2010) [8] recommendations.

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 IR and EDS spectroscopies

The EDS spectrum shown in figure 1a indicated that the fiber is composed by carbon atoms only. Since this technique cannot detect elements lighter than sodium (atomic number 11) [10], it can be concluded that the polymer is based on hydrocarbons. The IR spectrum is shown in figure 1b and the analysis on table 2. Analyzing the data of both spectra, it is possible to conclude that the polymer is composed by a mixture of polypropylene and polyethylene. These polymers are immiscible and promote the formation of a self-fibrillating fiber [6]. This condition is confirmed by the SEM analysis, as presented in item 4.3.

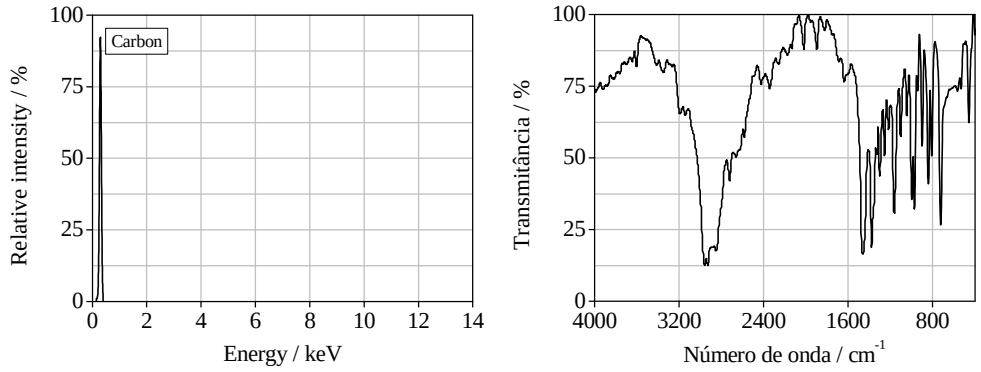


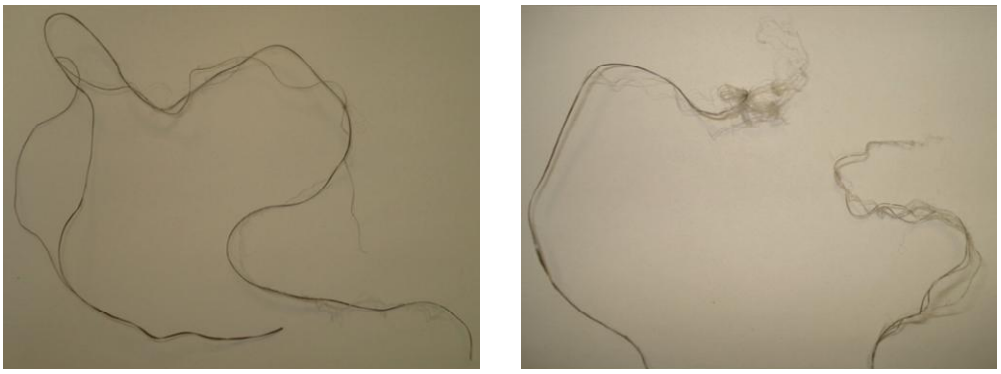
Fig. 1: EDS (a) and infrared (b) spectra of the polymeric fiber.

Tab.2: IR spectrum analysis (LDPE: low density polyethylene; iPP: isotatic polypropylene) [11].

Wave number / cm^{-1}	**Assignment**	**Polymer**
725	CH_2 rocking	LDPE
901, 997	Terminal vinyl groups	LDPE
810, 841, 974, 1220, 1260, 1300	CH_3 bending, wagging, twisting C–C stretching	iPP
2850	CH_2 symmetrical stretching	LDPE and iPP
2930	CH_2 asymmetrical stretching	LDPE and iPP
2960	CH_3 symmetrical stretching	iPP

4.2 Tensile test

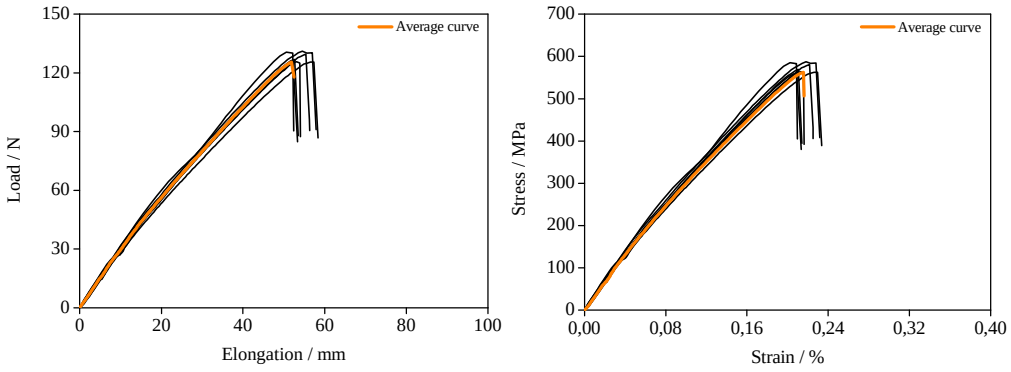
The filaments tested are shown in figure 2. This test was performed in a continuous filament prior to fiber cutting, as recommended by the standard ASTM D2256 (2010) [7]. It can be observed that the rupture mechanism does not occur by the necking of the fiber. Instead, the fiber is fibrillated by the rupture of the connections among the microfilaments.



(a) (b)

Fig. 2: Filaments after the tensile test: fibrillated (a) and ruptured (b) filaments.

The load-elongation and stress-strain curves are shown in figure 3. The results calculated from the curves are on table 3. It is possible to observe that the pattern of results is very uniform, presenting a very low variability. This condition may be related to the fact that the specimen used was the filament instead of the cut macrofiber. So, the anchorage of the filament at the test machine was in a very good condition. The problem is that the entire filament could only be obtained with the allowance of the manufacturer, which is not always possible to achieve.



(a) (b)

Fig. 3: Load-elongation (a) and stress-strain (b) curves.

Tab.3: Results calculated from the stress-strain curves.

Data	**Result**
Tensile strength	(572.1 ± 11.4)MPa
Modulus of elasticity	(2.63 ± 0.09)GPa
Ultimate deformation	(22.1 ± 1.0)%

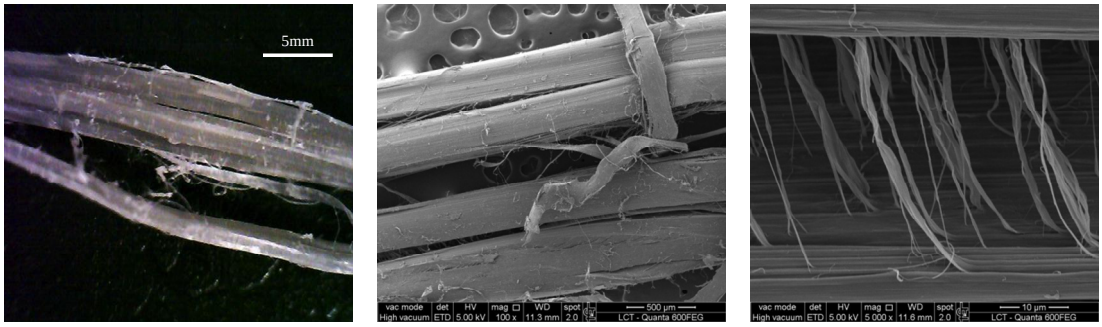
4.3 Analysis by SEM

The pictures taken during the SEM tests are shown in figures 4, 5 and 6. In figure 4, the pictures of the macrofiber in its original conditions, as delivered by the manufacturer, are shown. Then, the pictures of the macrofiber washed out from a fresh concrete mix are on figure 5. Finally, the pictures of the macrofibers after the toughness test are on figure 6.

In figure 4, it can be observed that the original macrofiber surface is rough and irregular and that each filament is composed by multiple microfilaments that are connected in a microscopic network.

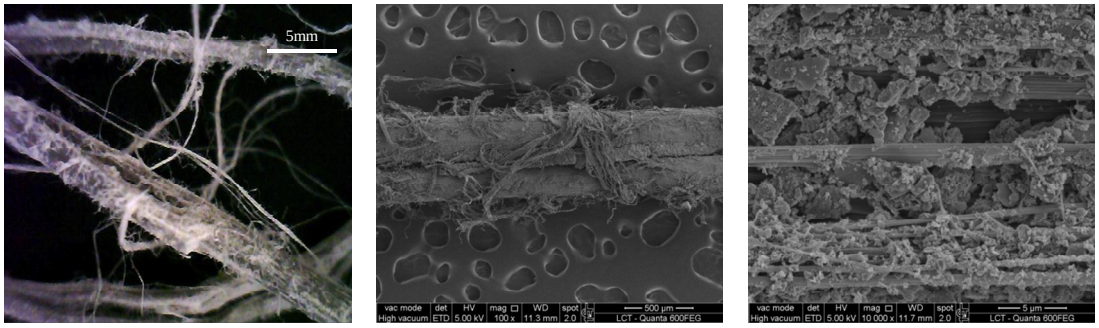
The fibers washed out from a fresh concrete mix (figure 5) present even rougher and more irregular surface, due to the abrasion caused by the mixing with the aggregates. Also, it can be observed that the macrofiber specific surface area increased in the process, when compared with the original macrofiber. So, the cement hydration products could penetrate in between the filaments, promoting a higher adherence between macrofiber and matrix.

In figure 6, it is possible to observe that the matrix surrounding the fiber is very dense, homogeneous and does not present any defects. The void left when the macrofiber is pulled-out is full of microfibers, indicating that two mechanisms may be present in this process: loss of adherence at the interface and rupture of the bond among all the filaments.



(a) (b) (c)

Fig. 4: Pictures of the fiber as received.



(a) (b) (c)

Fig. 5: Macrofiber washed out from a fresh concrete mix.

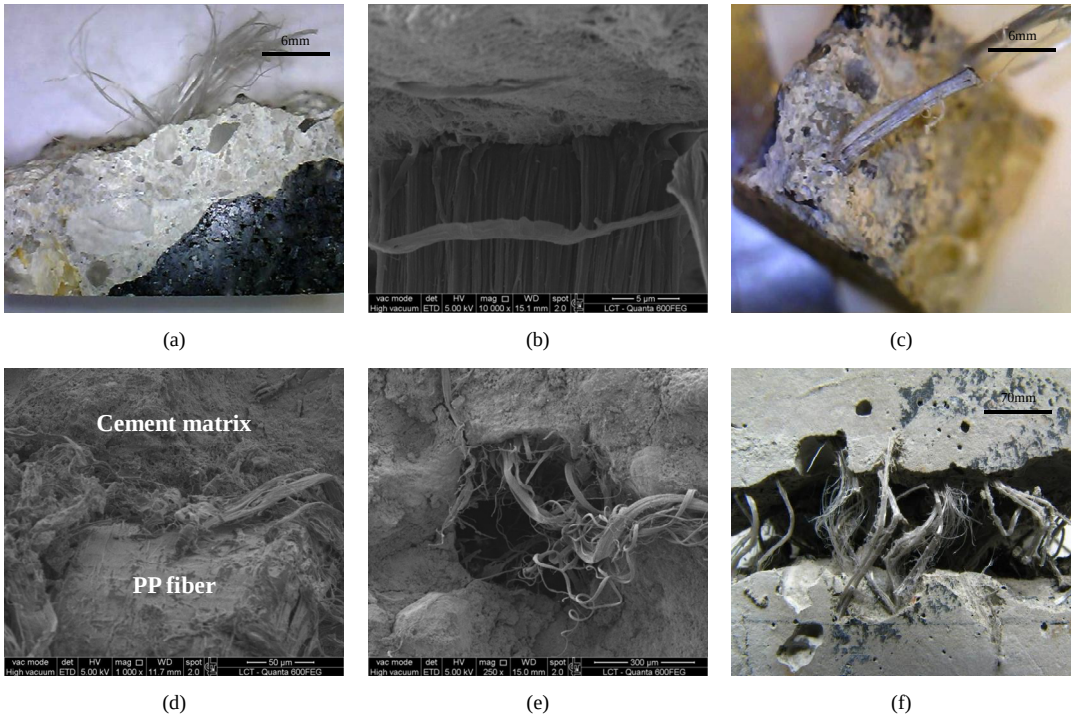


Fig. 6: Fibrillated fiber (a) and its interface with the matrix (b); Pulled-out fiber (c) and its interface with the matrix (d); void in the matrix generated by fiber pull-out (e); rupture section of a beam showing fibrillated fibers (f).

4.4 Compressive and flexural tests

The average compressive strength and the standard deviation of the compressive tests are shown in table 4. It can be observed that the results show a very low variability and that the presence of fibers does not alter that property, whatever the macrofiber content is.

Tab.4: Compressive strength results and standard deviation.

Fiber content / %	(f_{cm} ± sd) / MPa
0.22	31.9 ± 0.5
0.33	28.5 ± 0.2
0.50	30.9 ± 0.4

Fiber content / %	(f_{cm} ± sd) / MPa
0.66	31.3 ± 1.0
0.82	30.5 ± 0.3
1.0	28.7 ± 1.0

The load-deflection curves obtained during the flexural toughness tests carried out with the macrofiber-reinforced concretes with macrofiber contents of 0.22 and 1.0% are shown in figure 7. The average curves of all the series are shown in figure 8. From the analysis of the curves, the modulus of rupture and the residual strengths at the deflections of 0.75 and 3.0mm were calculated. These results are presented on table 5. The correlation between the macrofiber content and the residual strength graphically represented on figure 9.

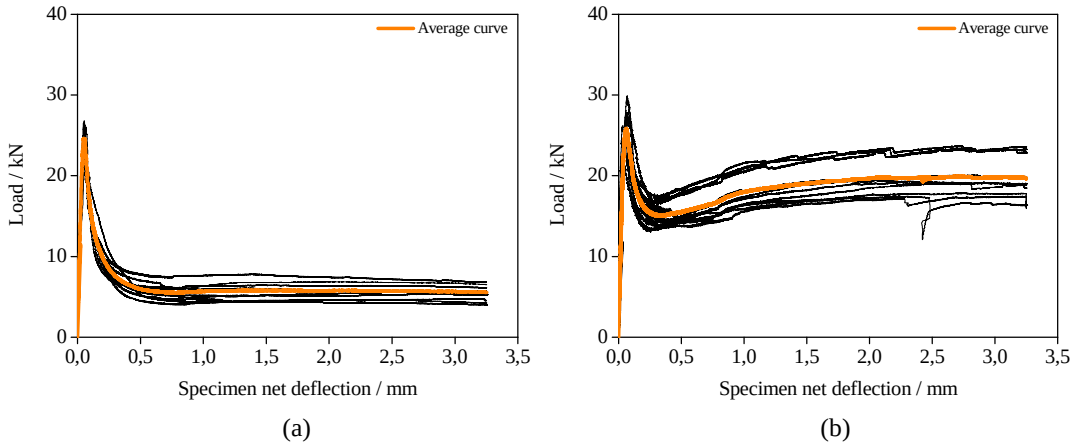


Fig. 7: Load-deflection curves obtained with concrete reinforced with fiber contents of 0.22% (a) and 1.0% (b).

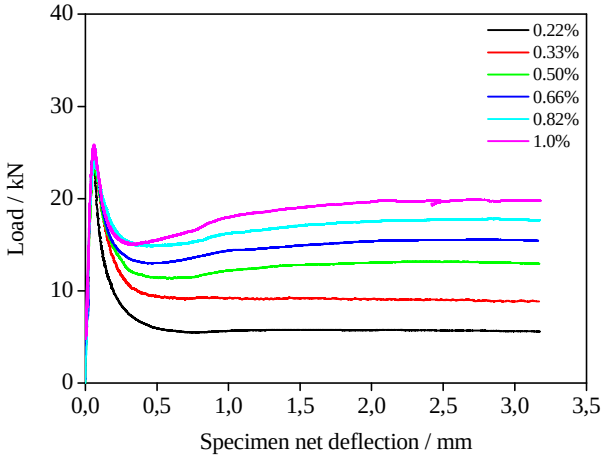
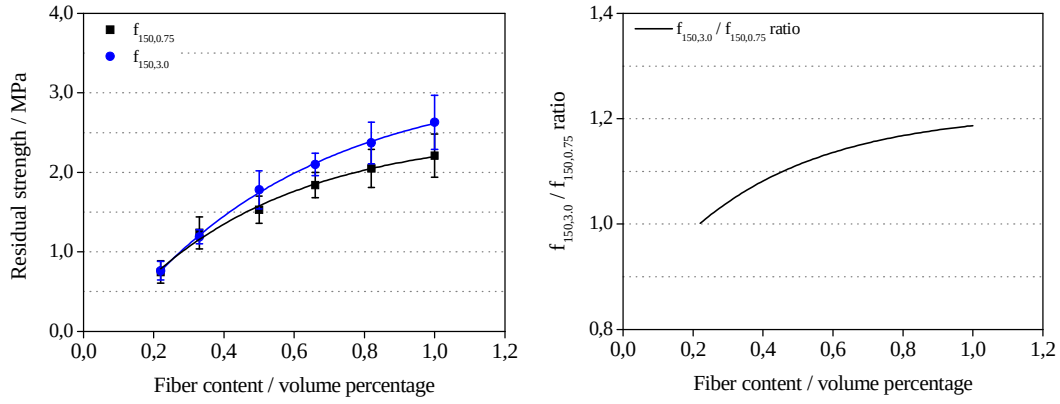


Fig. 8: Average load-deflection curves obtained with concrete reinforced with all the fiber contents used.

Tab.5: Results calculated from the load-deflection curves from figure 7.

Fiber content / %	**MOR / MPa**	**$f_{150,0.75}$ / MPa**	**$f_{150,3.0}$ / MPa**
0.22	3.46 ± 0.14	0.747 ± 0.14	0.764 ± 0.117
0.33	3.37 ± 0.33	1.24 ± 0.20	1.19 ± 0.09
0.50	3.18 ± 0.20	1.53 ± 0.17	1.78 ± 0.24
0.66	3.58 ± 0.12	1.84 ± 0.16	2.10 ± 0.14
0.82	3.36 ± 0.16	2.05 ± 0.24	2.37 ± 0.26
1.0	3.59 ± 0.21	2.21 ± 0.27	2.63 ± 0.34



(a) (b)

Fig. 9: Residual strengths at the deflections of 0.75 and 3.0mm (a) and ratio between them (b).

It can be observed that there is a low variability in the curves and that the post-crack behavior was very uniform, without post-peak instability. All the matrix ruptures occurred by the formation of a single crack, which propagated in a stable manner. Due to the low fiber content, all the beams presented a softening behavior [12].

There is an increase in the residual strength as the fiber content increases, as expected. As it can be observed in figure 9 and in table 5, the residual strengths at the deflections of 0.75 and 3.0mm for fiber contents of 0.22 and 0.33% are practically the same. However, for higher contents, a slip-hardening behavior can be observed. In other words, the residual strength at the deflection of 3.0mm is higher than that measured at 0.75mm. For the content of 1.0%, that difference reaches up to 20%.

The main reason for that behavior is that the load sustained by the cracked composite causes abrasion at the fiber surface due to the friction generated at the fiber-matrix interface during the crack opening. When the fiber content was increased, the probability of rupture of the filaments decreases and the effect of fiber defibrillation causes a more intense effect in the friction process. So, as the macrofiber fibrillation occurs, an increase of the friction effect enhances the pull-out strength of the macrofibers.

5. Conclusions

The present experimental study showed that it is possible to achieve an improvement in macrofiber characterization. In the case of the synthetic macrofiber analyzed in this work, it was possible to verify that its composition includes two immiscible polymers, which is the basis to explain its mechanical behavior. When that macrofiber undergoes a tensile stress, it was observed that the main mechanism that defines its strength is the rupture of the bond that connects all the microfilaments to form a macrofilament. The macrofilament rupture process is accompanied by an increase in its surface area, which improves fiber-matrix interaction when used in concrete. The SEM pictures confirm that behavior, showing that the fiber surface is increased by the abrasion caused when the macrofibers are

mixed in fresh concrete. Also, it was observed that fiber-matrix interface is very dense, homogeneous and presents a similar microstructure to the bulk matrix.

For this reason, an increase in the energy necessary for macrofiber pull-out is achieved. It was possible to observe on the flexural tests a slip-hardening behavior, which occurred for fiber contents above 0.50%.

Acknowledgements

The authors would like to thank the Microstructure Laboratory and the Technological Characterization Laboratory from the Polytechnic School of the University of São Paulo and the company Construquímica Indústria e Comércio de Produtos para Construção.

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