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EVALUATION OF FIBRE-CEMENT COMPOSITES REINFORCED WITH EXPERIMENTAL POLYPROPYLENE FIBRE

J. E. MEJIA[1], G.H.A. BARBIRATO[1], J. FIORELLI[1], H. SAVASTANO JR[1], J. CENSONI[2], C. BULHÕES[2], G.M. BASSO[2], M.A. OVIEDO[2]

[(1)]Department of Biosystems Engineering, University of São Paulo – Pirassununga, SP, Brazil.

[(2)]BRASKEM, Brazil.

Av. Duque de Caxias Norte, 225, Jardim Elite, CEP: 13635-900 Pirassununga-SP, Brazil.
e-mail: julian.mejia@usp.br

ABSTRACT

The use of polypropylene (PP) fibres for reinforcement of fibre-cement composites is a reality in the Brazilian market. As this has been the State of the Art since 2006 in Brazil for certain companies, the development of a new generation of PP fibres has been discussed in this paper. In particular characteristics such as physical-mechanical properties, dispersion in the cementitious matrix and interfacial adhesion engineered to improve the performance of the composite materials are reported in this paper. The experimental fibres are presented in comparison with PP and polyvinyl alcohol (PVA), synthetic fibres currently used in air-cured products. Physical and mechanical properties of these fibres (real density and contact angle) were determined. Fibre cement formulations were prepared using slurry dewatering followed by pressing as a crude reproduction of the Hatschek method in laboratory scale with the assessment of the physical and mechanical performance of the composite materials. The results indicated that the experimental PP fibre under consideration have a commercial competitive potential, based on its characteristics and a similar commercial performance when it is used as reinforcing material of cement based inorganic matrices.

KEYWORDS:

Cement-based matrix, fibre-cement, synthetic fibre.

INTRODUCTION

A wide range of components made of Portland cement has been used in various applications in the construction field (Bagherzadeh et al. 2012). However, the practical use of this type of material exhibits a number of disadvantages in terms of its fragile mechanical behavior, offering a poor deformation capacity, low fracture toughness (Bentur and Mindess 2007;. Fang et al. 2010) and low flexural strength (Bagherzadeh et al. 2012).

An alternative to minimize these effects in the cement performance is the introduction of reinforcing fibres within the cement matrix, producing toughening mechanisms in these cementitious composites that promote a ductile mechanical behavior. In other words, the inclusion of fibres in the cement structure improves the mechanical strength of the composite, and its energy absorption capacity (Bentur and Mindess 2007; Hejazi et al 2012.). The performance of the reinforcing elements depends on the volume, geometry and orientation of the

fibres, Fibre distribution in the cement matrix (Bagherzadeh et al. 2012) and the binding ability between the matrix and the fibres.

Historically, mineral fibres were used as reinforcement (e.g. asbestos) due to their technical performance associated with low cost, but their use has been banned in many countries because of problems related to public health (Jamshidi and Ramezanianpour 2011). An alternative refers to the vegetable fibres. However, due to the high alkalinity of the Portland cement matrix, a degradation of the cellulosic material can occur and these fibres lose their reinforcing capability. In addition, the natural hydrophilic characteristic of this type of fibre causes the detachment of the fibre from the matrix, caused by dimensional variations (Claramunt et al. 2010).

In this scenario, synthetic fibres become an attractive option as reinforcing elements for fragile composites (Motta et al. 2007), of which the most used are polyvinyl alcohol (PVA) and polypropylene (PP) fibres (Ikai et al. 2010). Both types of fibres can achieve good performance and durability within the cement matrix, which can be used individually or in combination with other fibres (e.g. vegetable fibres), with treatments on their surfaces or using reduced-alkalinity matrices (Motta et al. 2007).

PVA fibres have an efficient performance as a reinforcing element since the hydrophilic nature of the PVA resin, that provides adequate adhesion between fibre and cement matrix (Ali et al., 2014). Thus, when the fibre adherence in the matrix exceeds the strength of the fibre itself, its rupture takes place, becoming an undesired situation. In order to guarantee a ductile performance of the material, a gradual pull-out of the fibres is required, resulting in a higher specific energy.

In turn, composites reinforced with polypropylene fibres have shown a positive performance in terms of mechanical properties (Song et al. 2005), promoting ductility and toughness. Although the PVA fibre has higher mechanical performance when compared with PP fibres, PP fibres are widely accepted considering its cost-benefit ratio (Guo-Zhong and Shuai 2010). The explanation for its performance relies on the hydrophobic nature of PP fibres, which protects the fibres from wet cement paste environment (Kakooei et al. 2012). However, this characteristic restrain the formation of stronger links on the fibre-matrix interface originated during the hydration of cement (Mohammadkazemi et al. 2015). However, PP fibres present a favorable feature, which enhances its use as reinforcement: the lower fibre mass fraction (Alhozaimmy et al 1996; Ali et al. 2014).

In spite of the limitations of the use of PP fibres, they are worldwide traded and used, fostering the emergence of new generations of PP fibres that claim to expose a competitive or superior technical performance over commercial fibres. In this work, experimental results of physical and mechanical properties of cement composites reinforced with commercial PVA fibre (PVAcom), commercial PP (PPcom) and experimental PP (PPexp) are presented and evaluated.

EXPERIMENTAL

Materials

Commercial synthetic fibre of polypropylene (PPcom) and polyvinyl alcohol (PVAcom) were used in this research and compared with experimental polypropylene fibres (PPexp). Unbleached Softwood Kraft Pulp was mainly used as filtering element during the water drainage process. Unbleached pine pulp is presented in a dry sheet form with 5% RH. Cementitious matrix was constituted by high strength Portland cement CPV-ARI (NBR 5733 Brazilian Standards) and limestone filler. These mixes have been discussed in detail under the section title Composites Production.

Synthetic fibre characterization

Specific density

In order to determine specific density of the synthetic fibres, the technique by helium gas multi pycnometry, Quantachrom brand, MVP-6DC model was applied. This technique allows determining the density by the means of pressure change experienced by the gas into a known volume chamber.

A sample was weighed on an analytical balance and placed into the pycnometer chamber, where the gas was injected in the sample chamber and then released. Then the expansion valve was opened, releasing the gas into the sample chamber to attain a pressure of approximately 17 psi, thereby obtaining the pressure P1. The gas was released through the valve until pressure stabilization, thereby obtaining the value P2. The procedure was repeated 10 times. With the values of P1 and P2 (in psi), and the volume values of the sample chamber (Vc) and reference volume (Vr) in cm³, it was determined the sample volume (Vp) by using the equation (1):

$$\mathrm{Vp} = \mathrm{Vc} - \mathrm{Vr} * \left(\left(\frac{\mathrm{P1}}{\mathrm{P2}}\right) - 1\right) \qquad (1)$$

After finding the volume of the sample value (Vp), and using the mass of the dry sample, the specific density was determined following the equation (2):

$$Specific\ density: \frac{Mass}{Vp} \qquad (2)$$

Contact angle

For the determination of the contact angles, the Dynamic Contact Angle method of analysis was applied by using a DCAT11 tensiometer equipment, DATAPHYSICS brand, which is based on the Wilhelmy plate technique. This technique allows evaluating the molecular interaction between synthetic fibres under study and water contact. The technique uses the principle of wettability, defined as the macroscopic manifestation of the molecular interaction between solids and liquids in direct contact at the interface between them. The cohesive forces in the liquid tend to form a spherical drop, since the adhesive forces between liquid and solid latter tend to spread over solid. Thus, the contact angle is determined when these forces are competing.

Liquid bromonaphthalene (nonpolar) and deionized water (polar) were used to directly obtain the value of the dispersive component. A single synthetic fibre at a time was set in a nickel-chromium wire and hung on electro-balance tensiometer, (Single Fibre Measurement Method). Then, this fibre was introduced in the standard liquid (deionized water) and their contact angles were recorded by the equipment. It was performed 3 replicates for the PP and 8 replicates for the PVA fibres.

Dispersion grade

A volume of 0.03 cm^3 of fiber was placed in 1.5 L of alkaline water (pH = 11). The mixture was dispersed by mechanical stirring (3000 rpm – 3 min) and filtered over dark cloth to obtain an individual dispersion of fiber sample. The dark cloth was placed in an oven (100°C – 10 min), and after the sample was covered with contact plastic film. Visual comparison of samples was made by "Fiber Standard Dispersion Grade by Kuraray" and classified between levels 1, 2, 3 and 4 (from best to worst quality dispersion)(Figure 1).

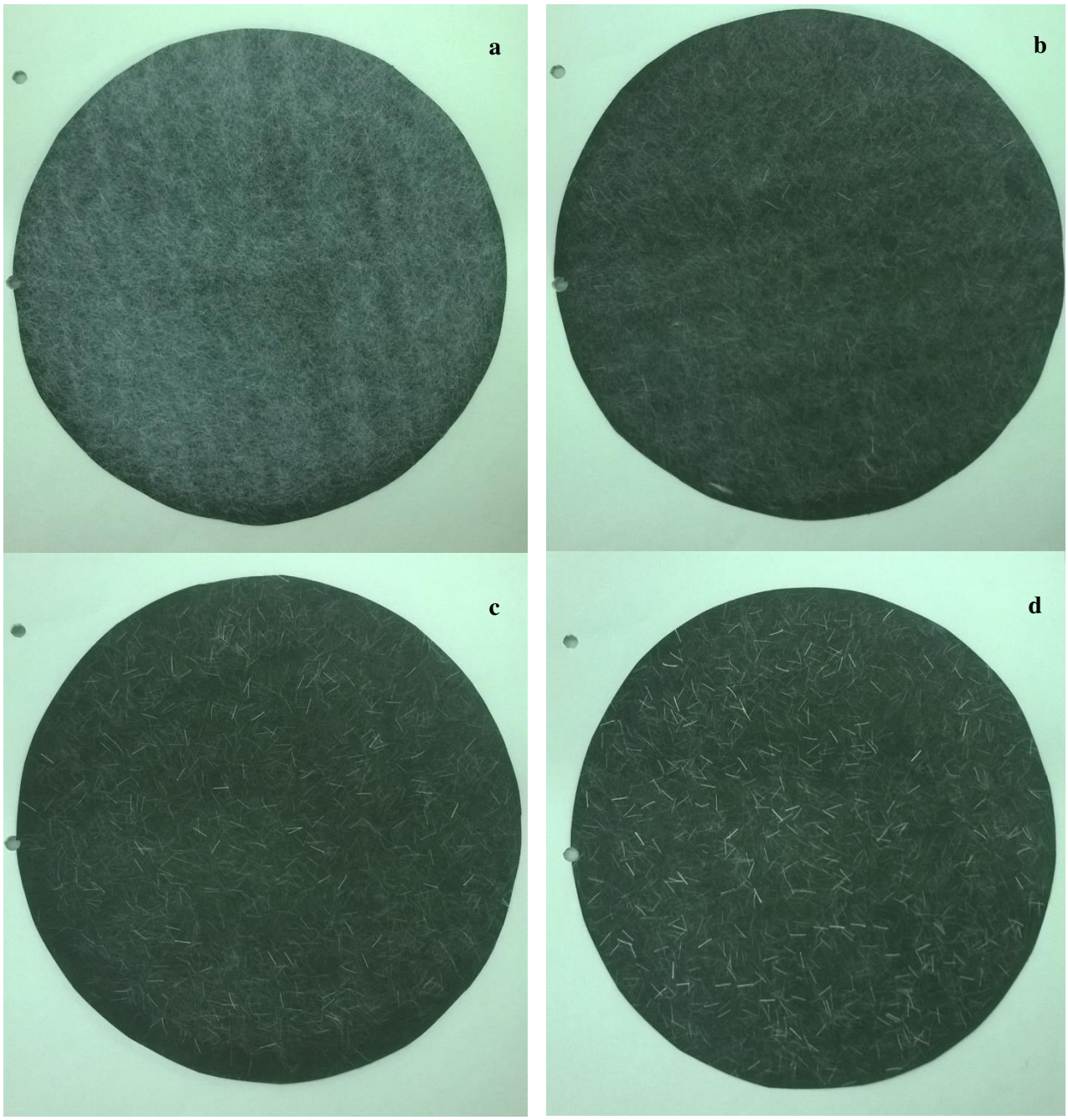


Figure 1. Levels 1(a), 2(b), 3(c) e 4(d) of fiber dispersion standard by Kuraray.

COMPOSITES PRODUCTION

Composition of cementitious matrix

Three sets of samples composed of 12 specimens each were prepared. The purpose of this test was to evaluate the behavior of composites reinforced with unbleached pinus pulp and synthetic fibres, according to their mechanical properties after 7 days of cure. The first set was prepared with PPcom fibre, the second set was prepared with PVAcom fibre and the third one was prepared with PPexp fibre. The experimental composition

of the specimens was fixed by mass percentages as follows: 64.3% Portland cement, 31.7% limestone filler, 3.0% cellulose pulp and 1.4% PP fibre. When using PVA fibre, the experimental composition of the specimens was fixed by mass percentages as follows: 64.0% cement, 31.1% limestone, 3.0% pulp and 1.9% PVA fibre.

Formation of flat plates

For the production and characterization of the composites, flat plates of cement with dimensions of 200 x 200 x 5 mm were molded. The method of production is based on the mixing of the raw materials, excess of water by vacuum drainage and subsequent mechanical pressing (3.2 MPa for 5 min) according to the procedure described by Eusebio et al. (1998). Wet pulps were previously dispersed in water in a disintegrator at 3000 rpm for 5 min. Synthetic fibres were added and homogenized by 4 min at 3000 rpm. Other inputs were added and homogenized for another 2 min at 2000 rpm. The formed suspension was transferred to a molding chamber. For each formulation, plates were pressed individually, and sealed in plastic bags in saturated conditions for two days and then submitted to cure by immersion in water at room temperature for 12 days. Upon completion of the cure period, the plates were cut into four specimens (160 x 40 mm) with water cooled diamond saw. Groups consisting of 12 specimens for each formulation were tested in the saturated conditions (immersed in water for 24 h before mechanical testing).

ESPECIMENS CHARACTERIZATION

Mechanical characterization

The specimens obtained from the flat plates were subjected to mechanical 4 point bending tests, using universal testing machine (Emic DL-30000 model), equipped with a 1 kN load cell and using a deflection speed of 1,5 mm/min. The values of module of rupture (MOR), elastic modulus (MOE) and specific energy (SE) were calculated from the flexural test. For determination of specific energy it was used the recommendation of the RILEM Committee 49 TFR procedure (1984) adapted by Savastano (2000).

Microstructural characterization

Samples of composite fracture surface section were fixed on metallic support (“stub”) using carbon double face tape. Then, the samples were placed in a low vacuum scanning electron microscope (SEM), brand Hitachi - model TM-3000, coupled with x-rays microanalysis system by energy dispersive spectroscopy. Working with low vacuum allows observing the samples without metallic covering. For this case, a voltage of 15 kV was used.

RESULTS AND DISCUSSION

Physical characterization of synthetic fibres

The results of specific density of the synthetic fibres, obtained by Helium (He) gas picnometry technique and contact angle are showed in Table 1.

Table 1. Physical characterization of synthetic fibres

Specimen of Synthetic Fibre	Specific density (g/cm^3)	Contact angle (º)
PVA commercial	1.320	118.1
PP commercial	0.910	90.4
PP experimental	0.954	83.2

The average value of specific density of the PPcom fibre was 0.91 g/cm^3 and 0.95 g/cm^3 for PPexp. It is evident that these values are below than 1.0 g/cm^3, as expected for polypropylene synthetic fibres. In turn, for synthetic PVA fibres, the average value of specific density was 1.32 g/cm^3, a typical value for this type of fibre.

The average contact angle value experimentally determined was 118.1° for PVAcom fibres, 90.4° for PPcom fibres and 83.2° for PPexp fibres. The PPexp fibres showed contact angle values below than 90°, indicating that this fibre surface exhibited the most hydrophilic behavior, and then presenting a good interaction with water. The PVAcom and PPcom fibres shown contact angles greater than 90°, suggesting a more hydrophobic behavior and, therefore, a lower affinity with water compared to PPexp.

As noted in Table 2, the results of fiber dispersion indicate a superior dispersion for PPcom synthetic fibers when compared to the fibers PPexp and PVAcom with 75% degree dispersion in grade 1. The PVAcom fiber showed results between levels 2 and 3 with 43% of the grade results for each grade 2 or 3. PPexp fiber showed 40% of results at level 3 after the dispersion procedure.

Table 2. Dispersion grade of synthetic fibres

Synthetic fibre	Dispersion grade (%)			
	1	2	3	4
PPexp	0	0	40	60
PPcom	75	25	0	0
PVAcom	14	43	43	0

Obs.: Grade 1 stands for the best dispersion and grade 4 for the worst.

Mechanical properties of composites reinforced with treated fibres

Typical tension-strain curves for the different studied formulations of cementitious composites reinforced with synthetic fibre of PVAcom, PPcom and PPexp can be seen in Figure 2.

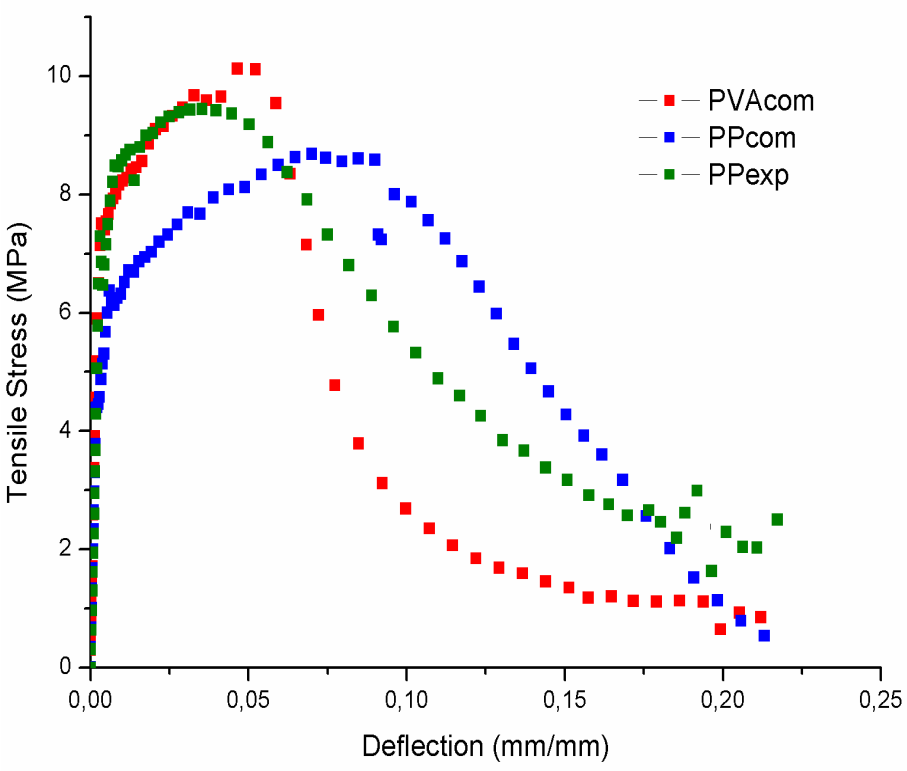


Figure 2. Typical stress-deformation curves for specimens reinforced by synthetic fibre PVAcom, PPcom and PPexp.

In Figure 2, the performance of the cement composite in the elastic region until the first cracking point may be observed. From this point, the bridging effect of fibre reinforcement is predominant and the composite exhibits ductile performance, presenting high deformation and absorption of energy (Hinoki et al. 2002).

Composites reinforced with PVAcom fibres have a typical performance for this kind of reinforcement, being subject to the highest value of flexural strength and lower specific energy. Synthetic fibres of PPcom have a similar performance within the cement matrix when compared to PPexp fibres, showing strength levels lower than those achieved by reinforcement with PVA and higher specific energy values.

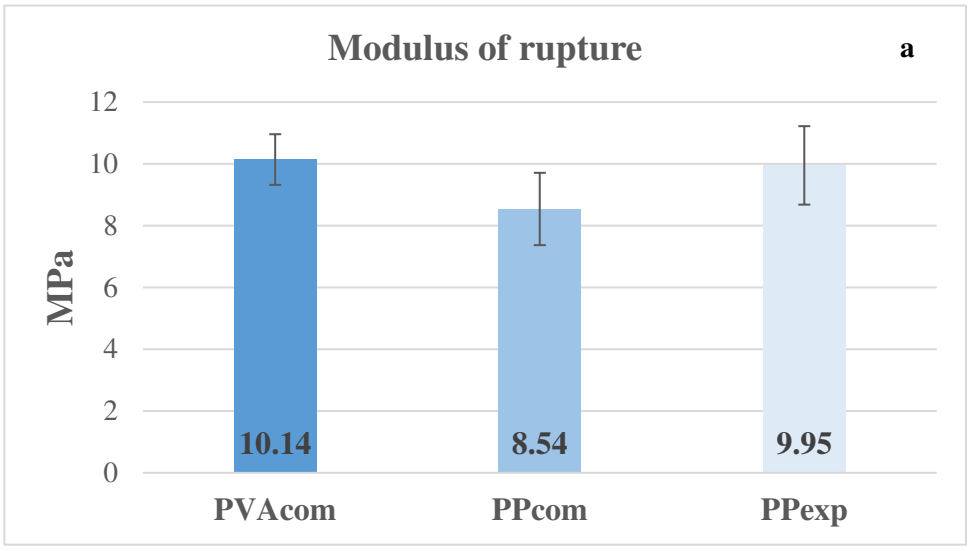
The results of the mechanical tests for the specimens studied are shown in Figure 3 and Table 3, where the average values for the modulus of rupture - MOR (a), modulus of elasticity - MOE (b), limit of proportionality - LOP (c), and specific energy - SE (d) were calculated. These results are seen as a way to evaluate the performance of the cement matrix and the reinforcing effect of the fibres.

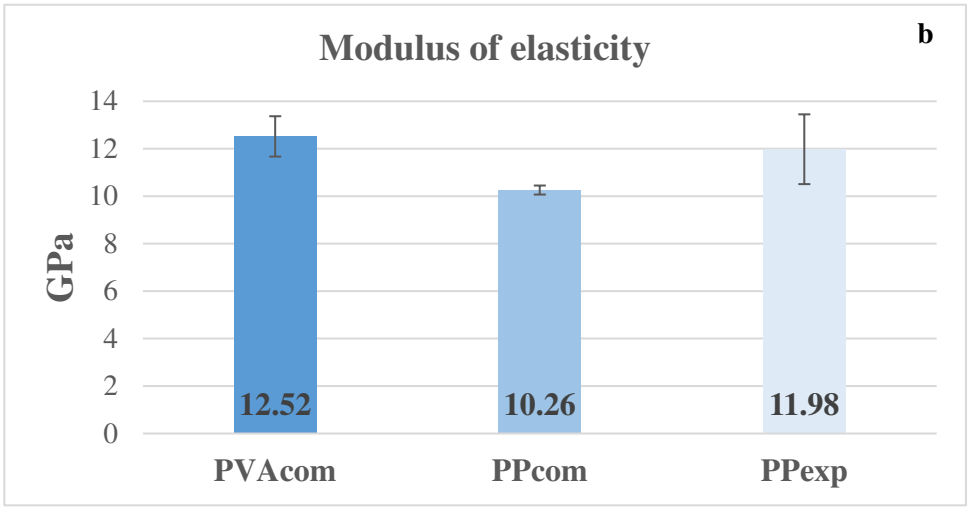
Table 3. Mechanical characterization of composites reinforced by synthetic fibres

Synthetic Fibre	MOR MPa	MOE GPa	LOP MPa	SE kJ m^{-2}
PPexp	9.95±1.27 ab	11.98±1.47 a	6.41±0.91 a	6.53±1.34 a
PPcom	8.54±0.82 b	10.26±0.85 b	4.22±0.77 b	7.66±1.06 a
PVAcom	10.14±1.17 a	12.52±0.19 a	4.87±1.07 b	4.62±0.69 b

*values with different letters in the same column have statistical difference for Tukey test ($p < 0.05$)

In Figure 3(a) and Table 3, composites with PVA fibres showed superior average values of MOR than the composites with PP fibres. Using Tukey statistical analysis, PPexp showed a significant difference when compared with PPcom (PPexp had superior MOR values). Regarding the properties whose performance depends on the cement composite behavior prior to the first crack (LOP and MOE), for MOE, as shown in Figure 3(b), the highest values were found for composites with PVAcom (12.52 GPa), followed by composites with PPexp fibres (11.98 GPa). As noted in Table 3, no significant difference appears ($p > 0.05$) comparing PPexp and PVAcom, different behavior was presented by PPcom with significant variation ($p < 0.05$) when compared with the other fibers under analysis. When the LOP is evaluated (Figure 3c), composites with synthetic PPexp fibres ($p < 0.05$) showed the best performance, with average values 24% and 34% greater than the composites reinforced with PVAcom and PPcom respectively. This can indicate a good interaction and dispersion of the synthetic fibers in the cement matrix leading to a stronger composite with lower cracking propensity. Regarding SE, (Figure 3d), composites reinforced with PVAcom fibres had lower specific energy (4.62 kJ/m^2) with statistic significant difference ($p < 0.05$) as compared to composites reinforced with PPexp and PPcom fibres, indicating the potential of PP fibres as reinforcing elements in fragile inorganic matrices. That can be understood by the higher stretching and lower modulus of elasticity of PP fibres in comparison with PVA fibre.





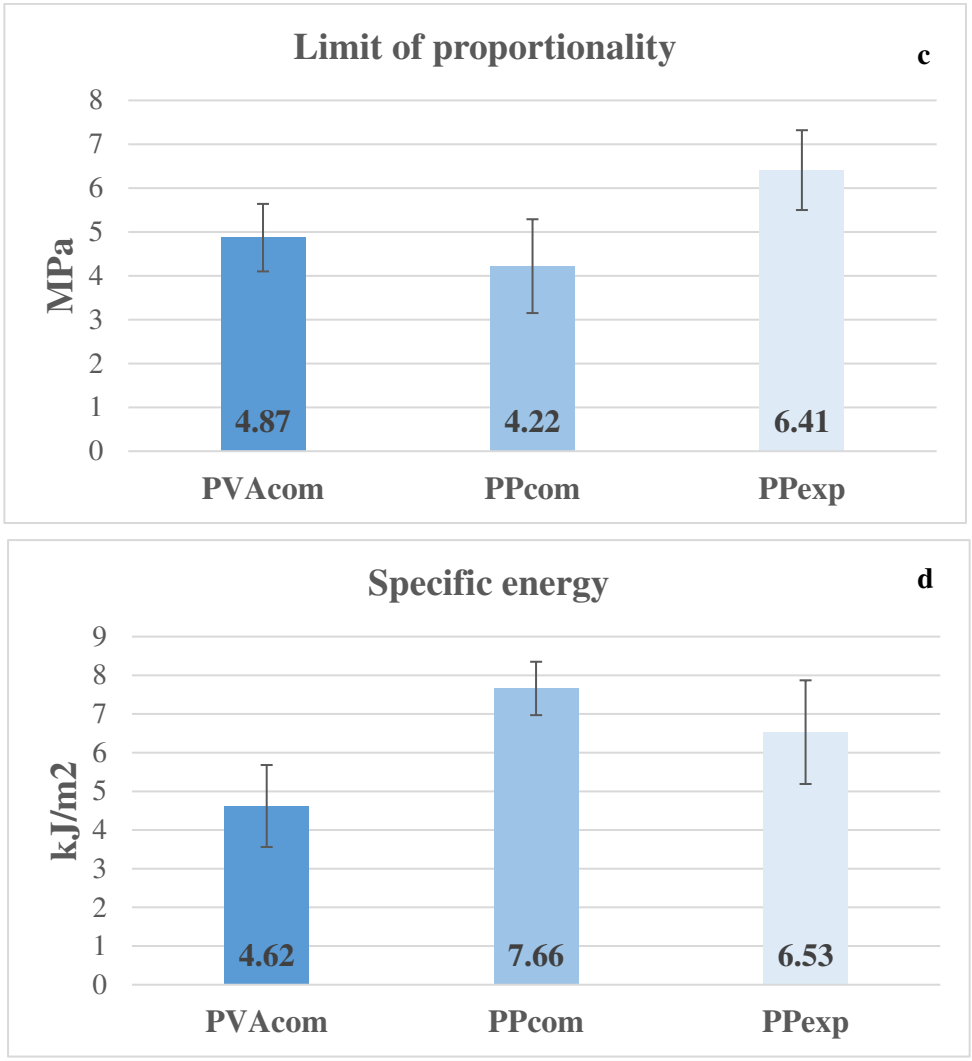


Figure 3. Average values and +/- standard deviation bars of the (a) MOR, (b) MOE, (c) LOP and (d) SE for composites reinforced with PVAcom, PPcom and PPexp fibres.

Microstructure characterization of composites with synthetic fibres

In Figure 4, SEM images magnified up to 500x are presented. Figure 4(a) shows a representative image of the region of fracture after mechanical testing of reinforced cement composites with PVAcom synthetic fibres. In this micrograph, it is possible to observe pine cellulose and synthetic fibres distributed throughout the matrix. The existence of hollows (black arrow) indicates the spots where the fibre pulled out from the matrix as a result of the areas of poor fibre-matrix interfacial bond (white arrow) with a low fiber-matrix adhesion and consequently an increase of the fibre pull out behavior. For the micrographs of synthetic fibres of polypropylene PPcom (Figure 4b) and PPexp (Figure 4c) reinforced composites, it is observed a high incidence of well-adhered fibres to the cementitious matrix and therefore less amount of porosity in the vicinity of the fibre surface. Furthermore, it is possible to observe a stronger fibre-matrix interface (red arrow), represented by a denser transition zone between the fibre and the cement matrix, indicating that the fibre is better adhered, favoring the mechanical performance of the reinforced composites.

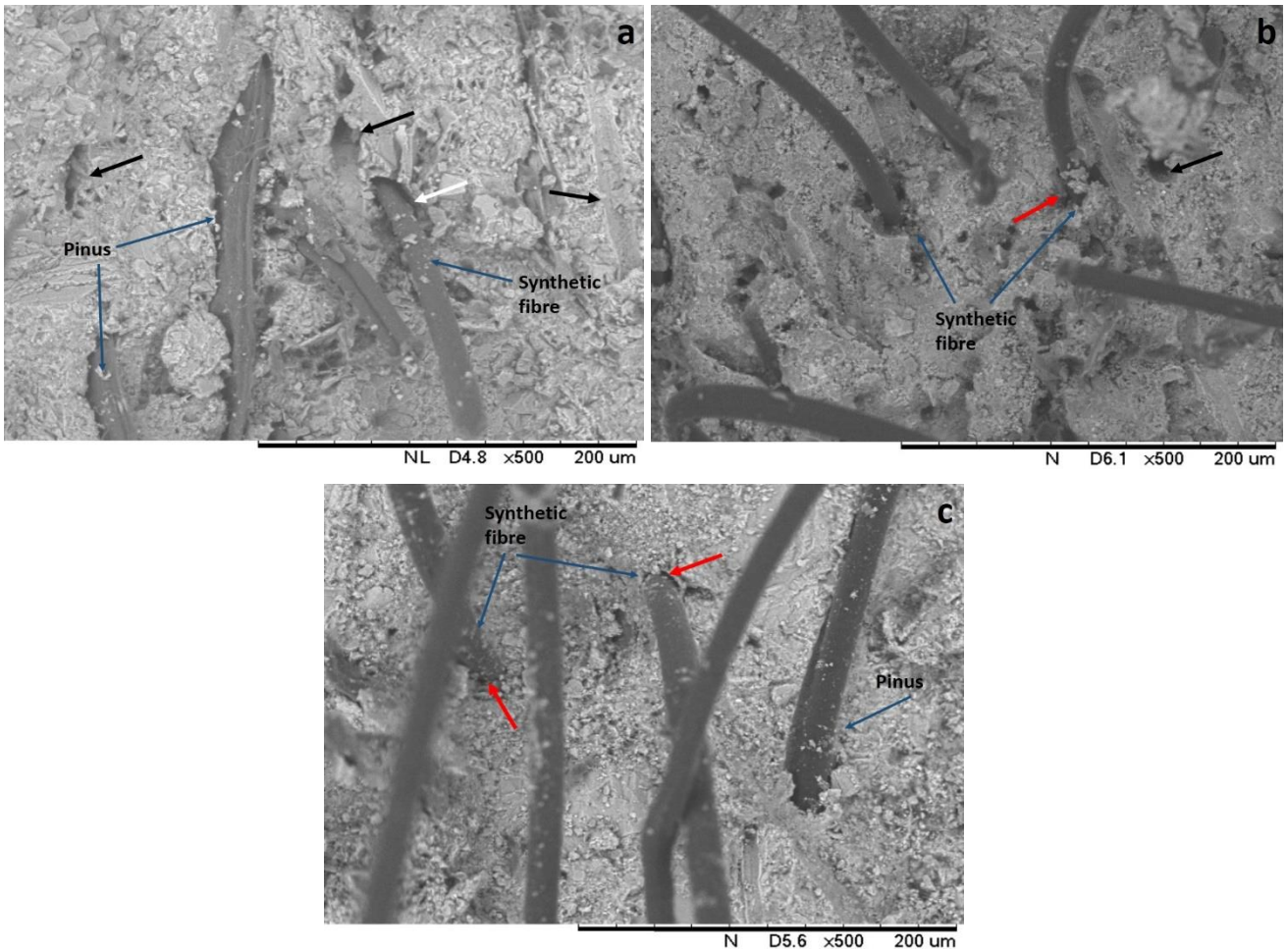


Figure 4. Scanning electron micrographs of composites reinforced with synthetic fibre of (a) PVAcom, (b) PPcom and (c) PPexp (magnification range of x500)

CONCLUSIONS

Based on the results obtained during the development of this work, it is possible to summarize some conclusions:

- The contact angle for PPexp fibres was lower than measured for PPcom and PVAcom fibres, indicating that the surface of that fibre has higher affinity with the water environment existing in the cement matrix.
- PPcom fibers showed the best dispersion behavior with the highest percentage of results for the grade, followed by PVAcom and PPexp fibers between levels 2 and 3.
- PPexp reinforced composites presented results of MOR and MOE statistically equivalent to PVAcom.
- LOP and SE was higher for PPexp than those composites reinforced with PVAcom. These results indicate a good capacity of reinforcement of the experimental PP fiber.
- PPexp reinforced composites presented higher MOE and LOP values when compared to PPcom reinforced composites. On the other hand PPcom composites had higher SE values than PPexp.
- The micrographs of the fracture surface of the composites indicate good adhesion of PPexp fibres with the cement matrix with less porous interfacial zone, favoring the mechanical performance of the composite.

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