EVALUATION OF THE MECHANICAL BEHAVIOUR BY MEASURING THE ENERGY OF FRACTURE AND IMPACT ENERGY OF FIBRE-CEMENTS

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ABSTRACT

The objective of this work is to discuss the measurement of energy of fracture and energy of impact as parameters to evaluate the mechanical behaviour of fibre-cement composites with hybrid reinforcement, before and after accelerated ageing test with exposure to 200 soak & dry cycles. Fibre-cement composites were formulated, reinforced with commercial fibres of polyacrylonitrile (PAN), polyvinyl alcohol (PVA) or polypropylene (PP) and refined pulp of short fibres (eucalyptus pulp, 55 ºSR). The energy of fracture, γ_{WoF}, was obtained with single edge notched specimens and three-point bending tests, to assure stable crack propagation. The behaviour of composite under dynamic load was evaluated by using Charpy impact test. The results were also used to analyse the interaction between the crack and microstructure. It was found a similar mechanical behaviour between composites with PVA and PP fibres, in relation to the composite with PAN fibres.

KEYWORDS

Energy of fracture, eucalyptus pulp; polyacrylonitrile fibres; polyvinyl alcohol fibres; polypropylene fibres; durability.

INTRODUCTION

Fibre reinforced cementitious materials are essentially brittle materials whose mechanical behaviour dependent on matrix, shape and distribution of the fibres and fibre-matrix interface. Therefore, fibre-cement, being a complex-structured composite, has presented a challenge to those who have used models base on fundamental mechanical properties. Similar problem exists in many other material systems such as metal and polymer. Furthermore, fundamental mechanical properties could bring more efficient application of various types of fibre and provide an insight into a microstructure influence on fracture process (Bentur and Mindess, 2007). Among the various mechanical properties of these materials, the energy approach stands out not only as the most suitable measurement for evaluating materials with complex microstructures, such as those of fibre reinforced cements (FRC), but also as an important parameter to indicate aspects of durability. For this reason, many experimental methods have been studied to evaluate toughness in FRC at various load conditions, dynamics and quasi-static.

Impact energy is a measure of the work employed in the fracture of a specimen under dynamic load condition. The specimen absorbs energy until it yields and, when it can absorb no more energy, fracture occurs. The impact tests can be divided into three categories: qualitative, semi-quantitative and quantitative, depending on the property measured rather than on the method by which the impact is applied (Bentur and

Mindess, 2007). The most common impact test categorized as semi-quantitative is the Charpy test. The energy involved in breaking the specimen is determined by the height difference of the pendulum before and after the impact, which implies that all of the energy lost by the pendulum is consumed to breaking the specimen. It is used more as a comparative test rather than a definitive test. Charpy tests were originally developed to test small metal specimens; they can be applied without any modification of the testing machine to thin fibre-cement sheets, as recommended by Rilem (1984). The values of total energy obtained in Charpy tests may be divided by the cross-sectional area of the specimen. This value is of physical significance on the face of the specimen, expressing the fracture energy per unit area of the material. Nevertheless, the results of such tests cannot be considered to represent a material property, since they are highly sensitive to the specimen´s geometry, testing configuration and rigidity of the testing machine (Bentur and Mindess, 2007).

In quasi-static load conditions, it can be determined the energy of fracture, γ_{WoF}. The γ_{WoF} can be defined as the average work per unit of area to propagate a crack, represented by the sum of distinct energies consumed during the fracture process, under stable crack propagation. The γ_{WoF}, can also be expressed as a sum in Equation 1 (Sakai and Bradt, 1993; Barinov, 1993).

$$\gamma_{WoF} = \gamma_0 + \gamma_p + \gamma_R \qquad (1)$$

where these energies are normally the intrinsic surface energy (breaking of chemical bonds), γ_0, the energy consumed as a result of plastic microstraining at the tip of the crack, γ_p, and the average energy dissipated by the interaction between the crack and the microstructure, this energy consumed by other irreversible processes, particularly in the wake region, γ_R. The γ_R represents the average energy consumed per unit of area, which is associated with mechanisms that are carried out in the bulk ahead of the tip of the crack. In the case of fibre-cement composites, bridging of crack surfaces by fibres and pull-out are the most relevant irreversible processes. Furthermore, γ_{WoF} can be obtained with specialized test using single edge notched bend specimens (SENB). Unfortunately, γ_{WoF} seems to be dependent on specimen size and geometry. For this reason, large specimens are recommended in an effort to avoid the so-called size effect.

The energy approach may be useful to evaluate durability; since the main mechanical property lost by FRC during the ageing process is toughness. This property is of fundamental importance for FRC because an increase in embrittlement may also result in reduced mechanical strengths. In addition the service life of FRC is expected to be at least more than ten years for general applications, and therefore there is a need to evaluate the long-term performance on the basis of accelerated ageing tests.

The present investigation addresses the use of impact energy and fracture energy, $\gamma_{WoF,}$ as parameters to evaluate the mechanical behaviour of fibre-cement composites reinforced with different polymer fibres before and after exposure to accelerated ageing cycles. This is a strategic approach for the evaluation of the mechanical performance for the understanding and differentiation of composites with three different types of reinforcement.

MATERIALS AND METHODS

Mix designs (Table 1) were established based on commercial formulations used in fibre-cement industry. Eucalyptus pulp and polymer fibres (Table 2) were both previously dispersed in water by mechanical stirring at 1,700 rpm for 1.5 h and 30 min successively. The mixture formed with approximately 20% of solids was stirred at 1,700 rpm for additional 20 min. The slurry was transferred to the evacuable casting box where vacuum (approximately 80 kPa gauge) was applied until a solid surface formed. The dewatering and pressing technique is described in details by Savastano Jr. et al. (2000). The nominal dimensions of the pad used here were approximately 200 mm x 200 mm x 15 mm. The pad of each formulation were pressed simultaneously at 3.2 MPa for 5 min, then sealed wet in a plastic bag to cure at room temperature for two days and subsequently immersed in water for 50 days. Pads were cut wet into four 165 mm x 40 mm flexural test specimens using a water cooled diamond saw. Samples were kept at ordinary laboratory conditions of 27 ± 2ºC and 65 ± 5% of relative humidity prior to mechanical testing, at 100 days after production.

Table 1 – Mix design of fibre-cement composites reinforced with eucalyptus pulp and polymer fibres.

Raw material	Content (% by volume)
Bleached eucalyptus pulp (CSF 220 mL) [a]	10.5
Polymer fibres	3.6
Ordinary Portland cement (CPII-E)	57.2
Carbonate filler	28.7

[a] The Canadian Standard Freeness test (CSF) is a widely recognized standard measure of the drainage properties of pulp suspensions (Coutts and Ridikas, 1982) and it relates well to the initial drainage rate of the wet pulp pad during the de-watering process.

Table 2 – Typical properties of the polymer fibres.

Fibres [a]	Length (mm) [b]	Diameter (µm) [b]	Aspect ratio	Modulus of elasticity (GPa) [b]	Density (g/cm^3)[b]
PAN	6	12	500	18	1.18
PVA	6	27	223	37	1.30
PP	6	26	231	6	0.98

[a] Commercial fibres used in the present work are polyacrylonitrile (PAN), Dolanit, type 10, produced by Kelheim Fibre, polyvinyl alcohol (PVA), Kuralon KII, REC 7, by Kuraray; and polypropylene (PP) by Saint Gobain. [b] Technical information from producers.

The impact energy tests were conducted in the impact testing machine, Heckert, model PSd 50/15. It was employed a swinging pendulum to strike bar without notch; heights before and after impact were used automatically to compute the energy required to fracture the bar. Consequently, the values of impact energy were obtained by dividing fracture energy by cross-sectional area of the specimen multiplied by two. The nominal dimensions of the samples of the three mix designs used here were approximately 10 mm x 10 mm x 50 mm. Five samples were used for each mix.

The γ_{WoF} was determined based on load versus displacement curves, *Fxd*, obtained under continuous loading in a three-point bending arrangement using a testing machine, Instron, model 5569, equipped with 1 kN load cell. Control by displacement rate of the actuator was adopted for imposing constant velocity as low as 50 µm/min. This low velocity is important to keep crack propagation stable during the test. Also, to ensure this stability, plan notches with 20% depth (in relation to the sample's height) were made using a 500 µm thick diamond disk. The nominal dimensions of the samples used here were approximately 165 mm x 40 mm x 15 mm.

In addition, the γ_{WoF} was calculated based on the work done by the test machine to completely break the sample under stable crack propagation conditions divided by the projected fracture surface area and multiplied by two. The *Fxd* curve was integrated up to the point where the load decreased to 10% of the maximum load reached during the test, thereby eliminating spurious consumption of energy produced by friction among grains in the upper part of the sample at the end of the test.

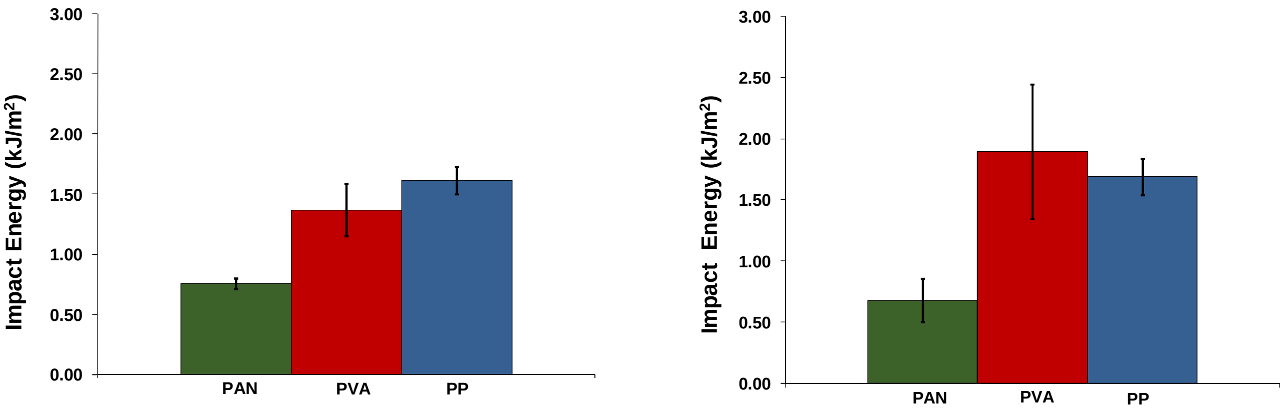
The mechanical performance of the composites was compared before and after exposition of the samples to accelerated ageing cycles. Specimens were successively immersed in water at 20 ± 5^{o}C during 170 min, followed by the interval of 10 min, and then exposed to the temperature of 70 ± 5^{o}C for 170 min in a ventilated oven and with the final interval of 10 min. This procedure was based on recommendations of the BS EN 494 (1994) Standards. Each soak/dry set represents one cycle and was performed for 200 times (i.e., 200 cycles).

Scanning electron microscopy (SEM) was applied for the characterization of fibre-matrix interface on polished and fractured surface of specimens, similarly to the procedures used in Savastano Jr. et al. (2005).

RESULTS AND DISCUSSIONS

The measurement of the impact energy (i. e. the energy absorbed during fracture to generate two new surfaces) of the composites was obtained as can be seen in Figure 1. Although, the impact machine has an instrumented test system, it is necessary to carefully analyse such results because the overall energy lost by the pendulum is expended not only in breaking the specimen, but also in other processes. The most notable of these additional processes is the one by which the specimen acquires kinetic energy due to inertial loading associated with its acceleration during the impact event (Bentur and Mindess, 2007).

Taking in account the significant standard deviation of the measurements, no difference of mechanical behaviour was found between FRC with PVA or PP fibres. However, it was eminent the superior performance of them in comparison with the ones reinforced with PAN fibres. An explanation for the difference in performance is that PAN fibres presented poor bonding with the matrix, probably due to their surface properties and chemical composition.



(a) (b)

Figure 4 – Comparison of the impact energy of fibre cement between (a) unaged and (b) after 200 ageing cycles.

Besides, it can be observed in Figure 1b that there is subtle more variation in the standard deviation of the measurement of impact energy of all treatments of FRC submitted to 200 ageing cycles. This fact can be related the random effects of accelerated ageing test on microstructure in each FRC sample.

Figure 2 shows SEM micrographs of fracture surface of the composites submitted to impact testing after 200 ageing cycles. Figure 2a depicts the fracture surface of the FRC with PAN fibres with indicatives that the fibres suffered bit degradation on their surface. In high magnification, it can be seen a structure of multifilament of the individual fibre with a tiny fibrillation (Figure 2b).

Figures 2c and 2d illustrate fracture surface of composites reinforced with PVA fibres. It was observed considerable number of fibres that were broken instead of the frictional slip provided by pull-out. Thus, it seems that there was sufficient bond between the PVA fibres and the cement matrix to induce a good mechanical anchorage. Therefore, it can suggest that the rapid cracking of the matrix did not permit a frictional fibre-matrix stress transfer, due to anelastic behaviour of the FRC. Assuming that pull-out mechanism needs a long time to occur a greater part of the energy of fracture was consumed to break the PVA fibres.

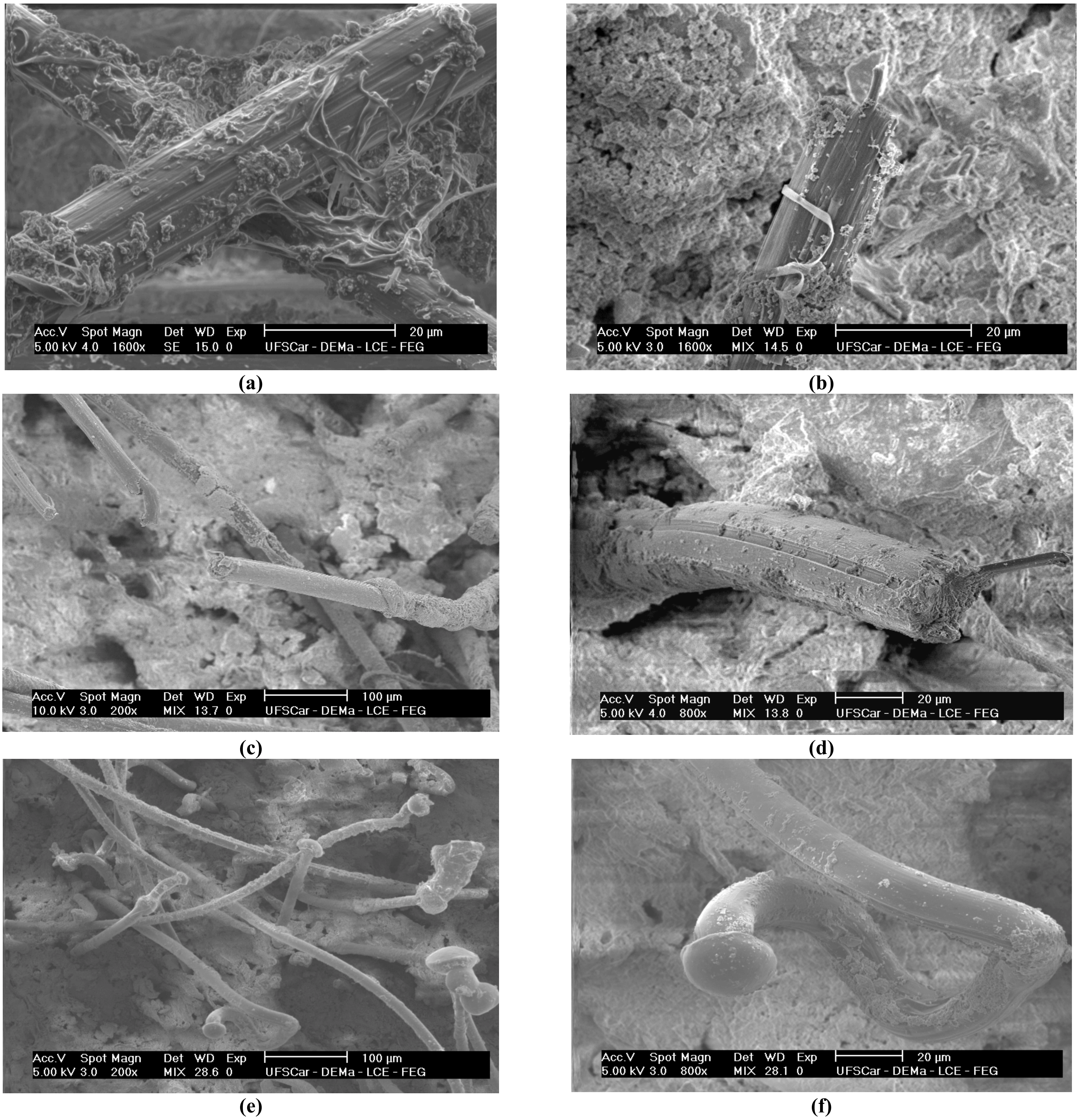


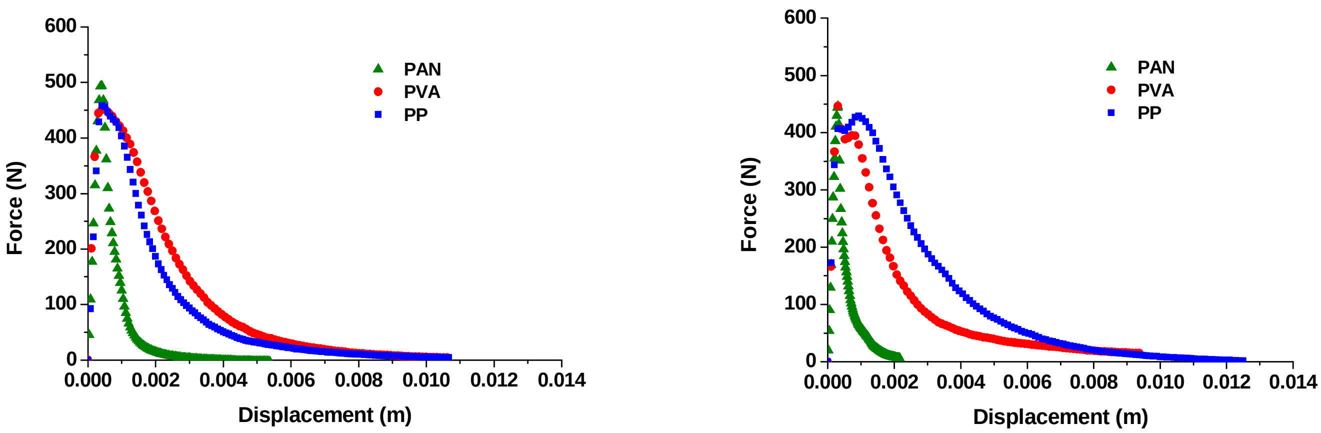
Figure 2 – SEM micrographs of the fracture surface of composites after 200 ageing cycles, reinforced with: PAN fibres (a) and (b); PVA fibres (c) and (d); PP fibres (e) and (f).

In FRC with PP fibres the bond with matrix was not only the result of interfacial adhesion. A considerable contribution may be due to mechanical anchoring and interlocking effects. It is reasonable to assume that such effects improve the bonding with the matrix by providing stretching of the fibres (Figure 2f). The most part of impact energy was due to the additional work required to stretch the PP fibres bridging the two surfaces of the crack (Figure 2e).

Despite of the matrix's brittleness, it was possible to achieve stable crack growth in the FRC for each mix design using single edge notched bend specimens (SENB) and displacement´s control with low test velocity. Figure 3 shows the shapes of the *Fxd* curves obtained from the stable crack propagation tests. The *Fxd* curves are important to characterize the mechanical behaviour, which reflect the effect of fibres on the toughness of the FRC and its capacity of crack control. The areas under those curves represent the total work done by the test machine to completely break the samples. It is observed the significant variation in the area of the curves between unaged and aged FRC, especially for those reinforced with PP fibres, revealing a subtle difference.

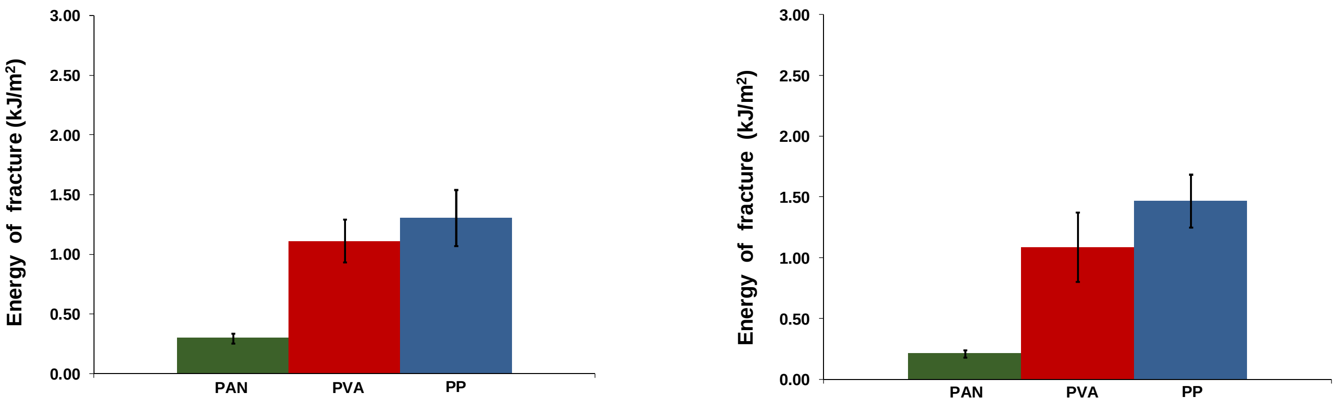
It can be seen in Figure 4 the average values of energy of fracture in unaged and aged FRC. According to the standard deviation of the measurements, there is no significant difference between unaged FRC and those submitted to 200 ageing cycles, except for FRC reinforced with PAN fibres. It is a similar behaviour if compared with impact energy in Figure 1.

The stable crack propagation tests indicate that the most part of energy of fracture was consumed by the combined elastic and frictional stress transfer mechanisms from matrix to fibres. This fact is illustrated in Figures 5a and 5c, which clearly show the pull-out mechanism in the fracture surface of the FRC reinforced with PAN fibres and PVA fibres respectively.



(a) (b)

Figure 3 – Typical load versus displacement curves for the composites: (a) unaged; (b) after 200 ageing cycles.



(a) (b)

Figure 4 – Comparison of the energy of fracture of fibre-cement: (a) unaged and (b) after200 ageing cycles.

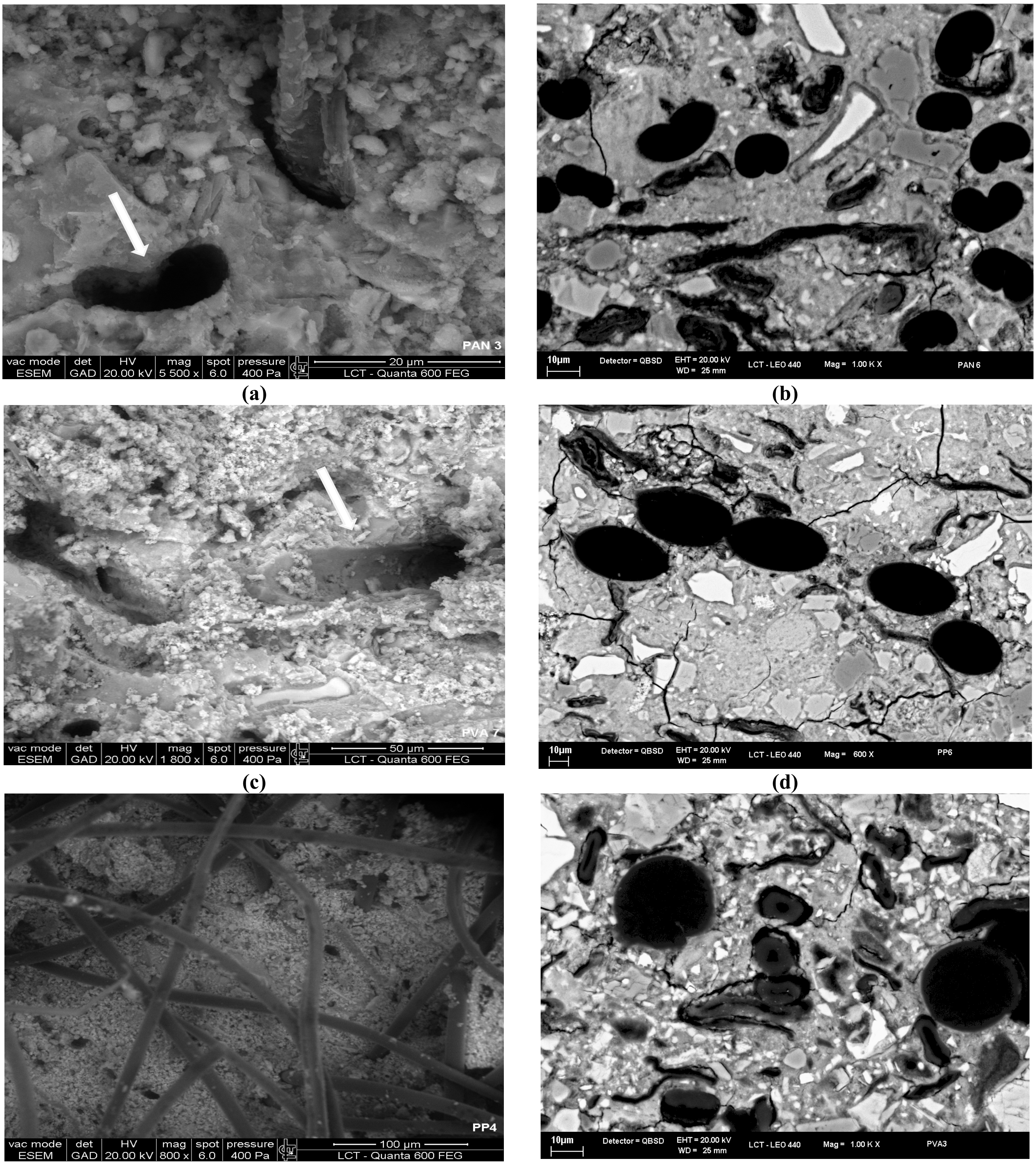


Figure 5 –SEM micrograph in the gaseous analytical detector (GAD) and back-scattered electron (QBSD) mode of the microstructure of composites reinforced with: PAN fibres (a) and (b); PVA fibres (c) and (d); PP fibres (e) and (f), after the stable crack propagation tests and accelerated ageing.

In the stable crack propagation tests, the additional work for the entire fracture of the sample of FRC with PP fibres was mainly used to stretch PP fibres as bridges between the two surfaces of the crack similarly to impact testing (Figure 5e). Thus, low modulus of elasticity of PP fibres combined with the considerable adhesion to the matrix improved its capacity to stretch during the slow fracture process.

There is no evidence of difference of interface between the fibres used in the current work and the matrix by SEM micrographs. Figures 5b, 5d and 5f show SEM micrographs in back-scattered mode of the microstructure of the FRC, respectively with PAN, PVA and PP fibres. Possible difference in the fibre-matrix interface could be clearly defined by measuring either porous transition zone or density of layer of calcium hydroxide in the space around the fibre in the interface (Bentur and Mindess, 2007).

The lower mechanical performance of FRC reinforced with PAN fibres in both conditions of load can be associated to their smaller diameter, higher aspect ratio and transversal section in bean shape (Table 2 and Figure 5e). These geometric characteristics can interfere strongly in the anchorage of the fibre in the matrix.

In fact, the quasi-static load test is highly different from the dynamic load test, but both can provide complementary information about fracture process of FRC. It is important to emphasize that the results of γ_{WoF} and impact energy presented here are not intrinsic mechanical properties of the composites because the methodologies applied (especially impact energy) were adapted in order to obtain comparative assessment of the effects of the different fibres.

CONCLUSIONS

The quasi-static load and dynamic load tests can provide complementary information about mechanical behaviour of FRC reinforced with different types of fibres. In the stable crack propagation tests the force versus displacement curves are important to characterize the mechanical behaviour, which reflects the effect of fibres on the toughness of the FRC and its capacity of crack control.

Composites reinforced with PVA and PP fibres presented similar energy of fracture and impact energy, both before and after accelerated ageing cycles. In the impact test PVA fibres were broken, but in the case of quasi-static test the pull-out was the main toughness mechanism. The low modulus of elasticity of PP fibres combined with the considerable adhesion in the matrix improved its capacity to stretch during the dynamic and quasi-static condition load. PAN fibres provided lower performance to the composites probably due to their geometric characteristics as well as tiny fibrillations on their surface after accelerated ageing tests.

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