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Degradation process of post-consumer waste bottle fibres used in Portland cement based composites

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Abstract: The degradation of synthetic fibres derived from post-consumer wastes and used in fibre-reinforced cement mortars was studied. Polyethylene terephthalate (PET) and high-density polyethylene post-consumer bottles were immersed in alkaline media: 1 M solution of sodium hydroxide and Portland cement paste. Then the fibres' surfaces were analysed by scanning electron microscopy (SEM). This study showed that the fibres have a progressive surface deterioration over time when immersed in either of the two media. Degradation of fibres immersed in pozzolan-Portland cement pastes was also observed. The results from the degradation process of these fibres show that it was stronger when they were immersed in the Portland cement system (pH = 12.9), even though the pH value was lower than that of the NaOH solution (pH = 13.8), suggesting than the degradation process in Portland cement also depends on the formation of solid hydration products. The preliminary results of this study show overall good behaviour of the studied fibres. This behaviour generally improves when using pozzolans. Considering that fibres and pozzolans are obtained from wastes, and therefore have low economic and environmental costs, the possibility of using these materials in developing countries should be considered. The use of these wastes would, on the one hand, improve the properties of construction materials, and on the other, enable proper management of waste that could otherwise eventually pollute the environment.

Author keywords: Waste PET bottles; Waste HDPE bottles; Degradation cement based composite, Fibre-reinforcement

Introduction

In civil engineering, the use of different fibres as reinforcement in cement matrix has been studied in recent decades, particularly since the use of asbestos was prohibited.

Different fibres are used to produce fibre-reinforced mortar (FRM) or fibre-reinforced concrete (FRC), such as metal, glass, and polypropylene fibres, but research continues on the use of recycled waste fibres, especially those that are difficult to manage (Wang et al. 2000; Siddique et al. 2008). Post-consumer plastic waste acquires more importance, especially due to the high volume needed for storage in landfills and its slow degradation. The use of these plastic materials continues to increase, mainly in the food industry, and they constitute a significant percentage of municipal waste. Thus, the use of post-consumer plastic wastes in the fabrication of construction materials would produce environmental and economic benefits.

Commercial uses of polyethylene terephthalate (PET) fibres and high-density polyethylene (HDPE) fibres in reinforced cement composites do not exist yet, but several groups are researching this topic. The objectives of some studies are related to the recycling of PET by thermal or chemical processes, where fibres are made from flakes of melted material extracted from post-consumer bottles (Ochi et al. 2007; Kim et al. 2008) but this thermal or chemical processes involves a high cost. Other researchers directly employ the flakes of these materials with amorphous geometry as aggregate replacement (Iucolano et al. 2013; Naik et al. 1996; Albano et al. 2009; Liguori et al. 2014; Akçaözoğlu et al. 2010; Hannawi et al. 2010; Frigione 2010; Rahmani et al. 2013) in a similar way that has been done with other plastic waste (Ismail et al. 2008; Saikia et al. 2012).

Post-consumer waste plastic fibres produced by mechanical methods such as short fibre reinforcements have been less frequently studied (Fernández et al. 2010; Foti 2011 and 2013). This process of obtaining fibres allows the method to be employed for self-construction, as occurs with other materials in South American countries like Uruguay.

In a different way, thermal or chemical processes have been used for polymeric matrix composites (PMCs) but these processes are not very common because of their high cost (Avila et al. 2003; Rebeiz 1995 and 1996).

Only a few investigations have focused on the degradation of these polymers when used in cement matrices. Silva et al. (2005) investigated the degradation of fibres in cement-based materials using a by-product obtained in a factory that recycles PET bottles to produce ropes. Their results showed that recycled PET fibres interact with alkaline solutions and the fibre-reinforced mortar resistance, after an initial increase, decreases with the degradation of the fibres. Won et al. (2010) evaluated the long-term durability of recycled PET fibre-reinforced concrete. In their studies, fibre-reinforced cement composites with recycled PET showed low compressive strength in alkaline and sulfuric acid environments. However, Machovič et al. (2008) found that the flexural strength of the composite was increased by alkali hydrolysis of PET fibres in sodium hydroxide and they considered that this degradation increases adhesion of the fibre to the cement matrix in the interfacial transition zone.

Therefore the purpose of this study is to investigate the degradation process of PET and HDPE fibres obtained from cutting up of post-consumer bottles when used in cement composites. To analyse this process, the fibres were embedded in cement-based matrices and were removed after different curing ages at given curing temperatures. Scanning electron microscopy (SEM) was used to examine the surface degradation.

Experimental procedure

Materials

Commercial Portland cement type CEM I-52.5R was used in the Portland cement paste elaboration and five pozzolans were also used in order to prepare cement-based mixtures. Rice husk ash (RHA), waste of fluid catalytic cracking catalyst (FCC), fly ash (FA), sewage sludge ash (SSA), and ceramic brick residue (CBR) were the pozzolans used. Their mean particle diameters are listed in Table 1, and the particle size distributions are shown in Fig. 1.

Four different types of fibres were used:

- Colour PET (CO-P) from post-consumer waste containers of carbonated drink, which has greater rigidity due to the need to withstand the filling pressure.
- Transparent PET (TR-P) from post-consumer waste containers of mineral water, which is more flexible than CO-P.
- Monofilament PET (MF-P) from the manufacture of brooms produced by processing of post-consumer bottle flakes.
- HDPE from post-consumer waste containers of milk drinks.

Fibres were obtained by mechanical cutting. All of them were 6 mm in length. They were selected based on previous experience and preliminary experiments. In fibres with a rectangular section, the equivalent diameter was determined as indicated in UNE-EN 14889-2:2008 (AENOR 2008). To determine the density of these materials, the liquid pycnometer method was used according to UNE-EN ISO 1183-1:2013 (AENOR 2013). Table 2 presents the characteristics of the fibres, and photographs of them are shown in Fig. 2.

Preparation of samples and methods

For the mechanical studies, a plastic mortar was used. In the preparation of the mortar, 450 g of Portland cement was mixed with 1350 g of sand with a water/cement ratio of 0.5, following UNE-EN 196-1:2005 (AENOR 2005). For fibre-reinforced mortars, transparent PET, monofilament PET, and HDPE fibres were added at 2% of the total volume. Specimens of 25 mm × 25 mm × 140 mm were manufactured. They were cured at 20 ± 1ºC and above 95% relative humidity during 28 days.

The flexural and compressive strengths were evaluated in cement-based mortar reinforced with these fibres and compared with those of unreinforced mortars. A three-point flexural test and prismatic compressive test were conducted on an Instron 3382 electromechanical testing machine. The flexural strength was determined on mortar specimens and for the compressive strength measurements the two broken pieces from flexural strength tests were used.

For durability studies of the fibres, two procedures were used. In the first, to analyse the degradation in an alkaline medium, the fibres were immersed in a one-molar solution of sodium hydroxide (1M NaOH, pH = 13.80) for two exposure periods: 15 and 270 days. Colour PET, transparent PET, monofilament PET, and HDPE fibres were employed.

The second method consisted of exposing the fibres to Portland cement pastes and pozzolan-Portland cement pastes (containing 20% by weight of different pozzolans). Transparent PET, colour PET, monofilament PET, and HDPE fibres were embedded in these pastes and were extracted after different curing processes. For extraction of the fibres, a notch was made in order to avoid putting extra tension on the fibre surfaces. In all cases, after extraction, the fibres were washed with a 1.2 M solution of hydrochloric acid to remove cement hydration products and pozzolanic products. Then the surfaces of the fibres were observed by SEM (JEOL JSM 6300). Accelerating voltages of 10, 15 and 20 kV were used to obtain the images and the surfaces were metallized with gold.

Results and discussion

Flexural and compressive strengths

As previously mentioned, flexural and compressive strengths were evaluated in Portland cement-based mortar reinforced with these fibres and compared with those of unreinforced mortars. The flexural modulus of rupture (MOR) and flexural elastic modulus were determined. The maximum resistance to compression and compressive elastic modulus were also obtained. In all fibre-reinforced mortars, the flexural strength increased by more than 10% compared to the unreinforced mortar. As expected, the compressive strength decreased, but by less than 20% compared to the unreinforced mortar. The results of the relative flexural strength and relative compressive strength are shown along with the corresponding elastic modulus in Fig. 3 and 4. The absolute values of the control sample were a flexural modulus of rupture of 7.1 MPa, a flexural elastic modulus of 4.6 GPa, a compressive strength of 57.7 MPa, and a compressive elastic modulus of 1.9 GPa. The results obtained shows that these fibres have great potential for use as reinforced cement mortar subjected to flexural loads. Taking into account this fact, it is very interesting to study the behaviour of fibres in an alkaline environment, produced by Portland cement matrix, in order to evaluate their durability.

Degradation studies.

Immersion in alkaline solution (NaOH)

Immersion of fibres in a one-molar solution of sodium hydroxide (1M NaOH, pH = 13.80) at room temperature was carried out. Degradation of the fibre surface was evident, as can be observed in Fig. 5.

The exposed fibres are compared with the corresponding control fibres (not attacked). Fibre degradation increases with exposure time, as can be expected. In order to study short and long exposure times, periods of 15 and 270 days were chosen. We can see that the magnitude of degradation is not the same for all fibres. TR-P fibres were the most degraded after exposure to the alkaline solution. It can be seen that CO-P fibres were less degraded than the TR-P fibres, but both types of fibres presented a change in surface texture that increased with the duration of exposure. HDPE fibres show less degradation than both types of PET fibres, although an increase in degradation from 15 to 270 days of exposure time was observed. However monofilament PET (MF-P) presents a different degradation. In contrast to CO-P and TR-P, these fibres suffered a progressive defibrillation process with immersion time.

This degradation of PET fibres produced in alkaline conditions is due to scissions at polymer chains by hydrolysis of esther bonds (Al-Sabagh et al. 2008). Apparently this attack is focussed on the fibre surface for CO-P and TR-P by means a topochemical reaction (Hosseini, 2007). Differently, MF-P showed a different degradation pattern: a defibrillation process was carried out probably due to a diffusion of alkaline compounds through the microfibrills which a debounding among them. The consequence was a swelling process of the fibre and total debounding of external microfibrills. HDPE behaviour was different: the greater stability of this polymer against alkaline media (Mark, 2007) made that slight changes were produced after treatment in alkaline medium.

Immersion in cement paste

The relationship between the fibre degradation process and the curing time and curing conditions of the cement paste was studied. In order to investigate the evolution of degradation, the surfaces of fibres embedded in Portland cement pastes were also observed by SEM.

The specimens were cured at a temperature of 20 ± 1ºC and above 95% relative humidity. Different curing times of between 1 hour and 28 days were studied, as shown in Figures from 6 to 9. On the other hand, fibres embedded in Portland cement paste under the same curing conditions were observed at 140 days to study the evolution of degradation (Fig. 10). In all Figures, micrographs of the control fibre (not attacked) are included as reference.

SEM allowed visualization of the fibre surfaces in all conditions. In these micrographs, it can be observed that the degradation occurs gradually during the hydration process of cement and can be clearly seen at 28 days of curing.

Again, transparent PET (TR-P) fibres showed higher degradation than the colour PET fibres (CO-P), and HDPE fibres showed the lowest degradation. Monofilament PET fibres (MF-P) again showed a different type of degradation and the defibrillation also occurs progressively with ageing up to 28 days of curing at 20ºC.

Additionally, all fibres were studied after 140 days in the cement paste. The degradation progressed as shown in Fig. 10. For CO-P, 30 μm wide scrapes are formed from 28 days to 140 days of aging, suggesting that the degradation significantly progresses for longer aging times. TR-P showed a small evolution in this interval, and the eroded sites barely changed in size and shape. MF-P had a different degradation pattern, showing micro-defibrillation of the monofilaments. In this case, the evolution in the 28-140 days was not significant. Finally, HDPE was the most resistant fibre against alkaline medium, and low and generalized surface corrosion was produced after 140 days of exposure.

In general terms, the fibre degradation in Portland cement pastes was stronger than that of fibres immersed in NaOH solution (pH = 13.8), despite the fact that the pH value was lower for the Portland cement system (pH = 12.4). This behaviour suggests that the degradation process in Portland cement is also influenced by the formation of solid hydration products. In this way, moreover the topochemical changes due to scission of macromolecules in the surface of the fibre, mechanical action of main hydrated products (calcium silicate hydrates C-S-H and portlandite) also influenced. This process was specially resulted in CO-P, TR-P and HDPE fibres, whereas for MF-P fibre the degradation level was similar or lower than that observed for NaOH environment. Probably, in the last case, the solid products from cement hydration sealed the voids among the microfibrills and impeded the diffusion of alkaline medium into the filaments.

Immersion in cement paste with accelerated ageing conditions

To study the process of degradation under accelerated ageing conditions, the procedure defined for glass-fibre reinforced cement (Purnell et al. 1999 and 2008) was used. Fibres were embedded in Portland cement paste and curing was performed for 15 days by immersing the cement in hot water at a temperature of 60ºC. Once again, after curing the fibres were removed from the paste and washed with 1.2 M hydrochloric acid solution. They were then observed by SEM and the images obtained can be seen in Fig. 11 (Line A).

Fibres that were embedded in Portland cement paste and subjected to the controlled ageing process (Fig. 11 Line A) showed greater degradation than those exposed to the alkaline solution (Fig. 5). It is observed that, as before, the degradation tendency is that colour PET fibres degrade less than transparent PET fibres and HDPE fibres degrade less than both fibres derived from waste PET bottles. The defibrillation of monofilament fibres can again be distinguished from the degradation of the other three types of fibres.

In fibres that were not washed with hydrochloric acid solution, the remains of the paste deposited within the cavities that were formed by degradation can be observed in Fig. 12.

Immersion in pozzolan-cement pastes

According to the results of the studies presented in previous sections, five Portland cement-based mixtures were made in which 20% by weight of Portland cement was replaced by the pozzolans listed in Table 1: RHA, FCC, FA, SSA, and CBR. The study of the durability of the fibres embedded in mixed paste was based on existing investigations of these materials. The construction studies show a better performance of these mixtures in terms of mechanical strength and durability (Payá et al. 2009). When pozzolanic materials react with calcium hydroxide, which is liberated in the Portland cement hydration, the reduction of portlandite occurs and therefore the pH decreases (Garcés et al. 2011). The purpose of this research is to

assess whether the degradation process decreases with this reduction of portlandite. The use of these pozzolans agrees with investigations on the effects of the addition to Portland cement. These studies confirm the technical viability of using these materials with pozzolanic properties (Payá et al. 2009; Garcés et al. 2011; Gener-Rizo et al. 2002; Puertas et al. 2008). In this work, the fibres were embedded in these pastes and were aged by immersion in water at 60ºC for 15 days to compare them with the same conditions sing Portland cement-based paste. SEM images of these fibres' surfaces are shown in Fig. 11 (lines B to F).

In this case, when the fibres were exposed to cement-pozzolan based pastes, they behaved differently depending on the matrix. This trend does not allow a comparison between fibres of different origins. The lowest degradation does not allow occur for the same cement-pozzolan paste, as shown in the images. Colour PET fibres (CO-P) were degraded less when ceramic brick residue (CBR) and rice husk ash (RHA) were used as pozzolan, and further deterioration occurred when spent catalyst from fluid catalytic cracking of petroleum (FCC) was used. However, the lowest aggression against TR-P is displayed with the use of fly ash (FA) and sewage sludge ash (SSA). However in all cases the degradation is less than that which occurs in the fibres in cement paste. The degree of degradation of HDPE fibres cannot be distinguished in the exposure to the different mixtures but it is also less than the degradation of fibres that were immersed in cement paste under the same curing conditions. This fact suggests that the portlandite reduction, together with the reduction in the hydration products of Portland cement and the generation of pozzolanic reaction products, enhances the durability of these fibres.

On the other hand, the monofilament PET fibres seem to present the same degradation in all cases. The defibrillation that they show is similar to that presented in cement pastes cured under the same conditions.

It can be assumed that the major mechanical effect on the surface of the fibre embbebded in OPC paste is due to the presence of portlandite crystals, which have a notching effect. Apparently, C-S-H and other hydration/pozzolanic products affected much lower to the integrity of the fibres.

Conclusions

From the results obtained, the following conclusions could be drawn:

- In all fibre-reinforced mortars, the flexural strength was increased by more than 10% compared to the unreinforced mortars. So, the contribution of the fibres to this property was significant. However, the fibres affected the compressive strength values negatively, but the decreases were less than 20% with respect to unreinforced mortars.
- The degradation process was stronger in Portland cement than in fibres immersed in NaOH solution (pH = 13.8), even though the pH value of the Portland cement system is lower (pH = 12.4), suggesting that the degradation process in Portland cement also depends on the formation of solid hydration products.
- For TR-P, CO-P, and HDPE fibres, a mixture of Portland cement and pozzolan (80:20 by mass) showed reduced surface degradation. This fact suggests that the portlandite reduction, together with the reduction in the hydration products of Portland cement and the generation of pozzolanic reaction products, enhances the durability of these fibres. However, monofilament PET fibres present similar degradation in all cases.

The preliminary results of this study show overall good behaviour of the studied fibres. This behaviour generally improves when pozzolans are used. Considering that fibres and pozzolans are obtained from wastes and therefore have low economic and environmental costs, the possibility of using these materials in developing countries should be considered.

The use of these wastes would, on the one hand, improve the properties of construction materials, and on the other, enable the proper management of waste that could otherwise eventually pollute the environment.

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330 **Table 1.** Mean particle diameters (μm) of pozzolans and cement.

CEM I	RHA	FCC	FA	SSA	CBR
17.49	9.86	17.11	15.94	52.82	19.83

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333 **Table 2.** Characteristics of fibres.

	Thickness (mm)	Width (mm)	Equivalent diameter (mm)	Density (g/cm^3)
CO-P	0.29	1.00	0.56	1.50
TR-P	0.23	1.00	0.59	1.63
MF-P	0.41 (diameter)	-----	-----	1.28
HDPE	0.48	1.00	0.64	1.07

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336 FIGURE CAPTIONS

337

338 **Fig. 1.** Particle size distribution.

339 **Fig. 2. Samples of fibres.**

340 Fig. 3. Relative flexural modulus of rupture (MOR) and relative flexural elastic modulus.

341 Fig. 4. Compressive strength and compressive elastic modulus.

342 **Fig. 5.** Micrographs of the fibres immersed in sodium hydroxide solution at room temperature.

343 **Fig. 6.** Micrographs of CO-P fibres immersed in cement paste cured at 20 ± 1ºC and above 95% relative humidity.

344 **Fig. 7.** Micrographs of TR-P fibres immersed in cement paste cured at 20 ± 1ºC and above 95% relative humidity.

345 **Fig. 8.** Micrographs of MF-P fibres immersed in cement paste cured at 20 ± 1ºC and above 95% relative humidity.

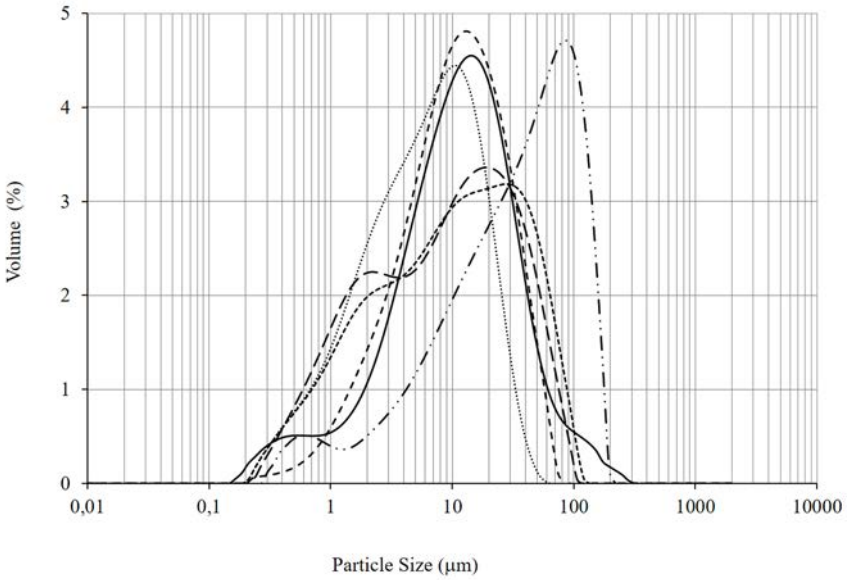
346 **Fig. 9.** Micrographs of HDPE fibres immersed in cement paste cured at 20 ± 1ºC and above 95% relative humidity.

347 **Fig. 10.** Micrographs of the fibres immersed in cement paste cured at 20 ± 1ºC and above 95% relative humidity during
348 140 days.

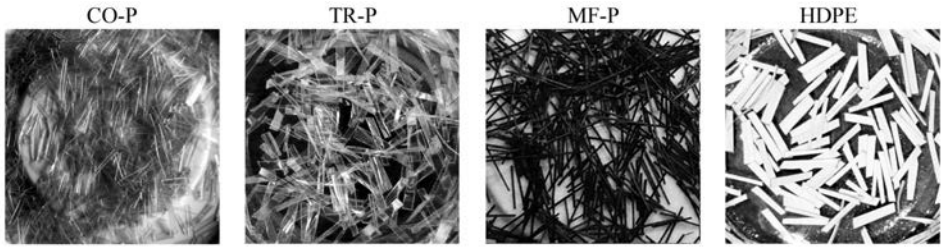
349 **Fig. 11.** Surfaces of fibres immersed in pastes and subjected to hot water controlled ageing at 60ºC during 15 days. (A)
350 Cement paste, (B) cement–RHA paste, (C) cement–FCC paste, (D) cement–FA paste, (E) cement–SSA paste, (F)
351 cement–CBR paste.

352 **Fig. 12.** TR-P fibre immersed in cement paste cured in hot water at 60ºC during 15 days, showing the deposited remains
353 of Portland cement hydration products.

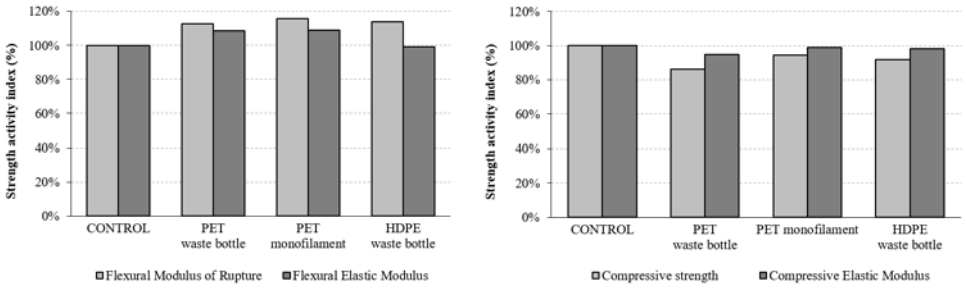
354


Volume (%)
Particle Size (μm)
0,01
0,1
1
10
100
1000
10000
CEM I
RHA
FCC
FA
SSA
CBR

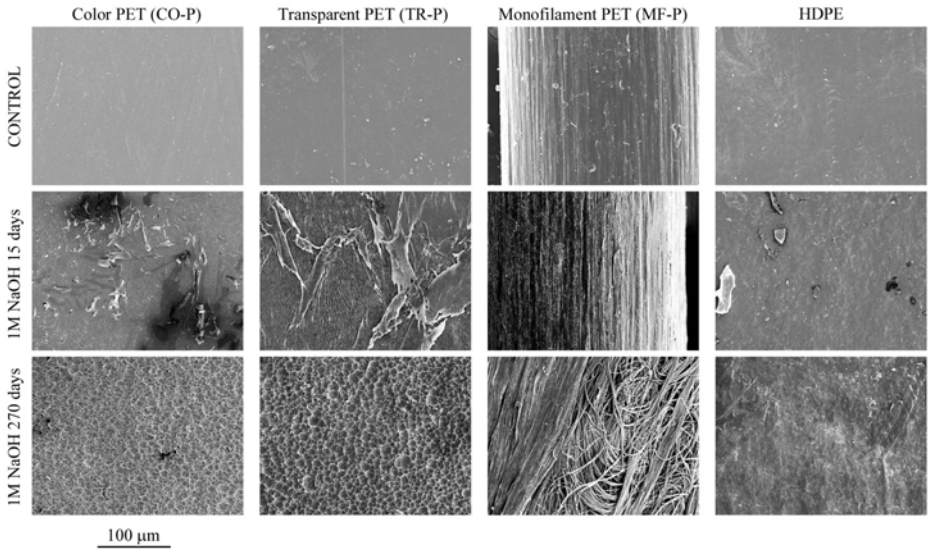
355


CO-P
TR-P
MF-P
HDPE
10 mm

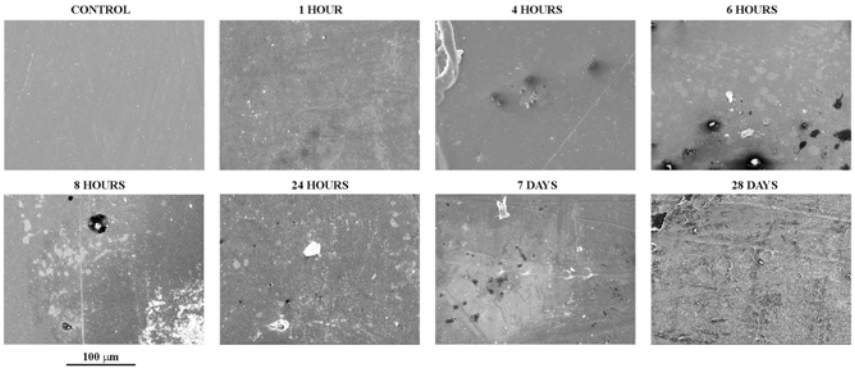
356


Strength activity index (%)
CONTROL
PET waste bottle
PET monofilament
HDPE waste bottle
Flexural Modulus of Rupture
Flexural Elastic Modulus
Compressive strength
Compressive Elastic Modulus

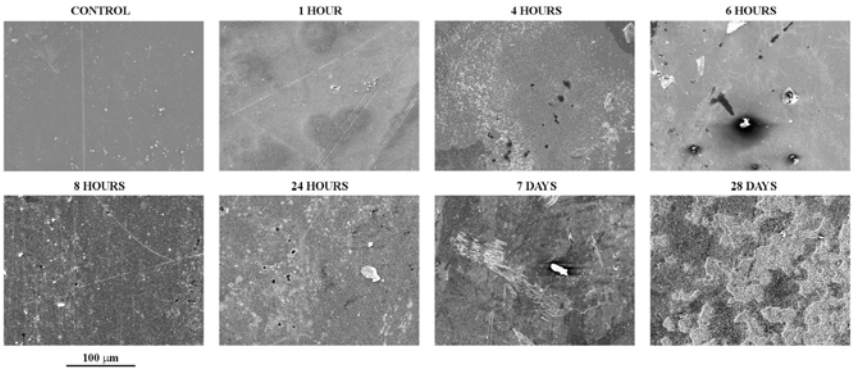
357



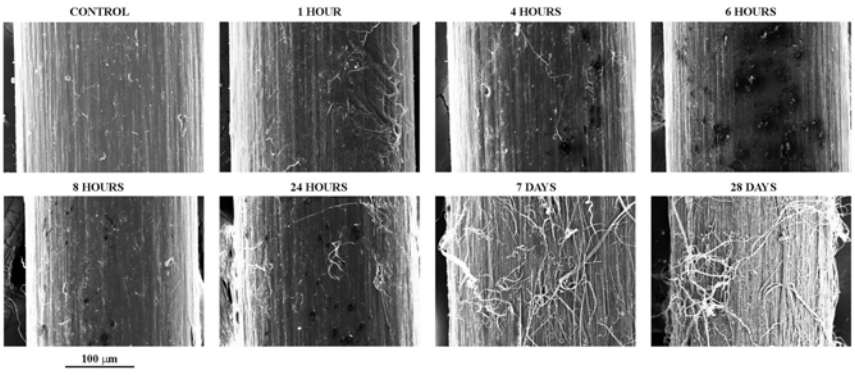
358



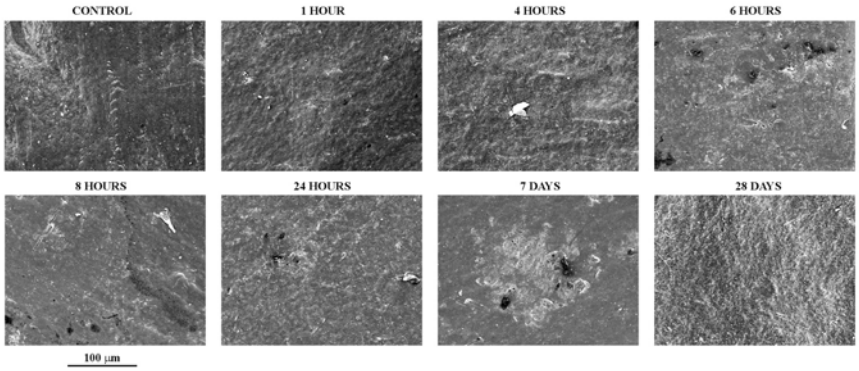
359



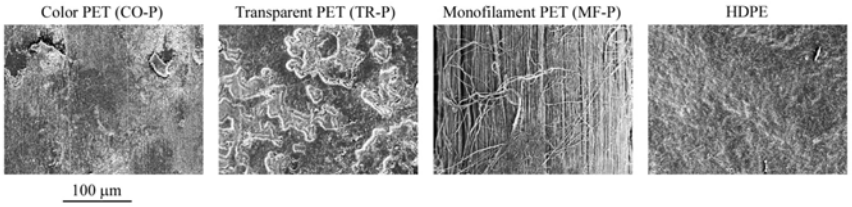
360



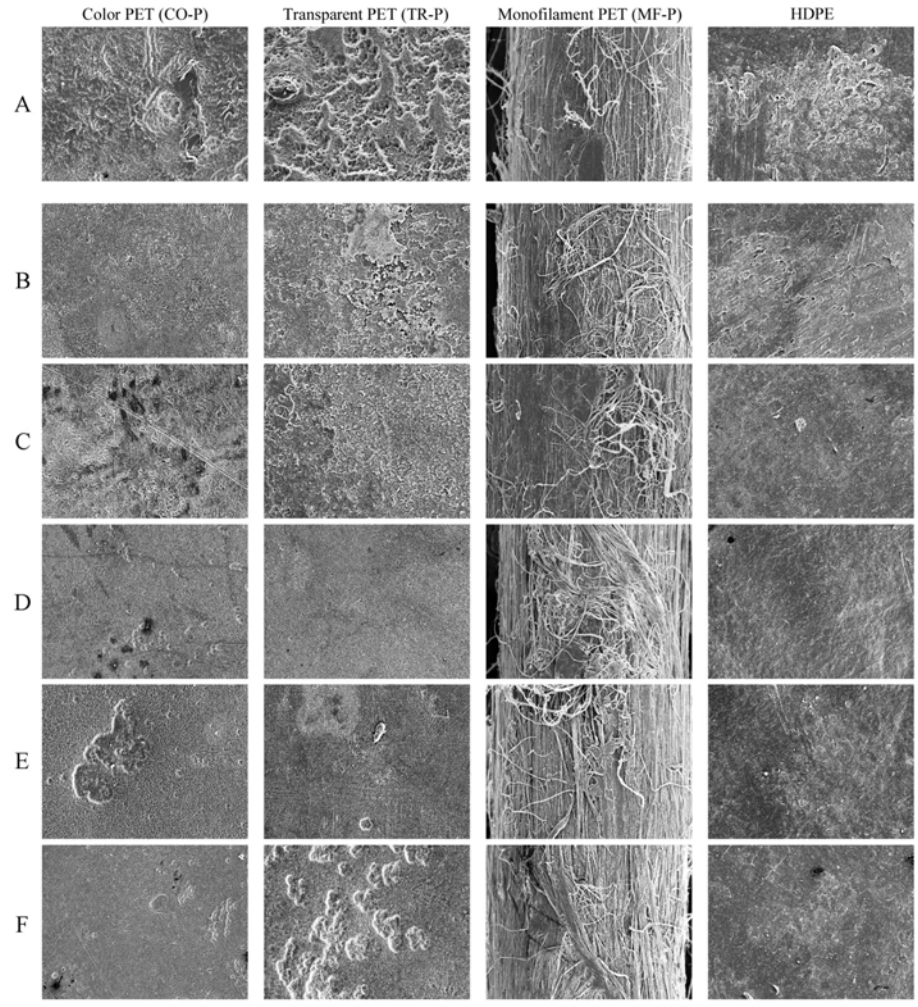
361


CONTROL
1 HOUR
4 HOURS
6 HOURS
8 HOURS
24 HOURS
7 DAYS
28 DAYS
100 µm

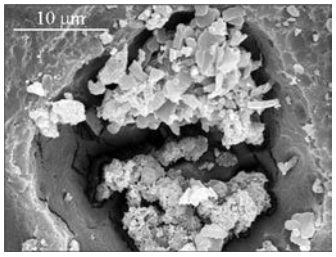
362


Color PET (CO-P)
Transparent PET (TR-P)
Monofilament PET (MF-P)
HDPE
100 µm

363


Color PET (CO-P)
Transparent PET (TR-P)
Monofilament PET (MF-P)
HDPE
A
B
C
D
E
F
100 µm

364


10 µm

365