J Mater Sci (2010) 45:2907–2915
DOI 10.1007/s10853-010-4282-1

Essential work of fracture testing of PC-rich PET/PC blends with and without transesterification catalysts

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Received: 3 November 2009 / Accepted: 27 January 2010 / Published online: 12 February 2010

Abstract 0.7 mm sheets of blends of polycarbonate (PC) with polyethylene terephthalate (PET) rich in PC in the presence and absence of three different transesterification catalysts have been obtained using reactive extrusion-calendering processing method in order to evaluate the fracture toughness of these materials applying the essential work of fracture (EWF) approach which has not been previously reported in the literature. The morphology has been characterized by scanning electron microscopy (SEM). In addition, the tensile properties of these materials were determined. There is a decrease on the essential term (w_e) values of PC/PET blends without transesterification catalysts while blends with transesterification catalysts present an increment in comparison with neat PC which may related to the product of the transesterification that plays like an emulsifier/compatibilizing agent to reduce the interfacial tension between the components of the blend and reduce the interfacial tension between the two immiscible or incompatible component phases to get a better fracture behavior. This is confirmed by the tensile test results obtained which demonstrate higher values for E and σ_y in the case of blends with transesterification catalysts compared with neat PC. For non-essential term of fracture (βw_p), blends without catalysts exhibit an increase compared with neat PC by increasing the amount of PET which may due to the lowering of the yielding stress. In contrary, the presence of transesterification catalysts and especially Zn-based shows decrease as a consequence of the restriction that occurred on the movement of PC segments during the transesterification reactions or as a decohesion of the dispersed phase during the test.

Introduction

Most of the purposes of blending processes are to enhance and modify the physical and chemical properties of the materials [1]. This has the advantage of being much cheaper, quicker, and the physical properties of a blend of known polymers can be easier to predict than the properties of a completely new polymer.

In the 1970s and 80s a series of papers were produced documenting the results of miscibility tests carried out on various pairs of polymers [2–4]. One of the common polymer blends was polyethylene terephthalate (PET) with Bisphenol-A-polycarbonate (PC). Extensive studies have been performed on the behavior of PET and PC blends focusing in, crystallization [5], morphology [6], transesterification reactions [3, 7, 8], compatibility [9], and thermal properties [10]. Blends of PET/PC found to be compatible in rich PET one due to transeterification or process or physical interactions (miscibility) [7]. Many studies confirm that the PET/PC pair results immiscible in the absence of transesterification reactions especially when the blends rich in PC.The miscibility is induced by transesterification reactions and, for this reason, the catalyst activity plays a crucial role on the control of the phase behavior and hence on the properties of the blends prepared by reactive blending [11, 12].

Much of the previous research has focused on the previous aspects when study PET/PC blends and few studies were done to investigate their mechanical properties

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[12, 13], however to our knowledge no work has been published on the fracture behavior of reacted PET/PC blends rich in PC.

In recent years, several researchers have successfully used the essential work concept to characterize the fracture toughness of polymer materials [14–18], so in this article we attempt to characterize and evaluate the tensile and fracture behavior at low strain rates of films of PET/PC blends in the PC-rich range by using or applying of the essential work of fracture approach in deeply double-edge notched tension (DDENT) geometry. This work is a continuation to previous study [19] where thermal, rheological, and thermodynamic mechanical behavior of PC/PET blends were evaluated.

Essential work of fracture (EWF)

EWF approach was developed by Cotterell and Mai [20] following an idea of Broberg [21]. It is used to characterize the fracture toughness of ductile materials. When a ductile specimen containing a crack is loaded, the plastic flow occurs in an outer plastic zone that borders the fracture process zone (Fig. 1). The total work of fracture, W_f, may be separated into two components:

$$W_f = W_e + W_p \tag{1}$$

where W_e is the essential work and is proportional to the ligament length (L) if it is assumed that the specific essential work of fracture (w_e) remains constant. W_p is the non-essential work in the rest of the plastic deformation zone which is proportional to L^2 if it is assumed that the specific non-essential work of fracture (w_p) remains constant. The total work of fracture can be written as:

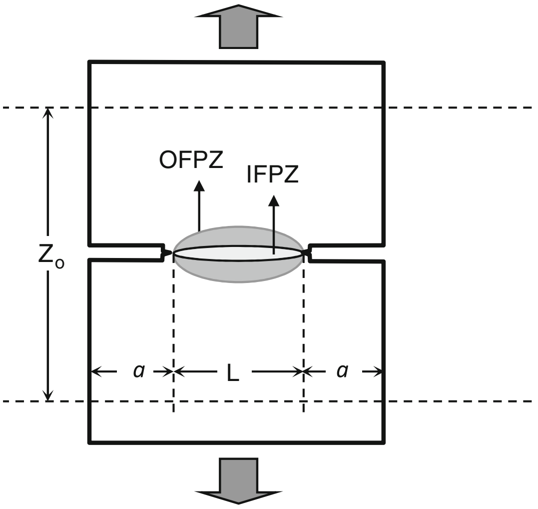


Fig. 1 Schematic diagram showing the deeply double-edge notched tension (DDENT) specimen

$$W_f = w_e BL + \beta w_p BL^2 \tag{2}$$

where β is a shape factor of the plastic deformation zone and B is the sample thickness. Dividing Eq. 2 by the ligament area ($L*B$), the specific total fracture work, w_f, is given by:

$$w_f = (W_f/BL) = w_e + \beta w_p L \tag{3}$$

Three critical assumptions behind Eq. 3 are that each of the specimens tested for different ligament lengths must deform in a similar fashion, the ligament should be completely yielded at the onset of crack growth and the ligament is in a state of plane stress. The assumptions are to ensure most of the work of fracture are consistently consumed prior to crack growth so that good linearity in plotting w_f vs. L can be obtained using Eq. 3.

In the EWF approach, it is common to employ for DDENT specimen's geometry [22]. For (DDENT) specimens (Fig. 1) that completely fail under stable crack growth and the ligament is entirely broken into two halves, self-similar deformation could yield a straight line relationship.

Experimental

Materials, blending conditions, and sample preparations

PET copolymer with IV = 0.80 dL/g and *Lexan 123R* PC with melt flow rate (measured at 260 °C and 2.16 kg) of 10 g/10 min were used as the original materials. By using a COLLIN ZK-25 co rotating twin screw extruder with a $L/D = 36$ and a screw diameter of 25 mm, PC and PET were blended in melt at temperature of 190–270 °C and a screw speed of 130 rpm for systems without transesterification catalysts and a screw speed of 160 when the transesterification catalysts were used. A catalyst (when used) is initially dispersed mechanically on polymer pellets at a concentration of 0.05% wt/wt with respect to the final product. Samarium acetyl acetonate ($Sm(acac)_3 \cdot xH_2O (x = 3\text{-}4)$), calcium acetate hydrate $Ca(CH_3COO)_2 \cdot xH_2O$, and zinc acetate hydrate $Zn(CH_3COO)_2 \cdot 2H_2O$ were purchased from Aldrich used as a catalysts.

Before melt blending the starting materials were dried. PET and PC pellets were held in a PIOVAN T30IX dryer at 120 °C for 4 h. Nitrogen and vacuum were applied to eliminate moisture. The PET amounts added were 10, 20, and 30% w/w and the following code will be used: PC##, where ## is the nominal PC content as shown in Table 1.

The blend films were developed using a Collin Teach Line 20T-E single screw extruder. It is 20 mm diameter with length to a diameter ratio of 25:1. The extruder is connected to large tube and a rectangular manifold die of

Table 1 Sample codes of materials used

Material	%wt of PC	%wt of PET	Remarks
PC	100	0	–
PC90	90	10	Without catalyst
PC90Ca	90	10	Ca-based catalyst
PC90Zn	90	10	Zn-based catalyst
PC90Sm	90	10	Sm-based catalyst
PC80	80	20	Without catalyst
PC80Ca	80	20	Ca-based catalyst
PC80Zn	80	20	Zn-based catalyst
PC80Sm	80	20	Sm-based catalyst
PC70	70	30	Without catalyst
PC70Zn	70	30	Zn-based catalyst
PET	0	100	–

0.7 mm lips opening (take-off unit). Take-off post extrusion which produces extrudates of film shape attached to chill rolls (Teach Rolls 72T). Before the extrusion process, PET, PC, and blended pellets which have the same code numbers as in Table 1 were dried in an oven at 120 °C for 4 h in the PIOVAN dryer.

The films were prepared in a melt at a temperature profile between 150 and 270 °C with screw rotation speed of 120 rpm at the extruder and 50 rpm at the chill rolls. The molten extrudate is forced through a pre-defined gap in the die and cooled down to below the melting temperature (70 °C) so that it permanently retains a closely uniform film thickness ($\approx$0.7 mm) as it passes through the take-off unit.

DDENT tensile specimens with a width of 60 mm, a clamping length of 60 mm, and a thickness of $\approx$0.7 mm were used for the EWF measurements. They were cut from the central zone of the films with the parallel direction (MD) to the melt flow direction. The initial sharp cracks were notched with a fresh razor blade. The ligament lengths between the pre-notched cracks ranged from 5 to 20 mm with an interval of 1 mm and were measured with a travelling microscopy after the tests.

Specimens destined for the tensile test were punched from the film central zone to form dumb-bell shaped with the following dimensions: 25 mm in width and 120 mm in length. The exact thickness of each specimen was measured at five different points using a micrometer and the average value used in subsequent calculations.

Testing

All tests were conducted at room temperature (23 °C) using a universal testing machine with a crosshead speed of 10 mm/min. The load applied during extension was monitored with a load cell of Galdabini material testing instrument.

In the case of fracture tests, for each ligament length, at least two specimens were fractured. The load–displacement curves were recorded and the absorbed energy until failure is calculated by a computer and all the data treatments followed the recommendations of European Structural Integrity Society (ESIS) for EWF technique [23]. The essential work of fracture (w_e) and plastic item (βw_p) were determined.

For tensile tests, the Young's modulus (E), engineering yield stress (σ_y), and yield deformation (ε_y) were obtained from the force–displacement curves.

A Joel JSM6400 scanning electron microscope (SEM) was used to study the morphology of the cryogenic fracture surfaces in the central zone of the sheet samples in a direction parallel to the flow direction which was gold-coated with a thickness of 0.3 mm.

Results and discussion

Tensile properties

According to results obtained as shown in Table 2, addition of PET for PC/PET blends without transesterification catalysts, does not lead to significant changes in these tensile parameters especially at PC80 and PC70 blends. However, there is an increase in E value at PC90 blend which defines a significant positive deviation from the rule of mixtures. Here the transesterification reactions generated during mixing could be enough to promote some "adhesion" between phases due to the low interfacial surface.

The tensile properties of PC/PET blends with transesterification catalysts are more or less similar to neat PC even it is slightly increased when the Ca- and Zn-based

Table 2 Tensile test results for the neat polymers and PC/PET blends

Material	E (GPa)	σ_y (MPa)	ε_y (%)
PC	2.18 ± 0.09	60.6 ± 1.0	5.73 ± 0.38
PC90	2.22 ± 0.11	60.6 ± 2.6	5.46 ± 0.31
PC90Ca	2.35 ± 0.08	64.5 ± 2.9	5.41 ± 0.42
PC90Zn	2.28 ± 0.11	62.8 ± 0.4	5.27 ± 0.13
PC90Sm	2.15 ± 0.15	59.9 ± 0.9	5.59 ± 0.08
PC80	2.20 ± 0.05	61.8 ± 0.7	5.07 ± 0.09
PC80Ca	2.30 ± 0.01	63.5 ± 1.2	5.18 ± 0.07
PC80Zn	2.46 ± 0.09	65.8 ± 1.9	5.20 ± 0.12
PC80Sm	2.20 ± 0.10	60.8 ± 1.0	5.21 ± 0.13
PC70	2.16 ± 0.12	59.4 ± 0.6	4.88 ± 0.37
PC70Zn	2.34 ± 0.10	63.9 ± 0.2	4.95 ± 0.11
PET	2.26 ± 0.12	64.1 ± 2.7	4.05 ± 0.08

catalysts were presented. In the case of PC80Zn and PC90Ca, it is found that the tensile modules were enhanced or increased by 13 and 8%, respectively. The enhancement of tensile parameters can be explained in term of strong interactions occurred between the PC and PET components. This indicates that an important paper plays the balance between interface adhesion, morphology of the mixtures, and extension of the transesterification. By this form, it would be reasonable to think that the copolymer formation, preferably located in the interface of the phases, would promote a mechanical compatibility for this type of imposed blends.

Fracture properties

Fracture types

Depending on the nature of polymer and the blend composition, two types of fracture were observed during the registration of load–displacement curves of the tested materials as shown in Fig. 2.

Post-yielding (PY) After achieving a full ligament yielding and blunting, starts the stable propagation of crack tip which is a necessary condition to the validity of the EWF technique [24]. The characteristic record corresponds to the Fig. 2a specifies this type of fracture which was observed exclusively on the neat PET that agrees with what Sánchez [25] mentioned in his study.

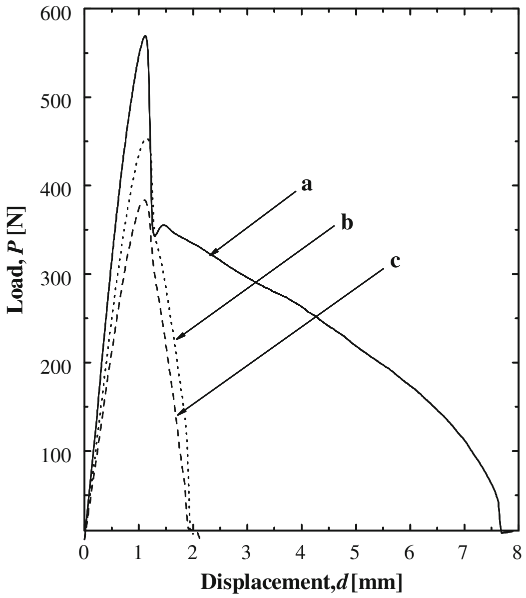
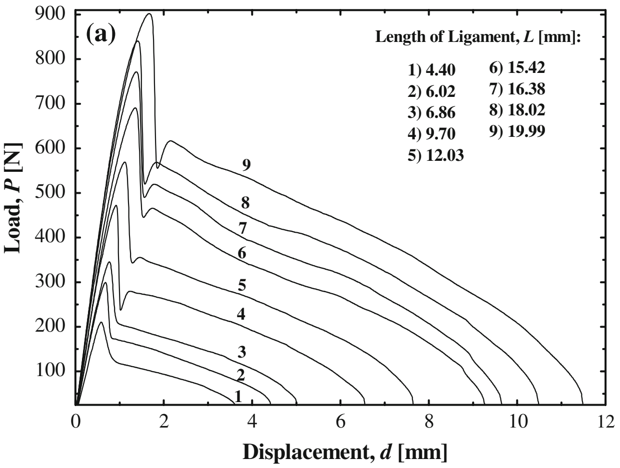


Fig. 2 Characteristic load–displacement curves of tested materials by using DDENT specimens and respective fracture behaviors of: *a* Post-yielding (PET sample, $L = 6.0$ mm), *b* rapid crack propagation (PC sample, $L = 13.0$ mm), and *c* rapid crack propagation (PC80Sm sample, $L = 13.0$ mm)



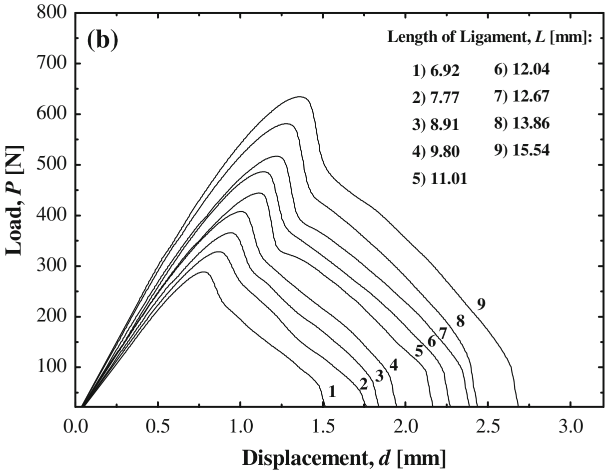


Fig. 3 Load–displacement curve (P–d) using EWF tests and DDNET specimens at 10 mm/min with different ligament lengths for **a** neat PET and **b** PC70Zn

Rapid crack propagation (RCP) It was observed on the neat PC and all PC/PET specimens. Initiation of crack propagation begins before the total ligament yielding (Fig. 2b, c).

The load–displacement curves of DDENT specimens for neat PC, PET, and PC/PET blends during EWF tests as a function of ligament length are shown in Fig. 3. These curves demonstrate that the load sustained by the specimen and its extension at break both increase, besides, the maximum load and ultimate displacement increase with the increase of ligament length. This is another crucial criterion for the application of EWF method, which ensures that the cracks propagated under similar stress conditions, being unchanged with the ligament length [24, 26].

Fracture parameters

Plots of the specific essential fracture work versus ligament lengths for the PC/PET blends were shown in Fig. 4. As

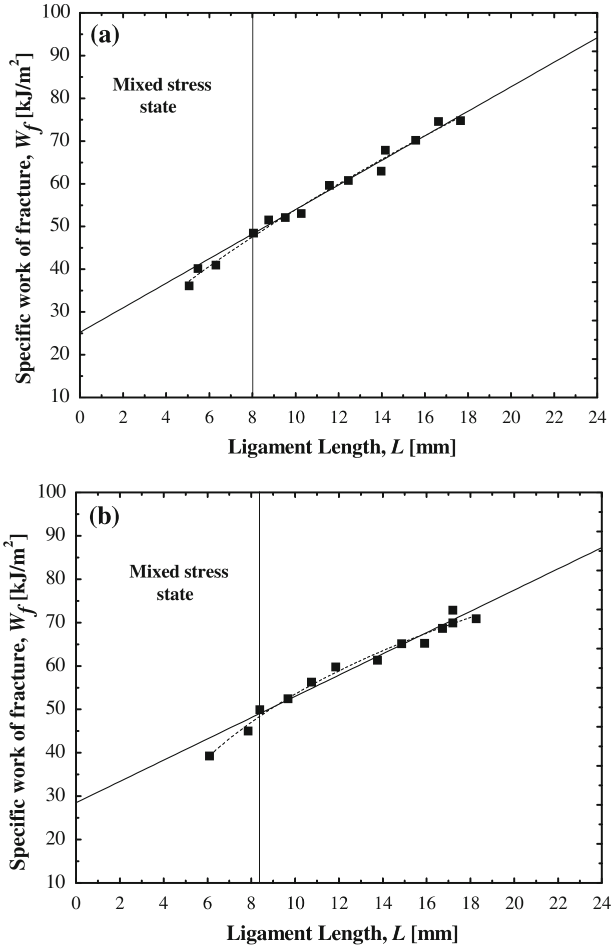


Fig. 4 W_f vs. L curves obtained for **a** PC90 and **b** PC90Ca. *Solid lines* represent the linear fit of curves while the *dash lines* represent the non-linear fit ones

expected, all neat polymers and PET/PC display very good linearity and minimal scatter in the specific total fracture work as proved by the linear regression coefficient (R^2) being in most cases higher than 0.96. According to plasticity theory of Hill [27]: the maximum stress values should be less or equal of 1.15 of yielding stress ($\sigma_{\max} \leq 1.15\sigma_y$) once $\sigma_{\max}$ is greater than σ_y. All the range of ligaments analyzed fulfils these conditions for all materials used.

The values of w_e and βw_p obtained from the interception and slope of the straight lines extrapolated to zero ligament length, together with the regression coefficient, are listed in Table 3. Values obtained for PET sheets are so similar to values that have been reported for PET elsewhere. Sánchez [25] has obtained values for w_e corresponding to 51.0 kJ/m^2 (same PET, but injected molded in 2 mm plates). Karger-Kocsis et al. [28] cite values of 45–47 kJ m^{-2} for w_e corresponding to a PET sample of 0.5 mm thick (PET bi-oriented). While Chan et al. [29] report a value of 54 kJ/m^2 for PET amorphous (copolymer). In the case of the neat PC, it has a fracture parameters similar to those obtained by Paton and Hashemi [30] for injected plates: w_e = 35–27 kJ/m^2, βw_p = 3.0–3.5 MJ/m^3), and even for films with thicknesses of 0.175–0.520 μm (w_e from 29 to 44 kJ/m^2 and βw_p between 4.2 and 2.3 MJ/m^3) [30–32]. Also, according to Yuan et al. [33] w_e and βw_p corresponding to 6.64 kJ/m^2 and 22.8 MJ/m^3, respectively while Sánchez [25] cites values of 31.0 kJ/m^2 for w_e and 3.1 MJ/m^3 for βw_p. The difference in w_e and βw_p values that can be found in the literature may be related to material specifications, i.e., molecular weight, molecular weight distribution, and viscosity or related to differences in sample thickness, testing speed, and bi-orientation degree, among others.

Table 3 Measured EWF parameters of neat polymers and PC/PET blends

Material	Parameters				
	w_e (kJ/m^2)	βw_p (MJ/m^3)	R^2	$\beta \times 100$	Fracture type
PC	22.4 ± 1.6	2.8 ± 0.1	0.983	8.7 ± 0.4	RCP
PC90	25.2 ± 1.8	2.9 ± 0.1	0.976	9.5 ± 0.2	RCP
PC90Ca	28.5 ± 2.1	2.4 ± 0.2	0.962	9.4 ± 0.3	RCP
PC90Zn	30.6 ± 2.1	2.5 ± 0.1	0.984	8.1 ± 0.2	RCP
PC90Sm	27.4 ± 1.9	2.7 ± 0.1	0.988	9.7 ± 0.3	RCP
PC80	24.3 ± 1.9	3.2 ± 0.1	0.984	9.5 ± 0.2	RCP
PC80Ca	28.1 ± 3.4	3.4 ± 0.2	0.975	9.1 ± 0.1	RCP
PC80Zn	35.0 ± 4.4	2.2 ± 0.3	0.962	8.5 ± 0.2	RCP
PC80Sm	30.9 ± 1.8	3.4 ± 0.1	0.991	9.1 ± 0.3	RCP
PC70	25.9 ± 0.8	3.8 ± 0.1	0.997	11.4 ± 0.2	RCP
PC70Zn	30.8 ± 2.4	3.6 ± 0.2	0.987	9.4 ± 0.2	RCP
PET	56.4 ± 7.2	11.1 ± 0.5	0.984	11.4 ± 0.2	PY

Blends without transesterification catalysts exhibit a slight increase on the w_e compared with neat PC. However, considering the error bars, it could be seen that although this trend, the values seem to no change between, giving an almost constant value of about 25 kJ/m^2. The non-dependence trend observed reflects the poor adhesion between the phases. Cavitation of the dispersed phase (PET) makes the crack propagation become dictated by the matrix, without apparent synergy. The fact that w_e value is greater than observed for neat PC (13% increasing), reveals that the work invested in the generation of new free surface is increased.

For PC/PET blends with transesterification catalysts, all w_e values of these systems lie in between of that values of neat PET and neat PC ones. In general, addition of transesterification catalysts to these blends enhances w_e fracture parameter values in comparison with neat PC or blends without catalysts. The presence of Zn-based catalyst in the blend promotes the greater enhance of w_e values compared with Ca- or Sm-based catalyst systems. Thus, compared with neat PC, the addition of Zn-based catalyst increases the w_e by about 37, 56, and 38% for PC90, PC80, and PC70, respectively. By the same trend, the existence of Ca- or Sm-based catalyst leads to increase w_e in general from 27% for PC90Ca to about 38% for PC80Sm compared with neat PC (22.4 ± 1.6 kJ/m^2). This is confirmed by the tensile test results obtained (Table 2) which demonstrates higher values for E and σ_y in the case of blends with catalysts compared with neat PC.

In the literature and regard to our knowledge, there is no work or study dedicated on the fracture behavior of PC/PET blends at low PET content. Therefore, to give an interpretation to the significant enhance of w_e values compared with neat PC or blends without transesterification catalysts, the role and the effect of the transesterification reaction in these systems should be analyzed. It is known that the copolymer product of the transesterification reactions works like emulsion/compatibilizer between the

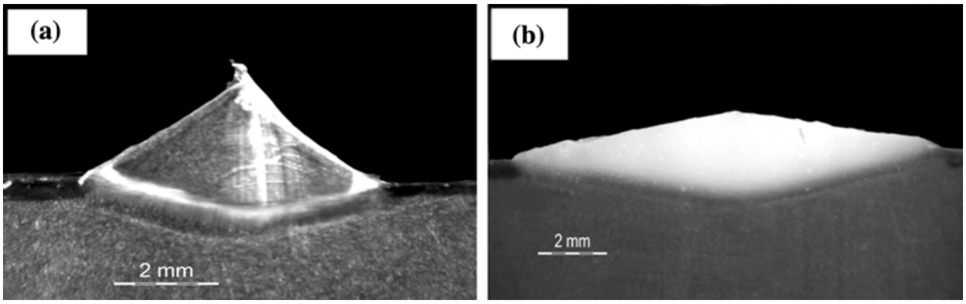


Fig. 5 An optical micrograph of the outer fracture process zone (OFPZ) on: **a** PET, and **b** PC80Sm

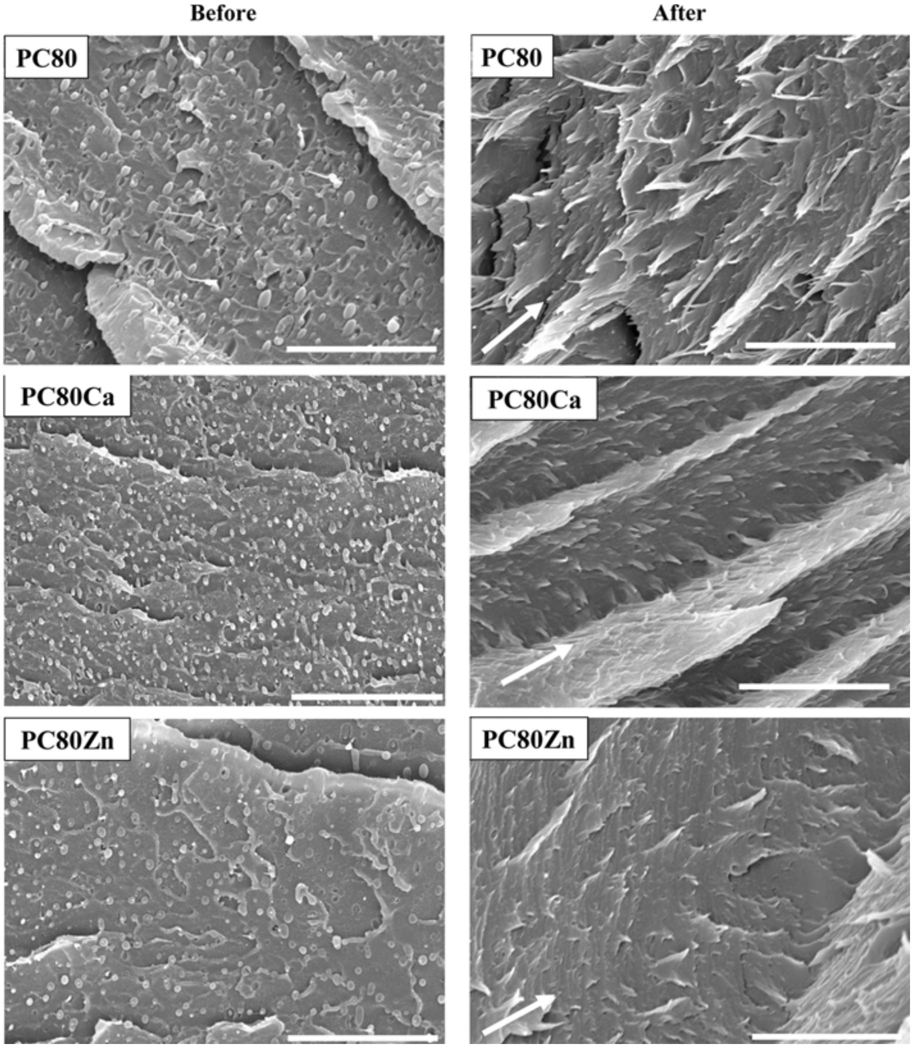


Fig. 6 SEM micrographs of the fracture surfaces of the OFPZ for PC80 blend before and after test. *Scale bar* 10 μm. *Arrows* indicate direction of loading

PC-rich phase and unreacted PET and so enhances the interfacial adhesions and reduces the interfacial tension between the two immiscible or incompatible component phases [9, 34, 35].

Although Sm-based catalyst was found the more active one, its activity may cause two effects: increase the degree of interchange reaction and so enhances the interaction between PC and PET phases by one hand but by the other hand, it may increase the degradation rate which could be supported by the evidences obtained with rheological measurements as explained in other study [19]. The competition between those two factors determines the fracture behavior of PC/PET blends containing Sm-based catalyst like PC90Sm system.

For non-essential term (βw_p) and as can be seen in Table 3, for blends without transesterification catalysts, there is a slight increase with increasing PET content compared with neat PC. It should be indicating that mechanically the system is working in "Iso-stress" conditions (each phase deforms independently). Addition of catalyst shows no clear trend. It seems that some morphological feature dependency is observed. The observed trend should be understood analyzing the geometrical factor of the outer fracture process zone (OFPZ) (β), associated to the volume of the influence zone where irreversible (dissipative) process takes place. According to the diamond shape of the plastic zone in all materials tested (Fig. 5), $\beta = h/2L$ where h is the height of the plastic zone.

As can be seen in Table 3, β values are maxima (and higher than PC one) for blends without transesterification catalysts, increasing with PET content. This could be related with the increase of PET phase size. As the size of dispersed phase increases the specific interfacial area between phases decreases. If adhesion between these particles is not enough, its cavitations probability increases. After cavitation the amount of matrix under triaxiality release condition is higher. As this stress state promotes shear yielding in the matrix the OFPZ should increase (higher β value).

The PC–PET copolymer generated by interchange reaction (transesterification) acts as emulsifier and compatibilizer, lowering the PET dispersed phase size and increase its adhesion to the PC matrix [19]. Under this situation it would be expected that cavitation and/or matrix shear yielding decrease. It seems that this trend is followed by PC80 and PC70 systems. Support of this effect would be observed by the observation of the fracture surface in the OFPZ region (Figs. 6 and 7), where the amount of tear surface is clearly lower with the addition of catalyst.

For PC90 no substantial effect is observed, excepting for Zn-based catalyst, in the β value, all of them higher than neat PC. Analyzing the fracture surface of the OFPZ zone (Fig. 8), it could be observed that the amount of cavitated PET phase is low (perhaps somewhat higher for system without transesterification catalysts), while a clear fibril feature is observed and associated to PET droplet deformation (excepting PC90Zn).

This observation leads us to think that the degree of adhesion in all these blends seems to be higher than other systems (including that without transesterification

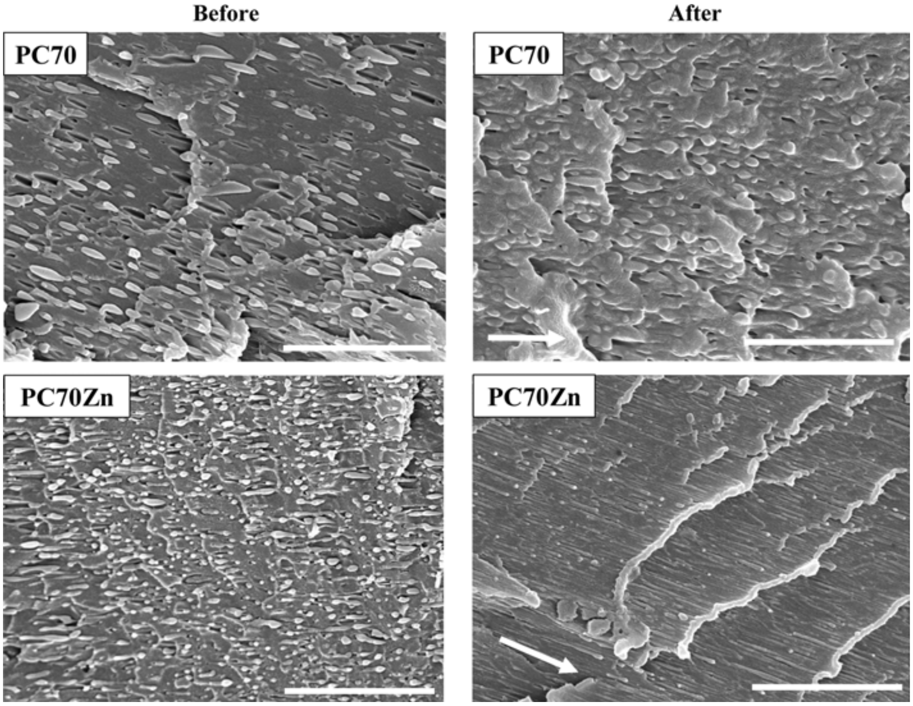


Fig. 7 SEM micrographs of the fracture surfaces of the OFPZ for PC70 blend before and after test. *Scale bar* 10 μm. *Arrows* indicate direction of loading

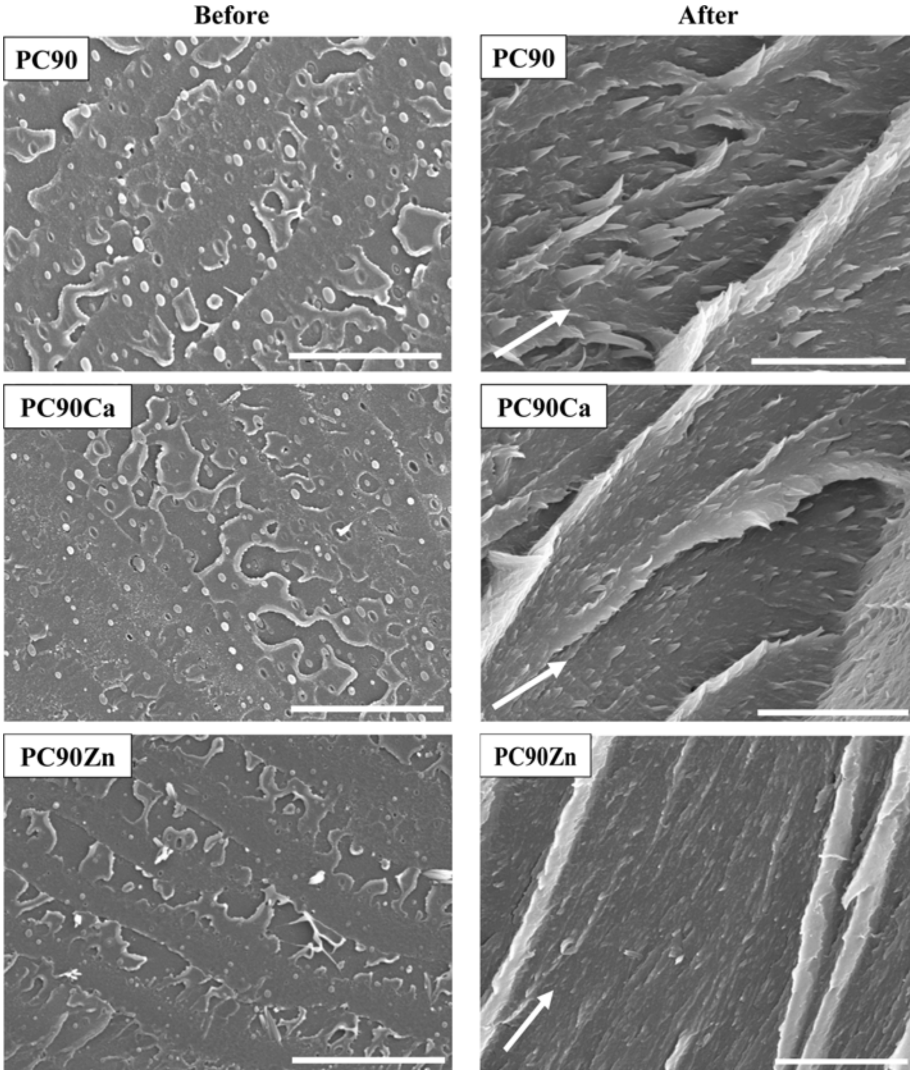


Fig. 8 SEM micrographs of the fracture surfaces of the OFPZ for PC90 blend before and after test. *Scale bar* 10 μm. *Arrows* indicate direction of loading

catalysts). At this point, it could say that, for this low PET content, the amount of PC–PET generated (even for blends without catalysts due to residual polymerization catalyst) is enough to assure the adhesion between phases due to the high specific area obtained in the dispersed phase. By other side, it could be inferred that the irreversible (dissipative energy) process in the OFPZ zone for this blends would be associated mainly with the PET droplet deformation, and in some extent would be influenced by its strain induced crystallization.

For PC80 and PC70 system, addition of transesterification catalysts clearly decreases the OFPZ size (decrease on β) clearly observed for PC80 and PC70 system. This could indicate that cavitation becomes restricted due to the better interaction between phases (higher adhesion): PC matrix and “hard” PET droplets. Evidences of these cavitation and adhesion effect of catalyst could be observed in Figs. 6 and 7.

Conclusion

EWF method using DDENT specimens has been successfully applied to study the fracture toughness for blends of PC with PET rich in PC in the presence and absence of three different transesterification catalysts.

The specific essential work of fracture and the specific non-essential work of fracture were both found to be strongly depending on the presence of transesterification catalyst and the amount of PET phase used. The modulus, yield stress, specific essential work of fracture, and specific non-essential work of fracture values of blends contain transesterification catalysts are higher than these without. Interestingly, the specific essential work of fracture did not exhibit the same tendency.

Acknowledgements The authors express their thanks to Ministerio de Asuntos Exteriores y de Cooperación – Agencia Española de

Cooperación Internacional (AECI) for the funding of a doctoral research scholarship.

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