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Analysis of the effect of the test rate on polymer essential work of fracture parameters for the small punch test

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ABSTRACT: In recent decades, one of the non-standard tests that has been consolidated as a viable alternative in those cases where there is not sufficient material to carry out standard tests is the small punch test. This test basically consists of deforming a miniature specimen using a high strength punch. It is possible for this miniature specimen to have an initial pre-notch with the aim of improving the fracture behavior estimation of the material analyzed. Recently, to characterize the fracture properties of polymer sheets under plane stress conditions, there has been an attempt to establish the feasibility of applying the essential work of fracture (EWF) method in polymer pre-notched miniature specimens. This article intends to go one step beyond and focuses on the test rate, which is an important aspect in the EWF application. Its effect on the EWF parameters in polymer pre-notched miniature specimens has been analyzed and its correlation has been established with the results obtained from standard specimens. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43314.

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INTRODUCTION

In recent decades, one of the non-standard tests that has been consolidated as a viable alternative in those cases where there is not sufficient material to carry out standard tests is the small punch test (SPT). This test basically consists of deforming a miniature specimen ($10 \times 10 \times 0.5\,mm^3$) whose edges are firmly gripped by a die using a high strength punch. It has been used by a great number of researchers in order to obtain mechanical and fracture properties when the material is limited,[1–5] and it is possible for this miniature specimen to have an initial pre-notch with the aim of improving the fracture behavior estimation of the material analyzed.[6,7] The experimental setup can be consulted in the CEN (European Committee for Standardization) code of practice for small punch testing.[8]

Recently, in previous articles by the authors,[9,10] to characterize the fracture properties of polymer sheets under plane stress conditions, there has been an attempt to establish the feasibility of applying the essential work of fracture (EWF) method in polymer pre-notched miniature specimens. This method was developed by Broberg[11,12] for metals and was later used for polymers by Mai and Cotterell,[13,14] and it has been widely used for more than a quarter of a century to assess fracture properties in thin polymer plates under plane stress conditions by testing deeply double edge notched tensile specimens (DDEN-T).[15–19] This method assumes that the energy consumed in the ductile tearing failure could be divided into two terms. One of them corresponds to the energy needed to create the new fracture surfaces and is called essential work (W_e), which is related to the inner fracture process zone (IFPZ) (Figure 1). The other one, geometrically dependent and called non-essential or plastic work (W_p), refers to the energy involved in more general plastic deformation and is related to the outer process dissipation zone OPDZ (Figure 1). Expressed as a function of the specimen ligament as shown in eq. (1), these terms can be rewritten into specific terms in eq. (2),

$$W_f = W_e + W_p = w_e \cdot L \cdot t + \beta \cdot w_p \cdot L^2 \cdot t \quad (1)$$

$$w_f = w_e + \beta \cdot w_p \cdot L \quad (2)$$

where t is the specimen thickness, L is the ligament length, and β is the shape factor related to the form of the outer plastic dissipation zone.[20]

One important aspect in the EWF application is the test rate. Over the years, several researchers have analyzed the effect of the test rate on different types of polymers using DDEN-T specimens.[21–24] All of the researchers have reached a common conclusion which is that the test rate does not affect the specific EWF, but it does affect the specific plastic work of fracture. So, the influence of the test rate on the miniature specimen results

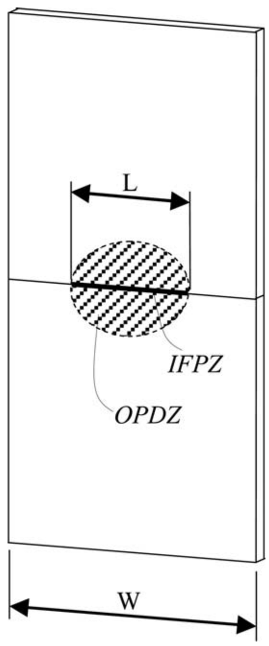

Figure 1. DDEN-T specimen.

has to be analyzed in order to find out whether the above conclusion can be extended to small punch specimens. This would contribute to the consolidation of the implementation of the EWF concept using the results from the SPT.

In this article, the effect of the test rate on the EWF parameters in polymer pre-notched miniature specimens has been analyzed, and its correspondence with the results obtained from standard specimens has been established.

MATERIALS AND TEST RATE EFFECT FOR STANDARD SPECIMENS

As previously mentioned, the EWF method postulates that in ductile tearing failure, it is possible for the total work of fracture (W_f) to be obtained from the area of the force–displacement curve and divided into two terms (W_e and W_p). Thus, as a function of the ligament length L, the specific work of fracture w_f can be plotted using eq. (2), where w_e and $\beta \cdot w_p$ can be determined by intercept with the w_f axis for $L=0$ and the slope obtained by its linear regression.[10]

Before EWF is applied to assess toughness, some different aspects such as full ligament yielding prior to crack initiation, prevalence of plane stress conditions, and self-similar load–displacement curves (with the same shape irrespective of the ligament length but whose size depends on the ligament length) should be fulfilled.[25] In any case, Bárány *et al.* suggested in their reviewed research[20] that the only aspect which is necessary for effective application of EWF is that of self-similar curves.

As a general rule, EWF is used for polymer films or thin polymer sheets (<2 mm). In the case of this study, an amorphous polyethylene terephthalate (A-PET) extrusion, type A-B-A 100% (layers A of 100% virgin material and layer B of recovered materials), with an initial extruded sheet thickness of 0.492 mm and a theoretical weight of 1.33 g/cm^3 was selected.

DDEN-T specimens with a width of $W=30$ mm were cut by laser from the polymer sheet to assess the toughness of the polymer analyzed, in all of which a razor blade was used to sharpen the notches. The range of ligament lengths obtained was $L \geq 3 \cdot t$ for the lower bound and $L < W/3$ for the upper bound, normal for the EWF method, which ensured plane stress conditions of the specimens.[20] The DDEN-T specimens were

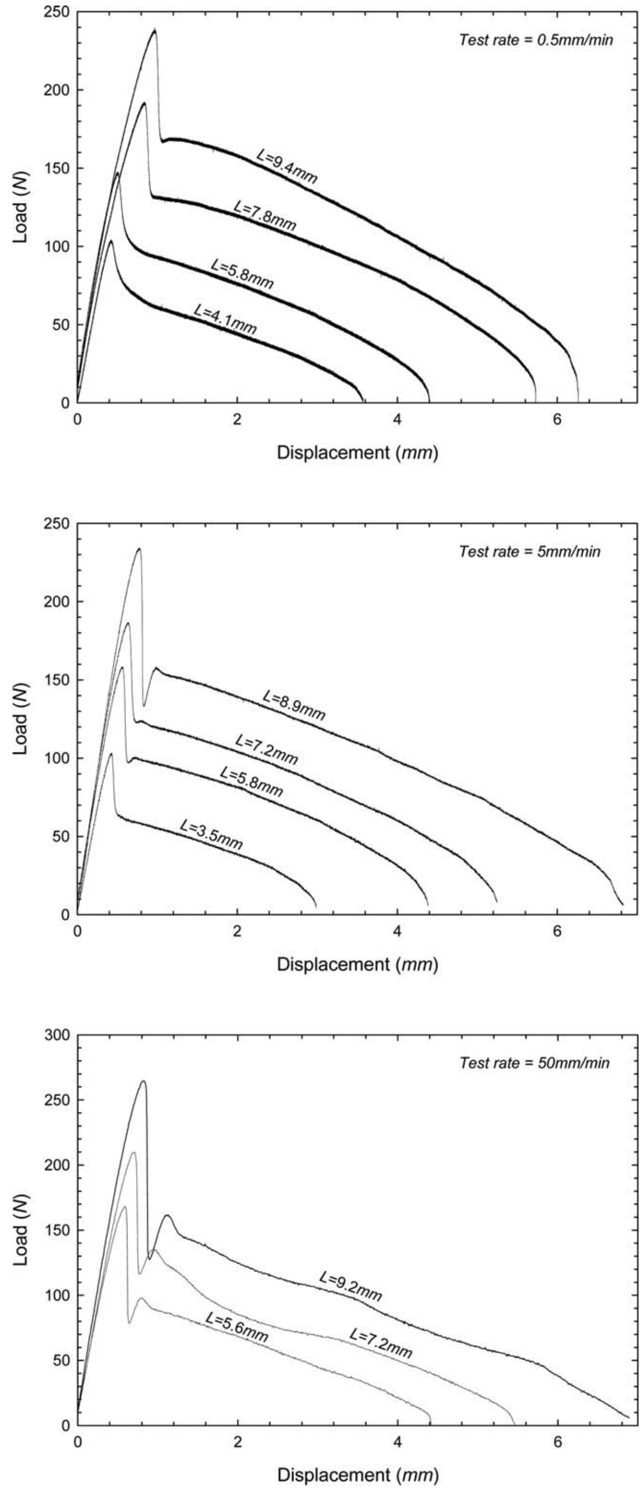

Figure 2. Load–displacement curves for DDEN-T specimens.

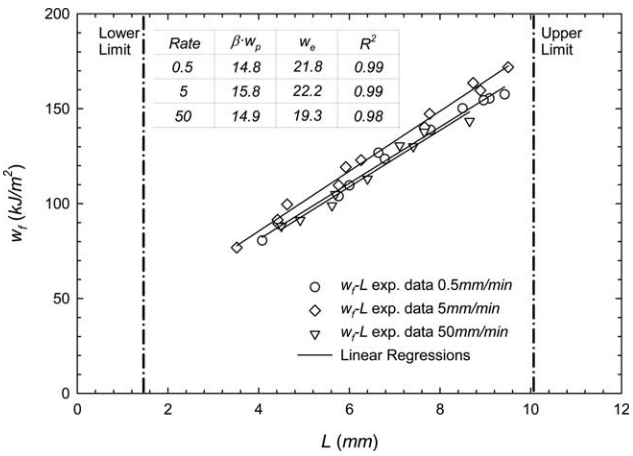

Figure 3. Specific work of fracture versus ligament length for DDEN-T specimens.

tested at room temperature, and three different test rates (0.5, 5, 50 mm/min) were used.

Figure 2 shows some of the load–displacement curve characteristics of the A-PET selected for the test rates. A similar shape can be clearly seen in all of them for each test rate, so EWF can be applied. Furthermore, if the ductility level (D_L), defined in eq. (3) and proposed by Gamez-Perez[26] to rationalize different fracture behaviors, is analyzed, it can be observed that this parameter takes values in all the specimens tested between 0.15 and 1, so the fracture behavior can be classified as post-yielding behavior. This is the type of behavior where the EWF method is fully applicable,[26] so the selected polymer is suitable for the application of EWF.

$$D_L = d_r / L \tag{3}$$

where d_r is the displacement at rupture

In all of the tested specimens, the shape of the load–displacement curve was verified in order to discard those with different shapes for each test rate. In Figure 3 w_f is plotted as a function of L, and as expected, the relationship can be adjusted in each case by a straight line where w_e, the slope $\beta \cdot w_p$ and the correlation coefficient R^2 can be consulted in the same figure as a function of the test rate. These results are consistent with those reported for this type of polymer in the review work by Bárány *et al.*[20] In all cases, the correlation coefficient value obtained is greater than 0.98 so it can be assumed that the fit is appropriate.[27] Not only that, the w_e values are very similar to each other, so it can also be considered that the test rate does not affect w_e, which is related to other research that can be found in the literature.[21–23]

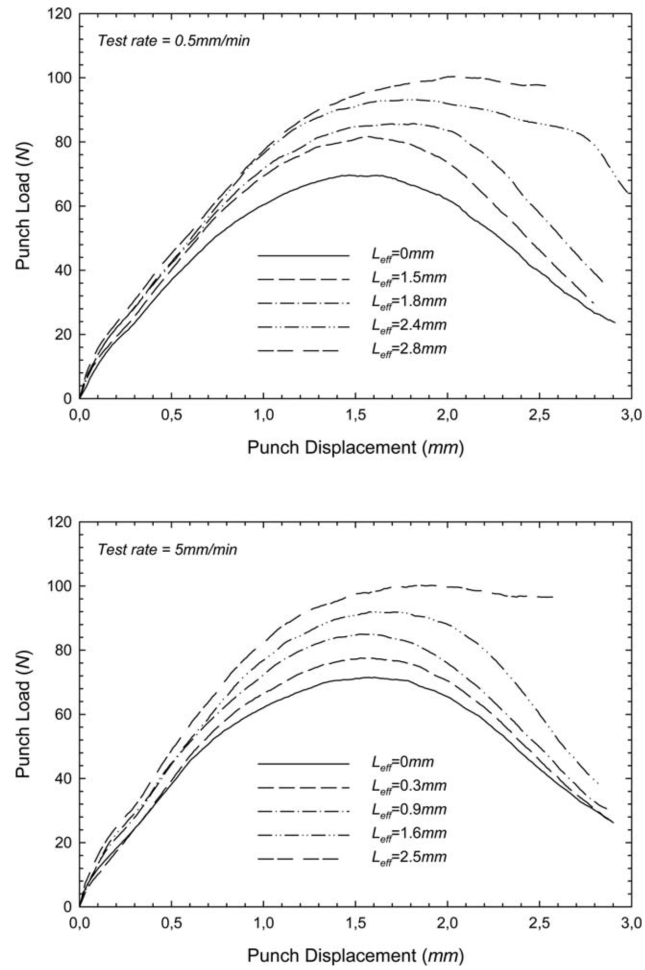

Figure 5. Load–displacement curves for P-SPT specimens.

EWF FOR PRE-NOTCHED SPT SPECIMENS

As mentioned above, the present research is focused on finding out the effect of the test rate when the EWF method is applied

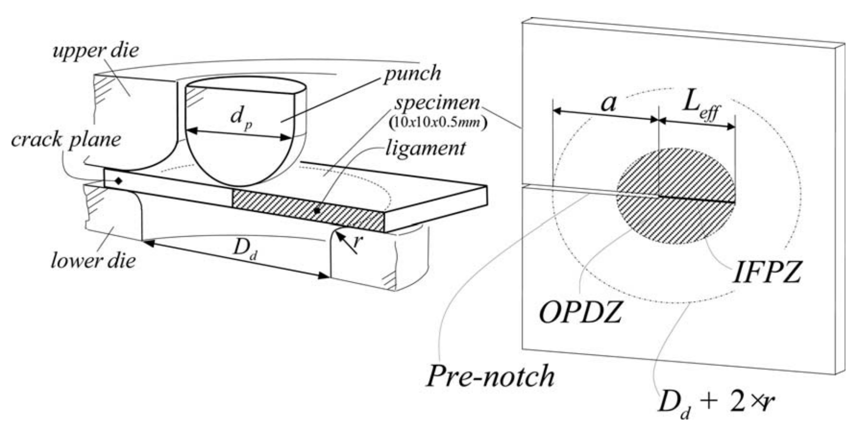

Figure 4. SPT device and pre-notched small punch specimen.

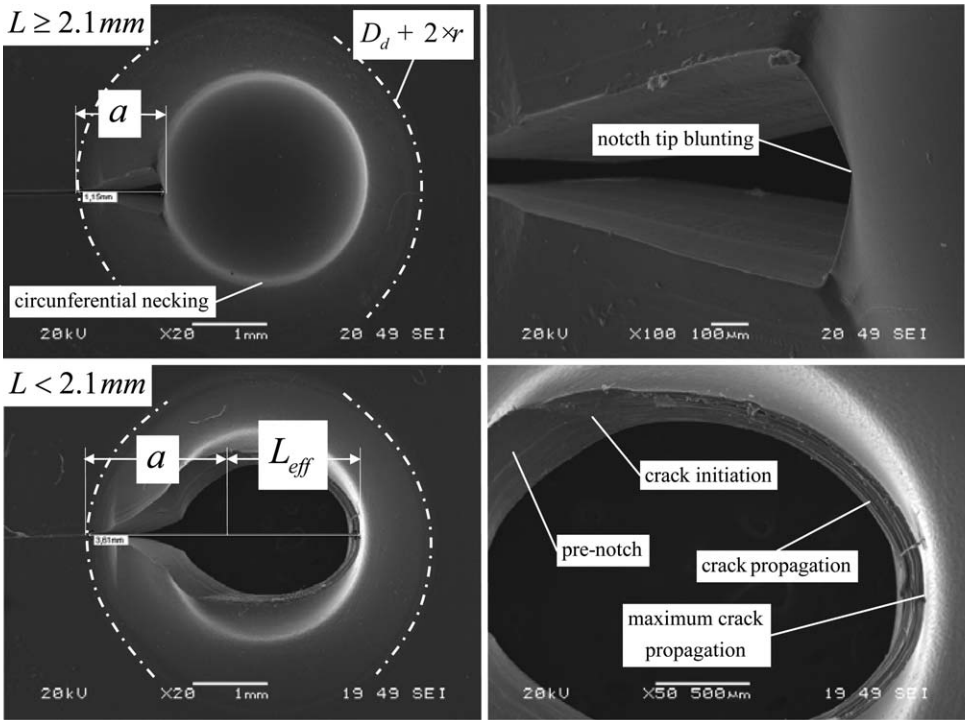

Figure 6. SPT specimens.

to pre-notched SPT specimens. It is to be expected that not all notch lengths used in SPT specimens have crack propagation in mode I.[9] However, mode I is necessary to compare the results with DDEN-T specimens, so this crack propagation mode has to be reproduced. To obtain this, the type of pre-notching used on the specimens was of the through-thickness notch type, from the middle point of one side to a point just passing the center of the specimen as shown in Figure 4. The range for the notch lengths (a) varied within the central area of the specimen that was not embedded between the two dies of the SPT device. In Figure 4, a cross-section of how a specimen with a specified value of a would be positioned in the SPT device is shown, and in the same figure, the distance regarded as the effective propagation ligament in mode I (L_{eff}) is defined.

Upper and lower bounds similar to those established for DDEN-T specimens for the effective ligament (L_{eff}) must be identified, taking into account the shape of the crack propagation, the shape of the load–displacement SPT curves and the tendency of w_f results as a function of the ligament length.[9]

Miniature specimens ($10\times10\,\mathrm{mm}^2$) were cut from the A-PET sheet and pre-notched using a razor blade following the same technique as for DDEN-T specimens. The SPT specimens were tested once the pre-notch was achieved. The tests were conducted at room temperature using a punch diameter of $d_p=2.5$ mm and the hole in the lower die had a diameter of $D_d=4$ mm and a fillet radius of $r=0.5$ mm (Figure 4).[10] For the selection of the two test rates used in this research, the rate usually employed in the SPT was taken into account. Thus, a value of v

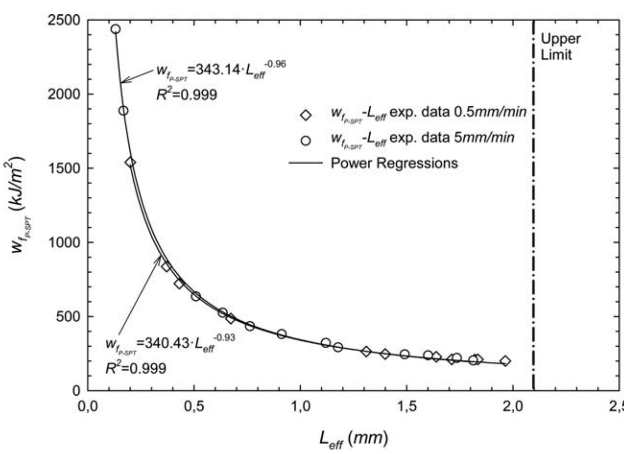

Figure 7. Specific work of fracture versus ligament length for P-SPT specimens.

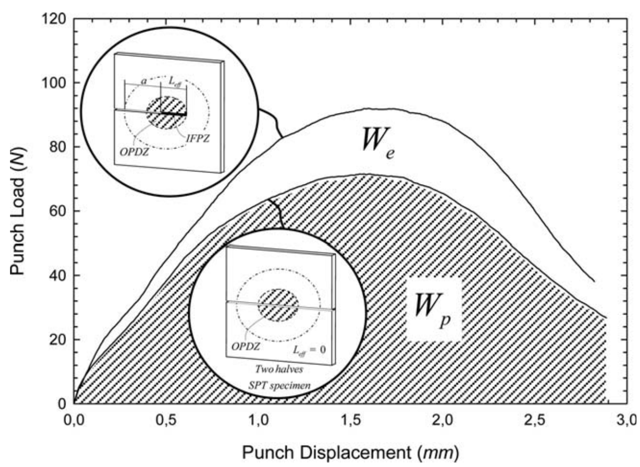

Figure 8. Essential work and plastic work in pre-notch SPT specimens.

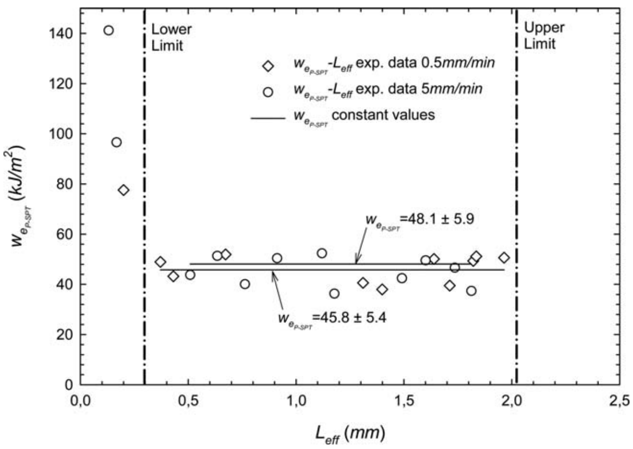

Figure 9. EWF method for P-SPT specimens.

$=0.5$ mm/min and another ten times greater, of $v=5$ mm/min, were used. A load–displacement curve was obtained for each specimen in which the area under the curve was calculated corresponding to the total work of fracture W_f.[10]

RESULTS AND DISCUSSION

In Figure 5, the load–displacement curves for some of the pre-notched SPT specimens corresponding to different effective ligament lengths are shown for both test rates. In both cases, it can be found that $L_{eff}=2.1$ mm marks a turning point in the curve shape. Not only that, if the failure in the SPT specimens is analyzed, it is possible to identify two failure modes independent of the test rate used. These failures modes had been observed and described in previous work.[9] Basically, the first corresponds to specimens with $L_{eff} \geq 2.1$ mm where a circumferential necking failure typically takes place in unnotched specimens.[28] For reasons previously mentioned, these specimens are discarded for the application of EWF. This turning point in the failure mode defines the upper limit of the $w_f - L_{eff}$ data. The second occurs after crack propagation in the notch direction. Figure 6 shows a representative specimen of both failure modes. For the specimen with $L_{eff} < 2.1$ mm, the crack propagation stops before reaching the embedded area due to the fact that the crack has already grown enough for the punch to push through the two halves of the specimen. This stop point is used to determine the effective ligament length L_{eff} so that $a+L_{eff} \approx 3.6$ mm in all specimens.

In Figure 7, the w_f values obtained as a function of the effective ligament length L_{eff} in which the data fit perfectly using a power law for both test rates are shown. But, there is a rejection in the EWF community with respect to the power law data representation in DDEN-T specimens.[20] However, when the effective ligament length approaches zero, the different behavior of P-SPT specimens with respect to DDEN-T specimens stands out. There are two cases: For DDEN-T specimens, when $L=0$, the load–displacement curve approaches zero ($W_f=0$), since the specimen is separated into two halves and the force during the test is zero. However, for P-SPT specimens, if $L_{eff}=0$, the load–displacement curve does not approach zero (Figure 8), as the punch must plastically deform the two specimen halves in order to pass through them to complete the test.[9,10]

To be able to compare EWF parameters for both DDEN-T and pre-notched SPT specimens, the following points developed in previous research[9] should be kept in mind:

- The W_p value can be considered constant in pre-notched SPT specimens since the size of the plastic zone on pre-notched SPT specimens is practically the same in all cases (regardless of L_{eff}) and is related to the punch size.
- The energy of a specimen can be separated into two terms: the essential work (W_e) and the non-essential or plastic work (W_p).
- An SPT specimen divided in two halves (the essential work is zero) must be tested in order to calculate the area under the curve, which represents the plastic work (W_p) required to deform the specimen halves for the punch to pass through them during the test (in Figure 8 the area filled in).
- The total work of fracture (W_f) can be easily separated for any pre-notched SPT specimen by subtracting the constant W_p value from the enclosed energy under the test curve (Figure 8).
- It is necessary to divide the values obtained from W_e by the product of the effective ligament and the specimen thickness to obtain the specific term of the essential work ($w_{e_{P-SPT}}$).

Figure 9 shows the $w_{e_{P-SPT}} - L_{eff}$ data obtained, and it can be seen that for low L_{eff} values, the $w_{e_{P-SPT}}$ values increase significantly, so the lower bound can be set to $L_{eff}=0.3$ mm. With values between the upper and lower limits, a $w_{e_{P-SPT}}$ mean constant value of 45.8 ± 5.4 kJ/m^2 for a 0.5 mm/min test rate and 48.1 ± 5.9 kJ/m^2 for a 5 mm/min test rate was obtained. These values are very similar to each other, as was the case with DDEN-T specimens, so once again it can be considered that the test rate does not affect these results. Furthermore, these values can be compared with the w_e values in Figure 3 from the DDEN-T specimens which are approximately three times those of w_e. This correlation is similar to that observed for a polypropylene in the same earlier research mentioned previously[9] and is justified because when the specimen failure does not occur purely in uniaxial traction, since in P-SPT specimens, the w_e value can increase by more than a factor of 2.[29] If the obtained results are compared with those from Ref. 9, it can be concluded that miniature specimens provide values between two or three times that of the D-DENT specimens. That is to say there is a proportional coefficient between the miniature specimens and the D-DENT specimens that falls somewhere between 2 and 3, depending on the material used.

CONCLUSIONS

This article has contributed to the consolidation of the EWF method using the results from pre-notched miniature specimens, because self-similar load–displacement curves are obtained, which are the most important of the basic principles for the application of the method. The methodology used was the same as that developed in previous research where it was necessary to establish upper and lower limits for the ligament length of P-SPT specimens. Again, it has been found that the $w_{e_{P-SPT}}$ value in pre-notched SPT specimens is approximately three times that of the w_e value for the A-PET polymer analyzed. Moreover, the effect of the test rate on the EWF parameters in A-PET polymer pre-notched miniature specimens was analyzed, in which the main conclusion is that the EWF parameters are not affected by the test rate, as is the case in conventional DDEN-T specimens. Obviously, this conclusion cannot be extended to other polymers, and more research is needed to know how the test rate affects the behavior of other types of polymers, but it does suggest that the effect of the test rate in EWF parameters for DDEN-T specimens can be extrapolated to pre-notched miniature specimens for a specific polymer. Furthermore, the notch used provides a potential relationship between w_f and L_{eff}, so it would be interesting to find a specimen or notch geometry that would provide a linear relationship similar to those of D-DENT specimens.

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