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Structure and properties of nanocomposites with a poly(trimethylene terephthalate) matrix

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ABSTRACT

Poly(trimethylene terephthalate) (PTT), and three organically modified montmorillonites were mixed in the melt state obtaining widely dispersed nanocomposites. The parameters varied in this study were the type and amount of the organic modification and the clay content. A higher polarity of the surfactant made intercalation easier, but neither the nature (included polarity) nor the amount of surfactant influenced the dispersion level. The latter was shown directly by TEM and indirectly by the values of the modulus of elasticity. Upon mixing, a maximum interlayer distance was observed regardless of the initial interlayer distance and the nature and amount of the organoclay used. This finding was also observed in other matrices but does not appear to be a general rule. The decrease in the break properties is mainly attributed to the clay addition and the increases in the modulus of elasticity were large, in agreement with the high degree of dispersion obtained.

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1. Introduction

In the last 10 years, the research on multicomponent polymer materials has increasingly focused on the development of nanocomposites with organically modified and laminar clays, obtained mainly in the melt state. The key for the success in terms of performance is to obtain a widely dispersed nanostructure that can lead to a maximum transmission of the organoclay performance to the polymer matrix.

Different procedures have been used to obtain a large degree of dispersion. For instance, the processing parameters can be chosen to optimize dispersion. In this way, it appears that both a high shear stress during blending [1,2] and a long processing time [3] without degradation help the dispersion of the organoclay in the melt. Another procedure used to improve dispersion is a chemical modification of the matrix to ameliorate the polymer/organoclay compatibility. Polymer blending has been also used to improve dispersion. This procedure involves mixing a masterbatch based on a polymer where a large degree of exfoliation is easily obtained, such as polyamide 6, with a miscible polymer where wide dispersion cannot be readily achieved. This technique was used to produce widely dispersed polyamide 6,6 [4] and polycarbonate [5] nanocomposites where small amounts of polyamide 6 or polycaprolactone (PCL), respectively, were present.

However, the most widely used technique to help dispersion is the organic modification of the clay by a surfactant whose structure matches that of the polymer matrix of interest. The most relevant parameters that are modified to promote positive interactions with the matrix are polarity and the amount of the surfactant. All these attempts have led to obtain significant organoclay dispersion by melt blending in many polymeric matrices. As it is known, the maximum dispersion achieved (close to full exfoliation) without modification of the matrix or the use of a masterbatch was obtained in polyamide 6 [6]. Very wide dispersion was obtained in PCL [7] and polypropylene, as well [8].
In the case of melt mixed poly(ethylene terephthalate) (PET) based nanocomposites, N-alkyl amines with different tail lengths were used to modify the pristine clay. The mechanical and rheological properties improved when the alkyl tail of the amine was long, but the lack of TEM pictures impeded the inspection of the exfoliation level [9]. Different organoclays were used in an attempt to find the organomodifier that was the best at promoting dispersion with PET [10]. The addition of an ionomer to PET enhanced both the dispersion and the mechanical properties due to an enhancement of the interactions between the matrix polymer and the organoclay [11]. The mechanical properties and the morphology of recycled PET based nanocomposites have been studied [12–14]. A nucleation effect of the organoclay [15] as well as enhancements in the barrier properties [16] of PET after organoclay addition were observed.

In the case of poly(butylene terephthalate) (PBT), an organoclay with a medium polarity surfactant achieved the best dispersion [17], while a higher polarity surfactant proved more adequate when an epoxy resin was added as a third component. A high extrusion rate resulted in a decrease in degradation and improved the organoclay dispersion [18] due to the higher stress involved. A higher molecular weight PBT showed higher mechanical properties due to its higher viscosity, which allowed for shear stress to be more efficiently transferred during melt blending [19]. Sulphonated PBT is another example of a matrix in which widely exfoliated nanocomposites were obtained [20].

In the case of poly(trimethylene terephthalate) (PTT)-based nanocomposites prepared in the melt state, some work was conducted on the crystal morphology [21,22] and on its dynamic mechanical properties [21–24], as well as on toughening by rubber addition [23]. The kind of organic modification that leads to the best dispersion and mechanical properties, mainly modulus, has been only sporadically studied. Studies of the tensile strength of PTT with one type of organoclay [25], and the nanostructure and flexural properties of PTT with one commercial organoclay and two organoclays prepared in the laboratory [24] have been reported. The best dispersion occurred when the organic modification was methyl tallow bis(2-hydroxyethyl) ammonium in detriment of either octadecylamine or dodecylamine.

In this study PTT has been mixed in the melt state with several OMMT’s to obtain nanocomposites (PN’s). The parameters studied were the chemical nature (polarity) of the organoclay, the surfactant content in the organoclay and organoclay content. The observed nanostructure was related to the mechanical properties of the PN’s. The different chemical nature of the OMMT’s will also allow us to shed some light into the chemical characteristics of the OMMT that lead to either wide intercalation or dispersion in PTT based clay nanocomposites.

2. Experimental

In this study, we employed a Corterra poly(trimethylene terephthalate) (PTT) developed by Shell Chemical with an intrinsic viscosity of 0.92 dl/g in a phenol/tetrachloroethane (50/50) mixture, and three different organically modified montmorillonites (OMMT) as fillers. The first clay (20A) (Cloisite®20A, Southern Clay Products) uses 95 meq/100 g clay of dimethyl, dehydrogenated tallow, quaternary ammonium. The second (15A) (Cloisite®15A, Southern Clay Products) uses 125 meq/100 g of the same modification. The third modification (30B) (Cloisite®30B, Southern Clay Products) was methyl, tallow, bis 2-hydroxyethyl, quaternary ammonium and the amount of surfactant was 90 meq/100 g.

Before processing, PTT was dried at 145 °C in an air oven for 4 h. Each of the OMMT’s and the pure PTT were fed to a Collin ZK25 co-rotating twin-screw extruder-kneader (screw diameter of 25 mm, and length to diameter ratio of 30/1). The barrel temperature was 260 °C, and the rotation speed was 400 rpm. After extrusion, the extrudates were cooled in a water bath and pelletized. Subsequent injection moulding was carried out in a Battenfeld BA230E reciprocating screw injection moulding machine to obtain tensile (ASTM D638, type IV, thickness 1.84 mm) and impact (ASTM D256, thickness 3.1 mm) specimens. The screw of the plasticization unit was a standard screw with diameter of 18 mm, L/D ratio of 17.8 and compression ratio of 4. The melt temperature was 280 °C and the mould temperature 18 °C. The injection speed and pressure were 11.5 cm3/s and 2300 bar, respectively. The clay (MMT) contents of the resulting nanocomposites were close to 2.00%, 4.00% and 6.00% MMT. The names of the PN’s used in this manuscript refer to their MMT content.

The MMT content of the PN’s was measured after calcination, introducing dry pellets into a furnace at 900 °C. The percent MMT in the PN’s was calculated from:

\[ \%\text{MMT} = \%\text{MMT}_{\text{ash}} / 0.942 \]

where \%\text{MMT}_{\text{ash}} is the mass after calcination relative to the original nanocomposite mass. The loss of structural water during calcination of the organoclays accounts for the 0.942 parameter, and it was calculated from TGA analysis performed on the three organoclays in a prior work [3]. As the reinforcing component is the inorganic clay only, all nanocomposite comparisons are made on the basis of the weight percent of MMT in the nanocomposite, instead of the amount of organoclay.

X-ray diffraction patterns were recorded in a Philips PW 1729 GXRD X-ray diffractometer that worked at 45 kV and 50 mA, using a Ni-filtered CuKα radiation source. The scan speed was 0.5°/min. The TEM samples were embedded in an epoxy resin and ultrathin-sectioned at 60 nm using an ultramicrotome. The micrographs were obtained in a Hitachi 600AB apparatus at an accelerating voltage of 100 kV.

Dynamic mechanical analysis was performed using a TA Instruments Q800 DMA that provided the glass transition temperature (\(T_g\)). The scans were carried out in bending mode at a constant heating rate of 4 °C/min and a frequency of 1 Hz, from 0 °C to 100 °C. Calorimetric analyses (DSC) of the neat PTT and of the PN’s were performed using a Perkin–Elmer DSC-7 differential scanning calorimeter. The crystalline content of the injection moulded specimens
was calculated from the crystallization and melting enthalpies measured from the areas of the correspondent peaks of the first heating scan and the melting enthalpy of 100% crystalline PTT (145 J/g) [26].

Rheological measurements were carried out in an extrusion rheometer (Göttfert Rheotester 1000) at shear rates between 100 and 2000 s⁻¹. Infrared spectra were performed on a Nicolet spectrometer, model Magna 560 FTIR, at a resolution of 8 cm⁻¹. The signal was averaged from a minimum of 64 scans. Attenuated total reflectance (ATR) experiments were carried out using an ATR objective (Spectra Tech) with zinc selenure crystal mounted on a microscope (Spectra Tech) attached to the FTIR spectrometer, in conjunction with a MCT detector.

Tensile testing was carried out using an Instron 4301 machine at a cross-head speed of 10 mm/min and at 23 ± 2 °C and 50 ± 5% relative humidity. The Young’s modulus was determined by means of an extensometer at a cross-head speed of 1 mm/min. The ductility, measured as the break strain (εₐₜ), was determined from the load–displacement curves. A minimum of five tensile specimens were tested for each reported value.

3. Results

3.1. Intercalating ability of PTT

Figs. 1a–c show the XRD patterns of the 30B, 20A and 15A clays and of their PNs with PTT. The corresponding d(001) before and after blending as well as the blending induced Δd(001) are collected in Table 1. As can be seen, the interlayer distance increased upon mixing with PTT in the case of the 30B and 20A organoclays, 1.17 and 0.57 nm, respectively. This indicates that intercalation of the polymer molecules occurred. In the 15A organoclay, the Δd(001) is negative. This negative Δd(001) indicates that the clay layers are closer to each other in the PNs’s, and therefore that some of the surfactant migrated to the surrounding polymer during mixing. However, this surfactant migration and the decrease in d(001) do not exclude intercalation because some surfactant could be substituted by the polymer chains. This phenomenon was described in a study of SAN nanocomposites [27] suggesting that the excess of surfactant not coordinated to a charged site was potentially mobile and could migrate to the polymer matrix.

One might expect that having a larger initial intergallery distance might lead to a greater degree of intercalation, since in principle, less energy is needed to separate the clay layers. However, the values of Δd(001) reported in Table 1 for the 20A and 15A organoclays show this is not the case. This was also reported [10] in the case of PET nanocomposites and PTT nanocomposites [24], where a larger initial interlayer distance (and therefore higher surfactant content) did not increase intercalation measured as Δd(001). Thus, it appears that an initial higher interlayer distance of the organoclay does not favour an ulterior intercalation.

The Δd(001) obtained for the Cloisite 30B of this paper (1.17 nm) is larger than the best previously [24] observed (0.94 nm). Usually this difference is attributed to a different interaction or miscibility between the polymer matrix and the organoclay. However, the organic modification of Cloisite 30B and that of a previous work [24] are the same. The amount of exchanged meq/g in Ref. 24 is unknown, but they report an initial interlayer distance of 1.21 nm. Cloisite 30B has a larger initial interlayer distance (1.91 nm), indicating it has a larger content of organic surfactant in its galleries. This could indicate that a larger surfactant content leads to an increased intercalation, but other parameters, such as the processing conditions [3,18,28] and the molecular weight of the matrix [19,29] also influence intercalation.

The intercalation ability of 30B and 20A can be understood in terms of structural differences in the modifications of the organoclays, since the surfactant content is the same (95 meq/100 g clay) for both of them. The main structural difference between the modifications of the two organoclays is that the modification of the 30B organoclay has a clearly polar character due to two hydroxyl groups, while the modification of the 20A organoclay, that has two aliphatic chains, is non-polar. As there is not other relevant structural difference between the two organoclays, this higher polarity is proposed as the reason for the larger increment of the interlayer distance of the 30B organoclay related to that of the 20A organoclay upon mixing with PTT.

Another interesting result seen in Table 1 and Fig. 1 is that upon mixing with clays with different organic substitutions, the final interlayer distance of the PNs was identical regardless of the initial interlayer distance of the organoclay. This final result occurs either by increasing the initial interlayer distance of the organoclays by means of intercalation, or by decreasing an initially large interlayer distance upon migration of surfactant. The occurrence of a final constant interlayer distance was reported in several polymeric matrices and is collected in Table 2. In PN’s with a PET matrix [10], the d(001) after melt mixing with three different organoclays was always 3.50 nm. In PN’s with a poly(styrene-co-acrylonitrile) (SAN) matrix most of the PNs had final interlayer distance of around 3.2 nm regardless of the organoclay used [27]. In PN’s with a PMMA matrix, all the PN’s presented final interlayer distances between 3.3 and 3.4 nm [30]. These results suggest that when a polymeric matrix is mixed with an organically modified clay and intercalation occurs, there is an ultimate constant interlayer distance in the intercalated PNs that depends on the polymer matrix and is independent of the organomodification used. However, this is not a general rule, since it has been observed that the change in d(001) after melt mixing in polycarbonate [31], poly(vinyl chloride) [32] and polypropylene [33] is dependent on the employed organoclay. At this time there is no evidence of whether this partially observed rule exists or not, or whether there are additional parameters that influence intercalation that lead to exceptions of its applicability.

3.2. Nanostructure

Figs. 2a–c show the TEM pictures of the 30B, 20A and 15A PNs with 2% MMT and Figs. 2d–f the correspondent
with 6% MMT. As can be seen, the organoclay particles are homogeneously dispersed in all the observed area in the three PN’s. Although the average number of layers per particle appears to be smaller in the case of the 30B PN, the dispersion level was very similar. This does not always occur [23], and indicates that the clay organomodifier did not affect the level of dispersion.

Intercalation (as measured by X-ray) and exfoliation (observed by TEM) are often considered related processes, intercalation being the first step towards exfoliation. In this way, the parameters that would favour one process should also benefit the other. Recent papers, however, have questioned this presumably reasonable possibility because sometimes a high degree of intercalation did not result in a high degree of exfoliation [3] and the parameters that lead to high intercalation and high dispersion are not the same. Additional discussion on the dispersion level and on interactions between the polymer and the

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**Fig. 1.** WAXD patterns of (a) 30B organoclay and 30B PN’s, (b) 20A organoclay and 20A PN’s and (c) 15A organoclay and 15A PN’s.

**Table 1**

<table>
<thead>
<tr>
<th>Clay</th>
<th>Initial (nm)</th>
<th>After blending (nm)</th>
<th>( \Delta d(001) ) (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30B</td>
<td>1.91</td>
<td>3.08</td>
<td>1.17</td>
</tr>
<tr>
<td>20A</td>
<td>2.51</td>
<td>3.08</td>
<td>0.57</td>
</tr>
<tr>
<td>15A</td>
<td>3.57</td>
<td>3.11</td>
<td>-0.46</td>
</tr>
</tbody>
</table>

**Table 2**

<table>
<thead>
<tr>
<th>Polymer matrix</th>
<th>Organoclay</th>
<th>Final interlayer distance (nm)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>PET</td>
<td>30B, 20A, 15A</td>
<td>3.50</td>
<td>[10]</td>
</tr>
<tr>
<td>PTT</td>
<td>30B, 20A, 15A</td>
<td>3.10</td>
<td>This work</td>
</tr>
<tr>
<td>SAN</td>
<td>30B, 20A, 25A, 6A, 15A</td>
<td>3.20</td>
<td>[27]</td>
</tr>
<tr>
<td>PBT</td>
<td>30B, 20A, 15A</td>
<td>3.75</td>
<td></td>
</tr>
</tbody>
</table>
organoclays will be made taking into account the modulus of elasticity.

3.3. Thermal properties

The $T_g$'s of the PN's (Table 3) were measured by DMTA and compared with that of the pure PTT. The $T_g$ of the pure PTT (47.5 °C) was lower than that of previous works [21, 24] indicating a higher amorphous nature of the PTT of this study, which has to be a consequence of the cooling conditions used. The $T_g$ of PTT, as that of other partially crystalline polyesters, changes widely depending on the crystalline content [22], and even two $T_g$'s have been observed corresponding to the amorphous phase far and

Fig. 2. TEM images of (a) 2% MMT 30B PN, (b) 2% MMT 20A PN and (c) 2% MMT 15A PN, (d) 6% MMT 30B PN, (e) 6% MMT 20A PN and (f) 6% MMT 15A PN.
close to the crystalline zones [21]. The T_g of PTT of this study did not change after OMMT addition. Both increases in T_g and lack of change of the T_g have been reported [21,24] in other PTT based PN’s, depending on the OMMT used. This effect has been attributed to different interactions between the OMMT and the matrix [24]; however, a possible change in the crystalline content of the nanocomposites as a result of the different OMMT should also be taken into account.

The crystalline characteristics of the nanocomposites and the pure PTT were analyzed by means of DSC measurements. The thermograms are shown in Fig. 3, the values of T_m and T_c are shown in Table 3 and the crystalline content in Fig. 4. As can be seen in Fig. 3, the T_c’s of the PN’s were lower than that of the pure PTT regardless of the nature of the organic modification. Similar results were seen at other MMT contents. This indicates a nucleation activity of the OMMTs that was corroborated by the decreases in T_m.

As can be seen in Fig. 4, a rather linear increase in crystalline content with the MMT content is shown. This increase was independent of the nature of the organic modification (estimated error 2–5%). This is in contrast to that seen in a previous work [22], where the crystallization and melting behaviour did not change at organoclay contents above 3%. The higher crystalline content indicates that the organoclays act as nucleation agents [22]. The crystalline content increased from 19% to roughly 27% in the case of the PN’s with 6% MMT. Nucleation activity of the organoclays was also observed in PET [10,34] and in PTT [21]. This increase in the crystalline content will hardly influence the modulus behaviour because a much larger increase is necessary to observe significant changes in the modulus of elasticity. Consequently, the modulus behaviour will not be a consequence of the nature of the PTT and will be attributed to the presence of the clay.

### Table 3

<table>
<thead>
<tr>
<th>Composition</th>
<th>T_g (°C)</th>
<th>T_c (°C)</th>
<th>T_m (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PTT</td>
<td>47.5</td>
<td>76</td>
<td>234</td>
</tr>
<tr>
<td>30B 2%</td>
<td>47.6</td>
<td>74</td>
<td>230</td>
</tr>
<tr>
<td>30B 4%</td>
<td>47.7</td>
<td>73</td>
<td>232</td>
</tr>
<tr>
<td>30B 6%</td>
<td>47.4</td>
<td>71</td>
<td>229</td>
</tr>
<tr>
<td>20A 2%</td>
<td>48.5</td>
<td>73</td>
<td>230</td>
</tr>
<tr>
<td>20A 4%</td>
<td>48.3</td>
<td>72</td>
<td>230</td>
</tr>
<tr>
<td>20A 6%</td>
<td>47.6</td>
<td>70</td>
<td>231</td>
</tr>
<tr>
<td>15A 2%</td>
<td>47.8</td>
<td>72</td>
<td>230</td>
</tr>
<tr>
<td>15A 4%</td>
<td>46.8</td>
<td>69</td>
<td>231</td>
</tr>
<tr>
<td>15A 6%</td>
<td>47.9</td>
<td>70</td>
<td>230</td>
</tr>
</tbody>
</table>

* T_g was measured by DMTA analysis.

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3.4. Rheological behaviour

Rheological results are shown in Figs. 5a–c (PN’s with the 30B, 20A and 15A organoclays, respectively). Rheological measurements were made with the double aim of detecting either a possible degradation of the polymer matrix during the processing or a plasticizing effect of the surfactant. As can be seen, the viscosity of the nanocomposites decreases as the clay content increases. Moreover, the decrease in viscosity does not depend on the organic modification of the clay, but appears to be related to the presence of small amounts of organoclay because it does not change appreciably with organoclay content.

Among the reasons for the observed drop in viscosity we can mention surfactant migration, change of the chemical nature of the PN’s by degradation and a decrease of the molecular weight of the matrix. A migration of the surfactant to the matrix can take place during mixing [5,35] and given its small molecular weight, it would lead to a plasticizing effect. However, the 15A OMMT had more surfactant than the 30B OMMT and there is no difference in viscosity between the corresponding PN’s. This rules out the migration of the surfactant as a reason for the viscosity decrease.

Pure PTT and the 6% 30B PN were analyzed by FTIR and the results are shown in Fig. 6. In agreement with previous results [26], it can be seen that the two spectra show the same peaks and the peaks are on the same position.
indicating that no major change of the chemical nature of the PN occurred during processing. This rules out a change of the chemical nature of the PN’s as the reason for the viscosity change.

With respect to a decrease in the molecular weight as the reason for the observed decrease in viscosity, the effects of the OMMT presence on the viscosity of polyesters are not clear as both increases [36] and decreases [37] have been reported. This could be due in part to the different type of viscosity measured. A decrease in the molecular weight has been reported to be the main degradation way of PTT during processing [26]. The molecular weight measured by intrinsic viscosity decreased from 69,000 to 54,000 after one extrusion cycle at 270 °C, and to 31,000 after four extrusion cycles. Besides this matrix degradation [26], the action of the surfactant cation could be another reason for observing a decrease in molecular weight [38]. Additionally, a decrease in viscosity was also observed in PA6 nanocomposites and was attributed to a decrease in molecular weight during the extrusion process [39]. The decrease in molecular weight was induced by the organoclays, since it was significantly smaller upon the extrusion of the neat PA6.

Therefore, having ruled out a plasticizing effect of the surfactant, and a significant change of the chemical nature of the PN’s as possible reasons for the observed behaviour, and being the decrease in the molecular weight the most often observed reason for the viscosity, a decrease in the molecular weight of the PTT matrix is proposed as the main reason for the observed viscosity behaviour.

3.5. Tensile properties

The tensile moduli of the PN’s are shown in Fig. 7. As can be seen the modulus of elasticity increases with the clay content. The increase in the modulus of elasticity is mostly linear attaining values roughly 50% greater than that of the PTT matrix upon 6 wt% clay addition. This increase in modulus is greater than those previously obtained in PTT with different organoclays both in the case of the tensile [23] and flexural [24] modulus. Table 4 compares our results to those observed in both modified and unmodified matri-
ces, such as maleic anhydride grafted polypropylene [8] and PA6 [6] with highly dispersed clays. The comparison of the relative modulus of the different matrices gives a valuable representation of the attained dispersion level of the PTT based nanocomposites of this study.

Fig. 7 shows small differences in the modulus, depending on the nature of OMMT; however, the differences are not significant. Therefore, the modulus results are in accordance with the TEM analysis, where the difference in the degree of dispersion between the different organoclays was not definitive. Other possible parameters that could influence the modulus are crystalline content or molecular weight. However, the observed changes of crystallinity were too small to significantly influence the mobility of the amorphous phase, mainly when compared with the effect on mobility typical of the OMMT addition. With respect to the molecular weight, its changes consequence of degradation are not large enough to influence the modulus [26].

The results for the modulus are clearly more conclusive with regards to the dispersion level than those of TEM pictures. This is because, even though the morphologies observed in TEM pictures were chosen as the most representative of a larger area, they represent scarcely one square micron instead of the full specimen in the case of the modulus of elasticity. The similar values for the moduli of elasticity of Fig. 7 indicate that neither the surfactant amount, nor its polarity drastically influence the overall dispersion level attained. Moreover, this also shows

<table>
<thead>
<tr>
<th>Polymer Matrix</th>
<th>Relative modulus</th>
<th>Reference</th>
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<tbody>
<tr>
<td>PET</td>
<td>1.41</td>
<td>[10]</td>
</tr>
<tr>
<td>PTT</td>
<td>1.53</td>
<td>This work</td>
</tr>
<tr>
<td>PA6</td>
<td>1.67</td>
<td>[6]</td>
</tr>
<tr>
<td>PP*</td>
<td>2.30</td>
<td>[8]</td>
</tr>
<tr>
<td>HDPE*</td>
<td>1.27</td>
<td>[2]</td>
</tr>
</tbody>
</table>

![Fig. 6. FTIR analysis of pure PTT and 6% 30B PN.](image1)

![Fig. 7. Young’s Modulus of pure PTT and 30B, 20A and 15A organoclay PN’s vs %MMT content.](image2)

![Fig. 8. Elongation at break of pure PTT and 30B, 20A and 15A organoclay PN’s vs %MMT content.](image3)
that, as observed before [3,27], the parameters that lead to high intercalation and high dispersion are not the same. Similar results were reported in PN’s based in PET[10].

The elongation at break of the PN’s is shown in Fig. 8. As can be seen, the PN’s with 2 wt% MMT are clearly ductile, as breaking occurred in the uniform elongation subsequent to the cold drawing. At higher clay contents (4 and 6 wt% MMT) the specimens mostly broke before yielding. As in the case of PET [10], the impact strength also decreased upon OMMT addition from 27 to 12–15 J/m whatever the OMMT nature and content. Besides the probable slight negative effect of the decrease in molecular weight of the matrix, the organoclay presence appears as the main responsible for the loss of impact strength and in the elongation at break of Fig. 8. These effects on the fracture properties are often observed when organoclays are added to polymers to produce nanocomposites [4,10,19,24].

4. Conclusions

The intercalation level of the PTT in the 30B OMMT was greater than in the 20A OMMT, and it was shown to be independent of the initial interlayer distance (surfactant content). As other structural characteristics were not significantly changed and the surfactant content was the same, the more polar nature of the 30B organoclay, that should lead to an easier interaction with the also polar ester groups of PTT, is proposed as the reason for the higher intercalation.

A maximum interlayer distance was seen regardless of the organic modification used. As this also occurred in other melt mixed polymer nanocomposites, it appears that a maximum interlayer distance previous to exfoliation exists. This is although other parameters seem to affect because exceptions are also contemplated.

The dispersion degree measured both directly by TEM and indirectly by the modulus of elasticity did not depend on either the polarity or the amount of surfactant. These results confirm previous findings on the fact that the parameters leading to large intercalation and wide dispersion are different. The increases in the modulus of elasticity were similar to those obtained in other matrices, but the break properties decreased in the nanocomposites probably due to the decrease in molecular weight of the matrix and mainly to the presence of clay.

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[10] U.G. also acknowledges the Basque Government for the award of a grant for the development of this work.


