

Article

Influence of a Multifunctional Epoxy Additive on the Performance of Polyamide 6 and PET Post-Consumed Blends during Processing

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Abstract: In this study, a commercial chain extender (Polyad PR 002) in concentrations of 0.5, 1.0, and 1.5% *w/w* was used as a compatibilizer in post-consumed PET/PA6 blends (25/75, 50/50 e 75/25) processed in a laboratory mixer. The degradation rate, phase miscibility, chemical interaction, crystallization, and melting behavior, as well as the thermal stability of the mixtures, were analyzed by torque rheometry, scanning electron microscopy (SEM), FTIR spectroscopy, differential scanning calorimetry (DSC), and thermogravimetry (TGA), respectively. The results showed that the addition of 1% chain extender was sufficient to increase the melt viscosity of the PET-rich mixtures and 1.5% for the PA6-rich samples. Clearly, adding Polyad PR 002 to PET/PA6 blends improves polymer compatibility; for the 25%PET/75%PA6 blend with 1.5% PR 002, complete miscibility was observed, indicating a new compatible blend formation, revealing a chemical reaction between the systems, and proving the synergism between them. This chain extender did not affect the thermal stability of the blends, and the low contents employed reduced the crystallization rate of the blends investigated.

Keywords: compatibilization; PET/PA6 mixtures; epoxy additive



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1. Introduction

The production of blends stands out as a way to obtain new polymeric materials without the need to invest in the development of new polymers or new polymerization processes [1]. Blends of virgin and post-consumer polymers are a viable solution for recycling polymeric waste, particularly PET [2–4].

Waste recycling is a strategic process that enables the expansion of the plastics market, which can generate new products, in addition to minimizing the environmental impact caused by the incorrect disposal of PET packaging [5–8].

By blending two engineering plastics such as PA6 and PET, which have low gas permeation and high thermal, chemical, and electrical resistance, one expects to obtain a material combining the toughness and high barrier properties of PA6 and the good finish and better dimensional stability of PET. Such material could be applied in several engineering areas, such as, for instance, in technical parts and special packaging [9–11]. A counterpoint to the feasibility of this mixture is that PET and PA6 are condensation polymers with different functional groups, so their mixture tends to be immiscible, resulting in polymer blends with inferior properties compared to the neat polymers, especially mechanical properties [4,6,12].

Several studies [3,13–16] show that mixing PET with polyamide 6 leads to an immiscible blend. Phase separation was clearly seen, while the mechanical properties of the blends were reduced. These authors suggested that interfacial adhesion between phases should be

improved for better mechanical properties to be achieved. Yan et al. (2019) [17] reported the importance of reversing the hydrolytic degradation process of blends with high PET content, as it has been shown that PET accelerates the hydrolysis of PA in PET/PA blends, and this can greatly degrade the mechanical properties of PA6 and, as a result, of the blend itself [18].

Therefore, to achieve the desired property in immiscible polymer blends, the addition of a compatibilizer is often necessary. The compatibilizer can function as an interfacial agent to decrease the interfacial tension and improve interfacial bonding adhesion between the polymers and, in this way, improve the properties of the mixtures [3,19,20].

Functional additives are mainly added to degraded condensation polymers with the aim of reconnecting polymer chains that have been broken during thermal, oxidative, or hydrolytic degradation [21]. Polymer recycling is a necessity in our consumer-intensive polymer world, but the nature of polymers is such that they are prone to thermal degradation when reprocessed, and this poses technical challenges for recycling [22,23].

A widely used chain-extender additive is Polyad, also known as Joncryl, which is a multifunctional reactive polymer containing nine epoxy groups that was developed to reverse thermal degradation and recover the molar mass of condensation polymers such as PET, PC, and nylon [24–26].

Joncryl was selected because it contains epoxy rings that can react with the terminal amine and carboxyl groups of PA6 and the hydroxyl and carboxyl groups of PET to elongate the macromolecular chains, leading to blends with a higher melting viscosity. Previous studies showed that Joncryl had performed dual actions, namely as a chain extender and compatibilizer for polymeric systems [15,27–30].

Hongsriphan et al. (2020) investigated various proportions of a Joncryl compatibilizer in a blend of 80 wt.% PET and 20 wt.% polyamide 11 (PA11) and showed the highest increase in tensile strength, impact strength, modulus Young's strength, and flexural strength for the system with the 2% chain-extender additive.

Khan et al. (2021) investigated the blend of Poly (butylene succinate)/biobased PA11 and an epoxy-based chain extender, Polyad ADR-4300, which was added to improve blend compatibility. The addition of the chain extender led to better chemical compatibility in the blends, as evidenced by smaller dispersed domain sizes and less pullout. The results of the tensile strength, Young's modulus, and flexural strength increased with the chain-extender addition in all concentrations: the tensile strength of the blend at 2 phr (joncryl®) remarkably increased from 26.8 to 46.24 MPa with a uniquely increased strain% from 3.56% to 196%. The Young's modulus was also significantly improved. The impact strength rose from 147.12 to 667.68 J/m.

One of the goals of chain extension is to improve the physical properties of polymers. Chain extension has the ability to reverse the loss of molar mass of post-consumed PET and other polyesters that is caused by hydrolysis and can modify the rheology of the polymer, increasing its melt strength. The use of chain extenders is quite simple, as they can be added during processing [16,31,32]. The reaction that occurs during mixing consists of the opening of the ring of the epoxides towards the carboxyl or secondary hydroxyl groups, creating new covalent bonds and increasing the molecular weight of the polymer [24,33].

The chain-extension effect can be evaluated by measuring the torque during processing in a torque rheometer, eliminating the temperature effects by correcting the torque to a specific temperature (reference temperature) to obtain the relative rate of degradation. In this work, the main objective is the development of new blends of polyamide 6 (PA6) and post-consumer polyethylene terephthalate (PET). The use of post-consumer PET will allow the sustainable production of new materials for packaging and technical parts in the automotive industry. The effect of the chain extender additive (Joncryl) was analyzed by torque rheometry to investigate the effect of the additive content on the viscosity variation and on the rate of degradation under processing. The crystallization and melting behavior were analyzed by differential scanning calorimetry and thermal stability by thermogravimetric analysis. The phase miscibility of the blends was analyzed by scanning

electron microscopy, and the chemical interaction was studied by Infrared absorption spectroscopy (FTIR).

2. Materials

Polyamide 6 was supplied by Thati Polymers (São Paulo, Brazil), with a density of 1.13 g/cm³. The post-consumed PET (PET) used in this study came from colorless soft drink bottles collected in the state of Paraíba and supplied as flakes by the company DEPET/PB (Brazil). The multifunctional epoxy additive Polyad PR 002 (a blend of Joncryl 4368 and Joncryl 4370), which was supplied by BASF (Brazil), was used as a chain extender for PET, and in this work, it is called “Joncryl”. With an average molar mass of 6800 g/mol, this additive contains 4 to 10 units of epoxy groups per molecule [34].

3. Methodology

3.1. Polymer Blends and Process for Preparation

PET and PA6 were dried in a vacuum oven operating at 80 °C for 24 h before processing.

PET/PA6 blends with 0, 25, 50, and 75%PET (by weight) and 0, 0.5, 1, and 1.5% Polyad PR 002 were mixed for 10 min in an Haake Rheomix laboratory mixer 600 equipped with high-intensity rotors (roller type rotors). Operating conditions were a nominal speed of 60 rpm with the chamber wall temperature maintained at 265 °C and a 70% fill factor at room temperature. The incorporation of the chain extender additive was carried out in a processing time of 7.5 min. A method proposed by Alves et.al [35] was used to investigate polymer degradation during processing in an internal mixer. The method allows for the quantitative estimates of the polymer degradation rate in real time.

3.2. Scanning Electron Microscopy (SEM)

Morphological analyses of the interface of the blends were conducted by using scanning electron microscopy with a MIRA 3 instrument, Tescan Mira, high-brightness FEG source in high vacuum mode, at an acceleration voltage of 15 kV. The samples consisted of pressed films fractured in liquid nitrogen and metalized with a thin layer of gold. Their fracture surfaces were observed at various magnifications.

3.3. Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) analyses were performed on a Mettler Toledo DSC-1 instrument, using a standard closed aluminum crucible with a volume of 40 µL. The crucible lid was perforated. The test took place under a nitrogen atmosphere with a flow rate of approximately 50 mL/min.

All samples, i.e., neat PA or PET and PET/PA6 blends with and without compatibilizer, were subjected to a three-stage thermal program: heating from 30 to 300 °C, cooling to 30 °C, and reheating to 300 °C. The heating/cooling ratio was 10 °C/min. Calculations were performed by using customized software (INTEGRAL).

The latent heat of crystallization/fusion per unit mass of the crystallizable polymer was evaluated as shown in Equation (1):

$$\Delta H = \frac{E_0}{w_p m_s} \quad (1)$$

where m_s is the sample weight, and w_p is the weight fraction of the polymer in question. The change in the degree of crystallinity during the event was estimated by using Equation (2):

$$\Delta X = \frac{\Delta H}{\Delta H_m^0} \quad (2)$$

where ΔH_m^0 is the latent heat of fusion of the 100% crystalline polymer. Values of 230 J/g and 140 J/g for PA6 and PET, respectively, as mentioned in the literature [19], were used.

3.4. Thermogravimetric Analysis (TGA)

Thermogravimetric tests were performed with a Mettler Toledo TGA 2 Star System instrument for neat and additivated polymers and PET/PA6 blends. Measurements were performed on samples weighing approximately 10 mg (placed in ceramic crucibles) and heated from 30 °C to 700 °C at 10 °C/min, under a nitrogen atmosphere, at a flow rate of 50 mL/min. The determination of mass loss (Δm), decomposition temperature ($T_{\frac{1}{2}}$), and mass loss rate (R) are presented in the Supplementary Materials.

3.5. Fourier-Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) was used to analyze the interactions between the functional groups that constitute the blends with the chain extender additive. The spectra in the infrared region were obtained in a Frontier Single Range-MIR FTIR Perkin-Elmer spectrophotometer operating in the region from 4000 to 220 cm^{-1} , with a resolution of 4 cm^{-1} . The measurements were carried out with the aid of the Attenuated Total Reflectance (ATR) accessory.

4. Results and Discussion

4.1. Analysis of Degradation during Processing

It is known that torque response is not a well-defined rheological property. However, the torque rheometer can be used to show how the polymer melts and different concentrations of chain extenders react with each other. The data regarding the incorporation of the extender additive in PET were discussed in the publications by Duarte [34] and Tavares [36] and indicate that the composition of 0.5% of the additive is sufficient to compensate for the degradation effect on PET after consumption. In this study, pure PA6/PET blends containing concentrations of 0, 0.5, 1, and 1.5% of Polyad PR 002 were processed in an internal mixer coupled to a Haake torque rheometer operating at 265 °C, with a rotor speed of 60 rpm and for 10 min.

Figure 1a,b illustrates the results of temperature and torque for 50%PA6/50%PET blends without additives and those containing 0.5, 1, and 1.5% of the additive. The curves for the other compositions can be found in the Supplementary Materials. Table 1 shows the average temperature and torque results during processing for neat and additivated blends in the last stages of processing (7–10 min).

In the temperature curves (Figure 1a), it is observed that initially there is a decrease in temperature, attributed to the exchange of heat with the environment during the opening of the chamber for feeding the polymer and the absorption of heat by the polymer during its melting, and then the temperature rises and tends to reach a plateau.

In the torque curves (Figure 1b), the torque increases rapidly when the material was fed into the mixer due to friction between the solid particles, then decreases as the systems melt and stabilizes at around 3 min of processing. This behavior was similar for all systems produced.

The curves also show the changes that occurred with the incorporation of the chain-extender additive at $t = 7.5$ min.

After adding the Joncryl chain extender, a small initial drop in torque is observed, which may be associated with a decrease in internal pressure when the piston that closes the supply port is released to introduce the additive. Once the torque stabilized in the final stage of 1 min (or 60 s), it was observed that the torque of the additivated blends were higher than that of the neat blend, which can be attributed to chain extension (Table 1).

Figure 2 shows in detail the adjusted torque results, that is, without the temperature effect (proportional to the viscosity at the reference temperature), for polyamide PA6, and its 25% and 75% PA6 blends without and with 0.5 and 1.5% Joncryl.

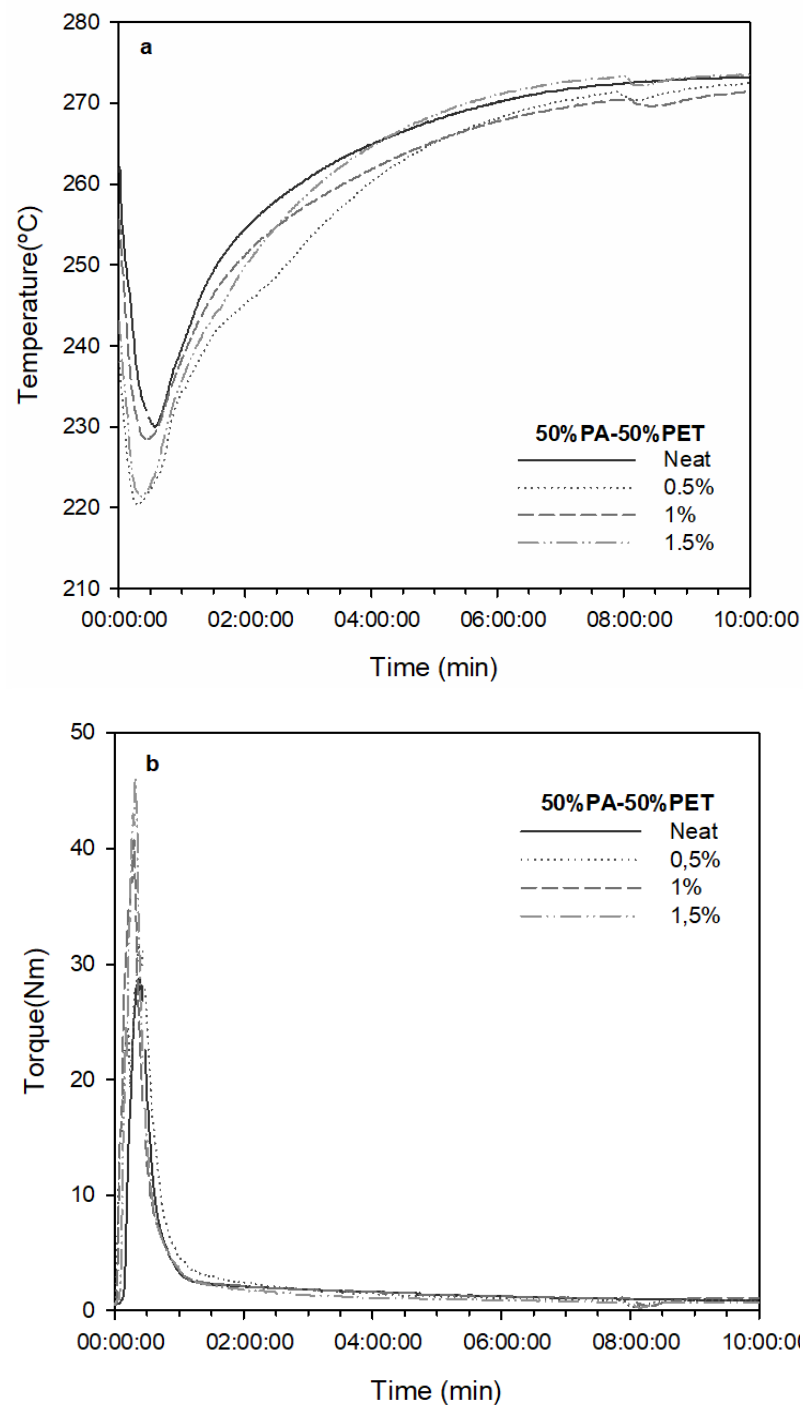


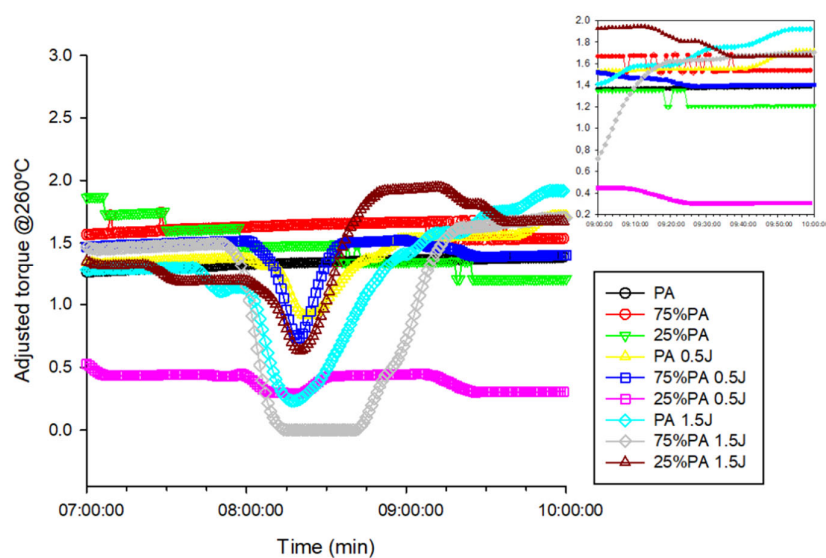
Figure 1. Temperature (a) and torque (b) curves as a function of time for pure polymers and pure and additive blends at 265 °C and 60 rpm for 10 min.

The data show that the incorporation of Joncryl led to an increase in torque for the neat PA6 and the blends and that the torque increased with the chain-extender concentration. This increase was more pronounced for the neat PA6 sample with 1.5% Joncryl. For blends with 1.5% of Joncryl (25% and 75% by weight of PA6), it is observed that the additive increased the torque when compared to blends without additives and other percentages of additive (0.5% and 1.0%).

Table 1. Parameters calculated during the processing of blends.

	Composition (PET/PA6)	T (°C)	Z (Nm)	Z* (Nm)	dZ/dt	Rz (%/min)
No additive	100%/0	271.1 ± 0.055	1.3 ± 0.004	2.1 ± 0.006	−0.1 ± 0.007	−5.6
	75%/25%	266.5 ± 0.535	0.8 ± 0.250	1.0 ± 0.081	−0.09 ± 0.018	−28.6
	50%/50%	269.2 ± 0.073	0.9 ± 0.004	1.4 ± 0.006	−0.1 ± 0.006	−7.4
	25%/75%	268.8 ± 0.091	1.1 ± 0.004	1.6 ± 0.005	−0.06 ± 0.008	−4.1
	0/100%	269.6 ± 0.085	0.9 ± 0.001	1.3 ± 0.001	−0.03 ± 0.008	2.5
0.5 J	0/100%	266.0 ± 0.656	1.0 ± 0.196	1.2 ± 0.043	0.46 ± 0.038	28.6
	25%/75%	273.4 ± 0.012	0.7 ± 0.001	1.1 ± 0.0464	0.0166 ± 1.1000 × 10 ^{−3}	1.5
	50%/50%	266.9 ± 0.6714	1.1 ± 0.2040	1.4 ± 0.041	−0.0716 ± 0.0178	−5.1
	75%/25%	271.5 ± 0.047	0.9 ± 0.007	1.3 ± 0.010	−0.2 ± 0.008	−13.6
1.0 J	0/100%	271.2 ± 0.059	1.1 ± 0.006	1.6 ± 0.011	0.2 ± 0.012	13.9
	25%/75%	268.7 ± 0.549	1.1 ± 0.165	1.4 ± 0.041	0.3 ± 0.011	15.6
	50%/50%	272.1 ± 0.028	0.8 ± 0.321	1.3 ± 0.058	−0.1 ± 0.027	7.3
	75%/25%	272.4 ± 0.056	2.9 ± 0.029	4.5 ± 0.047	0.5 ± 0.093	12.1
1.5 J	0/100%	270.6 ± 0.527	0.8 ± 0.151	1.1 ± 0.059	0.9 ± 0.023	50.1
	25%/75%	270.5 ± 0.064	0.6 ± 0.005	0.9 ± 0.009	0.1 ± 0.017	17.4
	50%/50%	270.5 ± 0.052	1.0 ± 0.015	1.5 ± 0.023	0.4 ± 0.029	28.2
	75%/25%	266.5 ± 0.535	1.3 ± 0.222	1.6 ± 0.065	−0.5 ± 0.044	−36.6

T = temperature; Z-torque; Z* = adjusted torque; dz/dt = torque variation; Rz = degradation rate.

**Figure 2.** Torque adjusted as a function of time in the last 3 min of processing.

A drop in torque and temperature is observed after adding the chain extender, which recovers after 30 to 60 s and then increases, revealing an increase in magnitude (molar mass) resulting from the chain extension. The degradation/chain extension process is a dynamic process in which the breakage or scission of the chains of the mixture occurs (decrease in molar mass/torque) occurs simultaneously with the formation of additive bridges between the chains (increase in molar mass/torque).

The sample with 75%PET showed a lower torque value than that with 75% PA, suggesting that a sample with a higher post-consumed PET content has a lower viscosity than a sample rich in PA6. This can be attributed to the origin of the PET used here, as it is a

post-consumption material and, as such, has a variety of contaminants that can alter this property.

Table 1 presents the data used to evaluate the degradation during the processing of the neat polymers and of the blends. T is the temperature in degrees Celsius ($^{\circ}\text{C}$), Z is the torque in Nm, Z^* is the adjusted torque in Nm at a reference temperature of 260°C , dZ/dt is the rate of change of torque in Nm/min, and R_z is the rate of change of torque at the final stages of processing (x-y min) in %/min.

Negative rates correspond to a decrease in viscosity (R_z) with time; that is, they are measures of the incipient degradation rate of the polymers during the last processing stage. On the other hand, positive rates correspond to an increase in viscosity and molar mass with time and reveal the recovery of the mechanical properties of the melt, possibly due to chain extension, branching, and/or crosslinking of the polymeric chains.

Figure 3 shows the comparison of results obtained for pure PA6 and blends without additives and additivated with Joncryl at concentrations of 0.5%, 1.0%, and 1.5%.

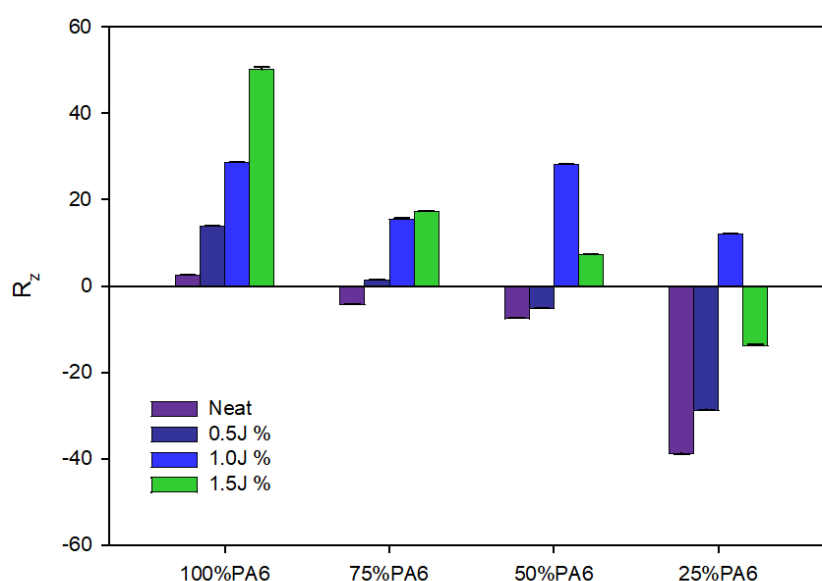


Figure 3. Relative variation rate of the adjusted torque R_z (%/min) for pure PA6 and its pristine and additive blends.

Figure 3 shows the degradation rate in the last minutes of processing in the internal mixer for the pure components and the pristine blends (without additives). We can also observe the absence of degradation for the PA6 sample and a moderate degradation for the blends; this can be attributed to the addition of PET, which introduced extra hydroxyl groups, causing greater degradation of PA. Comparing the additive compositions, it can be seen that the concentration of 0.5% was not able to reverse the degradation of the blends with 50%PA6–50%PET and 75%PA6–25%PET, as, for PA6 (100%), recovery is observed for all additive percentages, with 1.5% Joncryl being the most noticeable.

This shows that the chain extender epoxy groups (Joncryl) and $-\text{NH}_2$ groups of PA6 reacted to form branches in PA6, and then the increase in Joncryl content in PA6 increased the content of reactive epoxy groups to PA6. This increased the number of branches, resulting in an overall increase in torque.

For the blend with 75%PET/25%PA6, a particular feature is observed: the incorporation of 1.5% Joncryl does not compensate for the degradation of the blend. An explanation for this is that the same compound that acts as a stabilizer may act as a prodegradant when incorporated into the polymer chain. Once the additive undergoes the chemical changes inherent in the stabilization process and is consumed, it will transform into another chemical compound for degradation.

Thus, we concluded that the mixtures of chain extenders could react with both the amino and carboxyl terminal groups of PA6 and the hydroxyl and carboxyl groups of PET, as shown in the works by Duarte et al. [34] and Tavares et al. [36].

4.2. Scanning Electron Microscopy (SEM)

The addition of a chain extender with six epoxy functionalities, such as Joncryl, per polymer chain could introduce partial miscibility through the chain-extension reaction between these polymer molecules.

Figures 4 and 5 show SEM micrographs of 25%PET/75%PA6 and 75%PET/25%PA6 blends with 0, 1.0, and 1.5% Joncryl.

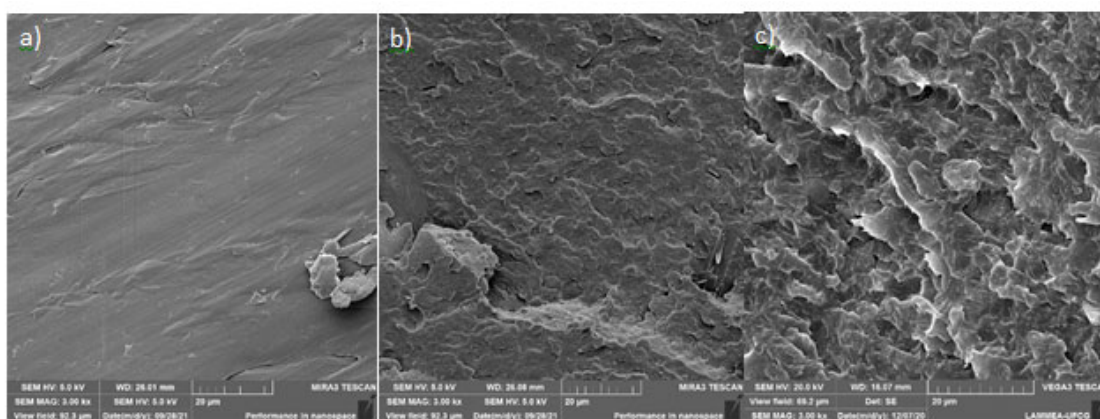


Figure 4. Scanning electron microscopy (SEM) images of blends with a composition of 25%PET/75%PA6 with 1.5% Joncryl (a) with 1% Joncryl (b); without additives (c) at 3000 $\times$ magnification.

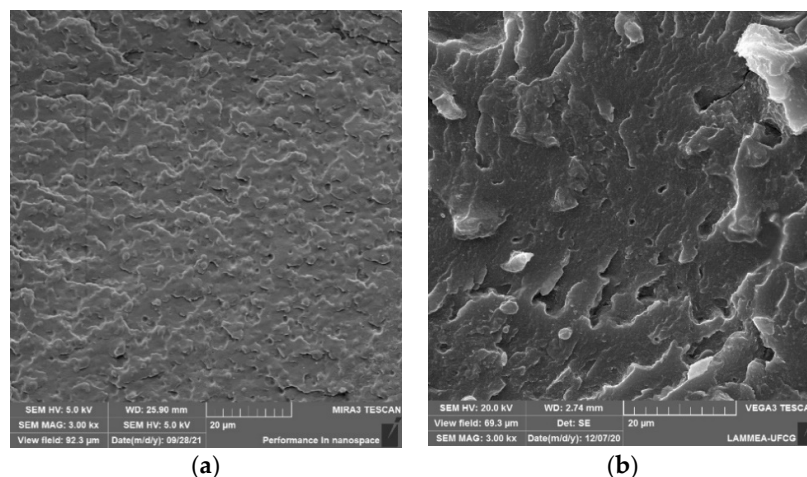


Figure 5. Scanning electron microscopy (SEM) images of blends with a composition of 75%PET/25%PA6 with 1.5% Joncryl (a) without additives (b) at 3000 $\times$ magnification.

The SEM micrographs clearly indicate that PET/PA6 blends without Joncryl are immiscible blends.

The micrographs of PET/PA6 blends with 1.5% Joncryl obtained in the internal mixer, as shown in Figures 4 and 5, illustrate that the incorporation of the additive led to good adhesion between the matrix and the dispersed phases.

This result is highlighted by the total disappearance of the gaps at the interface and the empty cavities corresponding to the extracted particles, indicating a decrease in the interfacial tension.

It was observed that this compatibilization effect was pronounced in the samples with PET as the dispersed phase in the PA6 matrix, and this can be attributed to the good

molecular interaction and better interfacial adhesion between the two phases since Joncryl can also act as a compatibilizer that reduces the interfacial tension of polyesters.

Nofar and Oğuz (2019) [27] reported the ability of Joncryl to reduce the interfacial tension between the PLA and PA11 mixtures.

This result is also similar to that reported by Zhou et al. [37]. According to these authors, the increase in melt strength influences compatibilization. Since chain extension reduces the difference between the viscosities of the polymers in the blend, the dispersed phases can be stretched more easily by the applied shear stress, and, therefore, they are divided into smaller domains distributed within the main matrix.

4.3. Fourier Transform Infrared Spectroscopy (FTIR)

Figure 6a–c show the FTIR spectra of the PA6/PET blends with 25, 50, and 75% *w/w* of PA6 additivated with 0.5 and 1.5% *w/w* of Joncryl. The spectra referring to Joncryl, neat polyamide 6, and with 0.5 and 1.5% *w/w* added are found in the Supplementary Materials.

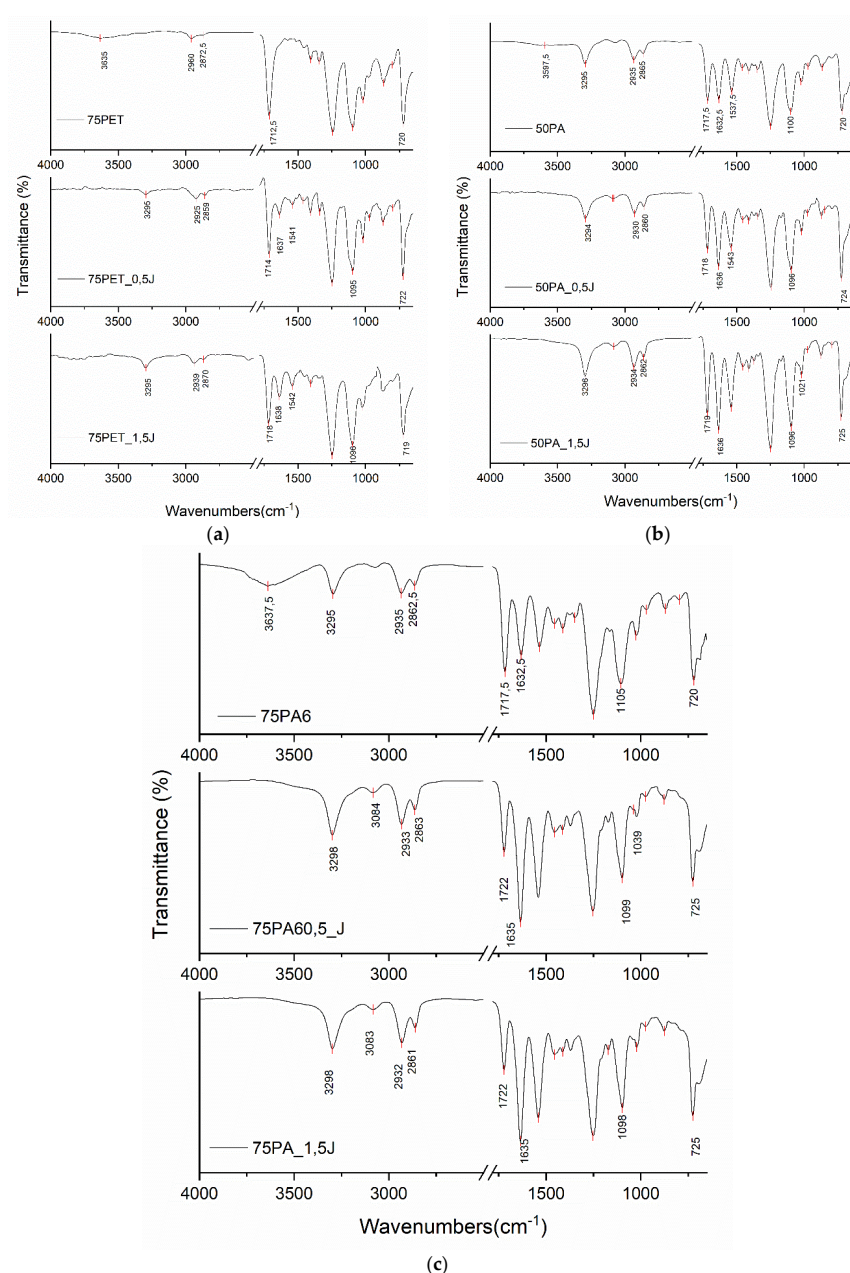


Figure 6. FTIR spectra of PET/PA blends with (a) 25% PA, (b) 50% PA, (c) and 75% PA blends additivated with 0.5 and 1.5% *w/w* Joncryl.

The FTIR spectra of the PET/PA6 blends without Joncryl were observed to be similar to those of neat PET and polyamide 6, as no peak shifts were observed. The spectrum of polyamide 6 shows a characteristic band of axial deformation of NH around 3300 cm^{-1} , at 2935 cm^{-1} , due to the axial deformation of aliphatic CH, and another strong band at 1633 cm^{-1} attributed to the deformation of amide I ($\text{C}=\text{O}$). This functional grouping (amide I) undergoes the resonance effect that increases the bond length and reduces the absorption frequency, with the band being observed at lower wavelengths; at 1538 cm^{-1} , there is a characteristic band of amide II (NH).

The characteristic peaks of PET were (a) a band at 1710 cm^{-1} that attributed to the stretching of the carbonyl group bond ($\text{C}=\text{O}$), which is predominantly found in the ester segments of the PET polymer chain or in the aromatic groups for degraded samples; (b) bands at 1100 and at 720 cm^{-1} that attributed to the out-of-plane deformation of two substituent carbonyls on the aromatic ring; (c) the band at 3428 cm^{-1} , which is attributed to the hydroxyl terminal groups O-H [38], and that at 1425 cm^{-1} , which refers to the CH groups of the aromatic ring. This implies that the PET and nylon 6 phases do not chemically interact to form covalent bonds between them, having only physical interaction via the hydrophilicity of functional groups. This correlates with the morphology of mixtures without Joncryl on SEM micrographs, showing an immiscible system with gaps between phases.

For Joncryl, the wave numbers at 1199 , 900 , and 765 cm^{-1} (epoxy ring) are assigned to the $\text{C}=\text{O}$ elongation, and that at 1726 cm^{-1} is assigned to the $\text{C}=\text{O}$ stretch.

The bands between 2953 and 2914 cm^{-1} are assigned to the $-\text{CH}$ elongation, confirming that Joncryl is an epoxidized ester. For the blends additivated with Joncryl, the spectrum of epoxy rings (765 cm^{-1}) is not observed, which means that either the concentration of Joncryl in the system was too low to be detected or that all epoxy groups in Joncryl were consumed during the compounding (processing) process. The torque and SEM results seem to indicate that Joncryl reacted with PET and PA6, as the torque increased, and better blend compatibility was observed in SEM micrographs.

Previous studies have suggested that, during chain extension, amide-terminal groups of PA6 react with anhydride groups to form an imide linkage.

The amide $\text{C}=\text{O}$ and $\text{C}=\text{N}$ stretching vibrations appear in both PA6 and PA6/Joncryl at their characteristic peaks ~ 1632 and $\sim 1538\text{ cm}^{-1}$, respectively, but in higher intensity. Essentially, the low Joncryl concentrations make it difficult to unequivocally detect these changes.

4.4. Differential Scanning Calorimetry (DSC)

The crystallization and melting parameters for PET, PA6, and their blends without and with 0.5% , 1% , and 1.5% *w/w* Joncryl can be seen in Table 2. ΔH_m is the melt enthalpy variation, ΔX_m is the degree of crystallinity, C_{max} is the maximum melt crystallization rate, and T_{pc} is the crystallization peak temperature.

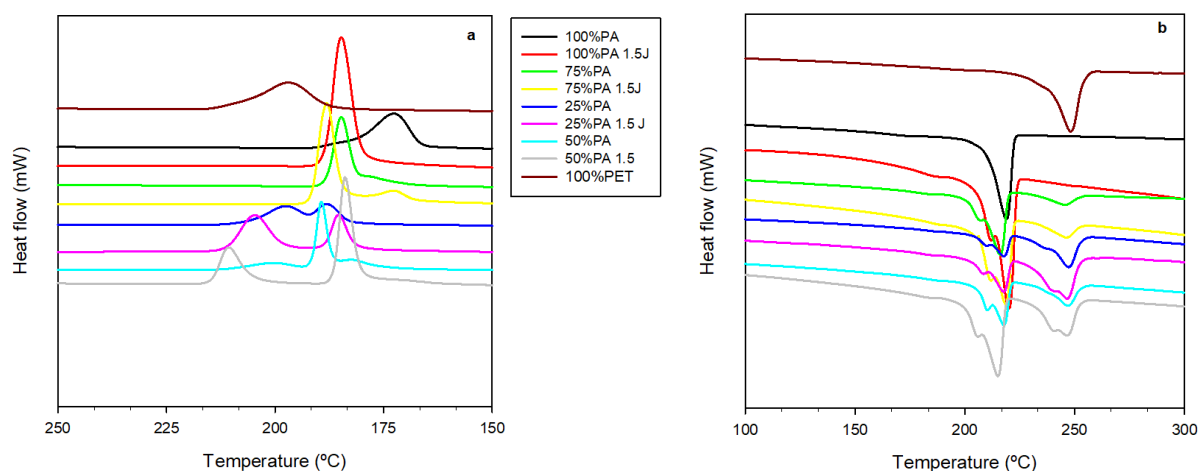
For the crystallization event, it is observed that, for pure blends (without additives), increasing the PA6 content in the blend results in the displacement of the peaks to higher crystallization temperatures. Two peaks are observed, which can be attributed to the polyamide and PET phases in the blends.

All samples of the PET/PA6 blends containing the chain extender exhibit higher crystallization temperatures compared to those without Joncryl. It appears that samples containing the additive result in having a steric obstacle to the close packing of PET chains, and this affects the crystallization capacity of the matrix [24].

The DSC curves of PA6, PET, and their blends with and without additive during reheating are shown in Figure 7.

Table 2. Melting and crystallization parameters for PET, PA6, and their blends with and without additives.

F2	Composition (PET/PA6)	T _{pc} (°C)	ΔH _m (J/g)	ΔX _m (%)	C _{maxm} (min ^{−1})	T _{pm} (PET) (°C)	T _{pm} (PA6) (°C)
No additive	0%/100%	172.6	48.3	34.5	0.7	248.2	-
	75%/25%	184.7	55.1 (PA6) 37.9 (PET)	23.9 (PA6) 27.1 (PET)	1.1 (PA6) 0.8 (PET)	247.3	209.9 217.7
	50%/50%	189.3 (PA6) 200.3 (PET)	62.0 (PA6) 38.2 (PET)	26.9 (PA6) 27.3 (PET)	1.0 (PA6) 0.8 (PET)	247	210.3 217.6
	25%/75%	188.1 (PA6) 197.4 (PET)	63.0 (PA6) 38.9 (PET)	27.3 (PA6) 22.8 (PET)	0.9 (PA6) 0.7 (PET)	245.5	207.1 216
	100%/0%	196.9	58	25.2	1.08	-	218.8
0.5 J	75%/25%	187.9 (PA6) 211.7 (PET)	70.9 (PA6) 109.4 (PET)	30.8 (PA6) 47.5 (PET)	0.8 (PA6) 0.7 (PET)	242.3	210.3 216.8
	50%/50%	189.8 (PA6) 210.7 (PET)	62.5 38	27.2 27.1	0.9 0.7	243.1 247	211.1 217.9
	25%/75%	175.8 (PA6) 188.9 (PET)	76.1 (PA6) 40.6 (PET)	33.1 (PA6) 29.1 (PET)	0.7 (PA6) 0.8 (PET)	246.1	210.2 217.4
	0%/100%	185.9	64.4	28	0.7	-	211.2 218.9
	75%/25%	186.8 (PA6) 201.8 (PET)	47.3 (PA6) 34.0 (PET)	20.6 (PA6) 24.3 (PET)	1.1 (PA6) 0.7 (PET)	248.3	210.3 219.1
1.0 J	50%/50%	188.7 (PA6) 203.7 (PET)	53.2 (PA6) 25.5 (PET)	23.1 (PA6) 18.2 (PET)	1.0 (PA6) 0.8 (PET)	246.2	211.2 218.4
	25%/75%	175.4 (PA6) 189.1 (PET)	61.3 (PA6) 29.1 (PET)	26.6 (PA6) 20.8 (PET)	1.9 (PA6) 0.9 (PET)	246.6	211.8 218.6
	0/100%	188.8	45.9	19.9	1.08	-	210.7 219.1
	75%/25%	185.3 (PA6) 204.7 (PET)	27.92 (PET) 49.03 (PA6)	19.9 (PET) 21.3 (PA6)	0.6 (PET) 0.9 (PA6)	246.5	208.4 217.6
	50%/50%	183.8 (PA6) 210.6 (PET)	65.2 (PA6) 38.9 (PET)	28.3 (PA6) 27.8 (PET)	0.9 (PA6) 0.7 (PET)	240.8 246.6	205.9 214.9
1.5 J	25%/75%	172.8 (PA6) 188.1 (PET)	61.3 (PA6) 28.5 (PET)	26.6 (PA6) 20.4 (PET)	0.8 (PA6) 0.8 (PET)	246.1	211.9 218.2
	0/100%	184.7	54.6	23.7	0.9	-	211.8 220

**Figure 7.** Curves of heat flow (mW) versus temperature (°C) in the crystallization during cooling (a) and melting during reheating (b) event for PA6, PET, and their blends.

For the PA6 melting peaks in the non-additivated blends, values close to the melting temperature of neat PA6 are observed. Furthermore, a higher C_{max} is observed for PA6 and blends that contain a higher percentage of this polymer. The double melting peaks attributed to PA are evidence that a change in the crystal phase has occurred, possibly the formation of a γ crystalline phase [10,39].

The DSC data shown in Table 2 reveal that the incorporation of Joncryl marginally decreased crystallinity during the reheating of PA6 in the 1% and 1.5% Joncryl compositions. Similar observations were made by Lu et al. [40], who used bisoxazolines, and Buccella et al. [41], who used CBC chain extenders. It is believed that this behavior is associated with the increase in polymer molar mass and the formation of branches that hinder structural organization and crystallization.

Comparing the temperature of the peaks of the blends with and without Joncryl, it was found that the presence of the extender led to higher values, which differed by about one or three degrees, and this approximation of the peaks may be indicative of compatibility.

This result is based on PET chain entanglement and is due to the high degree of branching that can result from a high amount of chain-extender additive. A similar result was observed when the additive methylene diphenyl diisocyanate was added to a blend of R-PET/LLDPE/SEBS-g-MA [42].

Furthermore, it was observed that, with the incorporation of the additive, both the crystallization rate and the degree of crystallinity of the blends that contain more PA6 presented lower values because they were hindered by the lower mobility of the branched chains.

4.5. Thermogravimetric Analysis (TGA)

The effect of chain-extender incorporation on the thermal stability of the resins and their blends is shown in Figure 8 and in Table 3.

Increased concentrations of the additive negatively affect the thermal stability of the material, as the degradation process initiates at lower temperatures.

The increase in the side groups causes structural disordering of the chains and inserts aliphatic segments, which are less thermally stable than aromatic segments [43]. It can also be observed that the increase in the carbonaceous residue is indicative of the reticulation of the studied systems. However, as can be seen, the effect on thermal stability is not very pronounced.

Table 3. Thermogravimetric analysis for blends PET/PA with 0, 0.5, 1, and 1.5% Joncryl.

9	Composition (%PET/%PA)	Mass Loss Range (°C)	T1/2 (°C)	Δm (%)	Residue at 700 °C
No additive	0/100	422.9–470.6	444.2	92.8	2.3
	25/75	370.3–414.3	392.3	84	7.9
	50/50	373.0–415.5	394.2	84.4	12.2
	75/25	379.2–443.8	411.5	85.4	12.7
	100/0	412.8–457.2	434.9	85.5	13
0.50%	0/100	414.4–457.6	436.0/442.2	81.7	13.4
	25/75	369.7–418.9	394.3/388.3	81.2	25.1
	50/50	363.6–420.2	391.9/386.8	91.2	7.3
	75/25	378.6–441.2	409.9/419.4	84.3	20.3
1.00% %J	0/100	406.1–462.6	434.3	91.1	12.7
	25/75	366.4–421.4	393.9	83.1	18.9
	50/50	362.4–421.6	392.0/388.2	85.2	10.2
	75/25	375.1–444.5	409.8	81.2	22.4
1.50%	0/100	411.4–450.2	430.8/435	86.5	14.8
	25/75	363.6–423.4	393.5/447.7	83.1	17.4
	50/50	363.7–421.9	392.8/386.8	87.2	9.4
	75/25	378.6–447.2	411.9	84.7	20.4

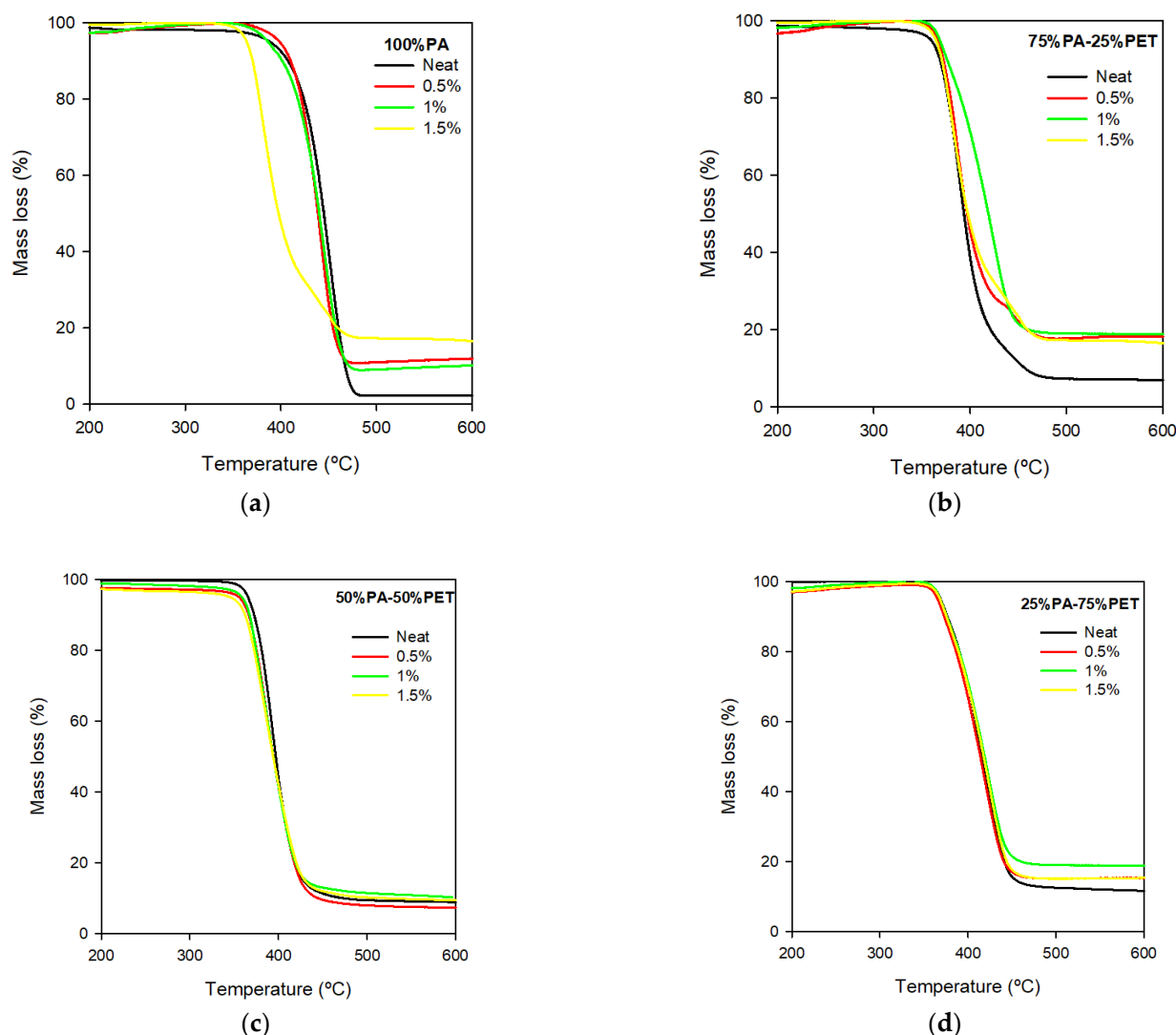


Figure 8. Thermogravimetric (TGA) plots of 100%PA (a), 75%PA–25%PET (b), 50%PA–50%PET (c), and 25%PA–75%PET (d) blends.

5. Conclusions

A chain extender with multiple anhydride functionalities, i.e., Joncryl, was used to test its potential as a compatibilizer, as well as compensating for the thermal degradation of PET/PA6 blends during processing in the laboratory mixer.

Joncryl was found to be a suitable compatibilizer for these blends, leading to the formation of completely miscible system (PET/PA 25/75 with 1.5% Joncryl), as evidenced by SEM morphological observations and reflected in the measured rheological and thermal properties.

The torque rheometry analysis showed that the viscosity increased with PET/PA6 ratio and the addition of Joncryl. The addition of 1% of the chain extender compensated for the degradation and increased the viscosity 2-fold compared to the pure polymer.

The FTIR spectra revealed that the epoxy groups belonging to Joncryl were consumed during blend processing, and this was attributed to the reaction of these groups with carboxyl groups from PET and amide from PA6.

The DSC data indicated that increasing the concentration of the additive and the consequent increase in the content of side groups lead to greater difficulty in packing, causing a reduction in intermolecular interaction forces, in the degree of crystallinity, and in melt enthalpy.

In addition to the practical benefit that results from this research, it also provides a strategy for developing chain extenders for other processing-sensitive polymers based on an understanding of their chemical degradation reactions and targeting the most reactive end groups arising from chain scission.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/su142416658/s1>, Figure S1. Temperature (a) and torque (b) curves as a function of time for pure polymers and pure and additive blends at 265 °C and 60 rpm for 10 min; Figure S2. Torque adjusted as a function of time in the last 3 min of processing; Figure S3. Curves of heat flow (mW) versus temperature (°C) in the crystallization (a) melting (b) event for PA6, PET and their blends; Figure S4. FTIR spectra of 100% of PA added with 0.5 and 1.5% by weight of Joncryl.

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