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Effect of adding new phosphazene compounds to poly(butylene terephthalate)/polyamide blends. I: Preliminary study in a batch mixer

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Abstract

Poly(butylene terephthalate) (PBT) and a sample of polyamide have been melt processed in the presence of two new phosphazene compounds, namely 2,2-dichloro-4,4,6,6-bis[spiro(2′,2″dioxy-1′,1″biphenyl)]cyclotriphosphazene (2Cl-CP) and 2,2-bis-(2-methoxy-4-methyleneoxy-phenoxy)-4,4,6,6,-bis[spiro(2′,2″dioxy-1′,1″biphenyl)]cyclophosphazene (CP-2EPOX).

The blends were prepared by using polyamide 6 (PA6) at 25/75 w/w and 75/25 w/w composition. In order to perform a preliminary analysis on the behaviour of the blends, the materials were prepared in a batch mixer.

The materials have been completely characterized from a rheological, morphological, mechanical point of view. The results indicate that the additives used cause an increase of the rupture properties and of the viscosity especially in the PA-rich blend. This result can be attributed to a chain extension effect on the PA phase, even if some compatibilisation with the formation of PBT/PA6 copolymers cannot be excluded as evidenced by the reduction of particle dimensions and by the improvement of the adhesion between the phases. Between the two compatibilisers, the CP-2EPOX displays the best results for both the investigated compositions while 2Cl-CP seems to act simply as a PA6 chain extender with very low compatibilising effect.
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1. Introduction

Blends of different polymers represent a valid alternative to synthesis in order to produce materials with new tailored characteristics. By using proper apparatus and procedures, it is possible to obtain, easily new materials with easier processes and lower costs with respect to the preparation of a completely new material.

Unfortunately, an important shortcoming in blending different polymers is that, as a rule, the couples form immiscible and incompatible blends that display a gross morphology with particles of dispersed phase badly adherent to the matrix and badly distributed. Consequently, the mechanical properties are poor thus often hindering any practical use.

In these cases, proper additives can significantly improve the compatibility with improvement of the morphology and of the mechanical properties [1–4].

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In this work, the effect of adding two new phosphazene compounds to polyamide 6 (PA6)/poly(butylene terephthalate) (PBT) blends will be studied.

These blends are particularly interesting for their applications in automotive, electronics and textile industries both to produce new high performance parts/items from virgin polymers and also to ease their recycling, eliminating the preventive separation of these materials or allowing the recycling when separation is not possible.

Several authors have studied blends of polyamide and polyesters. *p*-toluensulphonic acid has been used as a catalyst in polyester/polyamide blends to promote ester–amide exchange reactions [5–6]. The results show that it is possible to achieve an improvement in the impact strength and interference in the crystallization of the components.

Other authors studied the compatibilising effect of an epoxy resin on PBT/PA6, poly(ethylene terephthalate) (PET)/PBT and PBT/polyamide 6,6 (PA66) blends finding a remarkable improvement of impact and flexural properties and of the thermo-mechanical properties [7–11].

In other cases, maleic anhydride functionalized PBT [12] or a maleic anhydride modified ethylene–vinyl acetate copolymer (EVA-*g*-MAH) [13] has been successfully used in the preparation of compatibilised PA66/PBT blends and a sharp improvement of the mechanical properties has been observed. Similar results have been obtained by using phenoxy resins [14], and ionomeric polyolefins [15]. Finally, oxazoline derivatives [16] and biphenol [17] have been successfully used for compatibilisation of PBT/PA6 blends.

Cyclo(organo)phosphazenes (CP) are low molecular weight phosphazene derivatives, whose cyclic structure is formed by repeating –P=N– units, each phosphorus bearing two substituent groups (usually, but not necessarily, organic in nature). CPs find applications in several fields as biomaterials [18–19], photochemistry [20–25], sol–gel precursors [26–27], cores for dendrimers or star polymers [28–32], preparation of linear organic macromolecules with CP moieties [33], fire retardant additives [34]. Recently, CPs substituted with azide or 2-oxazoline groups have been used in the selective crosslinking of polyolefins and in the chain extension of PET and PA6 [35–40].

In this work, two CP compounds bearing, respectively, epoxy groups and chlorine as substituents, have been used to improve the morphology and the mechanical performance of PA6/PBT blends.

2. Experimental

2.1. Materials and preparation

The poly(butylene terephthalate) (PBT) used in this work was a sample of Arnitel T O8 200 kindly supplied by DSM. The polyamide 6 (PA6) was supplied by SNIA Tecnopolimeri. Its relative viscosity measured in sulfuric acid at a concentration of 1% w/w was 3.4.

The phosphazene compounds used in this work were a sample of 2,2-dichloro-4,4,6,6-bis[spiro(2′,2″-dioxy-1′,1″-biphenyl)]cyclotriphosphazene (2Cl-CP) and a sample of 2,2-bis-(2-methoxy-4-methyleneoxy-phenoxy)-4,4,6,6-bis[spiro(2′,2″-dioxy-1′,1″-biphenyl)]-cyclophosphazene (CP-2EPOX). The chemical formulae of the compounds are reported in Schemes 1 and 2. These compounds were prepared by functionalisation of the commercially available esachloro-cyclotriphosphazene (6Cl-CP) (Aldrich). Details on the preparation, the characterization and on some properties of these compounds can be found elsewhere [39–40].

The polymers were carefully dried prior to processing in order to avoid hydrolytic scission during the melt processing, keeping them overnight in a vacuum oven at 110 °C.

Binary and ternary blends were prepared in a Brabender batch mixer at 240 °C and a rotor speed of 64 rpm. In the preparation of the binary blends, the materials were fed together in the mixing chamber and processed for 6 min while, in the ternary blends with CP added, the PA6 and the PBT were processed for 5 min and in the last 60 s the additive (0.2 phr) was added.

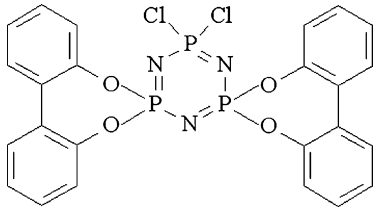
Tests to study the effect of the additives on the pure matrices were performed using the same processing parameters used for the blends.

In all the cases, to interrupt any possible reaction, the materials were put in liquid nitrogen immediately at the end of processing and then milled for further characterization.

2.2. Characterization

To analyse the activity of the CP additives as chain extenders, intrinsic viscosity measurements were performed using an Ubbelohde viscometer. PA6 was dissolved in 98% w/w formic acid at a concentration of 1% by weight at room temperature. PBT was dissolved in phenol/*o*-dichlorobenzene mixture at a concentration of 1% and at 50 °C. Testing temperature was in both cases 30 °C.

Turbidity tests were performed by preparing suspension of the binary and ternary blends with 98% w/w



Scheme 1. Chemical structure of 2Cl-CP.

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