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The effect of the interfacial assembly of nano-silica in poly(lactic acid)/poly(butylene adipate-co-terephthalate) blends on morphology, rheology and mechanical properties

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ABSTRACT

This work studies the effect of the interfacial assembly of spherical nano-silica particles on the morphology, rheology and mechanical properties of heterophase poly(lactic acid), PLA, and poly(butylene adipate-co-terephthalate), PBAT, blends. Through a controlled mixing strategy, nano-silica particles could be assembled in a stable fashion at the PLA-PBAT interface. Rheological analysis indicates that the interfacial assembly of nano-silica significantly diminishes the relaxation of the dispersed PBAT phase and 3 wt.% of nano-silica particles shifts the co-continuity region to a lower PBAT volume fraction. At co-continuity, the assembly of nano-silica at the interface changes the rheological behaviour of the co-continuous PLA/PBAT from a liquid-like to a gel-like behaviour. The interfacial assembly of nano-silica reduces the co-continuous phase size but maintains co-continuity. The results of thermal annealing indicate a remarkable stabilization effect of interfacially assembled nano-silica on the co-continuous morphology. A conceptual model is proposed to explain the observed effects of interfacially assembled nano-silica on the morphology which emphasizes the critical role of nano-silica content, the relaxation time of the dispersed phase and the migration time of nano-silica to the interface. The mechanical properties of the PLA/PBAT blends are clearly influenced by the observed shift in co-continuity in the presence of nano-silica. Most importantly, the mechanical properties of co-continuous PLA/PBAT are very sensitive to annealing due to morphological coarsening. Interfacially assembled nano-silica has the effect of completely stabilizing the co-continuous morphology without any diminishing of the mechanical properties. These results clearly indicate the significant potential of the interfacial assembly of nanoparticles as a promising strategy toward achieving polymer blends with highly robust and stable co-continuous morphologies.

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1. Introduction

The addition of solid inclusions to polymer blends is a promising method for achieving a balance between toughness and stiffness [1–3]. Moreover, with systems that display double and multiple percolation phenomena, the addition of conductive solid particles to polymer blends to produce a semi-conductive polymer nanocomposites has also received much attention [4,5]. Nanoparticles, in particular, have significant potential to improve the mechanical and/or electrical properties at much

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lower particle contents due to their much higher specific surface area. The localization of nanoparticles is a key parameter in achieving property control in multiphase systems incorporating nanoparticles [1,2,6–9]. It has been shown that the localization of nanoparticles in a dispersed rubbery phase increases the dispersed phase size by increasing the viscosity and elasticity of the dispersed phase [3,10]. On the other hand, the selective localization of nanoparticles in the matrix phase in a polymer blend reduces the dispersed phase size and increases not only the modulus and strength but also the impact properties [2,11].

The assembly of nanoparticles [8,12,13] and polymeric droplets [14,15] at the interface of polymer blends have recently received particular attention. From a morphological viewpoint, the interfacial assembly of nanoparticles in a blend with matrix-dispersed phase morphology considerably reduces the dispersed phase size and stabilizes the morphology through the formation of a solid shell around the dispersed phase [12,16–18]. However, the stabilization effect depends strongly on the wettability of the particles and their positioning at the interface. Nagarkar and Velankar [19] showed that in a blend of polyethylene oxide (PEO) and polyisobutylene (PIB), clusters of the dispersed phase (PEO) droplets bridged by silica particles were formed when silica particles were situated on the matrix (PIB) side of the interface. In addition, it has been shown that the localization of nanoparticles at the interface enhances the interfacial interactions between polymeric phases in the melt state [12,20–23].

In the case of polymer blends with co-continuous morphology, the interfacial localization of nanoparticles refines the morphology [18] and prevents coarsening of the phases during annealing and further processing through an interface jamming mechanism [20,24]. The interfacial localization of conductive nanoparticles in co-continuous blends is also considered as a promising method for enhancing electrical conductivity at much lower nanoparticle contents through multiple percolation phenomenon [5].

Despite previous studies on the effect of the interfacial localization of nanoparticles on the morphology and electrical conductivity of polymer blends, the mechanical properties of polymer blends with nanoparticles localized at their interface have been poorly investigated. This can be attributed to the complexity of this subject, which requires complementary analysis of the morphology, rheology and mechanical properties of the studied systems. For instance, Bitinis et al. [25] studied the effect of interfacial localization of organoclay on the mechanical properties of a PLA/Natural rubber (NR) blend and found that, depending on the surface modification of the particles, the addition of organoclay can increase either the elongation at break or the modulus of the blend.

In a previous study [26], nano-silica particles could be stably localized at the interface of a poly(lactic acid), PLA, and poly(butylene adipate-co-terephthalate), PBAT, blend. The morphology and miscibility of this blend system was characterized in detail in another study [27]. Considering the significant potential of PLA/PBAT as a fully compostable high performance bioplastic blend [27–29], the aim of this paper is to study the effect of the interfacial localization of nano-silica particles on the morphology, rheology and mechanical properties of PLA/PBAT blends. To this aim, the effect of the interfacial localization of nano-silica particles on the morphology and rheology of PLA/PBAT blends will be studied in three different regions of the PBAT phase composition: (a) in a blend with a matrix-dispersed morphology, (b) in a composition close to the boundary of the co-continuity region and (c) in the co-continuity region. The mechanisms responsible for the observed effects will also be examined in detail. Finally, the effect of the interfacial localization of nano-particles on the mechanical properties of PLA/PBAT blends will be evaluated.

2. Experimental methods

2.1. Materials

PLA 3001D (NatureWorks) and PBAT (Ecoflex FBX 7011) were purchased from Cargill and BASF, respectively. Spherical nano-silica particles (SEAHOSTAR KE-P10) with an average diameter of 100 nm were purchased from Nippon Shokubai, Japan. All the materials were dried under vacuum at 60 °C overnight to minimize their moisture contents.

2.2. Blend preparation

An internal batch mixer (Plasti-Corder DDR501, Brabender) with a total mixing chamber volume of 30 cm³ was used to prepare all samples. Mixing was carried out at 50 RPM and 180 °C for 10 min under a nitrogen blanket. The average shear rate at the mixing conditions was estimated as 25 s⁻¹ [30]. Two mixing strategies were used:

(Pr1) the addition of particles to a PLA/PBAT melt (one step process);

(Pr2) premixing nano-silica particles with PLA followed by mixing with PBAT (two-step process).

After mixing, samples were frozen in ice-water to freeze-in their morphology. The blend samples will be referred as PLA/PBAT(XX/YY) where XX and YY indicate the vol.% of PLA and PBAT respectively. To maintain the PBAT/nano-silica ratio in all samples, the wt.% of nano-silica particles added to blends was based on the weight of the PBAT phase.

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