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Effect of POSS on morphology and properties of poly(2,6-dimethyl-1,4-phenylene oxide)/polyamide 6 blends

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ABSTRACT

Poly(2,6-dimethyl-1,4-phenylene oxide)/polyamide 6 (PPO/PA6) (50/50 w) blends filled with epoxycyclohexyl polyhedral oligomeric silsesquioxane (POSS) were prepared via melt-mixing. The reactions between POSS and PPO/PA6 blends were studied by Fourier transform infrared spectroscopy, end group and gel content tests. The morphology of PPO/PA6/POSS composites was observed by field emission scanning electron microscope and transmission electron microscope. As a chain extender and a crosslinking agent for PA6, POSS largely affected the morphology of the composites, which was mainly dependent on the melt-viscosity ratio and interfacial tension between the components. With increasing POSS content from 2 to 4 phr, the morphology of the composites transformed from droplet/matrix to co-continuous morphology. The PPO/PA6/POSS composites with co-continuous morphology had the better mechanical properties than those with droplet/matrix morphology. Dynamic mechanical thermal analysis showed that the addition of POSS increased the T_g of PA6.

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1. Introduction

Poly(2,6-dimethyl-1,4-phenylene oxide)/polyamide 6 (PPO/PA6) blend is a typical incompatible blend and PPO has much higher melt viscosity than PA6 [1,2]. As a result, co-continuous morphology can be obtained only at high PPO content. Generally, it is considered that the blends with co-continuous morphology can exert the optimum efficiency of blend components [3]. Then polystyrene or synthetic fluorine mica has been added to PPO/PA6 blends to achieve co-continuous morphology by adjusting the viscosity ratio [4,5]. However, the addition of polystyrene makes the mechanical properties of PPO/PA6 blends worse, while the co-continuous structure of PPO/PA6/synthetic fluorine mica can not always be stable during melt-mixing. The melt viscosity of PA can be increased through the chain extension [6–8]. How to increase the viscosity of PA6 to match the viscosity of PPO is a key issue for obtaining

co-continuous morphology of PPO/PA6 blends. Aiming at significantly increasing the viscosity of PA6 to meet that of PPO, a high effective chain extender with multi-epoxy group different from bifunctional coupling agents, polyhedral oligomeric silsesquioxane (POSS), was introduced into the PPO/PA6 blends in this work, which could lead to branching or even crosslinking of PA6.

POSS molecule has a basic polyhedral silicone–oxygen nanostructure skeleton or cage (1–3 nm in size). 8 or 12 Si atoms (with a Si:O number ratio of 2:3) are located at the corner of the cage, which are surrounded by 8 or 12 organic groups, respectively (Fig. 1) [9,10]. By chemical tethering or physical blending, POSS can be incorporated into polymers such as polycarbonate [11], poly(ethylene terephthalate) [12], polyamide [13] for improved properties [11–17]. Specially, POSS as an effective chain extender has been used in Poly(butylene terephthalate) (PBT) [18]. For the amine end group is more active than the hydroxyl group in Poly(butylene terephthalate), the chain extension of POSS for PA6 should be more effective and then lead to a high viscosity of PA6. Thus co-continuous structure of PPO/

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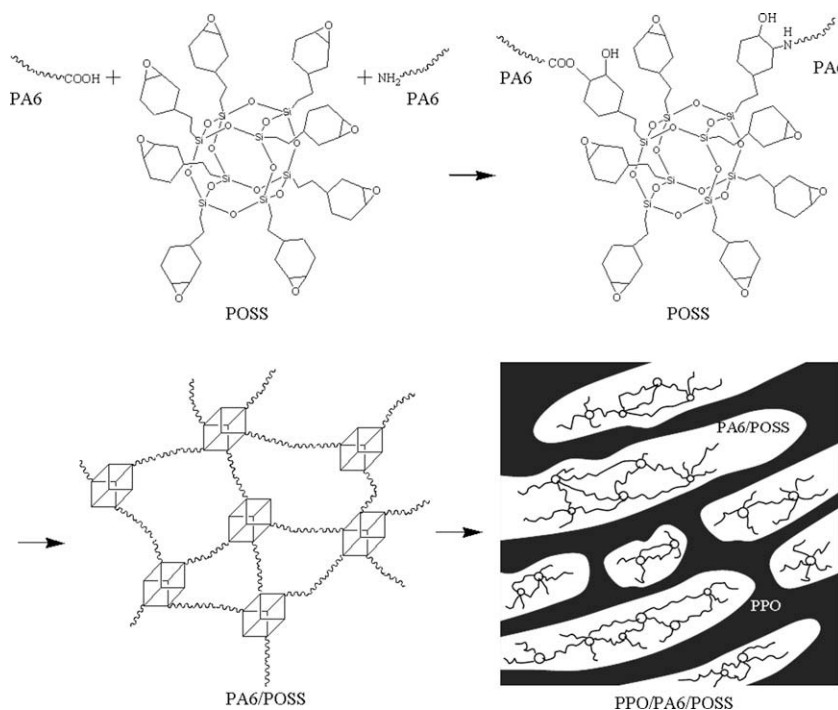


Fig. 1. Scheme of the preparation of PPO/PA6/POSS composites.

PA6 blends could be expected at a relatively low PPO content. In addition, as another key factor for the morphology, the interfacial tension between the components was also investigated. By analysis of the chain extension or cross-linking of PA6 and corresponding morphology of PPO/PA6/POSS, the mechanical and dynamic mechanical properties of the composites were studied.

2. Experimental

2.1. Materials and composites preparation

PPO, trademark Lupiace PX100L, produced by Asahi Kasei Corporation, Japan, had an intrinsic viscosity 0.47 ± 0.02 dl/g measured in trichloromethane at 25 °C. PA6, Ube1013B, with a relative viscosity of 2.3, was produced by Ube Industries, Ltd., Japan. POSS, EP0408, was purchased from Hybrid Plastic Company, USA.

Before blending, PPO and PA6 were dried at 80 °C under vacuum for about 12 h. A masterbatch of POSS was prepared by mixing PPO, POSS and trichloromethane, and dried at 80 °C for 12 h in a vacuum oven. PPO, PA6 and POSS were reactively blended in the mixing chamber of a Haake Rheometer (RC90, Germany) at 255 °C and 60 rpm for 8 min. The composites were compression molded in a press at 255 °C for 8 min, then cold pressed to get samples for testing. One of the reactions that might take place for the PPO/PA6/POSS composites at elevated temperatures is shown in Fig. 1. In this study, the composition of PPO/PA6 was fixed to be 50/50 (phr/phr).

2.2. Characterization

The Fourier transform infrared spectroscopy (FTIR) analysis was obtained using an FTIR spectrometer (Paragon 1000 from Perkin–Elmer Co., USA). The scan range was from 4400 to 400 cm^{-1} with a resolution of 4 cm^{-1} .

All samples for end group content test were pre-extracted by trichloromethane to take away PPO. The carboxyl contents were determined with the Waltz–Taylor method [19]. The sample (0.2 g) was completely dissolved in 12 ml of benzyl alcohol at 140 °C. The solution was cooled to 90 °C, and 3 ml of n-propanol and 10 drops of phenolphthalein were added immediately. The titration of this solution was performed with a 0.005 N NaOH/ethanol solution. The amine end chain concentration was determined by the Waltz–Taylor method [19]. The sample (0.2 g) was dissolved in 12 ml of metacesol at 140 °C. Five minutes later, 12 ml of methanol and 10 drops of thymol blue was added, and the titration of this solution was performed with a 0.005 N HCl/ethanol solution.

The gel content was determined as follows: 250 mg sample was dissolved in 50 ml trichloromethane at room temperature. The soluble part was removed by filtration until no deposition could be detected in trichloromethane solution by adding excess acetone. Then the insoluble component was dried and then dissolved in 50 ml formic acid at room temperature for 4 h. The soluble part was removed by filtration until no deposition could be detected in formic acid solution by adding excess alcohol. The insoluble gel was washed well with alcohol, dried, and weighted. The percentage of the insoluble gel was defined as the gel content.

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