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## Material Characterisation

# Characterization of PP/HDPE blend-based nanocomposites using different maleated polyolefins as compatibilizers

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### ABSTRACT

Nanocomposites based on a polypropylene (PP)/high density polyethylene (HDPE) blend were prepared using an organo-montmorillonite (15A) as a nano-filler and two maleated polyolefins (PE-MA and PP-MA) as compatibilizers. The phase morphology and typical physical properties of the prepared samples were examined. The nano-filler 15A was intercalated and/or partially exfoliated in the blend when PE-MA or PP-MA was present. The PE-MA facilitated the dispersibility of 15A to a better degree. The nano-filler 15A accelerated the crystallization of PP in the blends, whereas it hardly influenced the crystallization of HDPE. Moreover, at a slow cooling rate (i.e., 1 °C/min) the PP-MA induced a higher crystallization temperature for PP in the composite, while PE-MA impeded PP crystallization. On the other hand, the crystallization of HDPE in the composite was only slightly influenced by the presence of PE-MA or PP-MA. The thermal stability of PP/HDPE blend was enhanced after the addition of 15A regardless of the inclusion or not of PE-MA or PP-MA. The enhancement was more evident when the samples were scanned under an air environment than a N<sub>2</sub> environment. The stiffness of PP/HDPE blend increased marginally after adding 15A and was slightly altered with the further inclusion of PP-MA. The presence of PE-MA in the composite caused a slight decline in the stiffness. The impact strength of PP/HDPE blend declined after the formation of nanocomposites, especially for the sample incorporating PP-MA.

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## 1. Introduction

Research on the preparation and characterization of polymer/clay nanocomposite systems has attracted considerable attention during the past two decades. Numerous combinations of polymers and clays have been evaluated [1–9]. In spite of their potential to display advanced properties with only minor amounts of clay loading (e.g., 3–5 wt.%), polymer/clay nanocomposites may exhibit imbalanced properties. Among the various clays evaluated, montmorillonite (MMT) is recognized as an appropriate choice for preparing high performance

polymer/clay nanocomposites. To develop MMT-included polymeric nanocomposites successfully, layered-MMT has to be dispersed finely and uniformly (i.e., intercalated and/or exfoliated) in the polymer matrix. However, due to its hydrophilic feature, MMT does not generally disperse finely in hydrophobic polymer matrices. Thus, it is frequently modified with the inclusion of certain organic surfactants to improve its affinity to hydrophobic polymers (modified MMT is hereafter denoted as O-MMT). In addition to MMT modification, certain compatibilizers are further incorporated into the polymer/O-MMT system in some cases to ensure the formation of nanocomposites.

Polypropylene (PP) and polyethylene (PE) are widely used in commodity applications. To improve their properties and versatility further, PP/clay and PE/clay nanocomposites have been extensively studied [10–20]. Kawasumi et al. [10], Manias et al. [11], and Maiti et al. [12]

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conducted systematic work on the fabrication and characterization of PP/clay nanocomposites. Maleic anhydride-grafted PP (PP-MA) is ascertained to mix well with certain organoclays (O-MMTs) to form nanocomposites. The PP-MA can also play a compatibilizer role in promoting the formation of PP/clay nanocomposites. We [13] studied the combined effects of clay modifications and compatibilizers on the formation and properties of PP/clay nanocomposites. Rohlmann et al. [14] investigated the effects of different O-MMTs on the formation and rheological properties of PP/PP-MA/O-MMT nanocomposites. It was concluded that microstructure and flow behavior of the nanocomposites strongly depend on the nature of the modifier used in the O-MMT. With regard to the PE/clay system, Jeon et al. [16] employed a solution-mixing technique to prepare a nanocomposite with O-MMT partially intercalated in the PE matrix. Morawiec et al. [17] used maleic anhydride-grafted PE (PE-MA) as a compatibilizer to prepare PE/clay nanocomposites through melt-mixing processes. The crystallinity and crystallization kinetics of PE were not significantly affected by the presence of O-MMT. Meanwhile, Shah and Paul [18] disclosed the effect of the degradation of organoclays during processing on the structure and properties of the formed PE/clay nanocomposites.

Like most of polymers, various PP-based and PE-based blends have been prepared and investigated to upgrade their versatility [21]. Of all the known blends, PP/PE blend has been one of the most extensively studied over the years [21–24]. As both of the PP and PE components are crystallizable, a study on the crystallization kinetics and crystalline morphology of either component in the blend is essential in revealing the relationship between the structure and properties of the blend. From the reports on PP/clay and PE/clay nanocomposites, the addition of organoclays to the PP/PE blend may further improve the blend's properties and should result in a more complex phase morphology and crystallization kinetics for both of the crystalline components. Until now, only a few studies have been focused on the fabrication and characterization of polyolefin blend-based nanocomposites [25–27]. The aim of this study is to present and compare the effects of separately incorporating two maleated polyolefins in the preparation of PP/PE/O-MMT nanocomposites. The phase morphology, crystallization/melting behavior, and typical physical properties of the prepared (nano)composites were determined and compared with those of the PP/PE blend.

## 2. Experimental section

Isotactic PP with a melt flow index of 15 g/10 min (ASTM D1238) and a density of 0.9 g/cm<sup>3</sup> was supplied by Yung Chia Chemical Co., Taiwan. High density PE (HDPE) with a melt flow index of 6 g/10 min (ASTM D1238) and a density of 0.96 g/cm<sup>3</sup> was supplied by Formosa Plastics Corporation, Taiwan. An O-MMT clay (Cloisite® 15A, denoted as 15A) obtained from Southern Clay Products, Inc. was used as the nano-filler for the composites preparation. 15A was organically modified with dimethyl dihydrogenated tallow quaternary ammonium ion, with the following tallow composition: 65% C18, 30% C16, and 5%

C14. The following two maleated polyolefins were used separately as the compatibilizer for the prepared blend and composite. The maleic anhydride-grafted PE (PE-MA, Fusabond MB100D) with ca. 0.5–1.0 wt.% of MA was purchased from DuPont Co. The maleic anhydride-grafted PP (PP-MA, Fusabond M613) with ca. 0.55 wt.% of MA was likewise purchased from DuPont Co.

All the blends/composites were prepared through a melt-mixing procedure, which used an intermeshing twin-screw extruder (SHJ-20B, L/D = 40) in the co-rotating mode. The screw speed was maintained at 240 rpm. The barrel temperatures from hopper to die were kept at 140–220 °C. The 15A content of the composites was kept at 3 wt.%. The selected maleated polyolefin was loaded at 15 wt.%. The ingredients were weighed at a prescribed ratio and then dry-mixed before feeding into the extruder. After extrusion, the samples were pelletized and then oven-dried before further characterization. For comparison purpose, neat PP and HDPE were also melt-extruded under the same conditions. The formulation and sample designations for the prepared blends/composites are listed in Table 1.

X-ray diffraction (XRD) and transmission electron microscopy (TEM) were employed to assess the dispersibility of 15A in the composites. The crystal forms of PP and HDPE in the blends/composites were also determined by XRD. A Siemens D5005 X-ray unit operating at 40 kV and 40 mA was used to carry out the experiments at room temperature. The X-ray source was CuK $\alpha$  radiation with a wavelength of 1.54 Å. The TEM observations were performed on ultrathin sections of cryo-microtomed thin composite films with a JEOL JEM-1230 system using an acceleration voltage of 100 kV.

To evaluate the effects of adding 15A and maleated polyolefins on the phase morphology of the PP/HDPE blend, scanning electron microscopy (SEM) experiments were performed on cryo-fractured surfaces of the samples using a Hitachi S-3000N system. The samples were immersed in liquid nitrogen before fracturing. An Olympus BX-50 polarized light microscope (PLM), in conjunction with a Linkam THMS 600 hot stage, was employed for the crystalline morphology investigations. The thin-film specimens were prepared by melting the samples at 200 °C prior to compressing them between glass slides.

The crystallization and melting behavior of the samples was measured using a Perkin Elmer DSC 7 analyzer equipped with an inter-cooler. For the crystallization

**Table 1**  
Samples designation and formulations.

Designation	Composition	Parts (wt.%)
PP	PP	100
PP/C3	PP/15A	97/3
HDPE	HDPE	100
HDPE/C3	HDPE/15A	97/3
PP/HDPE	PP/HDPE	50/50
PP/HDPE/C3	PP/HDPE/15A	48.5/48.5/3
PP/HDPE/PE-MA	PP/HDPE/PE-MA	42.5/42.5/15
PP/HDPE/PP-MA	PP/HDPE/PP-MA	42.5/42.5/15
PP/HDPE/PE-MA/C3	PP/HDPE/PE-MA/15A	41/41/15/3
PP/HDPE/PP-MA/C3	PP/HDPE/PP-MA/15A	41/41/15/3

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