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Investigation of thermal, mechanical and gas barrier properties of polypropylene-modified clay nanocomposites by micro-compounding process

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ABSTRACT

Polypropylene (PP) nanocomposites were prepared by the melt extrusion of PP with modified clay in the presence of maleated polypropylene (PPgMA). Before melt extrusion, clay was modified by a sol–gel method with two different modifiers (amionpropyltriethyoxysilane and dimethyloctadecyl [3-(trimethoxysilyl)propyl] ammonium chloride) and further treated with tetraethyl orthosilicate. The modification of the clay was confirmed by X-ray diffraction (XRD), transmission electron microscopy (TEM), thermogravimetric analysis (TGA), and surface area analysis. The dispersion morphology of these nanocomposites was confirmed by XRD and TEM analyses. The storage modulus of PP nanocomposites was increased by 15.5%, and tensile strength improved by 45.9% compared to pure PP. Gas barrier properties were investigated, and the results showed that the O_2 permeability at 3 atm improved by a factor of 14.1 over pure PP. The surface contact angle was reduced from 97.90° to 67.72°, indicating increased hydrophilicity. These results suggest the potential of PP composites.

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1. Introduction

Polypropylene (PP) possesses an attractive combination of advantages-low cost, low weight, and is one of the most widely used in commodity polymers, successfully competing with other less environmentally friendly or versatile plastics. Furthermore, its physical characteristics such as low bulk density are advantageous [1–3]. Recently, significant research has focused on the study and development of polymer nanocomposites. Nanocomposites are of particular interests because their nanocomposites properties are superior to pure materials and regular composites [4–8]. Using a small amount of natural layered silicate clays, such as montmorillonite, or synthetic layered materials, such as layered double hydroxides, imparts similar properties to much higher volumes of conventional fillers such as talc, glass fiber, and calcium carbonate [9].

Montmorillonite type layered silicate clays are most widely used for the preparation of polymer nanocomposites [10–12]. Their unique properties, such as a large surface area to volume ratio, high interfacial reactivity, and nanoscale layered structure, MMT silicate layers can be used without any modification or can be organically modified with suitable reactants such as octadecylamine, stearylammonium, organic cationic surfactants, and alkylammonium, to reduce hydrophobicity and increase the clay interlayer spacing [13–16]. In this way, polymer chains can easily access into clay interlayers when the hydrophilic interlayer swells, producing polymer nanocomposites with highly dispersed clay. Intercalated and exfoliated nanocomposites are formed when PP and nanoclays mix via various methods such as in situ polymerization, solution blending, and melt blending methods [17–19]. Of these different processes, PP/clay nanocomposites were made by melt-blending in an extruder because it has better commercial feasibility, lower cost, and a shorter residence time, and is environmentally friendly and straightforward to use because it does not require any solvent or monomers. This process is also an accepted method in industrial research and development for new kinds of nanocomposites [20].

In this work, PP nanocomposites were obtained by the extrusion of PP with modified clay in the presence of maleated polypropylene (PPgMA). PPgMA acts as a compatibilizer, and most researchers have found that surfactants and organomodifiers containing active hydrogen atoms can react with the PPgMA between clay interlayers. Therefore, the structure of the surfactant influences the intercalation morphology of PPgMA [21,22]. The

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objectives of this study are to investigate the effect of clay dispersion in a PP matrix, and the effect of the thermal treatment on the structure, morphology, mechanical and gas barrier properties of nanocomposites. To this end, this work describes methods to (i) modify the clay with a long-chain organosilicate by a sol–gel method to ease exfoliation, (ii) thermally treat the obtained nanocomposites to obtain a cross-linked network, and (iii) improve the thermo-mechanical and gas barrier properties of polypropylene. The obtained results suggest the potential of PP composites for applications such as the production of containers for soft drinks as well as applications in the orchid industry and medical fields.

2. Experimental section

MMT types of clay CL120 (CEC: 168 meq/100 g) were supplied from China Glaze Co. Dimethyloctadecyl [3-(trimethoxysilyl) propyl] ammonium chloride (DTSACl, CH_3O_3Si $(CH_2)_3$ N $(CH_3)_2(CH_2)17CH_3)$ Cl) and tetraethyl Orthosilicate (TEOS, $SiC_8H_{20}O_4$) were purchased from Sigma Aldrich. Amionpropyltriethyoxysilane (APS, $C_9H_{23}NO_3Si$) was purchased from Acros. Polypropylene (PP, melting temperature 160 °C) and polypropylene grafted maleated anhydride (PPgMA) (maleic anhydride graft ratio 1 wt%) were supplied by LCY Chemical Corporation. Nitric acid (HNO_3) scharlan, hydrochloric acid (HCl) were purchased from Sigma and ammonium hydroxide (NH_4OH) was purchased from Pharmaco. All of these materials were used as received without further purification.

2.1. Modification of clay (CL120)

The CL120 was dispersed in 60 ml of deionized water with a magnetic stirrer for 24 h at room temperature to form swollen clay and divided into two equal parts of 30 ml each. The pH of the swollen clay was adjusted from 2 up to 3 and in this solution, 0.372 g of APS and 0.834 g of DTSACl solutions were added separately over a few minutes, and the mixture was stirred for 1 h at 60 °C. After 1 h, in each reaction mixture, pH was adjusted to 5. In these mixture solutions, 6.9 g of TEOS and 36 ml of ethanol solution were added separately. The resulting mixture was heated at 70–80 °C for 24 h. The mixture was then centrifuged and dispersed in deionized water, and the process was repeated 4 or 5 times. The modified clay was finally freeze-dried for 24 h. The obtained powders are denoted here as CL120-AT and CL120-DT, respectively.

2.2. Preparation of PP nanocomposite

To avoid hydrolytic degradation, all modified clays, PP, and compatibilizer PPgMA used in our experiments had to be dried in an air-circulating oven before initiating the compounding processes. The CL120-AT, and CL120-DT were blended with PP, and the modified clay content of the nanofillers were 1, 2, and 3 wt% of PP and the compatibilizer PPgMA (9:1, PP: PPgMA weight ratio and modified clay weight varies from 1to 3 wt%). The nanocomposites were prepared by a master-batch approach, and the nanocomposites were prepared via melt-extrusion in a laboratory scale DSM twin-screw microcompounder, whose internal structure is shown in Fig. 1, at 170 °C with rotation speed of 80 rpm, reaction or residence time of 10 min, and shear force of 4600 N. The obtained nanocomposites are represented herein as PP/CL120-AT-1 wt%, PP/CL120-AT-2 wt%, PP/CL120-AT-3 wt%, PP/CL120-DT-1 wt%, PP/CL120-DT-2 wt% and PP/CL120-DT-3 wt%.

2.3. Characterization

X-ray diffraction was carried out on a Bruker D8 with advance detection of the layered material structure, and the scan angle covered $2° < 2\theta < 70°$, step size 0.04°, 1 s per step. FT-IR spectra were recorded with a JASCO, FTIR-4200 Type A spectrometer with KBr pellets. The morphology of PP nanocomposites was measured by scanning electron microscopy (SEM), and was obtained on a JEOL JSM 6500F. Transmission electron microscopy (TEM) images were obtained on a Model JEOL JEM2010, 200 kV. A sample with a thickness of 80 nm was prepared by a Leica Ultracut-UCT to verify the physicochemical properties of nanocomposites. Thermogravimetric analysis (TGA) was carried out using SII TG/DTA6200, and the experiment was performed with a 10 mg sample of the PP nanocomposites under air gas flow from 40 °C to 900 °C at a scanning rate of 10 °C min^{-1}. The dynamic mechanical analysis (DMA) measurement was performed with a TA-Q800 instrument in air at a scanning range of 30–210 °C with a heating rate of 5 °C min^{-1} and with sample size: 35 mm × 12 mm × 3 mm. Tensile strength was measured with a Tensile Testing Machine Cometech D638 instrument. BET surface area was measured by a surface area and pore size analyzer model micrometrics Tristar 3000 instrument, with sample degas at 120 °C for 1 day. Thermal conductivity was measured by a Hot Disk Transient Plane Source via a ISO/DIS 22007-2 model with sample size: 5 cm × 5 cm. Gas permeability analysis was performed with a Yanco (GTR-31). UV–visible transmission and absorption spectra were obtained with a Cary-100 UV–vis spectrophotometer.

Fig. 1. Twin screw microcompounder and its internal structure.

2. Results and discussion

3.1. Characterization of clay and modified clay

The X-ray diffraction (XRD) patterns of pure clay and clay modified with organosilica (CL120-APS, CL120-AT, CL120-DTSACl and CL120-DT) are displayed in Fig. 2(A) and (B). The well-developed characteristic peaks of pure CL120 are shown in the figures, After modifying CL120 with organosilica via the sol–gel method, the (00*l*) reflection peaks were well developed and shifted to a lower 2θ angle observed. The basal spacing of CL120-APS and CL120-DTSACl are in good agreement with literature values [20] of 18.03 Å and 21.66 Å, respectively. The increased basal spacing compared to pure CL120 indicates that the large organosilicate molecules were successfully intercalated into the clay. After modifying the CL120-APS and CL120-DTSACl with TEOS to form CL-120-AT and CL120-DT, the basal spacing of CL-120-AT increased 25.98 Å. XRD patterns shown in Fig. 2(A) and (B) indicate complete delamination of the layer structure, which is evidence that

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