

Effect of processing conditions on morphology and mechanical properties of compatibilized polypropylene nanocomposites

M. Modesti*, A. Lorenzetti, D. Bon, S. Besco

Department of Chemical Process Engineering, Padova University, v. Marzolo 9, 35131 Padova, Italy

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Abstract

This work focuses on the influence of processing conditions on the nanocomposites structure, i.e. intercalated or exfoliated, and on the enhancement of mechanical properties of polypropylene (PP) nanocomposites. These nanocomposites were prepared using the melt intercalation technique in a co-rotating intermeshing twin screw extruder. In order to optimise processing conditions, both screw speed and barrel temperature profile were changed. The role of the compatibilizer (maleic anhydride grafted polypropylene) was also studied. The results obtained show that the barrel temperature is a very important parameter: using lower processing temperature, the apparent melt viscosity and, consequently, the shear stress are higher and, therefore, the exfoliation of the clay is promoted. Even using optimised processing conditions, exfoliation of clay can be achieved only when an high compatibility between polymer and clay exists: the PP nanocomposites containing maleic anhydride show an exfoliated structure and a sensible enhancement of mechanical properties while PP nanocomposites without compatibilizer show a structure mainly intercalated and a lower improvement of mechanical properties.

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1. Introduction

Manufacturers have long since filled polymers with particles in order to improve the stiffness and toughness of materials, to enhance their barrier properties and their resistance to fire and ignition. Addition of particulate fillers sometimes imparts drawbacks to the resulting composites such as brittleness or opacity. Nanocomposites are a new class of composites, that are particle-filled composites for which at least one dimension of the dispersed particles is in the nanometer range. The most interesting aspect of the use of nanofillers is the very low amount of filler that has to be added to the polymer in order to be effective. In this way, the undesired effects due to the introduction of the traditional amounts of inorganic additives, which are very high, can be avoided. Polymer layered silicate nanocomposites (PLSN) were reported in the patent literature as early as 1950 [1]. However, it was not until Toyota researchers [2,3] began a

detailed examination of polymer layered silicate composites that nanocomposites become more widely studied in academic, government and industrial laboratories. Since the early Toyota works, which are based on the formation of nanocomposites in situ during polymerisation of caprolactam, many works have been published on polymer nanocomposites. These works show that several strategies have been considered to prepared PLSN, i.e. intercalation of polymer or pre-polymer from solution, in situ intercalative polymerization and melt intercalation. Among them, the most versatile and environmentally benign technique is the melt intercalation owing to the absence of organic solvents, because it is compatible with current industrial processes, such as extrusion and injection moulding. It can be used for polymers, which were previously not suitable for in situ polymerisation or solution intercalation.

Many works on PLSN focus on the importance of chemical surface modification of the clay employed (usually montmorillonite) in order to increase the compatibility between clay and host polymer. The role and importance of processing conditions are the subject of very few papers. Until recently, the literature contains no summary conclusions about the optimum processing conditions needed in

* Corresponding author. Tel.: +39 49 8275541; fax: +39 49 8275555.
E-mail address: michele.modesti@unipd.it (M. Modesti).

order to obtain polymeric nanocomposites. Regarding polypropylene, several works on the preparation of nanocomposites via melt blending may be found in literature (reported in Ref. [4]), but these studies have not addressed the issue of how the resulting structure is affected by the design of the mixing device and processing conditions. Most studies were conducted for direct melt compounding process under intuitive processing conditions. Some works on the influence of the processing conditions on the properties of nanocomposites can be found in literature: Dennis et al. [5] studied polyamide 6 and showed that the potential properties improvement usually depends on a combination of the proper chemical treatment of the clays and optimized processing. Incarnato et al. [6] studied the influence of the extrusion rates on the properties of polyamide 6. About polypropylene, any exhaustive study cannot be found: Tidjani et al. [7] focused on the influence of the presence of oxygen during the preparation of polypropylene-*graft*-maleic anhydride nanocomposites while Wang et al. [8] analysed the effect of the shear stress on the dispersion of clay in polypropylene matrix processed via dynamic packing injection moulding (DPIM).

Today, polypropylene (PP) is one of the most widely used polyolefin polymers; it has no polar groups in the chain and then its direct intercalation or exfoliation in the silicate galleries is very difficult. Efforts were made to improve the mixing of clay in PP by using functional oligomers as compatibilizer. Usuki et al. [9] first reported a novel approach to prepare PP nanocomposites using a functional oligomer (PP-OH) with polar telechelic OH groups as compatibilizer. Several studies [10–12] reported the use of maleic anhydride grafted PP as compatibilizer. In both case the interaction between filler and polymer is enhanced by a strong hydrogen bonding between OH or COOH groups and the oxygen groups of silicate [13].

In this paper we report the effect of several processing conditions on the properties of OMLS-PP (organically modified layered silicate polypropylene) nanocomposites. The nanocomposites were prepared through melt intercalation in a co-rotating twin screw extruder, using several processing conditions: barrel temperature profile as well as screw speed were changed. Both pure polypropylene and polypropylene-*graft*-maleic anhydride (PP-*g*-MA) were filled with several amounts of an organically modified layered silicate. The morphology as well as mechanical properties of the materials prepared were characterised by use of transmission and scanning electron microscope (TEM and SEM, respectively), X-Ray diffraction (XRD) and

mechanical tests. Therefore, the aim of this work is the study of the influence of compatibilizer and processing conditions on the extent and degree of dispersion of the modified nanofillers in PP matrix.

2. Experimental part

2.1. Raw materials

Pure polypropylene (PPH 7062) and polypropylene-*graft*-maleic anhydride (Fusabond MD551D) were supplied by Total Petrochemicals and DuPont de Nemours Int., respectively. The melt flow index of such materials are 12 g/10 min (230 °C/2.16 kg) and 22.5 g/10 min (190 °C/2.16 kg), respectively. The polypropylene-*graft* maleic anhydride contains 1 wt% of maleic anhydride (MA). An organo-modified clay (Dellite 72T, powder), that is a ditallow-dimethyl-ammonium ion modified natural montmorillonite (MMT), with a mean particle size of 8 µm was supplied by Laviosa Chimica Mineraria (Livorno, Italy). Thermogravimetric analysis of OMLS, carried out in oxidizing atmosphere (from 30 to 900 °C, 15 °C/min), showed weight loss of about 35 wt% between 200 and 600 °C, due to water and to the organic fraction introduced as compatibilizer.

2.2. Preparation of the nanocomposites

PP nanocomposites were prepared using melt blending technology in an intermeshing co-rotating twin screw extruder ($D=42$ mm, $L/D=40$), set up with medium shear configuration modular screw. This kind of extruder gives higher degree of back mixing with respect the counter-rotating intermeshing or non-intermeshing extruders. The use of low or high shear intensity configuration screw have not been studied as Dennis et al. [5] already reported that excessive low or high shear intensity may cause poor delamination and dispersion. The filler used was previously pre-dry-blended in a turbo-mixer. Nanocomposites based on pure PP and PP containing 6 wt% of PP-*g*-MA were prepared. During extrusion, two different barrel temperature profiles (Fig. 1), screw speeds (250 and 350 rpm) as well as filler contents (3.5 and 5 wt%) were used. In the following we refer to the different processing conditions as high/low temperature profile or screw speed. The samples will be called as HL for the samples obtained at higher temperature profile and lower screw speed, as HH for the samples

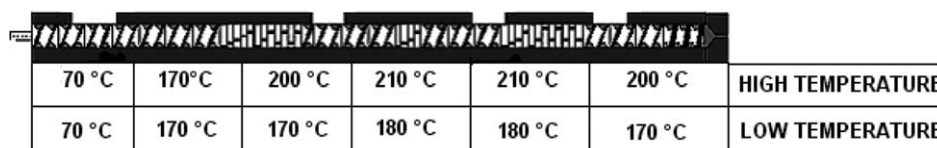


Fig. 1. Barrel temperature profiles.

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