



## Macromolecular Nanotechnology

## Effect of poly(tetrafluoroethylene) nanofibers on foaming behavior of linear and branched polypropylenes



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## ABSTRACT

The influence of poly(tetrafluoroethylene) (PTFE) nanofibers, generated *in situ* during shear extrusion, on foaming behavior of linear (L-PP) and long-chain branched (LCB-PP) polypropylenes has been investigated. PTFE nanofibers significantly improved both thermal and rheological properties of PPs studied. PTFE nanofibers formed an entangled network and induced the strain hardening of PP/PTFE nanocomposites. Foams of neat PP and PP/PTFE nanocomposites were produced using continuous extrusion foaming and autoclave-based bead foaming. The presence of PTFE nanofibers resulted in much smaller foam cells and higher cell concentration as compared to neat PPs. In addition, the autoclave-based bead foaming showed that PTFE nanofibers nucleated cells formation in LCB-PP and participated in controlling the cells growth. The entanglements between PTFE nanofibers prevented excessive expansion of cells. As the result, expanded beads of LCB-PP/PTFE nanocomposite possessed ten times smaller cells, narrower cell size distribution and much higher cell concentration as compared to neat LCB-PP.

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## 1. Introduction

Polymeric foams consist of at least two phases: solid polymer matrix and a gaseous phase that contributes to the formation of cells. In general, foamed polymer products are much lighter than their solid counterparts and possess various unique characteristics such as higher specific tensile strength, higher toughness, superior thermal and sound insulation properties [1–3].

Polypropylene (PP) is a semi-crystalline polymer having some unique advantages over polystyrene (PS) and polyethylene (PE) that makes it attractive for foaming applications. PP has high stiffness, excellent chemical resistance, good impact resistance, and the most important, can be used at higher service temperatures over PS and PE [4,5]. Unfortunately, low melt strength of linear PP (L-PP) near processing temperatures and lack of strain hardening, also known as extensional thickening during cell growth, cause difficulties in production of closed-cell L-PP foams with a high expansion ratio and uniform cell distribution [6,7]. Several ways to improve the foamability of L-PP are known: (1) modification of chain structure either by long-chain branching or chain grafting using electron-beam irradiation or chemical crosslinking methods [8–10], (2) modification of the molecular weight distribution by adding e.g. di-2-ethylhexyl peroxy carbonate [11], and (3) melt blending L-PP with long-chain branched polypropylenes (LCB-PP) [12,13]. Despite the fact that above methods are effective strate-

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gies to improve the melt strength of L-PP, broadening foaming window, retard cell coalescence, and increase expansion ratio, commercially available LCB-PP resins are at least twice as expensive as L-PP.

Alternative and very promising method to improve the foamability of L-PP is addition of nanofillers. Okamoto et al. [14] showed that the presence of nanoclay in L-PP matrix enhanced cell nucleation and increased extensional viscosity of the melt which resulted in a suppressed cell coalescence. Recently, poly(tetrafluoroethylene) (PTFE) has been used for broadening the foaming window and improving the cellular structure of L-PP foams [15] as well as increasing the expansion ratio [16]. The improvements of cellular structure have been attributed to the increased melt strength of L-PP as well as strain hardening behavior induced by presence of PTFE fibers generated *in situ* during extrusion process. This one-step manufacturing of composites reinforced with PTFE nanofibers was developed basing on the reports about the application of PTFE emulsions and powders at a low concentration, below 1 wt.%, as nucleating agent for crystallization of other polymers [17–20]. The nanofibrillation process is in fact a solid-state deformation of PTFE crystals embedded in molten polymeric matrix, i.e. a transformation of PTFE grains into nanofibers by shearing via various viscous molten thermoplastic polymers. The features of nanofibrillation have been described in details earlier by us [21–23]. It has been shown that the entangled network of PTFE nanofibers induced the strain hardening in linear polymers, which do not show the strain hardening themselves, as well as enhanced the strain hardening in branched polymers. The higher the content of PTFE nanofibers and the larger the strain rate applied, the more pronounced the strain hardening occurred. Additionally, the presence of PTFE nanofibers significantly improved the melt strength of studied thermoplastic polymers [22–24].

In this paper, we present a method to improve the foamability of L-PP as well as LCB-PP by 3 wt.% of PTFE nanofibers *in situ* generated during compounding PTFE powder with molten PP matrix. Continuous extrusion foaming of L-PP, LCB-PP and their nanocomposites with PTFE nanofibers was performed to produce foams in a form of strands and large chunks. The later form was obtained by filling a cylindrical cavity with foamed strand. In addition, the autoclave-based bead foaming was used to produce expanded beads of LCB-PP and LCB-PP/PTFE nanocomposite. The morphology and thermal properties of neat PPs and PP/PTFE nanocomposites as well as their rheological behavior under uniaxial extensional flow were studied. The effect of PTFE nanofibers on the cellular structure of PP-based foams, especially on the cell size and cell concentration, was examined.

## 2. Experimental

### 2.1. Materials

Nascent PTFE powder, Teflon 7C with average particle size of 28  $\mu\text{m}$  and melting temperature,  $T_m = 345.7\text{ }^\circ\text{C}$ , supplied by DuPont, was utilized as a deformable filler. Two grades of isotactic polypropylene differing in chain topology, were used as polymer matrices: (1) linear homopolymer, Adstif HA840K (L-PP) with a melt flow index, MFI = 3.5 g/10 min (230  $^\circ\text{C}$ , 2.16 kg) and density,  $d = 0.9\text{ g/cm}^3$  supplied by LyondellBasell; and (2) branched homopolymer, Daploy WB135HMS (LCB-PP) having MFI = 2.4 g/10 min (230  $^\circ\text{C}$ , 2.16 kg) and  $d = 0.9\text{ g/cm}^3$  obtained from Borealis. Polypropylene dedicated for rotational molding in the form of a fine powder, Icorene 4014 (PP<sub>powder</sub>) with MFI = 15 g/10 min (230  $^\circ\text{C}$ , 2.16 kg) and  $d = 0.9\text{ g/cm}^3$ , supplied by A. Schulman, was used for preparation of a masterbatch with 20 wt.% of PTFE. Porofo ADC/L-C2 with the active ingredient being azodicarbonamide (content of 99.1%) having peak decomposition temperature of 214  $^\circ\text{C}$  and volumetric gas yield of 228 ml/g at 210  $^\circ\text{C}$ , provided by Lanxess, was used as chemical blowing agent for the continuous extrusion foaming. *n*-butane with 99.9% purity, obtained from Air-Liquide, was used as physical blowing agent for autoclave-based foaming.

### 2.2. Preparation of PP/PTFE nanocomposites

The materials studied further in the paper were prepared by compounding PP with PTFE powder at temperature higher than the melting temperature of PP matrix but lower than the melting temperature of PTFE crystals. In order to reduce the aggregation of PTFE grains, a masterbatch based on PP<sub>powder</sub> and containing 20 wt.% of PTFE was prepared by compounding in a co-rotating twin-screw extruder 2x20/40D EHP (Zamak Mercator) operating at 100 rpm. The temperature zones in the extruder barrel were maintained at 175  $^\circ\text{C}$ , 180  $^\circ\text{C}$ , 185  $^\circ\text{C}$ , 185  $^\circ\text{C}$ , 190  $^\circ\text{C}$ , 190  $^\circ\text{C}$ , 195  $^\circ\text{C}$ , 195  $^\circ\text{C}$ , 200  $^\circ\text{C}$ , and 200  $^\circ\text{C}$ . Then the PP<sub>powder</sub>/PTFE masterbatch was diluted with respective PP by compounding using the same co-rotating twin-screw extruder operating at 100 rpm in order to obtain 3 wt.% of PTFE nanofibers in the final material. The temperature profile in the extruder barrel at 170  $^\circ\text{C}$ , 175  $^\circ\text{C}$ , 180  $^\circ\text{C}$ , 185  $^\circ\text{C}$ , 190  $^\circ\text{C}$ , 190  $^\circ\text{C}$ , 195  $^\circ\text{C}$ , 195  $^\circ\text{C}$ , 195  $^\circ\text{C}$ , and 195  $^\circ\text{C}$  was applied. PTFE nanofibers were formed during both preparation of the masterbatch and during diluting by shearing extrusion. It is estimated based on the screw-cylinder clearance and screw rotation speed that the material during diluting the masterbatch was subjected to shear with the rate of 1040  $\text{s}^{-1}$ .

### 2.3. Continuous extrusion foaming

Continuous extrusion foaming with a chemical blowing agent (Porofo ADC/L-C2) was used to prepare the foams of neat PP and PP containing 3 wt.% of PTFE nanofibers. A two-step processing route was applied for all studied materials. During first extrusion, 1 wt.% of the blowing agent was compounded with the PP-based materials, without foaming, in a single-

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