EXTRUSION COMPOUNDING OF POLYETHYLENE WITH BLOWING AGENTS

Carla I. Martins^{1*}, José A Covas², Tiago Ferreira³

Institute for Polymers and Composites, University of Minho, Guimarães, Portugal ¹ cmartins@dep.uminho.pt; ² jcovas @dep.uminho.pt; ³a68498@alunos.uminho.pt

* Correspondence: cmartins@dep.uminho.pt, IPC/DEP, University of Minho, 4800-058, Guimarães, Portugal

Key Words: Azodicarbonamide; polyethylene; compounding; extrusion; foams.

Abstract

Cellular plastics are very attractive for the production of lightweight, structural and/or large dimension parts, e.g., boats, floaters, decks, etc. For their production, polymers and chemical blowing agents are either mixed or compounded prior to processing by injection or rotational moulding. It is essential to ensure good dispersion of the blowing agent in the polymeric matrix, and prevent its activation from occurring during compounding, i.e., an optimal processing window must be used. The aim of this work is to produce medium density polyethylene with Azodicarbonamide (MDPE/ADCA) masterbatches in pellet form for further processing by rotational moulding. For that purpose, a set of experimental procedures was conducted to evaluate the correct processing window without premature expansion during extrusion. Upon melt compounding of the masterbatch in pellets of different sizes, foamed parts were produced and characterized in terms of visual aspect, expansion ability and morphology.

Materials and methods

The following materials were used in the experiments: (a) medium-density polyethylene (MDPE) Advancene EM-3405-UVH, manufactured by ETHYDCO (Egypt), with MFI of 5 g/10 min (190°C/2.16 kg) and density of 0,934 g/cm³ and (b) Azodicarbonamide (ADCA) LUVOPOR ABF/70 P-FF, supplied by Lehmann & Voss & Co (Germany), an exothermic chemical blowing agent (CBA) in yellow powder form, with a decomposition temperature at 200°C, gas yield of 220 ml/g and a recommended processing temperature above 220°C. In order to estimate the thermal conditions avoiding premature decomposition of the blowing agent, premixed MDPE with 1wt% ADCA were processed in a DAVENSTEST melt indexer (Davenport, UK). The temperature was varied between 150 and 170°C, and the load between 2.016 kg and 5 kg. Filaments were collected every minute during 10 min and imaged using a digital microscope Leica DMS 1000. Based on the data collected, MDPE/1% ADCA were compounded in a co-rotating LEISTRITZ LSM 30.34 (Leistritz, Germany) twin-screw extruder (screw diameter 30 mm; L/D=29), using the conditions presented in Table 1. The filaments obtained were cold pelletized using a LEISTRITZ rotary cutter into pellets with sizes of 1, 2 and 3 mm, which were subsequently exposed to temperatures of 220-240°C, during 10-60min, to evaluate the volume expansion and cell diameter of the foams formed. Samples were cut crosswise and then imaged with a digital microscope Leica DMS 1000, and analysed with a Leica Application Suite. The average cell diameter was computed from 100 measurements.

Results and Discussion

Compounding should be carried out above the melting temperature of the polymer and below the onset decomposition temperature of CBA, to prevent obtaining a foamed extrudate [1].

Figure 1 depicts images of filaments produced with the MFI equipment, demonstrating that at 150°C no expansion of the blowing agent is observed, whereas at 160 and 170°C cell nucleation and growth occur at different times, depending on temperature and applied load. The increase of temperature reduces melt viscosity, thus favouring gas diffusion and bubble-bubble interaction [2], and promoting cell coalescence and coarsening, as seems to be observed in some filaments. Also, a load of 5kg induces a higher melt pressure during flow of the molten polymer in the barrel, thus hindering cell nucleation and the ability to foam.

ADCA (wt%)	Barrel/Die Temp.	Screw speed	Flow rate (kg/h)	Haul-off (m/min)	Die diameter	Extrudate diameter (mm)
	(0)	(1)	2	6	(11111)	1,09±0,05
1	150	78	<u>6,5</u> 5,5	6	3	$2,10\pm0,05$ 3 03+0 05

Table1 – Processing conditions used for the production of MDPE/ADCA pellets



Figure 1 – Effect of processing conditions on the decomposition of ADCA in filaments.

Extrusion melt compounding was successfully carried out at 150°C, as shown in Figure 2. There were no signs of premature expansion of the blowing agent, the yellow colour resulting from the addition of ADCA.



Figure 2 – Filaments and pellets of MDPE/ADCA masterbatch.

Both PE/ADCA powder mixtures and masterbatches were submitted to a heat treatment to compare their ADCA decomposition rates above 220°C (processing temperature recommended for ADCA). As seen in Figure 3, foams were nicely produced in all cases, although the expanded volume was slightly lower for the masterbatch. This occurs because since the masterbatch pellets have lower heat transfer rate than the fine powder particles, the release of gas from blowing agent decomposition is less and, therefore, the foam expansion is smaller. Although the activation temperature of ADCA is approximately 200°C, time is needed for its nucleation and growth [3]. Increasing the temperature to 230°C and 240°C fosters cellular growth, resulting in a fully expanded white foam. However, the images also show foamed samples of different colours, due to thermo-oxidative degradation. As demonstrated in Figure 4, the latter occurs only externally, as it appears solely on the exposed surface of the sample.



Figure 3 – (top) Images of foamed samples after exposure to temperatures in the range 220 - 240°C during 10 - 60 min and (bottom) corresponding rate of volume change.



Figure 4 – Example of a cellular sample from Figure 3. Core (A); thermo-oxidized outer surface (B) and non-degraded bottom and side surfaces (C).

As depicted in Figure 5, the cell diameter increases generally with time. There seems to be no significant difference on cell size between 220 and 240°C. According to the literature, when foaming time and temperature increase, cell coalescence and rupture may occur, the gas can eventually escape resulting in a larger cell size [3]. Pellets with 3mm have coarser cells, given the higher proportion of available gas for the particle volume.



Figure 5 – Average cellular diameter as a function of heat treatment for powder samples and pellets of different diameters. Images A, B and C are for 220°C and 10, 30 and 60 min, respectively.

Conclusions

Successful compounding of MDPE/1wt% ADCA was performed at 150°C. Pellets of different sizes (1, 2 and 3 mm) were produced by cold pelletization. The pellets were yellow due to the presence of ADCA, with no signs of premature expansion of the blowing agent. Taking into consideration the typical processing cycle of MDPE by rotational moulding and the temperature needed for the decomposition of ADCA, the pellets were subjected to various temperatures (220-240°C) during different times (10-60min). Both powder mixtures and masterbatches were analysed. Foams were nicely produced in all cases, although the expanded volume was slightly lower for the masterbatch. Although the activation temperatures of 230°C and 240°C foster cellular growth, resulting in a fully expanded white foam. The possibility of producing masterbatches containing chemical blowing agents was proven, without loss of expansion ability. In future, they can be used to produce foamed parts by other processing techniques.

References

[1] R. Pop-Iliev, "Processing of fine-cell polypropylene foams in compounding-based Rotational Foam Molding", MSc thesis, Univ. Toronto, 1999.

[2] D.A. Klempner and K.C. Frisch, Handbook of Polymeric Foams and Foam Technology, Hanser, Munich, Germany (1991).

[3] Pop-Iliev, R., Dong, N., Xu, D., & Park, C. B. (2007). Visualization of the foaming mechanism of polyethylene blown by chemical blowing agents under ambient pressure. Adv. Polym. Tech., 26(4), 213-222. doi:10.1002/adv.20102

Acknowledgements



This project has received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No. 734205 – H2020-MSCA-RISE-2016.