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Polyoxymethylene foam: From an investigation of key factors related to porous morphologies and microstructure to the optimization of foam properties

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ABSTRACT

Polyoxymethylene (POM) foam based on using of a blowing agent, i.e. azodicarbonamide (ADCA) is proposed. The viscosity represented by melt flow index (MFI), which is related to the dioxolane content in POM, serves as the main factor to control the spherical cell size and its uniform distribution, including cell density. The POM foam with 13 wt% dioxolane content (V20HE) produced at 200 °C with ADCA content at 1 part per hundred parts of resin (phr) under 0.20 MPa is confirmed to be the optimal condition. At this condition, ADCA initiates cyanic acid with CO_2, and allows the monodispersed bubbles without significant degradation of POM. An increase in impact strength of V20HE foam confirms the role of the cell in absorbing the impact. The microstructure analysis based on the evaluation of folded chain crystal (FCC) and extended chain crystal (ECC) by FTIR reveals that the compression pressure applied primarily initiates the ECC. By simply varying the compression pressure, it is possible to fine tune the microstructure and control the impact strength, toughness, and tensile modulus of POM foam.

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1. Introduction

Polymeric foams have attracted much attention due to their excellent properties, including low density, light-weight structure, and good thermal and sound insulation. They are widely used in many applications such as packaging, insulation and sport equipment [1]. The foam can be formed following several processing methods such as phase separation [2], in-situ polymerization [3], extrusion [4], injection molding [5], and compression mold [6]. The use of blowing agents, e.g. azodicarbonamide (ADCA) and dicumylperoxide (DCP), to generate gas in the polymer matrices is also involved. Typically, polymers, such as polyurethane (PU), polystyrene (PS), polyethylene (PE) and polypropylene (PP), are known as polymeric foams [1]. However, polymeric foams with good thermal and organic solvent resistance, e.g. benzene, toluene, chloroform and acetone, are also needed. Furthermore, engineering thermoplastic foams, like poly(ethylene terephthalate) (PET) [6,7], polycarbonates (PC) [8,9], polysulfone (PSF) [10] and polyimides [11–13], are alternative choices for excellent chemical resistance in addition to mechanical and thermal properties.

Polyoxymethylene (POM) is one of engineering thermoplastics with high crystallinity, modulus, and tensile strength as well as specific wear and solvent resistant properties [14]. Thus, POM is good for moving parts used in automobiles, electronic appliances, and medical devices [15]. The fundamental understanding of POM microstructures related to the properties is still receiving much attention as it is considered as a guideline when considering other potential applications. Examples of current research are POM/organoclay nanocomposite [16,17], POM blends [18–20] and POM nanofiber [21,22].

As mentioned above, although the nanocomposites and nanofibers of POM along with the microstructure analyses have been proposed, it appears that POM foam processing is still unknown. It should be noted that POM tends to degrade and generates formaldehyde during processing when the temperature is in the range of 160 °C–180 °C [19,23,24]. Therefore, it is possible that when a bubbling agent was added to generate foam, an increase in chain degradation or, in other words, a decrease in thermal stability might occur at the same time.

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In fact, the copolymerization with oxyethylene chain such as ethylene oxide, 1,3-dioxolane (DOL), 1,3-dioxepane, or polyethylene glycol (PEG), so-called "copolymer POM" [25] are successfully developed to maintain thermal stability during processing. For example, Lüftl, S. et al. studied the thermal stability of the commercial copolymer POM and declared that CO_2, and fragments of CH_3COOH were generated at 225 °C before giving formaldehyde at 280 °C [26].

Based on this viewpoint, as the processing of foam via blowing agent is under the molten stage of polymer, the present work focuses on not only the process to obtain POM foam but also the key factors to control the porous morphologies, or mechanical properties, including the relationship between microstructure and macro-scale properties. The molten stage foam formation may lead to chain degradation during thermal treatment. Therefore, the optimal condition to avoid the degradation needs to be identified. The fact that the mechanical properties are directly related to pore size and dispersion as well as pore size distribution, the factors to control the foam formation and its morphology are important and are taken in the scope of the work. In addition, the present work also covers the clarification of foam microstructure since it leads to an understanding of how the foam processing influences the packing structure of POM.

2. Experimental section

2.1. Materials

A series of POM, i.e. A2003 (containing oxyethylene unit 1.5 wt% (A2003), V20HE (containing oxyethylene unit 13 wt%), and V20HT (containing oxyethylene unit 13 wt% and polyethylene glycol 1 wt %) were provided by Thai Polyacetal Co. Ltd., Thailand. The properties of each resin are shown in Table 1. Azodicarbonamide (ADCA) was purchased from USACO Co. Ltd., Thailand.

Table 1
Properties of POM resins.

Sample	DOL[a] (%)	T_m (°C)	χ_c	T_d (°C)	MFI (g/10 min)
A2003	1.5	172.2	47.4	338	~9
V20HE	13	155.3	34.7	421	~11
V20HT	13 + PEG 1 wt%	61.6, 157.2	1.9, 32.3	420	~12

T_m = melting temperature.
χ_c = crystallinity degree.
T_d = degradation temperature.
MFI = Melt flow index.
[a] Dioxolane content.

2.2. Preparation of POM foams

A2003 (70 g) was mixed with ADCA for 1 phr by using a Brabender® OHG Duisburg (Germany) (Fig. S1) under internal mixture temperature at 180 °C with screw speed 40 rpm. The mixture obtained was loaded into a square mold (10 × 10 × 0.2 cm^3) and compressed under 0.05 MPa at 200 °C for 15 min by using a Wabash V50H (U.S.A) compression mold. The foam obtained was cooled to room temperature. V20HE and V20HT were prepared by the same procedures. The ADCA contents were varied for 1.5 phr, 2 phr, and 3 phr. The compression pressure was varied for 1.0, 1.5, and 2.0 MPa.

2.3. Instruments and equipment

The thermal properties of samples were investigated by a 200F3 NETZCH (Germany) differential scanning calorimeter (DSC) under nitrogen with flow rate of 50 ml/min and a heating rate of 10 °C/min from 25 °C to 180 °C. The degree of crystallinity (χ_c) was estimated by assuming that the heat of melting per unit mass of crystalline material is identical to that of melting of 100% crystalline POM, i.e. 317.93 J/g [27]. The melt flow index (MFI) was measured according to the ASTM D1238 using a 4105 Zwick Extrusion Plastometer capillary canal melt viscometer (Germany). The melt temperature at 190 °C with a load of 2.16 kg was applied as the

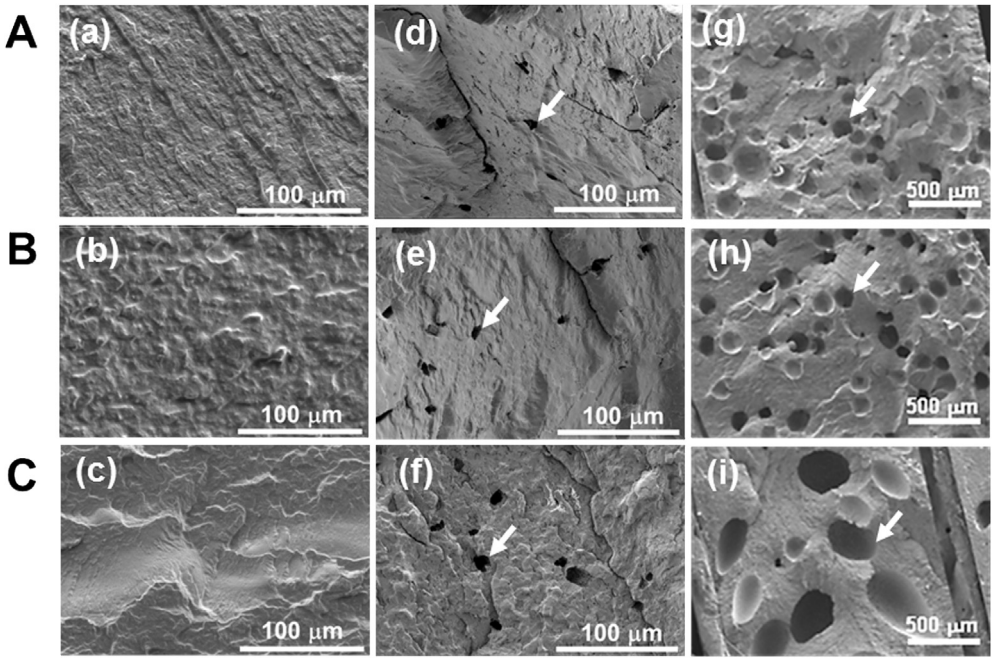

Fig. 1. Scanning electron micrographs of POM with MFI (A) 9 g/10 min (A2003), (B) 11 g/10 min (V20HE), and (C) 12 g/10 min (V20HT) for (a)–(c) POM bulks, (d)–(f) POM blends with ADCA (1 phr) before foaming. The samples were treated with DMSO to dissolve ADCA and the arrows indicating the traces of ADCA, and (g)–(i) the samples from (d)–(f) after foaming and the arrows indicating the bubbles in the samples.

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