The Effect of Nucleating Agents on the Foam Extrusion Characteristics

Today it is a well-established fact that the use of nucleating agents is absolutely essential for controlling the cell morphology (namely, the number of cells, the cell size, and its distribution) in thermoplastic foams. Without a nucleating agent, the number of cells is too small, and the cell size is too large to produce low density foams of thermoplastic resin.[1] On the other hand, there exists, unfortunately, no theoretical guidance, suggesting the type of nucleating agent to be used for a given combination of resin and blowing agent. Understandably, the problem at hand is very complex, because it involves heterogenous nucleation from a mixture of molten polymer and volatile liquid at high temperature and high pressure. Evidence indicates that the surface characteristics of a nucleating agent are of paramount importance in controlling the foam quality in the foam extrusion process.

In our previous study,[2,3] we investigated the effects of the processing variables, the type and concentration of fluorocarbon blowing agent, and the geometry of the cylindrical extrusion die on the foam extrusion characteristics of low-density polyethylene and polystyrene. We have continued the efforts by investigating the effects of the type and concentration of nucleating agent on the foam extrusion characteristics of low-density polyethylene. In this study, we have measured: (a) the expansion ratio of extrudate; (b) foam density; (c) cell size and open cell fraction. In this paper, we shall report some highlights of our findings.

EXPERIMENTAL

The polymer used is a low-density polyethylene (ElPaso Polyolefin, Rexene PE143), whose molecular characteristics and rheological properties are reported in our earlier paper.[4] As blowing agent, dichlorofluoromethane (FC-12) and dichlorotetrafluoroethane (FC-114) were used. Throughout the entire study, we used a fixed concentration (15 wt %) of the blowing agents.

As nucleating agent, we used (a) calcium carbonate, (b) calcium hydroxide, (c) calcium stearate, (d) zinc sterate, (e) aluminum stearate, (f) talc, (g) azodicarbonamide, (h) sodium bicarbonate, and (i) a 1.00/1.25 by wt mixture of citric acid and sodium bicarbonate.

Using a drum tumbler, a predetermined amount of nucleating agent was blended with the plastic pellets, which had previously been wet with mineral oil. In this way, a small amount of nucleating agent in the form of powder was uniformly distributed to the plastic pellets. The blend was then fed to the hopper of the extruder, where mixing of the ingredients was continued and the material was brought to a homogeneous melt.

The apparatus and experimental procedure employed are the same as that described in our previous paper.[2] Figure 1 of that paper shows a schematic layout of the foam extrusion apparatus employed in this study. The performance of the apparatus was checked by continuously recording the die pressure, and the flow rate of blowing agent. The injection pressure of the blowing agent (1000–2000 psi) was also recorded and provided an indication of how uniform the feed of the extruder was. Throughout the entire study, we used the following cylindrical die geometry: (a) entrance angle 60°; (b) reservoir-to-capillary diameter ratio 8; (c) capillary length-to-diameter (L/D) ratio zero (i.e., a converging die), where the capillary diameter was 3.175 mm.

During the experiment, we measured extrudate diameter and foam density. With selected foam samples, we measured the cell size and its distribution by using a magnifying lens and the open cell fraction by using an Air Pycnometer (Beckman Model 930).

RESULTS AND DISCUSSION

The effects of the nucleating agent concentration on foam density (ρ) are given in Figure 1, and the effects of the nucleating agent concentration on extrudate swell (d_j/D) ratio are

Journal of Applied Polymer Science, Vol. 29, 4465–4470 (1984)
 CCC 0021-8995/84/124465-06$04.00

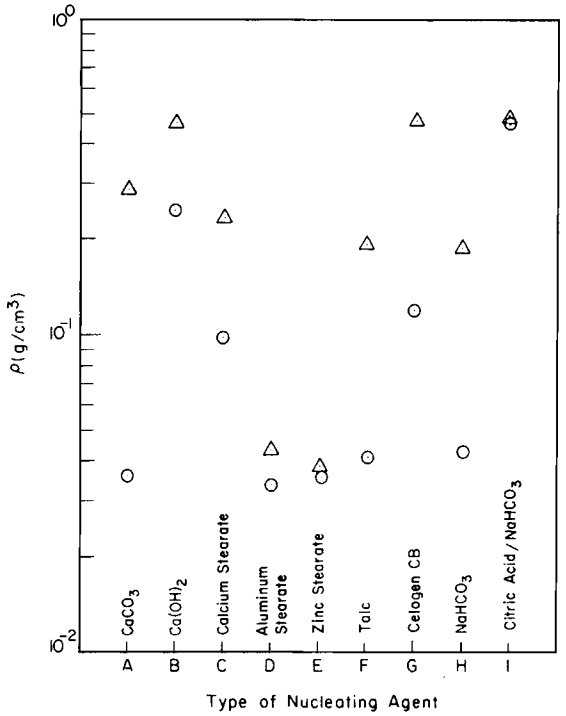

Fig. 1. Foam density vs. the type of nucleating agent in the LDPE foam extrusion at 100°C, using FC-12 (15 wt %) as blowing agent: (A) calcium carbonate; (B) calcium hydroxide; (C) calcium stearate; (D) zinc stearate; (E) aluminum stearate; (F) talc; (G) celogen CB; (H) sodium bicarbonate; (I) citric acid/sodium bicarbonate (1/1.25 by wt). Nucleating agent concentration (wt %): (⊙) 0.1; (△) 0.3.

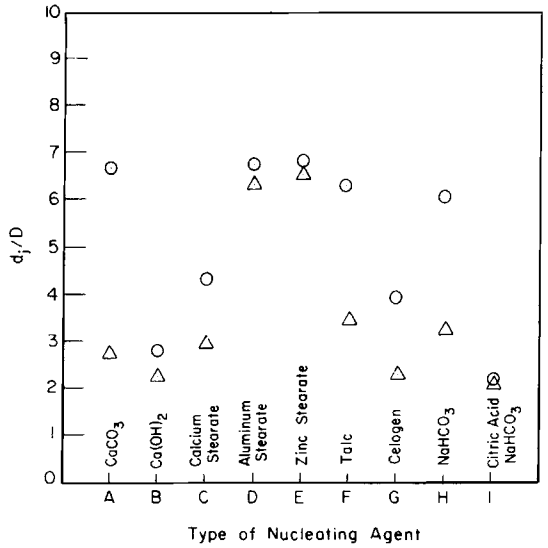

Fig. 2. Extrudate swell ratio vs. the type of nucleating agent. Processing conditions and symbols are the same as in Figure 1.

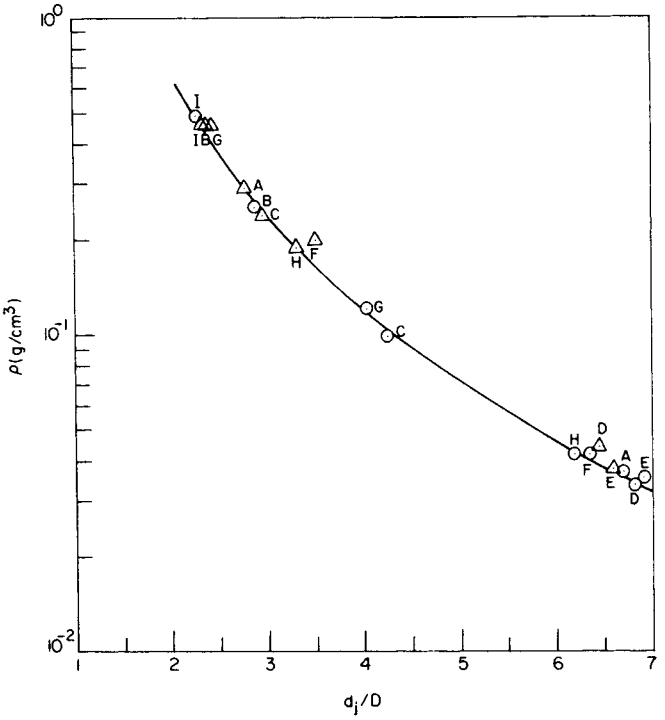

Fig. 3. Foam density vs. extrudate swell ratio. Processing conditions and symbols are the same as in Figure 1.

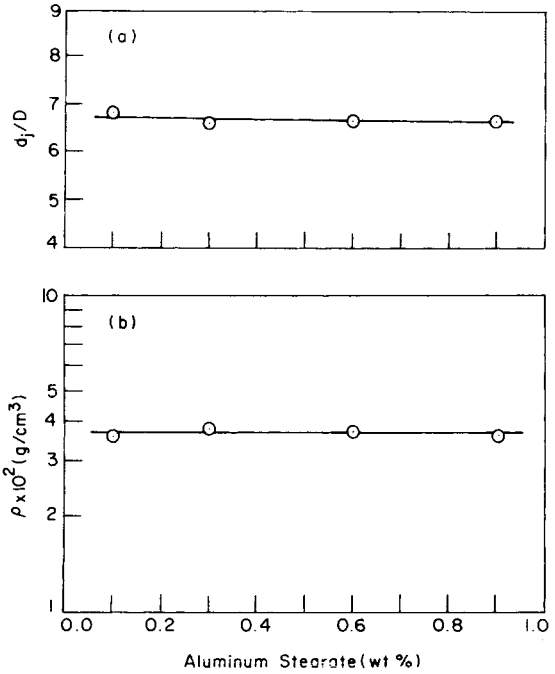

Fig. 4. Extrudate swell ratio and foam density vs. concentration of aluminum stearate. Processing conditions are the same as in Figure 1.

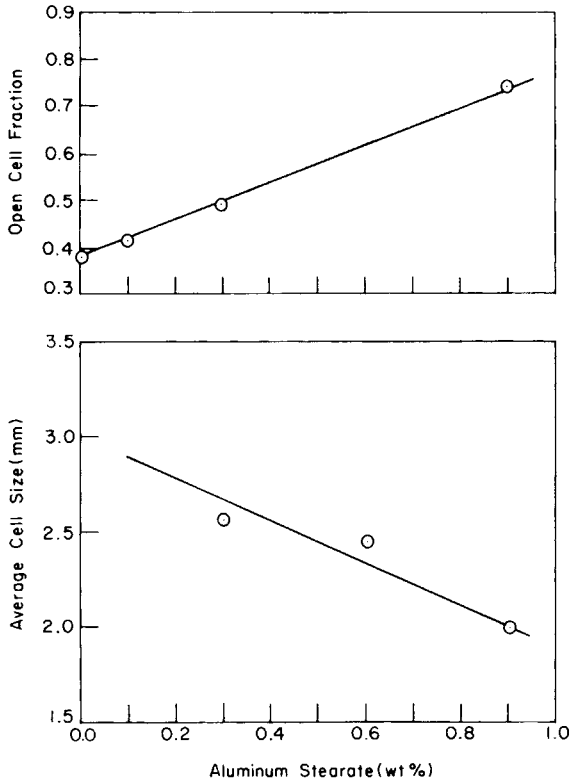

Fig. 5. Open cell fraction and average cell size vs. concentration of aluminum stearate. Processing conditions are the same as in Figure 1.

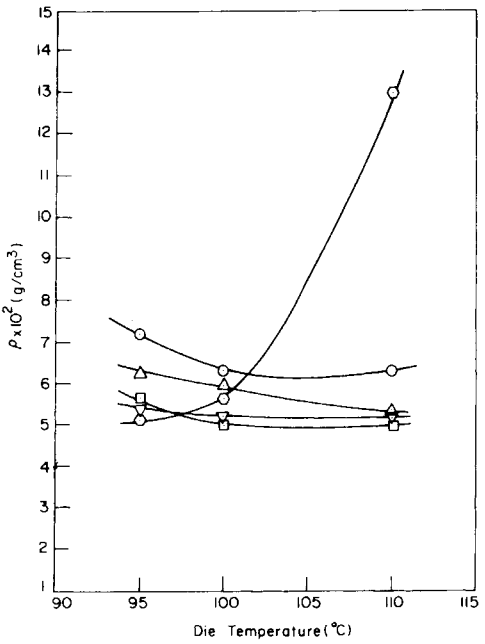

Fig. 6. Foam density vs. die temperature in the LDPE foam extrusion with FC-114 (15 wt %) as blowing agent. The type of nucleating agents investigated: (⊙) without nucleating agent; (△) 0.3 wt % calcium stearate; (⊡) 0.3 wt % zinc stearate; (▽) 0.3 wt % aluminum stearate; (⊙) 0.3 wt % talc.

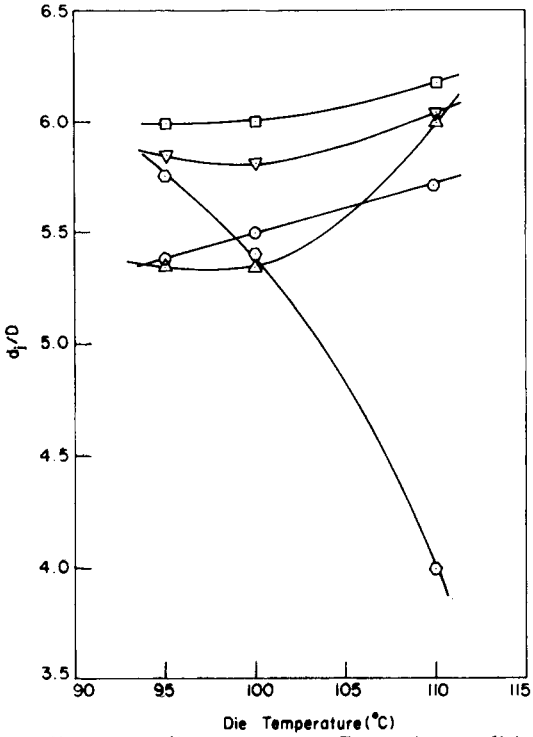

Fig. 7. Extrudate swell ratio vs. die temperature. Processing conditions and symbols are the same as in Figure 4.

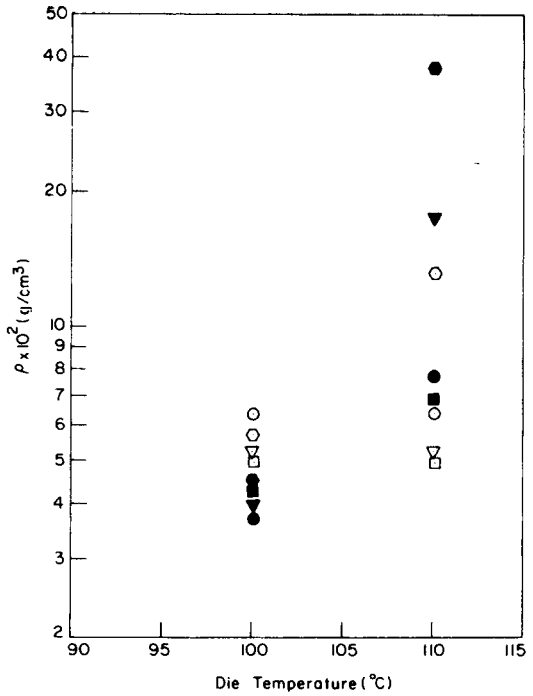

Fig. 8. Foam density vs. die temperature in the LDPE foam extrusion: (a) without nucleating agent: (●) FC-12; (⊙) FC-114; (b) with 0.3 wt % aluminum stearate: (▼) FC-12; (▽) FC-114; (c) with 0.3 wt % zinc stearate: (■) FC-12; (⊡) FC-114; (d) with 0.3 wt % talc: (⬢) FC-12; (⊙) FC-114

given in Figure 2, for the nine different nucleating agents investigated. With two exceptions (zinc stearate and aluminum stearate), in general, ρ increases and the d_j/D ratio decreases as the nucleating agent concentration is increased. In fact, in such cases the cell collapse was so severe that little extrudate swell was observed when the nucleating agent concentration was increased beyond 0.3 wt %.

Figure 3 gives plots of ρ vs. d_j/D ratio, demonstrating that, regardless of the type and the concentration of nucleating agents, an inverse relationship exists between ρ and d_j/D ratio. A similar observation was made in our earlier investigation.[2]

However, when aluminum stearate was used as nucleating agent, both the d_j/D ratio and ρ remain constant, even when the nucleating agent concentration is increased up to 0.9 wt %, as shown in Figure 4. It is of interest to note in Figure 5 that as the nucleating agent concentration is increased, the average cell size is decreased and the open cell fraction is increased. Similar observation was also noted in our earlier study,[2] which used talc as nucleating agent.

Figure 6 describes the effect of die temperature on ρ, and Figure 7 the effect of die temperature on the d_j/D ratio, for different nucleating agents, namely, calcium stearate, zinc stearate, aluminum stearate, and talc. It is of interest to note that ρ tends to decrease, while the d_j/D ratio tends to increase, as the die temperature is increased from 95 to 110°C. However, the opposite trends are seen with talc, i.e., ρ increases and the d_j/D ratio decreases very sharply as the die temperature is increased from 95 to 110°C. Note that an inverse relationship exists between ρ and d_j/D. The strong temperature dependency observed of ρ and d_j/D requires careful control of die temperature when talc is used as nucleating agent. For instance, when a high-viscosity grade of LDPE resin is chosen for foam extrusion, the die temperature may have to be set at a higher temperature (say, 110°C) because of the excessive pressure drops required otherwise. In such a situation, the use of talc as nucleating agent may not give acceptable foam quality. The results presented in Figures 6 and 7 suggest the use of calcium stearate, zinc stearate, or aluminum stearate.

Figure 8 describes the effect of the type of blowing agent on foam density, at two different die temperatures. It is seen that, at the die temperature 100°C, FC-12 gives lower density foams than FC-114 does, but, at the die temperature 110°C, FC-114 gives lower density foams than FC-12 does. It can then be concluded that the choice of correct combinations of nucleating agent, blowing agent, and die temperature is essential for controlling the foam extrusion process.

References

1. J. H. Saunders and R. H. Hansen, in *Plastic Foams*, K. C. Frisch and J. H. Saunders, Eds., Vol. 1, p. 23, Marcel Dekker, New York, 1972.
2. C. D. Han and C. Y. Ma, *J. Appl. Polym. Sci.*, **28**, 2961 (1983).
3. C. Y. Ma and C. D. Han, *J. Appl. Polym. Sci.*, **28**, 2983 (1983).
4. C. D. Han and C. Y. Ma, *J. Appl. Polym. Sci.*, **28**, 831 (1983).

HENG-HUEY YANG
CHANG DAE HAN

Department of Chemical Engineering
Polytechnic Institute of New York
Brooklyn, New York 11201

Received November 21, 1983
Accepted May 15, 1984