
Extrusion of Microcellular Foams Using Pre-Saturated Pellets and Solid-State Nucleation

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ABSTRACT

A novel process for continuous production of microcellular foams has been developed and applied to make flat microcellular strips from PVC. The first novel aspect of the process consists of the way in which the blowing agent, or gas, is delivered to the extruder. We pre-saturate the solid PVC pellets with carbon dioxide. The gas-saturated pellets are then put in the extruder hopper. The second novel aspect of the process is in applying nucleation in the solid state to continuous extrusion. As the pellets are crushed and heated in the extruder, a large number of bubbles nucleate as the temperature reaches the T_g of the CO_2 plasticized PVC. The melt thus is full of nucleated bubbles that are not able to grow in size due to the pressure that develops in the barrel. When the melt exits at the die, where the pressure is atmospheric, the bubbles have a chance to grow larger and create foam. The process employs effective cooling strategies at the die exit, including lower melt temperatures, to keep the bubbles in the microcellular range.

1. INTRODUCTION

Microcellular plastics refer to closed-cell thermoplastic foams with a very large number of very small bubbles. Typically, the cells are of order $10\ \mu\text{m}$ in diameter, and there are 10^8 or more cells per cubic centimeter (cm^3) of the foam. The idea to introduce such small bubbles in plastics was first introduced by Martini *et al.*⁽¹⁾ as a means to reduce the density of solid plastics, and thus to save on material costs in applications where the full mechanical properties of solid plastics were not needed. The applications envisioned were mainly for the packaging industries with a typical wall thickness in the range of 0.5 to

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1.5 mm. For such applications the microcellular foams are novel materials, since conventional polymer foams that have cell sizes of 0.25 mm or larger are not suitable due to excessive loss of strength. An early review of microcellular foams can be found in Kumar⁽²⁾.

The basic solid-state microcellular process is a two-stage batch process. In the first stage, the polymer is placed in a pressure vessel with a high-pressure non-reacting gas. This step is usually conducted at room temperature. Over time, the gas diffuses into the polymer, and attains a uniform concentration throughout the polymer specimen. When this specimen is removed from the pressure vessel and brought to the atmospheric pressure, a "supersaturated" specimen that is thermodynamically unstable due to the excessive gas dissolved into the polymer is produced. In the second stage, the specimen is heated to what is termed the foaming temperature. This step is typically carried out in a heated bath with temperature control. The dissolved gas lowers the glass transition temperature of the polymer⁽³⁾ and the foaming temperature needs only to be above the glass transition temperature of the polymer-gas system in order for the bubbles to nucleate and grow. Since the polymer is still in a solid state, the foams thus produced are called "solid-state foams" to distinguish them from the conventional foams that are produced from a polymer melt. The solid-state microcellular process relies on heating a supersaturated polymer specimen to nucleate cells and create microcellular structures.

2. PREVIOUS WORK ON EXTRUSION OF MICROCELLULAR FOAMS

Since the invention of a process to make microcellular plastics using inert blowing agents (specifically N₂ and CO₂) was reported by Martini *et al*⁽¹⁾ a number of efforts have been made to enable large-scale production of these novel materials. The strongest emphasis has been on development of extrusion processes.

Extrusion of microcellular polymeric materials using supercritical CO₂ was first demonstrated by Park and Suh⁽⁴⁻⁶⁾ at the Massachusetts Institute of Technology. Park and Suh proposed an extrusion process in which three functional requirements would have to be satisfied in order to achieve the microcellular structure. These are – (1) plasticate the polymer, (2) create a polymer-gas solution at industrial rates and (3) promote high cell nucleation rate in the polymer-gas solution to achieve microcellular structure. This proposed process was experimentally verified for the HIPS-CO₂ system. The experimental setup consists of a single screw extruder with a compression screw

to plasticate the polymer and deliver it under pressure. Once the polymer melt is formed, a gas pump injects supercritical CO₂ into the barrel of the extruder at pressures in the range of 10 ~ 40 MPa. Static mixers at the front end of the barrel enable effective mixing of the polymer and supercritical CO₂ gas thereby enhancing the dissolution of gas into the molten polymer. This ensures uniform distribution of the blowing agent inside the polymer melt. Past this point in the barrel the polymer-gas solution enters a custom designed nozzle to achieve a predetermined pressure drop rate. This pressure drop creates a thermodynamic instability that nucleates millions of micro sized bubbles. The nucleated polymer melt is then pushed through a filamentary die to shape the extruding microcellular plastic foam.

Other variations of the above approach have been reported in literature. Baldwin⁽⁷⁾ used a two-stage compression single screw extruder with static mixers at the delivery end of the screw. There is also a secondary diffusion zone, which helps to disperse the injected bubbles and creates fine bubble morphology within the polymer matrix. Extrusion of microcellular polymers in a twin-screw extruder using high-pressure carbon dioxide was reported by Seibig *et al*⁽⁸⁾. A co-rotating twin screw was used to mix the molten polymer effectively and homogenize it. A melt gear pump was used to pressurize the polymer-gas solution to a required level before allowing it to exit the nozzle. Shimbo *et al*⁽⁹⁾ developed a two stage extrusion process to obtain stable polymer-gas solution. Uniform dispersive mixing was achieved using a static mixer between the two extruders to help in mixing of the supercritical carbon dioxide gas. Finally, Huang *et al*⁽¹⁰⁾ produced microcellular foams using chemical blowing agents. They used standard extrusion setup to extrude microcellular polysulfone with azodicarbonamide and 5-phenyltetrazole as the blowing agents. There are certain key processing aspects and related challenges common to all the microcellular foam extrusion methods that use supercritical gas as described below.

- Blowing agent is delivered at supercritical pressures. High pressure and high temperature seals are required on the extruder barrel at the injection port of the supercritical fluid. This would normally require significant modifications to the existing extrusion lines or using extruding equipment specifically designed for microcellular foam processing.
- Blowing agent is injected directly into the extruder barrel while the polymer is in its molten state. The injected blowing agent is in a two-phase mixture with the molten polymer before the formation of a polymer-gas solution. For uniform dissolution mixing enhancers and complex screw designs are required in the extruder. Also, a blowing agent metering device external

to the extruder is required to monitor the amount of supercritical fluid delivered to the polymer.

- Cells are nucleated in the molten phase of the polymer. This is achieved by creating a large pressure drop across a specially designed nozzle.

In this paper we propose a novel method to achieve microcellular morphology in a continuous process using pre-saturated pellets. First, we present the idea of using saturation and nucleation in the solid polymer pellets along with lower melt temperatures as processing strategies for extruding microcellular foam. Second, we describe the experimental procedure in which the above processing strategies were utilized for the PVC-CO₂ polymer-gas system. Finally we present results showing that the proposed strategies can indeed produce microcellular PVC foams in an extrusion process using pre-saturated pellets and standard extrusion equipment.

3. PROPOSED PROCESS FOR EXTRUDING MICROCELLULAR POLYMERS

Figure 1 shows the schematic of the proposed microcellular polymer extrusion process using pre-saturated pellets. It consists of saturating polymer pellets with CO₂ gas in a pressure vessel until they achieve a uniform gas concentration level

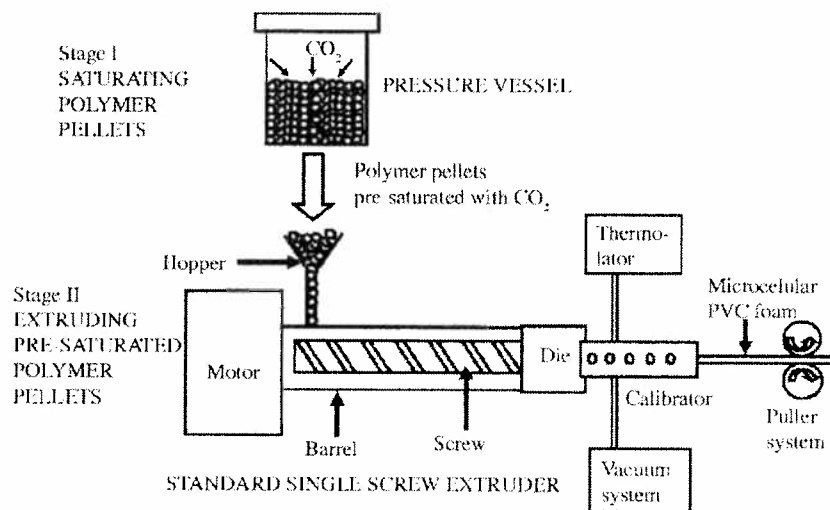


Figure 1. Schematic of the microcellular extrusion process using pre-saturated pellets. Cells are nucleated upon initial heating in the barrel, and cell growth is controlled in the calibrator

throughout the cross-section and then transferring the gas-saturated polymer pellets into the hopper of a standard compression single screw extruder. In the extruder the polymer pellets are heated, crushed and delivered under pressure to a shaping die. At the exit end of the die, cell growth occurs in the gas-saturated polymer melt. The novel aspects of this process are using pre-saturated pellets to deliver gas to the polymer melt, using cell nucleation in the solid polymer pellets to achieve microcellular morphology and using lower melt temperatures to achieve controlled cell growth. The following subsections describe these strategies in greater detail.

3.1 Delivery of CO₂ to the Process at Sub-Critical Pressures

Pre-saturating the polymer pellets with CO₂ gas before extrusion allows achieving a uniform polymer-gas solution through the cross-section of each individual pellet. This in turn would allow homogeneous dispersion of blowing agent throughout the volume of the molten polymer in the extruder. The amount of uniformly dispersed CO₂ gas that each individual pellet carries within itself will be a function of the saturation pressure and saturation temperature used in the pressure vessel. There are several advantages of using this approach of gas delivery compared to injecting supercritical CO₂ into the extruder barrel directly. It eliminates the need for a gas metering pump that is designed for supercritical fluid handling. In an attempt to achieve a uniform polymer-gas solution, complex mixing elements and screw designs are not required for mixing the injected gas with the molten polymer in the extruder barrel. High pressure and high temperature seals used on the extruder barrel at the supercritical CO₂ injection port are no longer required because the gaseous blowing agent is introduced through the hopper. The proposed method of gas delivery will require large pressure vessels in which the pellets can be saturated.

3.2 Cell Nucleation in the Solid-State

In the microcellular foaming process, cell nucleation and growth occur in the solid state when the gas-saturated polymer specimen is heated close to its effective glass-transition temperature. A number of mechanisms have been proposed to explain this nucleation phenomenon⁽¹¹⁻¹⁵⁾. Using this phenomenon we propose nucleating the cells in the gas-saturated polymer pellets while they are still in the solid state. When the polymer pellets are saturated with a high-pressure gas, a large number of microvoids get 'charged' with the gas, and act as sites where bubbles will nucleate later when the polymer is heated. As the pellets are crushed and heated in the extruder, a large number of bubbles nucleate due to drop in solubility of the gas as the temperature increases. The heating of the pellets in the feed section of the extruder barrel would

be sufficient for nucleating the large number of cells required to achieve the micro size. The melt thus is full of nucleated bubbles that are not able to grow in size due to the pressure that develops in the barrel. Also, we believe that as the polymer pellets are melted, compressed and pushed through the barrel, the nuclei created in the solid-state are sustained without been re-dissolved into the polymer melt at the high pressures experienced in the barrel. This is because at points of highest pressures in the barrel the melt temperatures are also very high resulting in a reduction of gas solubility. For example, in the case of the PS-CO₂ polymer-gas system, the solubility of CO₂ in PS at 35 C and 5 MPa pressure is 6.5% by weight whereas at 215 °C and 5 MPa the gas solubility drops to 2.2% by weight^(16,17). Similarly for other polymer-gas systems such as PVC-CO₂ at a given pressure gas solubility at melt temperature will be much lower compared to room temperature. Hence, only a small portion of the gas in the nuclei (30% for PS-CO₂) will be dissolved back into the molten polymer at the pressures experienced in the barrel. This low gas solubility at melt temperatures sustains the existing nuclei, formed in the solid state, until they reach the die exit.

There are two key advantages of using nucleation in the solid-state as opposed to nucleation in the molten state in processes which use supercritical CO₂ injection into the polymer melt. First, the proposed method eliminates the need for devices such as melt pressure building gear pumps and nucleating nozzles. Another advantage is that since the concentration of dissolved gas will be uniform in the melt the nucleation density can be expected to be equivalent in all parts of the melt. This in turn results in a consistent product with uniform foam density.

3.3. Cell Growth Control Using Lower Melt Temperatures

When the gas-saturated polymer pellets are extruded, the well documented plasticizing action of the dissolved CO₂ gas allows the polymer chains to achieve the mobility required for extrusion at lower temperatures. This means that the melt temperatures can be lowered without dangerous increase in melt pressures. These lower melt temperatures provide control over the bubble growth at the die exit. This can be explained with the aid two concepts. First, at lower melt temperatures a larger amount of the dissolved CO₂ gas will remain in solution than at higher melt temperatures. Hence, the transport of CO₂ gas from the polymer-gas solution to the nucleated cells is lower at lower extrusion temperatures thereby limiting cell growth. Second, the ideal gas law states that for a given pressure the volume of the gas is directly proportional to the temperature of the gas. Assuming that CO₂ obeys the ideal gas law in the temperature and pressure conditions experienced in the extruder and at the

die exit, decreasing the extrusion temperatures reduces the expansion of the CO₂ present in the nucleated cells. Using this cell growth at the die exit can be arrested in the microcellular range. After exiting from the die the extrudate can be immediately solidified by cooling to prevent any further cell growth. Hence, in addition to improving the economics of the foaming by lowering energy consumption, lower melt temperatures also provide control over cell growth.

4. EXPERIMENTAL - MICROCELLULAR PVC EXTRUSION

4.1 Materials

Poly-vinyl chloride (PVC) pellets from Mikron Industries, Kent, WA were used for these experiments. The resin called PVC foam formulation was designed to have higher melt strength and better foaming characteristics such as finer filler particles. 99.9% pure CO₂ gas supplied by Airgas Norpac was used for saturation of PVC pellets in the pressure vessel.

4.2 Equipment

A standard single screw extruder with a 22:1 L/D ratio, 31.75 mm dia., 3:1 compression ratio screw supplied by BBS Corporation, Spartanburg, SC was used to conduct the microcellular foam extrusion experiments. Figure 2 shows the cross-sectional dimensions of the shaping die used for these experiments. It has a rectangular exit of dimensions 38.1 mm x 3.18 mm. After the shaping die, a calibrator (shown in Figure 3) was used to regulate the temperature of the extrudate and improve its surface finish. The calibrator was fabricated from two 304.8 mm long x 101.6 mm wide x 25.4 mm thick stainless steel blocks with 38.1 mm x 1.59 mm slot machined in the center of the surface along the length. When assembled together the two steel plates formed a slit with the same dimensions (38.1 mm x 3.18 mm) as the shaping die through which the extrudate can pass as it is exiting the shaping die. Also, the calibrator blocks have 38.1 mm x 0.71 mm rectangular through slots machined at 1 in intervals in the top and bottom profile surfaces perpendicular to the flow of the extrudate. These slots are all connected to a vacuum source so that vacuum could be applied on the extruded PVC foam surfaces. This is to enhance contact between the strip and calibrator thereby increasing the cooling rate and improving the surface finish and shape of the extruded strip. The calibrator temperature was regulated using Neslab's Endocal refrigerated circulating bath that circulates cooling water through the flow channels. In addition to the calibrator the downstream equipment consists of a pulling system with adjustable speed.

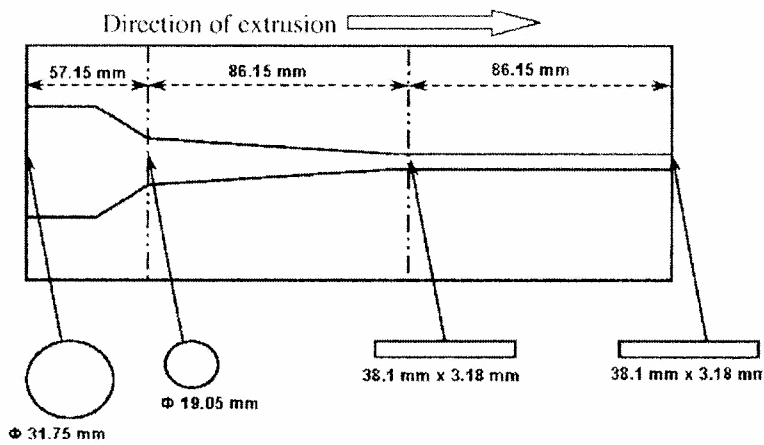


Figure 2. Cross-section of the die supplied by Mikron Industries. The geometries at the bottom, shown by leader arrows, indicate the shape and dimensions of the flow path at those points in the die. Note the transition from circular geometry to a flat geometry in the middle zone

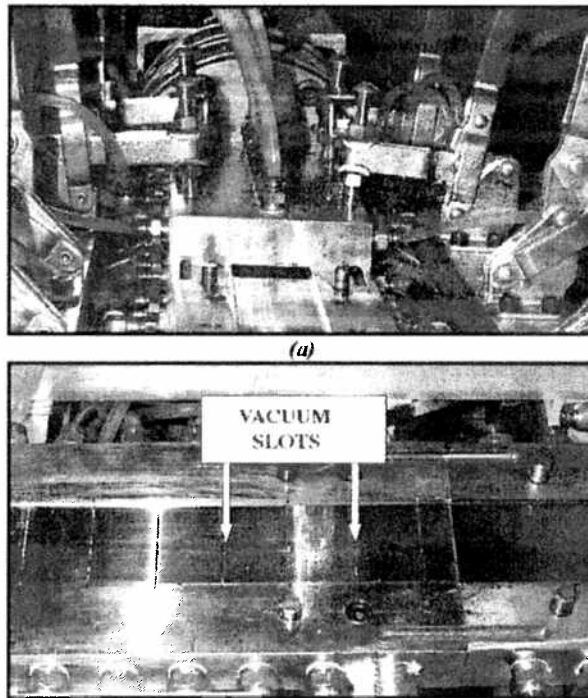


Figure 3. (a) Close-up photograph of the calibrator showing vacuum hoses on the top and plumbing for cooling water on the sides (b) Photograph of the calibrator showing the rectangular slots to apply vacuum on the extrudate

The pulling system was built in house using two rubber lined 42.67 mm dia. and 609.6 mm long stainless steel slip rolls from ENCO with an adjustable gap in between them to allow for feeding and gripping of the extruded strips. The slip rolls were driven by a chain drive using a 0.37 kW, 70:1 gear ratio, DC gear motor. Spring-loaded screws under the two rollers supports were used to adjust the gap between the rollers depending on the friction needed to pull the strip through the calibrator.

4.3 Gas Delivery - Sorption and Desorption

PVC pellets were packed in 11.5 kg lots in a porous cloth bag and were placed in a pressure vessel for saturation with CO₂ gas. The PVC pellets were cylindrical with an average diameter and thickness of 4.06 mm and 6.1 mm respectively. The pellets were saturated with CO₂ gas at a pressure of 5 ± 0.01 MPa. After saturation, the pellets were removed from the pressure vessel and were allowed to desorb at atmospheric pressure for 1 to 3 hours.

4.4. Cell Nucleation

The gas-saturated pellets were then introduced into the hopper of the standard single screw extruder. The temperatures of the feed section of the extruder for the different experimental runs are as listed in Table 1. Each of these temperatures is above the effective glass transition temperature of the PVC pellets saturated at 5 MPa pressure with CO₂ gas⁽¹⁸⁾. This allows cell nucleation to occur as soon as the pellets enter the barrel. This phenomenon occurs at the entry of the feed section before the pellets are plasticated and compressed by the heat and crushing action of the screw. Early growth of the nucleated cells is then arrested by the pressure that is developed in the barrel. Also, the pressure

Table 1. Experimental conditions and density results for microcellular extrusion experiments on PVC

S. No.	Die Temp °C	Extruder Barrel Zone Temperatures			Die Pressure	Average Density	Average Relative Density
		Metering Section C	Compression Section C	Feed Section			
1	179.4	179.4	173.9	162.8	4.48	0.53	0.37
2	173.9	176.9	168.3	154.4	4.48	0.54	0.38
3	168.3	168.3	162.8	148.9	4.48	0.73	0.51
4	162.8	162.8	157.2	140.6	4.48	0.87	0.61
5	157.2	151.7	146.1	121.1	3.45	0.91	0.63
6	148.9	143.3	132.2	110.0	3.45	1.04	0.73

in the die for various extrusion conditions is recorded in Table 1. It can be seen that the die pressure is lower than the saturation pressure (5 MPa) used for the pellets. The combinations of melt temperatures and die pressures, as listed in Table 1, are not sufficient to dissolve all the CO₂ gas (7% by weight), introduced through the PVC pellets, back into solution with the molten PVC. Hence, the nuclei formed in the solid-state will be sustained until they reach the die exit.

4.5 Controlled Cell Growth

The cell growth in the gas-saturated melt was initiated and controlled using both lower melt temperatures and effective cooling in the calibrator. The extruder screw speed was set at 14 rpm and with strip extrusion rate of 1.5 mm/s. All experiments were conducted at this screw speed. The temperatures used for the barrel zones and the die are listed in Table 1. A die temperature of 179.4 °C was recommended by the material supplier for extruding the virgin PVC that is not saturated with CO₂ gas. Starting from 179.4 °C the die temperatures were dropped in four steps of 5.5 °C and a final drop of 8.3 °C to reach 148.9 °C. The corresponding barrel zone temperatures were also dropped by the same amount. Once the extrudate reached steady state at each of the temperature conditions listed in Table 1, at least four extruded foam strips of 1.83 m length were collected. These samples were long enough such that a minimum of four 177.8 mm long tensile specimens and four 25.4 mm long density specimens can be cut from them. For all the extrusion conditions listed in Table 1, a vacuum of 0.012 MPa was applied and the calibrator temperature was maintained at 15.56 °C.

The densities of the microcellular PVC strips were determined using the ASTM D792-91 standard test method⁽¹⁹⁾. For determination of cell morphology the procedure included freeze fracturing the PVC strip samples to obtain a clean brittle fractured surface, sputter coating of the clean surface to make it conductive, and finally conducting microstructure investigation using a JOEL JSM 5200 scanning electron microscope (SEM).

5. RESULTS AND DISCUSSION

Figure 4 shows that the time required for PVC pellets with an average diameter and thickness 4.06 mm and 3.05 mm to reach equilibrium gas concentration was 3 days. Figure 5 shows the plot of gas concentration as a function of desorption time for PVC pellets saturated with CO₂ gas at 5 MPa. It can be seen from this plot that for five hours after removing the PVC pellets from the

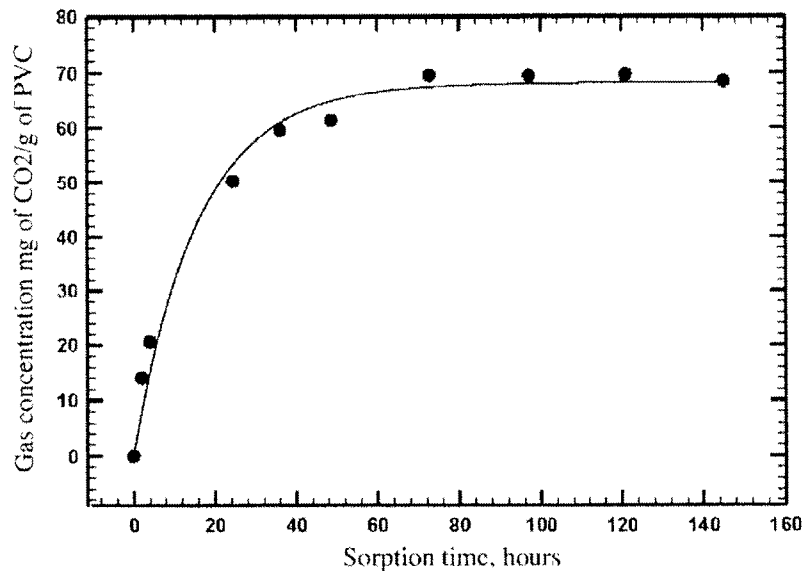


Figure 4. Plot of gas concentration versus saturation time for PVC pellets at 5 MPa. The average diameter and thickness of the pellets was 4.06 mm and 3.05 mm respectively. At equilibrium PVC pellets have 7% by weight of CO₂ dissolved in the polymer

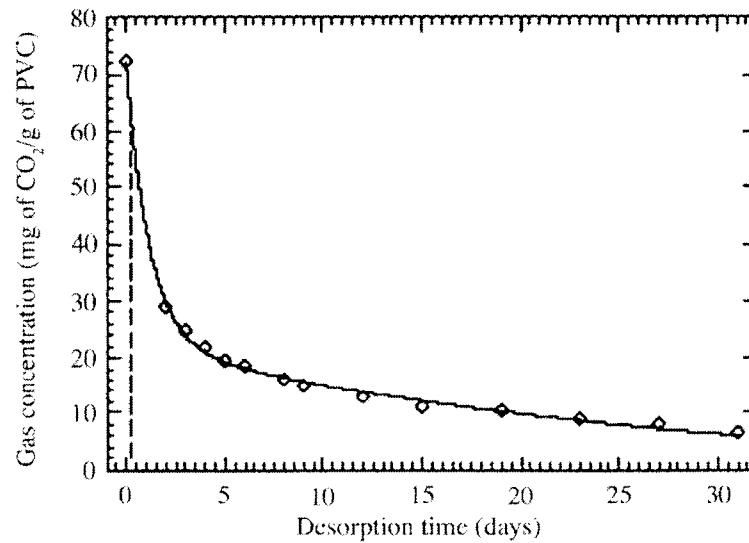


Figure 5. Plot of gas concentration versus desorption time for PVC pellets saturated with CO₂ at 5 MPa. The average diameter and thickness of the pellets was 4.06 mm and 3.05 mm respectively. Note that under 5 hours desorption time (dashed line) there is still at least 6% by weight of CO₂ dissolved in the PVC pellets

pressure vessel, there is still at least 6% by weight of blowing agent available to the polymer melt for foaming to occur.

Figure 6 shows the plot of relative density as a function of die temperature for the extruded PVC foams. The curve shows that as the die temperature decreases from 179.4 °C to 148.9 °C the relative density (ratio of density of foam to density of solid polymer) increases from 37% to 73%. Figure 7 shows the development of the through thickness cell morphology as the die temperature is decreasing. At die temperatures of 179.4 °C, 173.9 °C and 168.4 °C the resulting PVC foam strips have similar cell structure, with 20-80 μm cells on the surfaces and 100-500 μm in the core (Figures 7a, 7b and 7c). However, at a die temperature of 162.9 °C a dramatic change in microstructure can be observed. The cross-section of the PVC foam strip has a uniform distribution of microcellular bubbles of size 20 – 80 μm (Figure 7d). At a die temperature of 157.4 °C the same structure resulted with bubbles of size 20 – 80 μm (Figure 7e). However at 148.9 °C the foam structure had crushed plate-like structure closer to the surface and uniform 20 – 80 μm microcellular structure in the core (Figure 7f). Figure 8a, 8b and 8c show the magnified cores of the PVC foam strips extruded at 162.9 °C, 157.4 °C and 148.9 °C respectively. As seen there is a uniform distribution of microcells in all three samples. In terms of surface finish, the first three extrusion conditions shown in Table 1 resulted

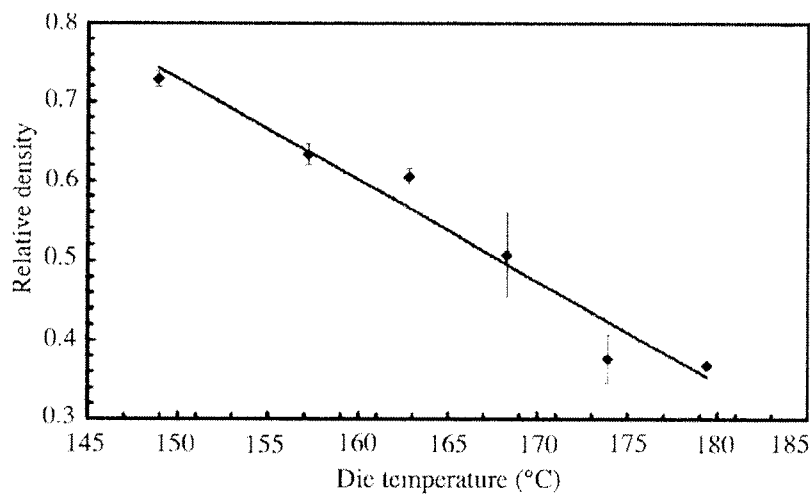


Figure 6. Plot of relative density of microcellular PVC foam as a function of temperature of the extruder die. The microcellular structure was achieved at die temperatures of 162.8 °C, 157.2 °C, and 148.9 °C

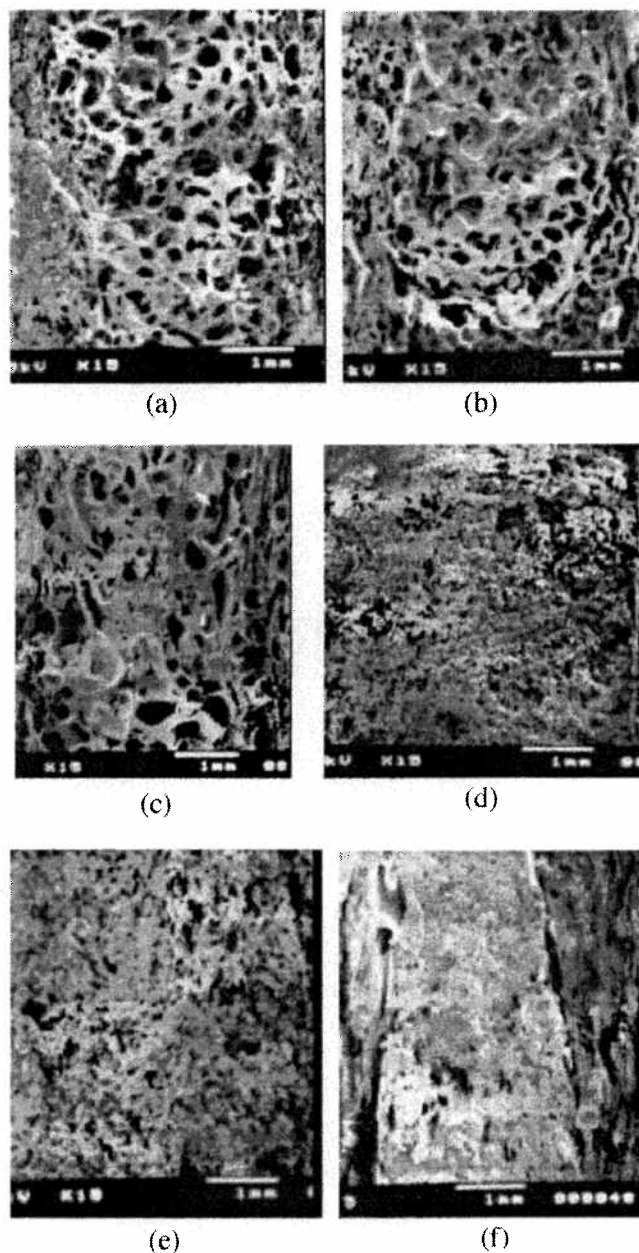


Figure 7. Scanning electron micrographs showing the development through thickness cell morphology for the extruded PVC strips as die temperature is decreasing. Die temperatures are (a) 179.4 °C, (b) 173.9 °C, (c) 168.3 °C, (d) 162.8 °C, (e) 157.2 °C, (f) 148.9 °C. As seen from figures (c) and (d), when the die temperature is decreased from 168.3 °C to 162.8 °C the bubble size in the core drops from 100-500 μm to 20-80 μm

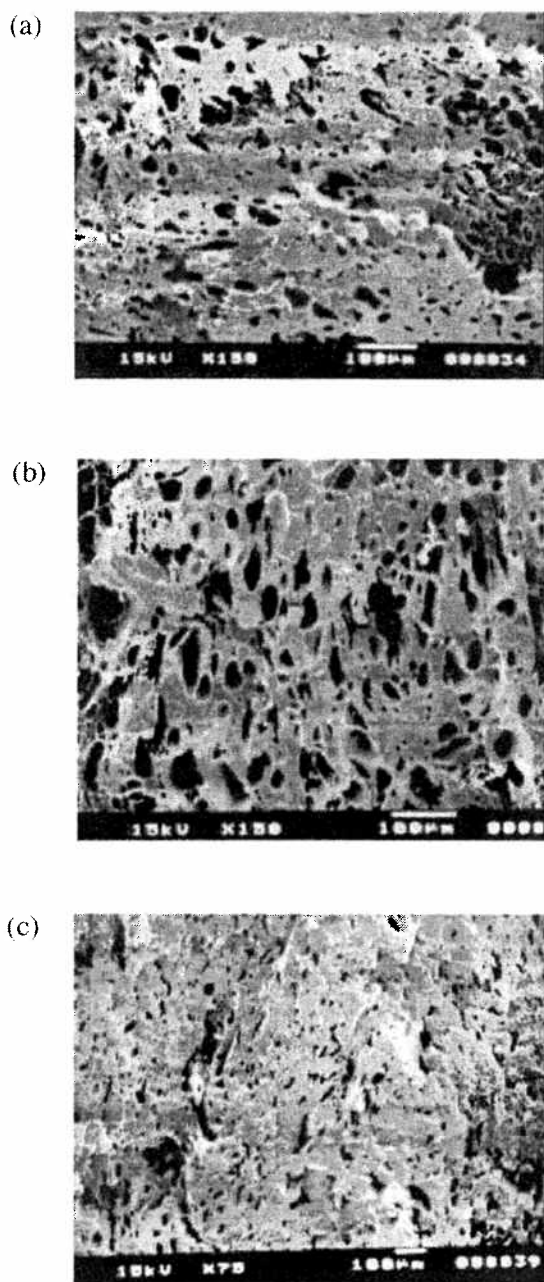


Figure 8. Higher magnification scanning electron micrographs of extruded microcellular PVC strips at die temperatures of (a) 162.8 °C, (b) 157.2 °C, (c) 148.9 °C. In all the three samples 20-80 μm bubbles are uniformly distributed in the core of the strip cross section

in foams which had a shiny surface but when the die temperature dropped to 162.9 °C and below the surfaces of the foam strips developed a dull look.

6. CONCLUSIONS

A novel microcellular extrusion process using pre-saturated pellets and solid-state nucleation has been developed. The process can be conducted on existing extrusion equipment without modifications. This is in contrast to supercritical process where typically gas at several tens of MPa is introduced in the melt and significant modifications to the standard extrusion equipment are required. Innovative gas delivery and cell nucleation strategies were proposed and demonstrated. To implement these strategies requires adding to the production line only a pressure vessel in which the polymer pellets are saturated with CO₂. Results show that as the die and barrel temperatures and thereby melt temperatures decrease, relative density of the PVC foam increases and the bubble size decreases. The extrusion was conducted at temperatures 20-30 °C below the normal temperature for PVC extrusion. Microcellular structure with cell size in the range of 20-80 μm was achieved in the PVC-CO₂ system.

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REFERENCES

1. Martini, J. Waldman F.A., and Suh N.P., "The Production and Analysis of Microcellular Foam", SPE Tech. Papers: XXVIII (1982), pp. 674-676
2. Kumar, V., "Microcellular Polymers: Novel Materials for the 21st Century", Progress in Rubber and Plastics Technology: Vol. 9 (1993), No. 1, pp. 54-70.
3. Zhang Z. and Handa Y. P., "An In-Situ Study of Plasticization of Polymers by

- High-Pressure Gases*", J. of Polymer Science: Part B: Polymer Physics: Vol. 36 (1998), p. 977-982.
4. Park C. B., PhD Thesis, M.I.T. Dept. Mech. Eng (1993)
 5. Park C. B., and Suh N. P., "*Extrusion of Microcellular Polymers Using a Rapid Pressure Drop Device*", Proceedings of SPE ANTEC 1993, New Orleans, p. 1818-1822.
 6. Park C. B. and Suh N. P., "*Extrusion of Microcellular Filament: A Case Study of Axiomatic Design*", Cellular Polymers, Vol 38, (1992), p. 69-91.
 7. Baldwin D. F., PhD Thesis, M.I.T. Dept. Mech. Eng (1994)
 8. Seibig B., Huang Q., and Paul D., "*Design of a Novel Extrusion System for Manufacturing Microcellular Polymer*", Cellular Polymers, Vol 19, No.2 (2000), p. 93-102.
 9. Shimbo M., Nishida K., Nishikawa S., Sueda T., and Eriguti M., "*Foam Extrusion Technology of Microcellular Plastics*", Porous, Cellular and Microcellular Materials, ASME, Vol. 82 (1998), p. 93-98.
 10. Huang Q., Klotzer R., Seibig B., and Paul D., "*Extrusion of microcellular polysulfone using chemical blowing agents*", J. of Applied Polymer Science, Vol. 69 (1998), p. 1753-1760.
 11. Holl M. R., Kumar V., Garbini J.L., and Murray W. R., "*Cell nucleation in solid-state polymeric foams: evidence of a triaxial tensile failure mechanism*", J. of Materials Science, Vol. 34 (1999), p. 637-644.
 12. Colton J.S., "*The nucleation of microcellular thermoplastic foam*", Massachusetts Institute of Technology, Cambridge (1985).
 13. Ramesh N.S., et al, "*An experimental study on the nucleation of microcellular foams in high impact polystyrene*", SPE ANTEC (1992), Detroit.
 14. Kweccder J.A., et al. "*The nucleation of microcellular polystyrene foam*", SPE Technical Papers, Vol. 37 (1991), p. 1398.
 15. Ramesh N. S., Rasmussen D. H., and Campbell G. A., "*The nucleation of microcellular foams in polystyrene containing low glass transition particles*", SPE ANTEC (1993), New Orleans.
 16. Dey S. K., Zhang C., Faridi N. and Xanthos M., "*Measurement of Gas Solubility in Thermoplastic Melts During Foam Extrusion*", Proceeding of the 55th SPE ANTEC, Vol. 2 (1997), No. 43, p. 1988-1990.
 17. Wong B., Zhang Z., and Handa Y. P., "*High-Precision Gravimetric Technique for Determining the Solubility and Diffusivity of Gases in Polymers*". J. of Polymer Science: Part B: Polymer Physics, Vol. 36 (1998), p. 2025-2032.

18. Kumar V., Weller J. E., Juntunen R. P., and Bezubic W. P., "*Impact Strength of high density microcellular poly vinyl chloride foams*", *J. of Vinyl & Additive Technology*, Vol. 6., No. 2 (June 2000), p. 93- 99.
19. ASTM D-792, "*Standard Test Methods for Density and Specific Gravity of Plastics by Displacement*", ASTM International (2002), West Conshohocken, PA.