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EXTRUSION OF CO₂/N-PENTANE BLENDS IN POLYSTYRENE

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Abstract - Blends of CO₂ and n-pentane were extruded in polystyrene. Rheological characterization of the system using the on-line slit die showed that n-pentane is a more effective plasticizer than CO₂ on an equivalent molar basis. This is consistent with previous findings indicating that larger gas molecules create more internal spacing at the molecular level. Solubility and homogeneity of the blends was also probed using ultrasonic monitoring. It was observed that degassing pressure in CO₂/n-pentane blends was CO₂-dependant only. This feature enables to increase the global gas loading in a foaming system in order to improve the expansion ratio without compromising on the gas pressure exiting the die, which may cause extrusion defects. Moreover, adding a lower diffusing agent such as n-pentane is expected to decrease the overall diffusion rate of the blend. Foam characterization results revealed that incorporating n-pentane to CO₂ lowered the density further while producing foam samples containing virtually no open-cells as compared to its pure CO₂ counterpart.

Introduction

The ban on CFCs and the pressure to develop alternatives has certainly generated lots of efforts in the development of blowing agents blends to meet required performance. Carbon dioxide has been since the very beginning targeted as an interesting candidate due to its inertness and low cost. However, because CO₂ is inherently more difficult to process than other gases due to its high volatility and low solubility, blending it with a minor low volatility component may provide a viable solution for the production of low-density commodity foams.

This paper reports on polystyrene foam formulations extruded with pure CO₂ and blends of CO₂ and n-pentane. The foaming process was investigated using measurements such as rheology and ultrasonic monitoring of the polymer-gas solutions. The results give valuable information on the plasticizing and solubility behavior of the mixtures. Samples were collected at the end of the extrusion line and analyzed in terms of density and morphology.

Experimental

Experiments were conducted on a 50 mm counter-rotating twin-screw extruder equipped with an instrumented slit die and a gear pump to control flow fluctuations. The standard extrusion setup and screw configuration was reported previously [1]. Crystal polystyrene (Fina PS 535, MI= 4.0, Atofina) was fed at a rate of 20 kg/hour and the blowing agents (n-pentane and carbon dioxide) were directly injected in the barrel.

Shear stress and degassing pressures were monitored at a constant temperature of 165°C. Foam samples were produced at a lower temperature (115-146°C). For all foam formulations, barrel temperatures were adjusted in order to maintain viscosity constant (i.e. pressure drop) at the die. A 4 mm strand die (1 mm die land) was used to produce the foam samples. Density of the foam samples was measured by water displacement. Morphology and cell size distributions were obtained from scanning electron micrographs. The nucleate cell density (β , the number of nucleation sites per volume unit of unfoamed polymer) was calculated according to Moulinié *et al.* [2]:

$$\beta = \frac{6 (\rho_{PS} / \rho_{foam} - 1)}{\pi d_{cell}^3} \quad (1)$$

Results and Discussion

Figure 1 depicts shear stress measured at 165°C for various mixtures of n-pentane and CO₂ in polystyrene. The global mixture concentration was increased while maintaining the proportion of each blowing agent constant. The curves for pure n-pentane and CO₂ are also represented. All concentrations are reported on a total weight basis. It can be seen that all the data scale up to a single master curve indicating that on a weight basis, CO₂ and n-pentane plasticize the polymer melt solution in a similar fashion. However, because CO₂ has a lower molecular weight than n-pentane, the actual quantity of gas molecules is actually higher for a given weight. When the same data is represented on an

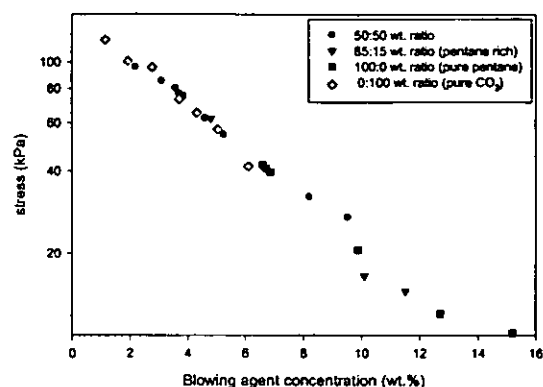


Figure 1 – Shear stress measured at 165°C

equivalent molar basis (not shown here), it can clearly be observed that n-pentane is a better plasticizer than CO₂. This feature is in agreement with previous work done on other gases [1]. Clearly, larger molecules are more effective in creating chain internal spacing at the molecular level.

Figure 2 depicts the degassing pressure measured during a typical pressure drop where a gear pump is accelerated to gradually decrease the melt pressure in the die. When the pressure is no longer sufficient to keep the solution homogeneous, a degassing pressure is recorded as previously reported elsewhere [3].

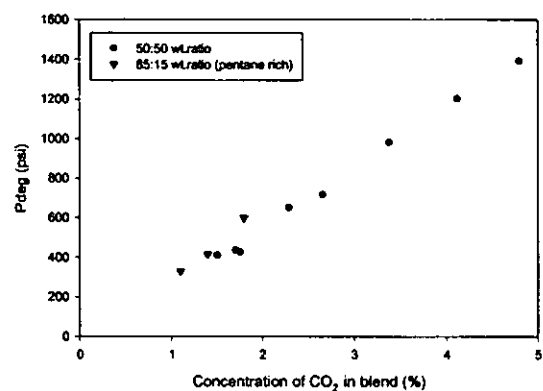


Figure 2 – Degassing pressure measured at 165°C

This pressure corresponds to the dynamic solubility conditions encountered in the extrusion line which accounts for temperature and pressure as well as shear flow. It is generally well known that solubility recorded by this method is slightly lower than that measured at equilibrium. The data reported in Figure 2 are represented as a function of proportion of CO₂ in the blends. It is clear that the pressure is function of the CO₂ fraction only. N-pentane is so much less volatile

than CO₂ that its presence does not impact vapor pressure. This feature is of interest for foam processing as it indicates that more blowing agent may be incorporated in a blend to increase blowing power without being detrimental to gas pressure. The later generally limits the extent to which density can be efficiently lowered.

Foam samples made from pure CO₂ were compared with foams made from a blend of CO₂ and n-pentane. Characterization results are summarized in Table 1. Generally, n-pentane containing formulations have a larger cell diameter than its CO₂ counterpart. Furthermore, higher blowing agent loadings may be globally added. Processing conditions would have not allowed keeping the equivalent concentration in pure CO₂ dissolved under pressure. One interesting feature is the drastic reduction of open cells. Because the global diffusion rate of the gas mixture is lowered, foam expansion is now possible without encountering cell wall rupture.

Table 1 – Foam characterization results

CO ₂ Wt. %	n-pent. Wt. %	Total Wt. %	Density (kg/m ³)	Diameter (μm)	β (cells/cm ³)	Open cell	T _{melt} (°C)
2.8	-	2.8	44.3	216	4.27 x 10 ⁶	70	146
3.2	-	3.2	50.5	174	7.07 x 10 ⁶	63	146
3.6	-	3.6	41.2	149	1.41 x 10 ⁷	76	146
3.87	-	3.9	34.7	69	1.70 x 10 ⁶	81	139
3.86	-	3.86	34.3	58	2.87 x 10 ⁶	77	138
4.32	-	4.32	30.1	72	1.71 x 10 ⁶	79	134
4.46	-	4.46	30.5	63	2.48 x 10 ⁶	76	132
4.74	-	4.74	29.6	36	1.40 x 10 ⁷	81	130
2.2	2.6	4.8	67.2	1183	1.67 x 10 ⁴	<5	131
2.6	2.8	5.4	33.7	310	1.90 x 10 ⁵	<5	121
2.8	3.0	5.8	38.0	546	3.09 x 10 ⁵	<5	123
3.5	3.7	7.2	34.6	196	7.26 x 10 ⁶	<5	115

Conclusion

Results presented in this paper show the numerous advantages of blending CO₂ with n-pentane in terms of viscosity, degassing pressure and final foam morphology.

Acknowledgements

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