EXTRUSION FOAMING OF TPVs USING WATER-FILLED POLYMERS

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Abstract

A novel, physical foaming agent, 'water filled polymer,' has been proven successful in producing low density foamed TPV profiles on standard single screw extruders. Extrusion processing parameters and physical properties (cell size, cell shape, tensile properties, specific gravity, compression load deflection, compression set, surface smoothness and water absorption) of TPV foamed profiles are reviewed. These results are compared to results from other types and forms of foaming agents used to produce foamed TPV profiles.

Introduction

Thermoplastic vulcanizates (TPVs) are engineered materials that have many of the properties associated with elastomers but can be processed similarly to thermoplastics. The most important family in this class is blends composed of EPDM/polypropylene (PP). The rubber/thermoplastic blend is cured under shear in an in-situ dynamic vulcanization process that creates a multiphase material consisting of crosslinked rubber particles in a thermoplastic matrix (1).

The foaming of TPVs using physical blowing agents (PBAs) and chemical blowing agents (CBAs) has been known for some time (2,3,4,5,6). CBAs are individual compounds or mixtures of compounds that liberate gas as a result of chemical reactions, including thermal decomposition, or as a result of chemical reactions of CBAs or interaction of CBAs with other components of the formulation. CBAs are typically solids. PBAs are compounds that liberate gases as a result of physical processes (evaporation, desorption) at elevated temperatures or reduced pressures. These usually are liquids but can include gases (7).

There are advantages and disadvantages of using a CBA or a PBA to create extruded TPV foam. The reasons for choosing a certain type of blowing agent depend on a number of factors. These include desired properties, application, cost and available processing equipment. Choosing a specific CBA or PBA, along with process settings, has a large impact on the cell structure, cell size and surface aesthetics of TPV foam. One benefit of using a CBA is the ability to foam TPVs using a typical thermoplastics extruder with an L/D ratio as low as 24:1. Typical extruders used for foaming TPVs using water as the blowing agent need an L/D of at least 30:1 and require sophisticated pumps to deliver water directly into the melt stream of the process. This also requires a specially designed screw to not only allow the injection of the water, but also the distribution of the water in the melt prior to the die exit. One benefit of water is the ability to foam to very low densities, below 0.10 g/cm^3 (8). Densities this low have been difficult to achieve with CBAs. Another advantage of using water as the blowing agent is the relative cost of water compared to CBAs and the environmental friendliness of water-produced foams. Recyclability of TPV foams made with water is much better than that of foams made with CBAs because the CBAs leave residuals from the decomposition reactions.

For the purposes of this paper, comparisons between a CBA, a PBA (water), and a new type of PBA, a water-filled polymer (WFP) in pellet form, are made. The water-filled polymer combines the advantages of the two types of blowing agents. TPV foam can be produced with WFP in the same type of extruder used with CBAs. Therefore, the capital investment necessary for a specially designed extruder or to retrofit an existing extruder is not necessary. Additionally, the TPV foam has characteristics similar to TPV foam created using direct water injection and the other advantages associated with foam made with this PBA as mentioned above.

Material, Equipment and Process

The base material used for creating all of the foamed TPVs was a fully cured TPV with a Shore A hardness of 68 that is specifically manufactured for foaming applications by Advanced Elastomer Systems, L.P. Water was injected at three rates to produce three different densities of foam. Typically, the higher the water concentration the lower the density of the extrudate foam. The three densities were created by injecting 171, 195 and 253 ml/hour of water. The water-filled polymer used in this study was a PP that contained 45 to 50% water by weight, Cellfoam™ H-2911A-50 from Polymer 5. However, it is believed that similar results could be seen using other base carriers such as EVA, SEBS, etc. with the same loading of water to produce TPV foam. Also, different loadings of water, 30 to 70% in a PP carrier, are available and have been proven successful in creating TPV foam, but results are not presented here. The various densities of

TPV foam were made with WFP added at 1.3%, 1.6% and 2.4% by weight to obtain a foam density near 0.55g/cm^3, 0.45 g/cm^3 and 0.35 g/cm^3, respectively. The CBA used for this study was a bicarbonate/citric acid blend in a polyethylene carrier known as Hydrocerol® BIH-40E from Clariant Masterbatches. This CBA is endothermic, that is, it absorbs heat during the extrusion process and has been found to create a more stable foaming process than exothermic CBAs. The TPV foams were created using the CBA at 5% and 8% loading by weight to form the comparable densities for this study.

The TPV foams were created on a 30:1 L/D, 2½ inch NRM extruder. TPV foams have been prepared on a 24:1 L/D extruder using WFP and CBA as the blowing agents. However, for direct process comparative purposes, samples were not prepared on this extruder. For the foam made with CBA, water was injected at 18D on a screw that had a mixing section the last 8D of the screw. The water-filled polymer and CBA were each added at the feed throat with the TPV pellets in a tumbled blend at the loadings mentioned above and in Table 1. The screw used with the CBA and the WFP had a special mixing section from 18 to 20D of the screw and two rows of pins at the end of the screw. After exiting the die, the TPV foam was cooled in a 40 feet long spray mist conveyor tank. The best process conditions known for each specific blowing agent were used to produce the foam samples. The screw speed was set slightly above the midpoint of the extruder range, which was 65 rpm. The tooling used was a 0.35 inch diameter bulb seal die with an inside diameter of 0.25 inch. Foamed TPV profiles were then made at the targeted densities of 0.35 g/cm^3, 0.45 g/cm^3 and 0.55 g/cm^3 for each type of blowing agent. The process settings used with each blowing agent are also listed in Table 1. A TPV foam could not be produced at a 0.35 g/cm^3 density with the chosen CBA and die design.

Testing Discussion and Results

The samples were then tested for various physical properties. Micro-tensile properties, such as ultimate tensile strength (UTS), ultimate elongation (UE) and modulus at 100% (M100), were tested using methods based upon ASTM D 1708-96. These results are shown in Table 2. The TPV foams created with a CBA had higher UTS and modulus values. This may be attributed to the higher loading of the CBA, which increases the overall plastic content of the foam. The UE results show a decrease in elongation with decreasing density regardless of the blowing agent used. The TPV foams created with WFP showed very similar properties to those of water and, in most cases, slightly better tensile properties. This again might be due to the addition of more plastic via the carrier.

Compression set (CS) and compression load deflection (CLD) testing were completed to compare sealing performance. For compression set testing the samples were compressed 40% at 100°C for 22 hours. The actual method used was based upon ISO 815-1972, ASTM D 395 and ASTM D 5056-85. The CS was calculated according to the following formula:

1) $CS = 100*(H_i-H_f)/(H_i-H_c)$

Where H_i is the initial sample height,
H_f is the final height measured after 22 hours, and
H_c is the height during compression.

The CLD method used for testing was based upon ASTM D 1667. The results for both CS and CLD are shown in Table 3. The CS results are very similar for the TPV foams created by each blowing agent at the various density ranges. The compression set was better for the foams created with water and WFP at a density of 0.44 g/cm^3 than the foam created by CBA. The CLD measures the resistance to sealing in such applications as a door seal. As expected, the CLD decreases consistently with foam density for each type of blowing agent, although the foam created by CBA had a higher CLD than that created by WFP or water. One factor that can influence the CLD values is the profile height, which is also reported in Table 3. Profile heights were higher for foams made with CBA than the other two blowing agents. This, along with the cell structure and increased plastic content contributed by the carrier, may have led to the higher CLD values for foams made with the CBA.

Water absorption results were compared on all samples under atmospheric conditions for 24 hours using methods based upon Chrysler MS-AIC87 and Ford ESB-MZD189A, and under vacuum conditions of 17 Kpa pressure for 5 minutes using methods based upon ASTM D 1056. The actual value for the water absorption is given as a percentage and was calculated using the following formula:

2) $Ab = 100*(W_f-W_i)/(W_i)$

Where Ab is the water absorption %
W_f is the final weight after the absorption, and
W_i is the initial weight prior to testing.

The results in Table 4 compare the water absorptions for the various foams under both test conditions. Under atmospheric conditions, the foam made with WFP absorbed the least amount of water, followed by foam made with water and the CBA. However, all three foams absorbed water below the automotive industry limit of 5%. It is easy to see that the blowing agent did not have a significant impact in the vacuum absorption percentages across the same densities. These results give some indication of the cell structure of the foamed samples. Lower densities had higher water absorption which indicates a more open, ruptured cell structure. Low water absorption indicates a foam structure that has mostly closed cells or less open cells that are connected. It appears that the cell structures, although different in size and shape, absorbed very similar amounts of water and have similar open cell volume between the TPV foams.

Cross-sections of the profile were taken for each foamed sample. Micrographs of these cross-sections were evaluated to compare the cell sizes and structures of the various foamed samples. Figures 1, 2 and 3 show micrographs of the different cell structures associated with each type of foam at a density near 0.44 g/cm^3. It is clear from Figures 1 and 2 that the cell size is very similar for the foam made with water injection and the foam made with WFP, although the cells seem to be more evenly dispersed in the foam created with the WFP. The cell size is much smaller for the foam made with a CBA, as shown in Figure 3, and the number of cells is much higher comparatively. Cell size variation and cell shape irregularities appear in all three TPV foam micrographs. These trends could also be seen at the other densities.

The surface roughness, Ra, was measured on the foamed samples to compare the surface quality. Ra results can be found in Table 5. A stylus profilometer was used to measure this Ra. The formula can be found below.

3) $Ra = \Sigma Yi/n$

Where Ra (roughness average) = the arithmetic average of roughness irregularities measured from a mean line within the sample length. Yi is the roughness irregularity and n is the number of irregularities.

The higher the Ra is the rougher the surface. Typically, the lower the foam density, the higher the Ra is. This is caused by the increase in cells and cell collapse near the foam surface. The surface of the foams created by WFP showed the highest Ra values and the foam created with a CBA had the lowest Ra values. However, all the TPV foam surfaces were well below an Ra value of 9, which has been established as the usable foam surface upper limit (9) for weatherseal applications.

Conclusions

It has been shown that a new physical blowing agent, a WFP, can be used to produce TPV foams with properties similar to those of TPV foams made with water or with a CBA. This technology combines the advantages of processing that a CBA has with the properties of a TPV foam made with water injection and its associated benefits of environmental friendliness and recyclability. It is hoped that this study can be expanded with further optimizations of the process and more comparisons to other foams can be made.

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Key Word/Phrase Index

TPV foams
Extruded foam
Water foam

Figure 1. 0.44 g/cm^3 density, TPV foamed bulb made with water on a 2½ inch 30:1 L/D extruder.

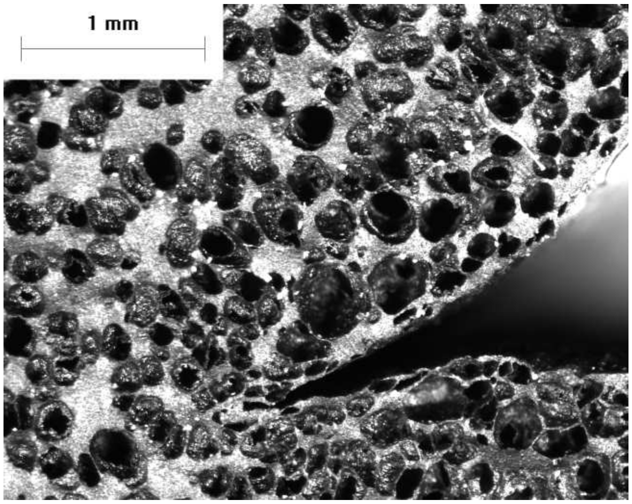

Figure 2. 0.43 g/cm^3 density, TPV foamed bulb made with WFP on a 2½ inch 30:1 L/D extruder.

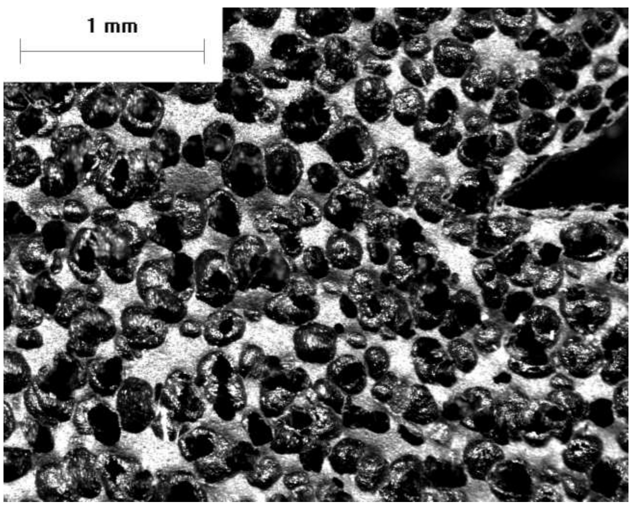

Figure 3. 0.43 g/cm^3 density, TPV foamed bulb made with CBA on a 2½ inch 30:1 L/D extruder.

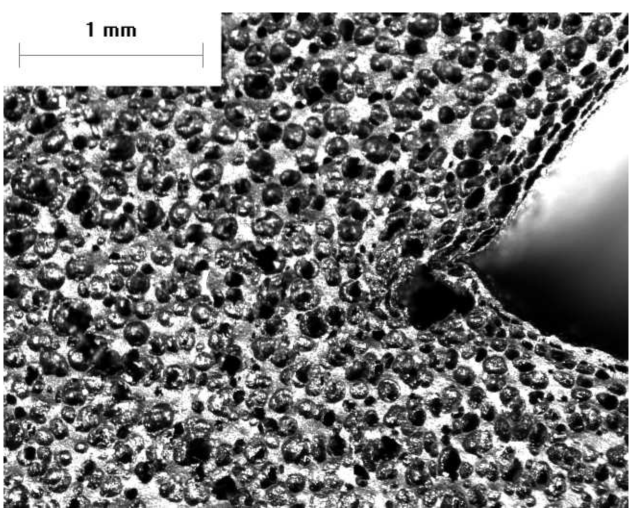

Table 1. Process settings for creating TPV foamed bulbs at various densities using identified foaming agent on a 2½ inch 30:1 L/D extruder.

Parameter	*Blowing Agent - Water*		
Extruder zones, feed to exit, °C	163 to 168	163 to 168	163 to 171
Die, °C	174	174	177
Melt temp, °C	178	178	181
Die pressure, MPa	4.97	4.90	4.55
Water flow rate, ml/hr	171	195	253
Foam density, g/cm^3	0.53	0.44	0.34

Parameter	*Blowing Agent – Water Filled Polymer*		
Extruder zones, feed to exit, °C	171 to 179	171 to 179	171 to 177
Die, °C	171	171	168
Melt temp, °C	184	184	182
Die pressure, MPa	5.72	5.59	5.79
Blowing agent, % wt.	1.3	1.6	2.4
Foam density, g/cm^3	0.55	0.43	0.35

Parameter	*Blowing Agent – Endothermic Chemical*	
Extruder zones, feed to exit, °C	204 to 171	202 to 166
Die, °C	177	171
Melt temp, °C	182	179
Die pressure, MPa	8.48	8.28
Blowing agent, % wt.	5.0	8.0
Foam density, g/cm^3	0.57	0.43

Table 2. Micro-tensile properties of TPV foamed bulbs using various foaming agents tested according to ASTM D 1708-96.

Blowing Agent	**Density, g/cm^3**	**UTS, MPa**	**UE, %**	**M100, MPa**
Water	0.53	2.09	248	1.32
WFP	0.55	2.35	246	1.61
CBA	0.57	2.58	270	1.90
Water	0.44	1.47	208	1.06
WFP	0.43	1.61	195	1.26
CBA	0.43	2.32	172	2.01
Water	0.34	1.30	160	1.16
WFP	0.35	1.62	207	1.21

Table 3. Compression set, compression load deflection and profile height data for TPV foamed bulbs.

Blowing Agent	Density, g/cm^3	40% Comp. Set*, 22 hr/100°C, %	40% Compress. Load Deflect.**, N/100 mm	Avg. Profile Height, mm
Water	0.53	34	50.8	11.90
WFP	0.55	38	44.5	11.64
CBA	0.57	37	75.1	13.56
Water	0.44	30	43.3	12.70
WFP	0.43	35	35.5	12.91
CBA	0.43	40	50.6	17.16
Water	0.34	34	25.8	12.22
WFP	0.35	33	25.7	14.38

*Compression set testing was based on ISO 815-1972, ASTM D 395 and ASTM D 5056-85.

**Compression load deflection was tested according to ASTM D 667.

Table 4. Water absorption percent at 1 bar and 17 Kpa for various TPV foamed bulbs.

Blowing Agent	Density, g/cm^3	Water Absorption, 1 bar *	Water Absorption, 17 Kpa **
Water	0.53	1.5	75.8
WFP	0.55	1.1	66.3
CBA	0.57	3.1	64.8
Water	0.44	2.4	109.7
WFP	0.43	1.8	113.7
CBA	0.43	4.0	114.4
Water	0.34	2.6	167.3
WFP	0.35	1.8	174.5

*Tested according to Chrysler MS-AIC87 and Ford ESB-MZD189A.

** Tested according to ASTM D 1056.

Table 5. Surface roughness of various TPV foamed bulbs.

Blowing Agent	Density, g/cm^3	Surface*, Ra
Water	0.53	3.9
WFP	0.55	5.7
CBA	0.57	4.0
Water	0.44	5.6
WFP	0.43	6.4
CBA	0.43	4.7
Water	0.34	6.6
WFP	0.35	7.5

*Tested using a stylus profilometer.