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BDX-613-869

FABRICATION OF OPEN CELL POLYETHYLENE FOAM

Technological Spinoff Report

March 1973

Bendix Corporation
Kansas City, Missouri

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FABRICATION OF OPEN CELL
POLYETHYLENE FOAM

BDX-613-869

March 1973

Project Leader:
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Department 814

Technological Spinoff Report

Contract Number AT(29-1)-813 USAEC

Technical Communications

**Kansas City
Division**

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FABRICATION OF OPEN CELL POLYETHYLENE FOAM

Prepared by D. J. Fossey

Open cell polyethylene foam was fabricated using sodium chloride as the pore-former. A solvent blending/leaching process was used which allowed easy control of cell size and open cell content.

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CONTENTS

Section	Page
SUMMARY	9
DISCUSSION	11
SCOPE AND PURPOSE	11
ACTIVITY	11
<u>Blending Process</u>	11
<u>Molding Process</u>	17
<u>Leaching and Drying Cycles</u>	18
<u>Test Results</u>	18
ACCOMPLISHMENTS	24
FUTURE WORK	24

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ILLUSTRATIONS

Figure		Page
1	Polyethylene Resin Density Versus Weight Percent NaCl for 70 percent Porosity Foam	14
2	Nomogram for Polyethylene Resin (0.96 g/cm^3)/NaCl Blends	14
3	Effect of Partial Mold Loading on Foam Density	15
4	Radiographs of Polyethylene Foam Test Specimens at Various Times During Leaching	19
5	Comparison of Foam Compressive Modulus and Base Polyethylene Resin Density	20
6	Typical SEM Pictures of Open Cell Polyethylene Foam at Magnifications of 100, 300, and 1000	23

TABLES

Number		
1	Polyethylene Resin Data	12
2	Polyethylene Foam Test Data	21

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SUMMARY

Open cell polyethylene foam with a desired cell structure was successfully fabricated from polyethylene resins whose densities ranged from 0.92 to 0.96 g/cm³, using the solvent blending process recommended by Los Alamos Scientific Laboratories. Some modifications had to be made to the process for the higher density resins. Sized sodium chloride crystals were used as the pore-former and were subsequently leached out with water after compression molding to a desired shape.

Foam specimens fabricated from the high density resins had a higher compressive modulus and lower creep value than the foam specimens fabricated from the low density polyethylene resins.

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DISCUSSION

SCOPE AND PURPOSE

This investigation was undertaken to mold open cell polyethylene foam of controlled density and cell size. A wide range of different types of polyethylene resins, varying primarily in density and melt index, were investigated.

Los Alamos Scientific Laboratories (LASL) developed a process for fabricating open cell polyethylene by the following method:

Solvent blending sodium chloride and a polyethylene resin in hot toluene at the required ratio,

Compression molding the resultant blend into the desired form, and

Leaching the sodium chloride with hot water, leaving the desired polyethylene foam structure.

In their investigations, LASL used toluene as the solvent for a low density polyethylene (LDPE) resin, Microthene FN 510. Bendix was requested to adapt the solvent blending-leaching process to high density polyethylene (HDPE) resins and to determine the physical properties of the resultant foams.

Porosity (free volume content) and cell size requirements were placed on the open cell polyethylene foam. Also, the foam was to have the highest compressive modulus and the lowest creep obtainable. The porosity and cell size are determined by the quantity and size of the pore-forming sodium chloride crystals. The compressive modulus and creep are a function of the polyethylene resin. It was theorized that foam fabricated from HDPE resin would have higher compressive modulus and lower creep values than foam produced from LDPE resins.

ACTIVITY

Blending Process

Sixteen polyethylene resins were chosen for evaluation. These resins, shown in Table 1, covered a wide range of melt indices and densities. Some

Table 1. Polyethylene Resin Data

Resin Identification	Density* (g/cm ³)	Melt Index**	Softening Temperature By TMA	
			Melted ^{††} (°F) (°C)	As Received ^{†††} (°F) (°C)
EHM6003***	0.959	0.3, 0.34	302 150	279 137
EHM6009***	0.959	0.9, 1.02	302 150	289 143
EMB6035***	0.962	3.5, 3.6	302 150	292 144
EMB6050***	0.959	5.0, 5.8	304 151	280 138
EMN6065***	0.961	6.5, 6.7	306 152	284 140
EMNTR885***	0.962	30.0, 30.4	304 151	280 138
BMB5065***	0.949	6.5, 7.61	298 148	275 135
MA219†	0.933	- -	237 114	248 120
FN510†	0.925	5.0	259 126	
MN703-06†	0.917	1.2	235 113	
MN711-20†	0.916	22.0	237 114	
MN715 †	0.933	8.0	239 115	
MN718 †	0.916	8.0	235 113	
MN752-19Y †	0.922	2.0	248 120	
MN754-18 †	0.921	12.0	250 121	
MT759-41Y †	0.948	5.0	293 145	
*Determined on sample melted for 1 hour at 350°F (177°C) **First value reported in manufacturer data sheet. Second value reported by Bendix Test Laboratory. ***Manufactured by Phillips Petroleum Company, Bartlesville, Oklahoma †Manufactured by U. S. Industrial Chemical Company, New York ††Melted into specimen from pellet or powder form. ††† Powder form. No as-received test possible.				

were supplied in powder form and others in the form of small pellets. Sodium chloride was the only pore-former investigated.

The structure and number of pores in the finished foam result from the shape and size of the sodium chloride crystals used and the amount of sodium chloride blended with the resin. To obtain a desired pore size of approximately 0.002 inches (0.05 mm) diameter, the sodium chloride crystals had to be screened or sized between a 270 U.S. standard mesh (0.053 mm opening) and a 400 U.S. standard mesh (0.037 mm opening) sieve.

Initially, common table salt had to be ground repeatedly to reduce its crystal size below a 270 mesh sieve opening. This very time-consuming procedure yielded few crystals of the proper size because a large percentage of the crystals passed through the 400 mesh sieve. A source of extra-fine powdered sodium chloride was located which eliminated the need to grind the material. The supplier, Morton Salt Company, Hutchinson, Kansas, sells the powdered salt in four different size ranges.

Screening was still required to obtain the desired size. An 18-inch-diameter (457 mm) SWECO and an 8-inch (203 mm) Rotap material sizer were used to screen the sodium chloride. The sodium chloride had to be screened four or five times to remove the 400 mesh material.

The porosity or open volume content of the foam can be controlled by the amount of salt blended with a given amount of resin. That is, for the foam to have a 70 percent porosity value, 70 percent-by-volume of the resin must be replaced with sodium chloride. Thus, the weight of sodium chloride required to obtain a given porosity will vary with the density of the base polyethylene resin. Figure 1 shows this relationship for a 70 percent porous foam along with the weight percent required. Figure 2 shows the relationships between the volume and weight percent loadings and the unleached and leached density of the foam. These relationships are given for a base polyethylene resin with a density of 0.960 g/cm^3 . Figure 2 can be used to determine the required sodium chloride loading for any given foam density. Also, it can be used to determine the final foam density for a desired porosity.

Another method of adjusting the porosity or final foam density is by changing the amount of material charged into a given mold volume. That is, for a given ratio of resin and sodium chloride, the maximum unleached and leached density is achieved by charging the theoretically required amount of material into a known volume and molding the material under enough pressure to remove all of the entrapped air. By charging only 80 percent of the theoretical amount of material, the unleached and leached density of the

Figure 1. Polyethylene Resin Density Versus Weight Percent NaCl for 70 Percent Porosity Foam

Figure 2. Nomogram for Polyethylene Resin (0.96 g/cm³)/NaCl Blends

foam will be reduced to 80 percent of the theoretical. A corresponding increase in porosity will also occur. Figure 3 shows a typical example of this method for adjusting the foam density.

Two additional factors have to be considered when obtaining a desired foam density; they are material flash and shrinkage. Material flash occurs during the molding cycle and becomes a factor when high pressures-3000 psi (21 MPa)-are required to remove the entrapped air. This flash is usually rich in resin content. This, in effect, increases the percent of sodium chloride and increases the porosity, thereby, lowering the final foam density. Shrinkage of the foam occurs during the leaching and drying cycles which reduces the volume of the foam with a resulting increase in its density. These two factors have opposite effects on the foam density and, in some cases, they cancel each other. However, the flashing is difficult to control and could cause a density gradient in the foam. The shrinkage has been found to depend on the polyethylene resin and may vary from 2 to 6 percent of the unleached volume. Figure 3 shows the actual densities (dashed line) of a series of foam specimens molded at various percents of theoretical loading. Also shown in Figure 3 is the corresponding theoretical leached

Figure 3. Effect of Partial Mold Loading on Foam Density

foam density (solid line), which is approximately 1 percent lower than the actual density. This is attributed to a 5 percent shrinkage in volume determined for that particular blend. A 5 percent volume reduction for a 0.20 g/cm^3 foam at constant weight would produce a 1.1 percent increase in density. It may also be noted in Figure 3 that this difference between the actual and theoretical densities becomes smaller when 100 percent loading is approached. This is due to the flashing previously mentioned, which results in a decrease in density.

Two blending methods, other than the solvent blending process recommended by LASL, were tried. One method was simply dry blending the powdered polyethylene resin with the sized sodium chloride in a twin shell blender. It was found that a homogeneous blend was difficult to obtain when using this dry blending method. The foam produced from dry blended polyethylene foam had a lower modulus and was more friable than the polyethylene foam that had been solvent blended using the same base polyethylene resin.

The second method, blending the sodium chloride with the polyethylene resins received in pellet form, was successfully accomplished on a rubber mill with heated rollers. This method produced a very rigid sheet of homogeneous material, which was difficult to mold into test specimens or parts.

The recommended solvent blending procedure was successfully repeated at Bendix. However, some changes were necessary to obtain an acceptable molding powder from the HDPE resin. The major change to improve the LASL process was the use of xylene as the solvent in place of toluene. This allowed the use of a higher dissolving temperature, 260°F (127°C) versus 190°F (88°C) which helped to break up the HDPE pellet.

In the solvent blending method, the sodium chloride and polyethylene resin are added to the solvent (at least 15 ml of solvent per 1 gram of resin), heated to within a few degrees of the boiling point of the solvent and maintained under constant agitation at that temperature long enough for the resin to be dissolved. The sodium chloride crystals are not dissolved into the solvent but are uniformly dispersed by constant agitation. The mixture is then allowed to slowly cool to room temperature, during which time the polyethylene precipitates out of solution and coats the sodium chloride crystals. The solids are then removed by filtration, followed by an acetone wash to remove the residual solvent. This acetone wash was another change to the recommended procedure. It allowed a shorter drying cycle and usually produced a molding powder free of hard lumps and very uniform in texture. After evaporating the residual acetone in an oven at 150°F (66°C), the blend is ready to be molded.

This solvent blending procedure produced molding compounds from both LDPE and HDPE that could be processed into acceptable foam. However, HDPE resins with low melt index values resulted in non-uniform foam. Additional changes to the procedure, such as longer dissolving times of up to 3 hours and the use of cymene as the solvent, did not improve the resultant foam. It is felt that if these resins, EHM 6003 and EHB 6009, had been supplied as powders instead of pellets, acceptable foam could have been produced.

The batch size for this investigation was approximately 100 grams of sodium chloride and polyethylene resin. However, for another project the batch size was successfully scaled up to 125 pounds (56.7 kg) with the use of a 50 gallon (0.225 m³) reactor. The major problem encountered with the scale-up was the filtering and drying of that large quantity of material. Several batches of 400 and 1200 grams have also been blended.

Molding Process

The sodium chloride-resin mixtures were compression molded into the desired shape. Test specimens, 1.25 inches (31.7 mm) in diameter by 0.25-inches (6.35 mm) thick, were chosen for this investigation. A predetermined amount of the molding powder was charged into the mold. The mold was then placed in a press and enough pressure ($\approx 5,000$ psi [34.5 MPa]) was applied to close the mold. While sufficient pressure was maintained to keep the mold closed, the mold was heated to 250°F (121°C) for the LDPE or 300°F (149°C) for the HDPE and held at that temperature for 30 minutes. The molded part was then cooled to below 120°F (49°C) under pressure before being removed from the press.

It was found that the molding powder could be cold pressed (compressed without applying heat) and subsequently molded using applied heat and lower pressure. This cold pressed technique resulted in a more efficient molding operation. A four-cavity mold was designed and fabricated in which four test specimens could be molded at one time. This reduced by approximately one-fourth the time required to make a set of four test specimens.

An extensive investigation of the effect of molding temperature was not undertaken. Molding temperature of a few degrees above the melting points of polyethylene resins were used. The molding pressure, as previously mentioned, does affect the properties of the resultant foam. When too low pressure is used, a larger test specimen is obtained, with a lower density and a higher porosity. When the required pressure is applied to compact the molding powder and remove the air, resin rich flashing causes the resultant foam to be low in density and high in porosity. This flashing

is hard to control, but its effects on density are offset by an increase in density due to shrinkage during the leaching and drying cycles as previously mentioned.

Leaching and Drying Cycles

After the test specimens were removed from the mold and stabilized to ambient conditions, the weights and dimensions of the specimens were recorded. The specimens were then placed in hot water (180° to 190°F (82° to 88°C)) for 3 to 5 days, and the water was changed periodically. The exact time required to remove all of the salt was not determined; however, the 3 to 5 days leaching period was chosen to ensure that all the salt had been removed, as determined by X-ray. Additional analytical testing will be performed at a later date to verify that the elemental requirements are met. After leaching, the specimens were dried in a vacuum oven at 180°F (82°C) overnight to remove the water. The specimens were then ready for evaluation.

The leaching time required to remove the salt is a function of the specimen size and the pressure used in the molding cycle. Lower pressures do not compress the material as much as higher pressures, thereby making it easier to remove the salt. In one case, a 0.5-inch-thick specimen was leached for 14 days before all the salt was removed.

The open cell structure of the foam is obtained during the leaching cycle. Calculations by LASL have shown that the osmotic pressure produced when the salt is ionized by the water is great enough to burst the thin cell walls. If this bursting action did not occur it would take months to remove the sodium chloride by diffusion through the polyethylene resin. Although the solubility of sodium chloride does not increase very much between room temperature and 190°F (88°C), it is believed that heating the water to 190°F aids in the removal of the sodium chloride because the polyethylene becomes weaker at the elevated temperature and bursts at a lower osmotic pressure.

Test Results

After drying and returning the specimens to ambient conditions, they were X-rayed to determine the presence of excess salt. Figure 4 shows a radiograph picture of two specimens after various leaching times. These specimens, shown full size, were 0.5-inch (12 mm) thick and were molded under extremely high pressures, approximately 20,000 psi (138 MPa). These pictures indicate the pattern in which the salt was removed. The radiograph pictures were also helpful in determining the overall homogeneity of the foam structure, as indicated in Figure 4. Specimen 40 appears to have resin-rich

Figure 4. Radiographs of Polyethylene Foam Test Specimens at Various Times During Leaching

high-density lines throughout. The radiograph of Specimen 41 was typical of the majority of the radiographs from the various resins and indicates that the foam has a uniform structure. The density, porosity, compressive modulus, and short-term creep data for the foam were then determined. The test data for these tests are given in Table 2. The compressive modulus shows a definite increase with increasing resin density (approximately 1500 psi [10.3 MPa] per 0.01 g/cm³), as shown in Figure 5.

The short term creep data did not correlate as well but did indicate that the foam from HDPE resins had a lower creep value than the foams from the LDPE.

Scanning electron microscope (SEM) pictures were obtained from a foam specimen made from each polyethylene resin. A typical SEM picture of the foam structure is shown in Figure 6 at 100X, 300X, and 1000X magnifications. There was no noticeable difference in the foam structure and pore size obtained from the various polyethylene foam specimens. However, the SEM pictures of the foam from EHM 6003 and EMB 6009 did indicate some non-homogeneity of the foam.

Figure 5. Comparison of Foam Compressive Modulus and Base Polyethylene Resin Density

Table 2. Polyethylene Foam Test Data

Group and Specimen Number	Blend Number	Polymer Identification	Polymer Density (g/cm ³)	Foam Density (g/cm ³)	Porosity (Percent)	Compression Modulus (psi) (MPa)	Creep Rate (μ -in/in/min)**
1-54	R2	FN-510	0.925	0.268	69.1	1588 (6.9)	71.7
55				0.273	70.7		
67				0.270	71.6	1948 (13.4)	39.8
2-83	6	EMB6050	0.959	0.261	72.6		
84				0.269	74.3	9093 (62.7)	15.8
85				0.273	71.8	7722 (53.2)	15.8
3-80	4	MN703-06	0.917	0.284	70.3		
81				0.284	69.0	1625 (11.2)	23.3
82				0.288	67.7	1909 (13.2)	31.2
4-76	3	EMN6065	0.961	0.286	70.6	8755 (60.4)	35.2
98				0.264	73.6		
99				0.263	73.6	9216 (63.5)	11.7
5-88	8	EMB6035	0.962	0.285	69.6	4761 (32.8)	43.1
110				0.278	71.6		
111				0.279	71.2	5423 (37.4)	38.9
6-112	114	MT759-41Y	0.948	0.258	74.1	4316 (29.8)	38.3
113				0.256	70.6		
114				0.259	73.9	4533 (31.3)	19.3
7-121	17	NA219	0.933	0.278	70.2	4096 (28.2)	23.4
122				0.280	69.3		
123				0.277	72.3	3953 (27.3)	15.7
8-126	18	MN715	0.933	0.274	70.8	3701 (25.5)	35.0
127				0.273	72.7	3767 (26.0)	27.1
128				0.269	70.7		
9-130	16	EMB6035	0.962	0.266	72.9	8476 (58.4)	7.7
131				0.265	71.2		
132				0.264	72.4	7748 (53.4)	7.7
10-134	22	BMB5065	0.949	0.284	70.3		
135				0.284	69.7	6610 (45.6)	30.9
136				0.284	69.8	6035 (41.6)	11.6
11-142	21	MN752-14Y	0.922	0.284	72.8		
143				0.283	70.3	2356 (16.2)	27.5
144				0.282	69.4	2406 (16.6)	23.4
12-146	19	MN718	0.916	0.292	68.0	1670 (11.5)	43.5
147				0.290	68.0		
148				0.290	68.4	1658 (11.4)	39.2
13-149	25	MN711-20	0.916	0.275	70.8		
151				0.279	69.9	1503 (10.4)	23.7
152				0.274	70.6	1323 (9.1)	39.2
14-169	34	MT759-41Y	0.948	0.272	72.7	4895 (33.8)	23.0
170				0.269	73.1	5033 (34.7)	7.7
171				0.271	71.9	5621 (38.8)	15.3
15-173	32	NA219	0.933	0.278	69.6	3920 (27.0)	15.5
174				0.276	69.4	4750 (32.8)	15.5
175				0.275	70.9	3864 (26.6)	23.2

Table 2 Continued. Polyethylene Foam Test Data

Group and Specimen Number	Blend Number	Polymer Identification	Polymer Density (g/cm ³)	Foam Density (g/cm ³)	Porosity (Percent)	Compression Modulus (psi) (MPa)	Creep Rate (μ-in/in/min)**
16-183	31	EHM6003	0.959	0.290	68.4	6880 (47.4)	7.6
184				0.286	71.5	6120 (42.2)	30.4
185*				0.284	70.5	2832 (19.5)	23.0
17-187	35	EMNTR885	0.962	0.277	70.5	9746 (67.2)	22.9
188				0.270	72.9	9810 (67.6)	23.0
189				0.267	72.6	10280 (70.9)	15.3
18-176	30	MN754-18	0.921	0.279	70.5	2045 (14.1)	38.9
177				0.270	72.3	1805 (12.4)	54.3
178				0.278	69.7	2130 (14.7)	23.3
19-194*	43	EHR6009	0.959	0.303	71.7	2144 (14.8)	64.0
197*				0.290	70.9	2501 (17.9)	52.6
198*				0.277	72.8	1895 (13.1)	68.4

*Specimen had numerous soft spots close to surface
**1 μ-in/in/min = 1 μ-mm/mm/min

GROUP NO. 4
SPECIMEN 98
RESIN, EMN 6065
DENSITY, 0.264 g/cm³
POROSITY, 73.6 PERCENT

Figure 6. Typical SEM Pictures of Open Cell Polyethylene Foam at Magnifications of 100, 300, and 1000

ACCOMPLISHMENTS

Solvent blends of NaCl and polyethylene resins which vary in density from 0.916 to 0.962 g/cm³ were processed into open cell polyethylene foam. High density, low melt index resins were the most difficult to make into acceptable specimens with uniform density, and still require some investigation.

It was determined that the foam compression modulus increases with increasing density of the base polyethylene resin at a rate of approximately 1500 psi (10.3 MPa) per 0.01 g/cm³. The short term creep rate appeared to decrease with increasing base resin density.

The pore structure of all the acceptable solvent blends was essentially the same and is controlled by the size and shape of the NaCl crystals. Molding pressure will influence the density and porosity of the polyethylene foam specimens.

FUTURE WORK

Additional investigation of solvent blending the high density, low melt index resins will be conducted. Experiments to determine effects of the variables of the molding, leaching, and drying processes on the physical properties of the foam will be conducted.