Rheological Behavior of a Microcellular, Oil-Extended Ethylene–Propylene–Diene Rubber Compound: Effects of the Blowing Agent, Curing Agent, and Conductive Carbon Black Filler

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ABSTRACT: The rheological behavior of microcellular, oil-extended ethylene–propylene–diene rubber (EPDM) compounds was studied in extrusions containing a blowing agent. The cell morphology development and rheological properties were studied for unfilled and conductive carbon black (Vulcan XC72, Cabot Corp., Ltd., Alpharetta, GA) filled compounds with variations of the blowing agent, extrusion temperature, and shear rate. The apparent shear stress, apparent viscosity, die swell (%), and total extrusion pressure of the Vulcan XC72 filled, oil-extended EPDM compounds were determined with a Monsanto processability tester (St. Louis, MO). The effects of the curing agent and blowing agent on the rheological properties of the compounds were also studied. A significant reduction in the stress and viscosity with the blowing agent was observed in the compound in the presence of the curing agent in comparison with those without the curing agent. The viscosity reduction factor was found to be dependent on the blowing agent loading, shear rate, and temperature. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 1022–1030, 2008

Key words: blowing agents; foam extrusion; microstructure; rheology; vulcanization

INTRODUCTION

The rheological characterization of polymeric compounds is one of the important steps in polymer processing. The extrusion of polymeric materials, especially those containing a blowing agent, is an important industrial processing operation.[1,2] Studies of bubble morphology development and apparent rheological properties of thermoplastics in foam extrusion processes and the effects of processing variables on the quality of foam produced have been reported by several authors.[3–5] They concluded that the bubble size and void fraction increase with temperature, reducing the extrusion rates and die length/diameter ratio under constant extrusion rate conditions. An important aspect of foam extrusion is the mechanics of bubble growth in the polymer matrix. Numerical and experimental studies of bubble growth during the microcellular foaming process and the influence of the temperature, saturation pressure, molecular weight, and nature of physical blowing agents have been reported.[6] Shear effects on thermoplastic foam nucleation have been studied on a twin-screw foam extruder,[7] and it has been concluded that both the shear rate and viscosity contribute to foam nucleation in the continuous foam extrusion process. A phenomenological model of the flow in an extrusion die[8] describes the measurements of pressure profiles and visualization of the phase change in the extrusion dies with respect to the rheological aspects of foam extrusion. Experimental studies of bubble growth in elastomers[9–11] have led to the conclusion that the number of bubbles that form depends on the temperature, supersaturation pressure, solubility, and diffusion coefficients for the gas and surface tension of the polymer. Bubble growth and physical properties of thermoplastic elastomers have also been reported with respect to extrusion foaming.[12] An extensive study of the rheological properties of homopolymers in the molten state, polymer blends, and filled polymers has been carried out.[13] Rheological properties of mixtures of a molten polymer and a blowing agent have been reported,[14,15] and it has been found that the viscosity reduction factor (VRF) is blowing-agent-dependent. Studies of effects of the pressure drop rate on cell nucleation in a continuous process of microcellular polymers have also been carried out,[16,17] and they indicate that the gas solubility increases as the processing pressure increases; this results in a larger number of cells being nucleated. The rheology of molten polystyrene with dissolved supercritical and nearly critical gases has been reported.[18] When a blowing agent is added to a polymer composite to

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TABLE I
Formulations of Unfilled and Vulcan XC72 Filled Compounds

Ingredient (phr)	G_1	G_2	G_3	G_4	EB_1	EB_2	EB_3	EB_4	EB_5	EB_6	EB_7	EB_8
Keltan 7341A	120	120	120	120	120	120	120	120	120	120	120	120
ZnO	5	5	5	5	5	5	5	5	5	5	5	5
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Vulcan XC72	0	0	0	0	40	40	40	40	40	40	40	40
Paraffinic oil	0	0	0	0	4	4	4	4	4	4	4	4
Dicumyl peroxide (98% pure)	0	1	0	1	0	1	0	0	0	1	1	1
ADC-21	0	0	4	4	0	0	2	4	6	2	4	6

produce a microcellular polymer, several phenomena can influence the extrusion behavior of the mixture, and the blowing agent will also affect the rheological behavior of the compound.[19] The effects of blowing agents and several fillers on microcellular ethylene–octene copolymer composites have also been studied.[20–22] Those authors explained changes in the cell morphology, number of cells, apparent shear stress, die swell, and changes of VRF with the shear rate, blowing agent loading, curing agent, and temperature.

The morphology, physicomechanical properties, compressive deformation, and energy absorption characteristics of closed-cell microcellular ethylene–propylene–diene rubber (EPDM; Keltan 7341A, DSM Elastomers, Singapore) filled with excess conductive carbon black (Vulcan XC72, Cabot Corp., Ltd., Alpharetta, GA) have been reported,[23,24] and those reports describe the effects of the filler and blowing agent on the aforementioned properties. Recently, the dynamic mechanical properties of conductive carbon black reinforced, closed-cell, microcellular, oil-extended EPDM vulcanizates have been studied.[25]

The objective of this work was to study the rheological properties of a closed-cell microcellular rubber compound based on conjugated, long-chain-branch-grade, oil-extended EPDM (Keltan 7341 A) with the variation of the blowing agent loading in both unfilled and conductive carbon black filled systems. The effect of temperature in the extrusion was also studied. The morphological studies of the extrudate were performed with scanning electron microscopy (SEM) to characterize the cellular structure of the extrudates.

EXPERIMENTAL

Materials

An oil-extended EPDM [Keltan 7341A (a new conjugated long chain branching (CJCB) grade rubber) with 7.5 wt % ethylene–norbornene, 20 phr oil, and a Mooney viscosity of 53 (at 150°C)] was used. Vulcan XC72 (excess conductive carbon black) was used as the filler. The curative used was dicumyl peroxide with a purity of 98%; it was manufactured by Aldrich Chemical Co. (St. Louis, MO). The blowing agent used was azodicarbonamide (ADC-21) manufactured by High Polymer Lab (New Delhi, India), and the paraffinic oil used was Sunpar oil supplied by Sun Oil Co. Pvt., Ltd. (Kolkata, West Bengal, India).

Compounding and sample preparation

The rubber was compounded with the ingredients according to the formulations of the mixes given in Table I, and the blowing agent was loaded at the end. Compounding was done in a laboratory-size two-roll mixing mill at room temperature according to ASTM D 3182. The mixed items were considered samples for testing.

Measurement of the rheological properties

Rheological properties of the samples were studied with the help of a Monsanto no. 83077 processability tester (St. Louis, MO) at temperatures of 120, 130, and 140°C. The capillary had a 2-mm diameter with a length-to-diameter ratio equal to 16:1. The test samples were charged into the barrel and preheated for 3 min for uniform temperature distribution and were extruded with different piston rates of 0.02, 0.05, 0.08, 0.10, and 0.15 in./min with time. The piston rates correspond to the apparent shear rates at the wall ($\dot{\gamma}_{wa}$) of 3.074, 7.685, 12.296, 15.37, and 23.055 s^{-1}, respectively.

$\dot{\gamma}_{wa}$ and the apparent shear stress at the wall (τ_{wa}) were calculated with the knowledge of the barrel diameter, piston speed, length, and diameter of the capillary. These are expressed as follows:

$$\tau_{wa} = \frac{d\Delta P}{4l} \tag{1}$$

$$\dot{\gamma}_{wa} = \frac{32Q}{\pi d^3} \tag{2}$$

where d is the capillary diameter, l is the capillary length, ΔP is the pressure drop over the capillary length (measured as a pressure gauge reading at the inlet of the capillary), and Q is the volumetric flow rate of the compounded material. Q is calculated with the following expression:

$$Q = S\pi R_B^2 \tag{3}$$

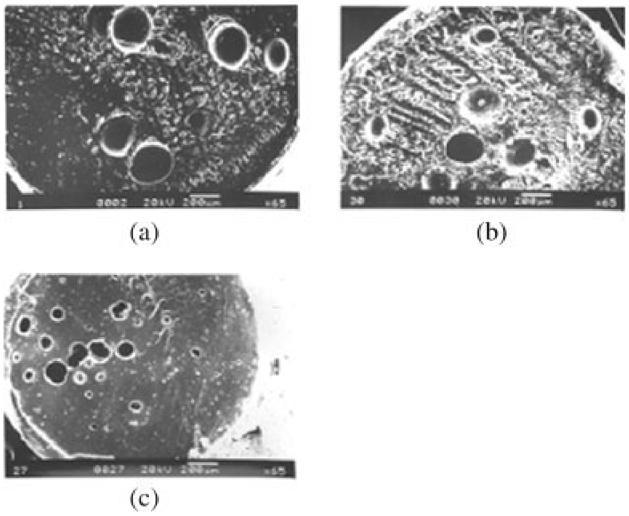
(a) (b) (c)

Figure 1 SEM photomicrographs of unfilled extrudates at 65× magnification: (a) G_3 (3.074 s^{-1} at 130°C), (b) G_3 (23.055 s^{-1} at 130°C), and (c) G_4 (23.055 s^{-1} at 130°C).

where S is the linear piston rate and R_B is the barrel radius [i.e., 9.53 mm (constant for the whole experiment)].

The apparent shear viscosity (η_a) is given by the following relation:

$$\eta_a = \frac{\tau_{wa}}{\dot{\gamma}_{wa}} \tag{4}$$

The data for the running die swell (die swell at the exit of the die as soon as the extrudate comes out) were directly recorded with a built-in laser scanning system.

SEM

SEM photomicrographs of razor-cut surfaces of the microcellular extrudates were obtained with a JEOL JSM 5800 scanning electron microscope (Tokyo, Japan). The samples were gold-coated with a vacuum gold-sputter machine for the SEM studies. The average cell size and number of cells were calculated with quantitative image analysis by consideration of the scale of the SEM microphotographs.

RESULTS AND DISCUSSION

Morphologies of the unfilled and Vulcan XC72 filled extrudates

SEM photomicrographs of unfilled and Vulcan XC72 filled, microcellular extrudates are shown in Figures 1 and 2, respectively. The figures show the effects of the blowing agent, curing agent, shear rate, and temperature on the cell size and number of cells of the extrudates. The gas bubbles that form inside the capillary depend on the pressure, temperature, and concentration of the blowing agent. Table II shows the average cell size of the extrudates with variations of the shear rate, temperature, and blowing agent loading. In the case of unfilled compounds, the cells of the extrudates are more or less spherical in nature. Figure 1(a,b) shows that in the absence of the curing agent, an increase in the shear rate increases the number of cells and decreases cell size. In the presence of the curing agent, the number of cells also increases, as shown in Figure 1(c), with a slight decrease in cell size.

In the case of a Vulcan XC72 filled compound, cells are nonspherical in nature. In the absence of the curing agent, an increase in the blowing agent loading increases the average cell size and number of cells, regardless of the shear rate, at 130°C in Figure 2(a,b) and at 140°C in Figure 2(c,d) in the case of filled extrudates. From Figure 2(a,c) and Figure 2(b,d), it is found that with an increase in the temperature, the number of cells increases at any shear rate. In the presence of the curing agent, the cell size decreases, and cells are more uniformly distributed in the rubber matrix, as shown in Figure 2(e–g). The addition of the curing agent increases the viscosity of the compounds, so the dissolved gases are unable to escape from the mass. It is also observed that in the presence of the curing agent, an increase in the shear rate increases the number of cells and decreases the average cell size of the Vulcan XC72 filled extrudates, as shown in Figure 2(h,i) at 140°C.

Die swell

Effects of the blowing agent and curing agent in the unfilled and Vulcan XC72 filled compounds at different temperatures

The die swell of unfilled compounds at 120, 130, and 140°C after a 3-min extrusion time is shown in Figure 3. The figure shows that for the unfilled compound, the die swell increases with the shear rate and with decreasing temperature. On the addition of the blowing agent and curing agent, the die swell increases with the shear rate and temperature. The rate of increase is more pronounced at a higher shear rate.

The die swells of Vulcan XC72 filled compounds at different temperatures are shown in Figure 4 in semilog plots. Both the curing agent and blowing agent enhance the die swell at all temperatures in comparison with the base compound. In the presence of the curing agent, the rate of increase in the die swell for the compounds with the blowing agent is higher. However, at a higher shear rate, the rate of increase in the die swell decreases. With the increase in the shear rate, the die swell increases at all temperatures. At a higher shear rate, the rate of

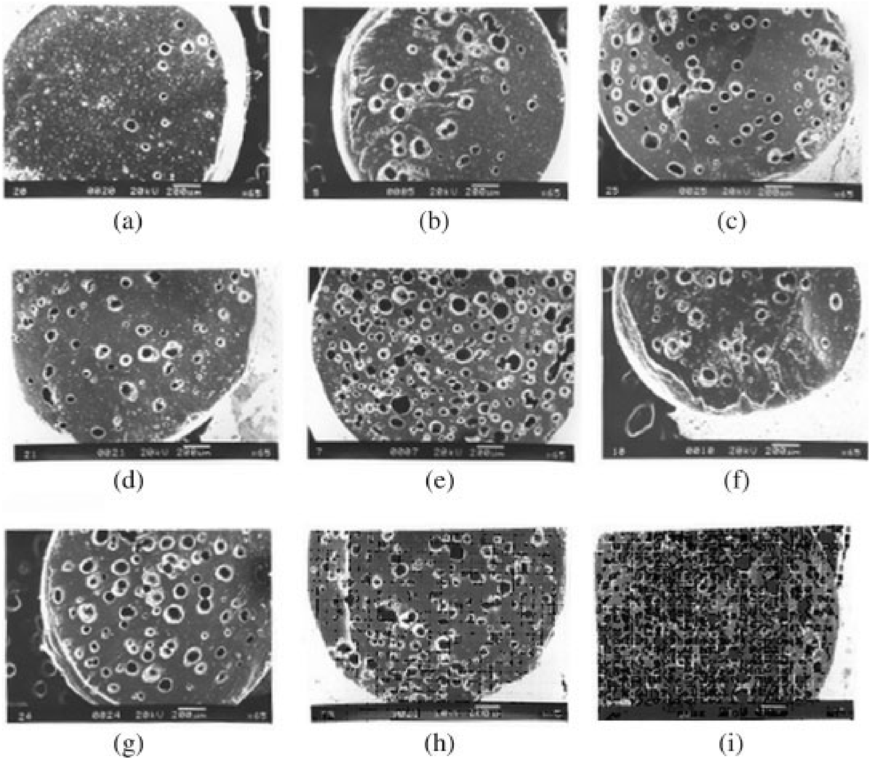

Figure 2 SEM microphotographs of Vulcan XC72 reinforced extrudates at 65× magnification: (a) EB_4 (12.296 s^{-1} at 130°C), (b) EB_5 (12.296 s^{-1} at 130°C), (c) EB_4 (12.296 s^{-1} at 140°C), (d) EB_5 (12.296 s^{-1} at 140°C), (e) EB_6 (12.296 s^{-1} at 140°C), (f) EB_7 (12.296 s^{-1} at 140°C), (g) EB_8 (12.296 s^{-1} at 140°C), (h) EB_7 (7.685 s^{-1} at 140°C), and (i) EB_7 (23.055 s^{-1} at 140°C).

increase in the die swell is less in the presence of both the curing agent and blowing agent. In the presence of the curing agent, at a higher shear rate, skin breakdown occurs, and this leads to the release of decomposed gas from the compound; thus, die swell decreases.[19] The die swell of Vulcan XC72 filled, microcellular compounds with the variation of the blowing agent loading and temperature in the absence of the curing agent is shown in Figure 5. The die swell increases with both the blowing agent loading and temperature. Figure 6 demonstrates the effect of the blowing agent loading in the presence of the curing agent at all temperatures. The relative difference in the die swell of the compounds with both the curing agent and blowing agent is remarkable at all shear rates. However, the trend is the same at all temperatures.

Total extrusion pressure

The total extrusion pressures of Vulcan XC72 filled, microcellular, uncured and cured compounds are

TABLE II
Average Cell Size of the Extrudates: Variation of the Shear Rate, Temperature, and Blowing Agent Loading

Mix name	Shear rate (s^{-1})	Temperature (°C)	Average cell size (μm)
G_3	3.074	130	267
G_3	23.055	130	200
G_4	23.055	130	120
EB_4	12.296	130	62
EB_5	12.296	130	80
EB_4	12.296	140	87
EB_5	12.296	140	113
EB_6	12.296	140	54
EB_7	12.296	140	76
EB_8	12.296	140	88
EB_7	7.685	140	93
EB_7	23.055	140	60

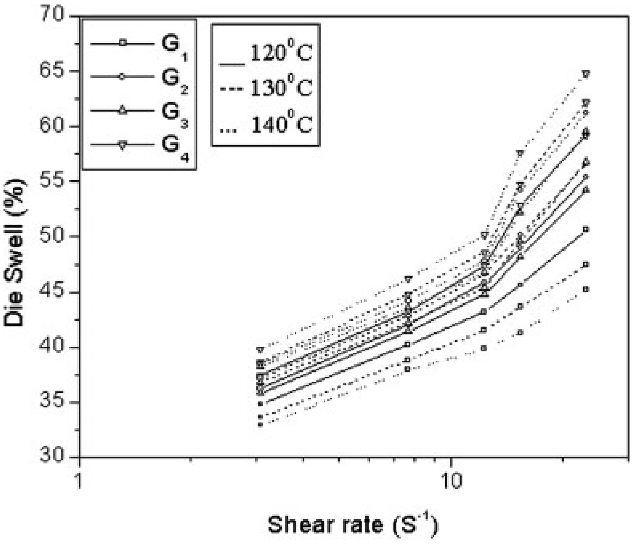

Figure 3 Plots of the die swell (%) versus the shear rate of unfilled, microcellular, oil-extended EPDM vulcanizates at different temperatures, showing the effects of the curing agent, blowing agent, and both: (□) gum, (○) gum with curing agent, (△) gum with 4 phr blowing agent, and (▽) gum with both curing and blowing agents.

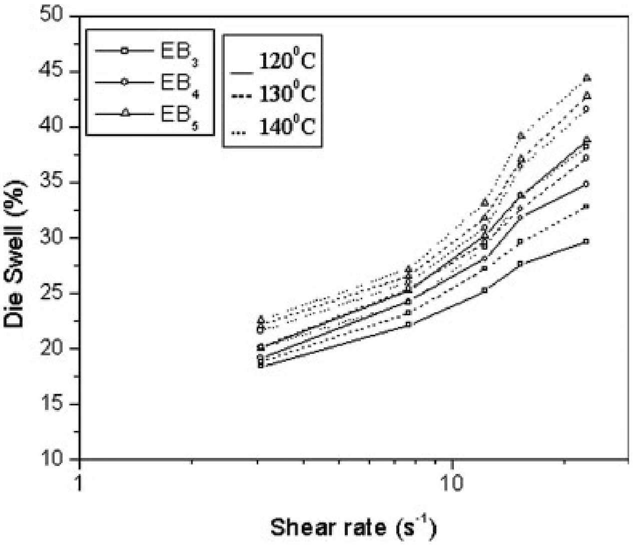

Figure 5 Plots of the die swell (%) versus the shear rate of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates without the curing agent at different temperatures, showing the effects of the blowing agent: (□) 2, (○) 4, and (△) 6 phr.

plotted as a function of the blowing agent loading at various shear rates and temperatures in Figures 7 and 8, respectively. The total pressure decreases with an increase in the blowing agent loading at all shear rates and temperatures. The incorporation of the blowing agent causes a sharp decrease in the pressure followed by a slow decrease with an increase in the blowing agent loading. The initial sharp pressure drop is due to decomposition of the blowing agent. However, with an increase in the blowing agent loading, the change in the pressure drop is not very effective. In the presence of both the curing agent and blowing agent, the initial pressure drop (Fig. 8) is more due to the effective entrapment of decomposed gas.[19]

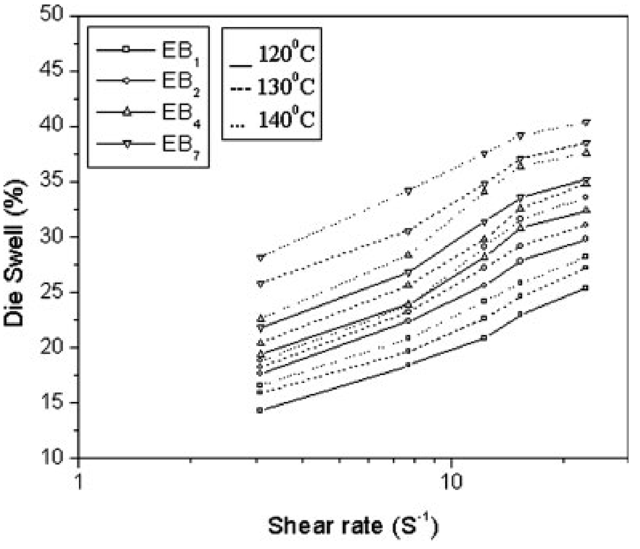

Figure 4 Plots of the die swell (%) versus the shear rate of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates at different temperatures, showing the effects of the curing agent, blowing agent, and both: (□) filled vulcanizate, (○) vulcanizate with curing agent, (△) vulcanizate with 4 phr blowing agent, and (▽) vulcanizate with both curing and blowing agents.

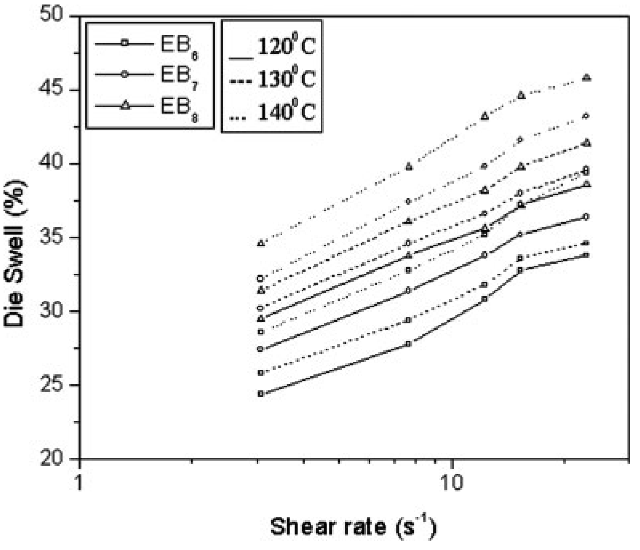

Figure 6 Plots of the die swell (%) versus the shear rate of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates with the curing agent at different temperatures, showing the effects of the blowing agent: (□) 2, (○) 4, and (△) 6 phr.

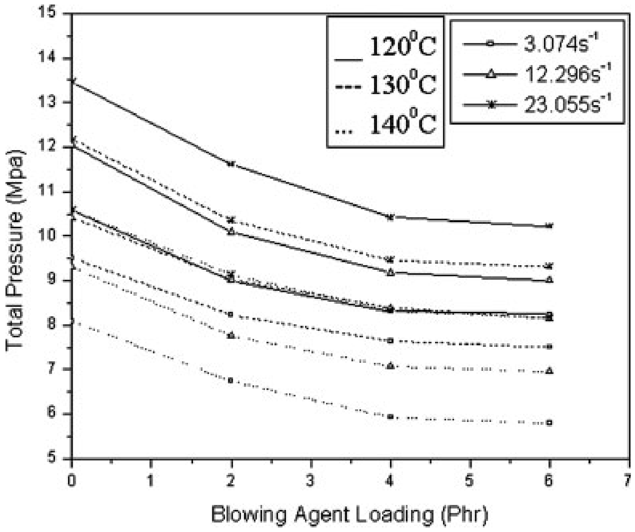

Figure 7 Plots of the total extrusion pressure of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates as a function of the blowing agent loading at various shear rates (without the curing agent) at different temperatures.

Stress behavior

Effects of the blowing agent and curing agent of the compounds at different temperatures

Logarithmic plots of the apparent shear stress at the wall versus the apparent shear rate are shown in Figure 9, which illustrates the effect of the curing agent and blowing agent on unfilled compounds at different temperatures. Figure 9 shows the gradual increase in the shear stress with the increase in the shear rate. In the absence of the curing agent and blowing agent, the unfilled compound follows the power law, regardless of the change in temperature. The compounds having the blowing agent, curing agent, or both show a nonlinear relationship between the shear stress and shear rate. The presence of only the blowing agent restricts the stress rise with the shear rate, regardless of the temperature. At a higher shear rate, retardation in the stress rise occurs because of the decomposition of the blowing agent. The decomposition of the blowing agent produces gas bubbles, which dissolve inside the mass of the compound, or forms microbubbles and restricts the stress rise. However, in the presence of both the curing agent and blowing agent, the decomposed gas is unable to be released from the compound, and this restricts the increase in the shear stress with the shear rate in comparison with the compound containing only the curing agent.

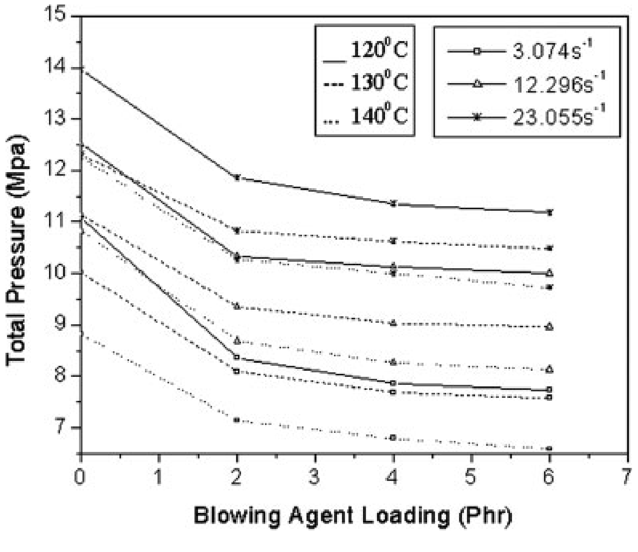

Figure 8 Plots of the total extrusion pressure of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates as a function of the blowing agent loading at various shear rates (with the curing agent) at different temperatures.

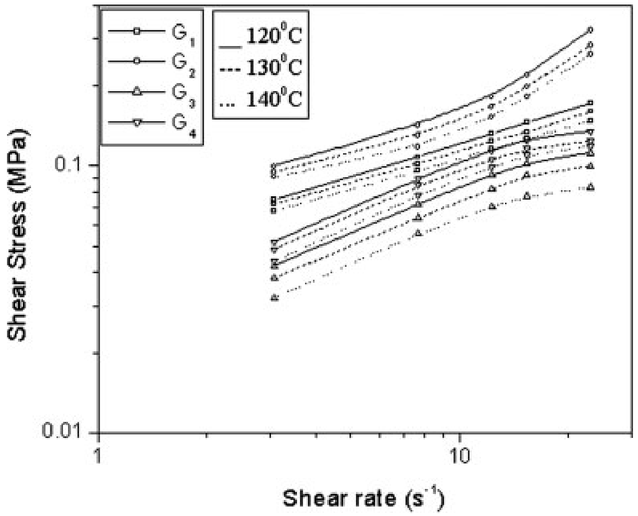

Figure 9 Plots of the apparent shear stress versus the shear rate of unfilled, microcellular, oil-extended EPDM vulcanizates at different temperatures, showing the effects of the curing agent, blowing agent, and both: (□) gum, (○) gum with curing agent, (△) gum with 4 phr blowing agent, and (▽) gum with both curing and blowing agents.

Figure 10 shows logarithmic plots of the apparent shear stress at the wall versus the apparent shear rate, illustrating the effects of the curing agent and blowing agent on Vulcan XC72 filled, microcellular compounds at different temperatures. Figure 10 shows the gradual increase in the shear stress with an increase in the shear rate. In the absence of the curing agent and blowing agent, the compound follows the power law, regardless of the temperature. In the presence of the blowing agent, curing agent, or both, the microcellular EPDM compound shows a nonlinear relationship like the unfilled compound. In the presence of the blowing agent at a higher shear rate, retardation of the stress rise occurs because of

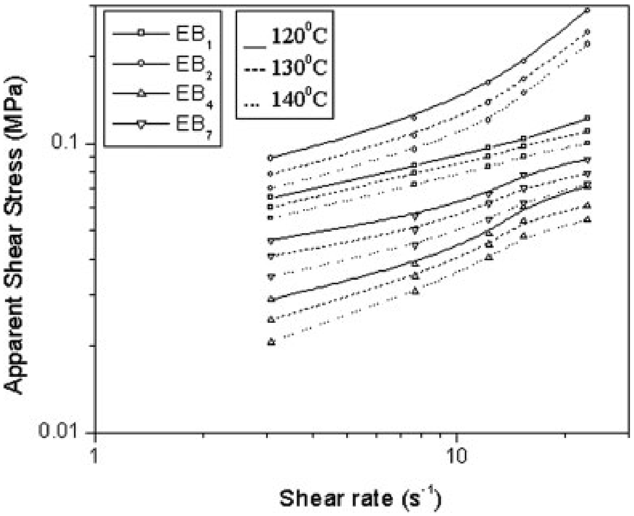

Figure 10 Plots of the apparent shear stress versus the shear rate of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates at different temperatures, showing the effects of the curing agent, blowing agent, and both: (□) gum, (○) gum with curing agent, (△) gum with 4 phr blowing agent, and (▽) gum with both curing and blowing agents.

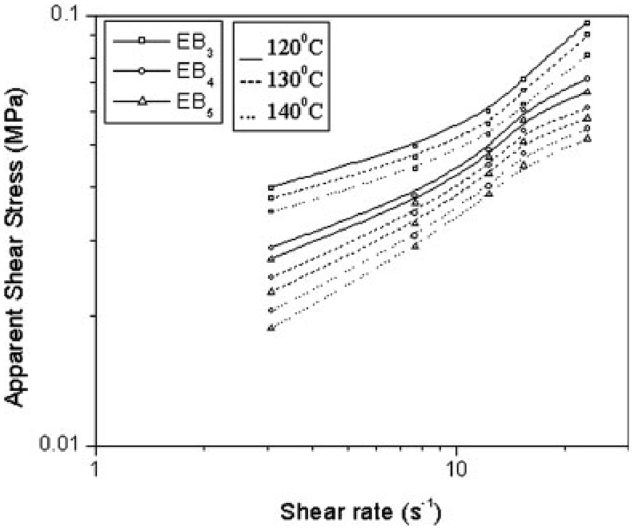

Figure 11 Plots of the apparent shear stress versus the shear rate of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates (without the curing agent) at different temperatures, showing the effects of the blowing agent: (□) 2, (○) 4, and (△) 6 phr.

the formation of gas bubbles, which dissolve inside the mass of the compound and restrict the stress rise. In the presence of both the curing agent and blowing agent, the decomposed gas is unable to be released from the compound, and this restricts the increase in the shear stress with the shear rate like the unfilled compound.

Effect of the blowing agent loading in the absence of the curing agent

Plots of the shear stress at the wall versus the shear rate with the variation of the blowing agent loading in the absence of the curing agent of Vulcan XC72 filled, microcellular compounds at different temperatures are shown in Figure 11. Figure 11 shows that with the increase in the blowing agent loading, the shear stress has a pronounced effect at a higher shear rate versus a lower shear rate. At a higher shear rate, the rate of the stress increase is restricted with an increase in the blowing agent loading because at a high shear rate, blowing agent decomposition is facilitated. Hence, the rate of the shear stress rise is predominantly reduced at a higher shear rate.

Effect of the blowing agent loading in the presence of the curing agent

Figure 12 shows plots of the apparent shear stress at the wall versus the shear rate with different loadings of the blowing agent in the presence of the curing agent of Vulcan XC72 filled, microcellular compounds at different temperatures. The stress decreases with an increase in the blowing agent loading. At a higher shear rate, the decrease in stress is slightly more compared to that of the uncured compound, and this may be due to the breakdown of gas bubbles, which is enhanced at a high shear rate and temperature.

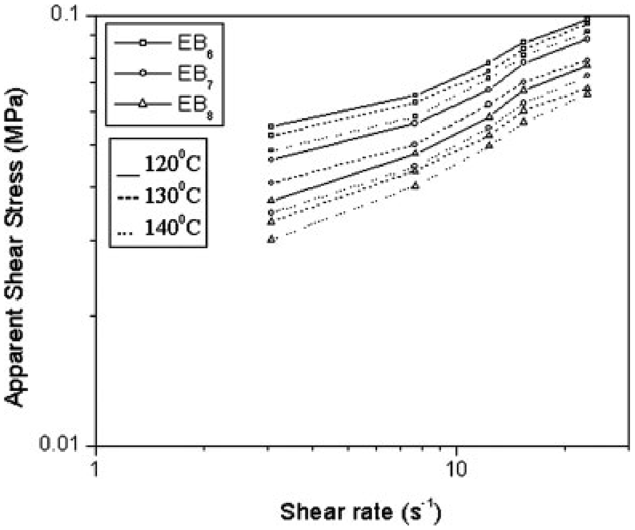

Figure 12 Plots of the apparent shear stress versus the shear rate of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates (with the curing agent) at different temperatures, showing the effects of the blowing agent: (□) 2, (○) 4, and (△) 6 phr.

VRF

The correlation between the extent of viscosity reduction and the loading of the blowing agent is of practical interest. VRF is defined as the ratio of the viscosity of the mixture of the compound with the blowing agent [$\overline{\eta}(\dot{\gamma},T)$] to the viscosity of the compound [$\eta(\dot{\gamma},T)$]. Mathematically

$$\mathrm{VRF} = \frac{\overline{\eta}(\dot{\gamma},T)}{\eta(\dot{\gamma},T)} \tag{5}$$

The VRFs at different shear rates and with various combinations of the blowing agent and curing agent at different temperatures are presented in Figures 13 and 14. For a given blowing agent loading, VRF is dependent on the shear rate and temperature. Moreover, VRF decreases with the shear rate and temperature. VRF shows a decreasing trend with an increase in the blowing agent loading. The decrease in the VRF value is more in the presence of a curing agent, which is enhanced at a higher temperature. Figures 13 and 14 give predictions of the viscosities of mixtures of the microcellular EPDM compound with the blowing agent from the viscosities of the virgin compound at the desired shear rate and temperature.

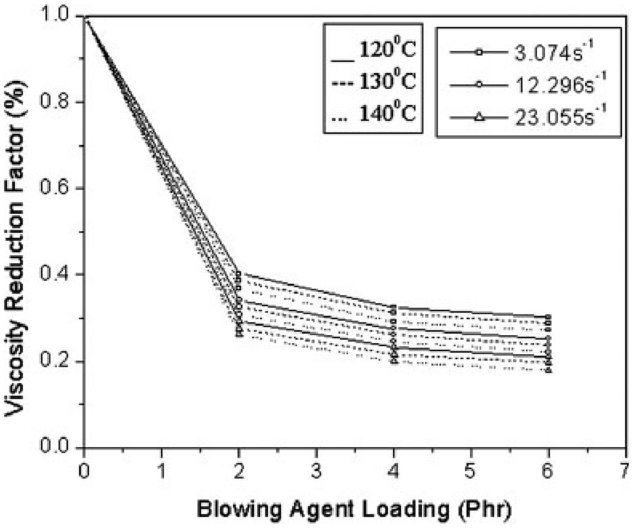

Figure 14 Plots of VRF (%) of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates as a function of the blowing agent loading at various shear rates (with the curing agent) at different temperatures.

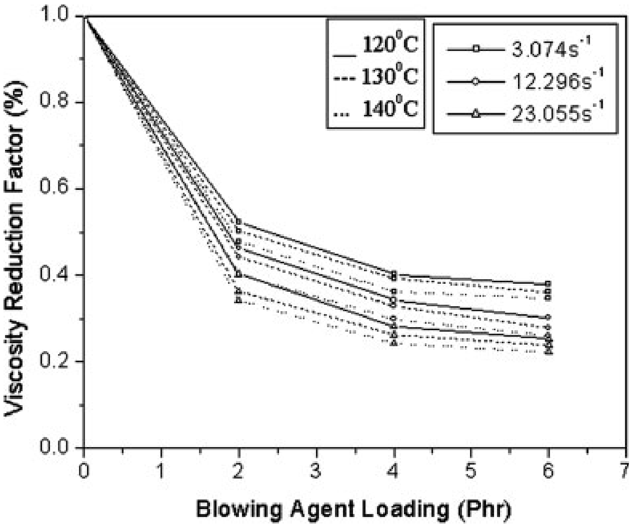

Figure 13 Plots of VRF (%) of 40 phr Vulcan XC72 filled, microcellular, oil-extended EPDM vulcanizates as a function of the blowing agent loading at various shear rates (without the curing agent) at different temperatures.

CONCLUSIONS

1. The morphology of the extrudates reveals that cells are nearly spherical in the case of the unfilled compound and nonspherical in the case of the Vulcan XC72 filled compound. The presence of the curing agent decreases cell size and distributes cells uniformly throughout the matrix in the case of the Vulcan XC72 compound because of the increase in the viscosity. The number of cells increases with an increase in the shear rate, blowing agent loading, and temperature.
2. In the case of the unfilled compound, the die swell increases with the shear rate and with decreasing temperature. On the addition of the blowing agent and curing agent, the die swell increases with the shear rate and temperature. The rate of increase is more pronounced at a higher shear rate. In the case of the Vulcan XC72 filled compound, the die swell increases with the curing agent, blowing agent, and temperature in comparison with the base compound. At a higher shear rate, the rate of increase in the die swell decreases.
3. The total extrusion pressure drop of the Vulcan XC72 filled, microcellular EPDM compound decreases with an increase in the blowing agent loading at all shear rates and temperatures. However, the incorporation of the blowing agent, curing agent, or both causes a sharp decrease in the pressure followed by a slow decrease.
4. The apparent shear stress increases with the shear rate. In the absence of the curing agent and blowing agent, the unfilled compound follows the power law, regardless of changes in the temperature. However, in the presence of the curing agent, blowing agent, or both, the increase is nonlinear. The apparent shear stress decreases with the blowing agent loading and

temperature for unfilled and Vulcan XC72 compounds.

5. VRF decreases with the increase in the shear rate, temperature, and blowing agent loading. The decrease in the VRF value is more pronounced in the presence of the curing agent and is enhanced at a higher temperature.

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