

Relationship Between the Cell Structure and Mechanical Properties of Chemically Crosslinked Polyethylene Foams

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ABSTRACT: The main objective of this work was to study the effect of the controlling parameters on the morphology and mechanical properties of the peroxide crosslinked low-density polyethylene foams. The relationship between the morphology and mechanical properties was also considered. Using different Dicumyl peroxide (DCP) and azodicarbonamide (ADCA) concentrations, various foams with different cell structures were prepared. Gel content and density of the foams were measured according to the standard methods. The morphology was examined using SEM technique. The mechanical properties of the foams were evaluated by means of compression and creep recovery tests. The results showed that the gel content and the density are mainly controlled by

DCP and ADCA concentration, respectively. The results also showed that the cell size distribution is mainly controlled by DCP concentration. Increasing of DCP increased the gel content and decreased the cell size and cell size distribution. Foam density was mainly controlled by ADCA concentration, whereas the morphology was less affected with ADCA concentration. The foams with small cell size and narrow cell size distribution showed higher mechanical strength and lower plastic strain. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 2789–2797, 2012

Key words: polyethylene; foam; crosslinking; morphology; mechanical properties

INTRODUCTION

Closed-cell polyethylene (PE) foams have extensively been used in many applications such as packaging, transportation, sports, construction, and agriculture because of their variety of properties including light weight, chemical resistance, thermal and electrical insulation.^{1–4} To achieve suitable melt strength, foamability, and a wide temperature range of rubbery plateau region, crosslinking is already applied either by chemical agents or by radiation processes.^{4–9} Although the most focuses have been concerned in irradiated foams,^{4,5,10,11} the chemical crosslinking is more economical, and it is applied in melt state, which causes to more uniform crosslinking. In chemical crosslinking, the crosslinking and foaming agents are selected on the basis of their decomposition temperature relative to PE melting temperature.¹ Dicumyl peroxide (DCP) and azodicarbonamide (ADCA) have frequently been used in many studies as a crosslinker and foaming agent, respectively.^{7,9,11,12–14} The experiences have showed that the mechanical properties of these foams are strongly dependent on their density, which is a complicated function of different parameters such as

crosslinking degree, foaming agent content, and process condition.^{6–9,15–18}

Although controlling of the cell structure of the foam has found a great interest in recent researches,^{15–20} but in most studies, mechanical characterization of the final produced foams is considered.^{21–24} Recently, Zakaria et al.⁷ studied on the formulation of PE foam compounds and also on the effect of foaming temperature. Although it has been shown for other polymeric foams such as polyurethane foams that the cell morphology and cell density can affect the mechanical properties,²⁵ there is a lack of information about the effect of controlling parameters on the cell structure of PE foams and the effect of cell structure on the final foam properties. Therefore, the main objective of this work was to study the effect of the controlling parameters on the morphology and mechanical properties of the peroxide crosslinked low-density polyethylene (LDPE) foams. The relationship between the morphology and mechanical properties was also considered. For this propose, different foams with different cell structure were prepared using different DCP and ADCA concentrations via a single-step compression molding method.

EXPERIMENTAL

MATERIALS

LDPE-0020 (MFI = 2.0 g/10 min; 2.16 Kg, 190°C) from Bandar Emam Petrochemical Company, Iran, was

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used. Dicumyl peroxide (DCP) as a crosslink agent from AkzoNobel (Amersfoot, Netherlands) and Azodicarbonamide (ADCA) as a foaming agent from Foco Company, South Korea, were used as received.

Sample preparation

The compositions and corresponding codes of the different compounds prepared in this study are listed in Table I:

Compounding of all the samples was carried out in an internal mixer (Brabender W50EHT) at temperature of 120°C with a rotor speed of 60 rpm to inhibit the thermal decomposition of ADCA and DCP during compounding process. PE was first filled into the chamber, and ADCA and DCP were added to the melt mixture after 3 and 5 min, respectively, and compounding was continued up to 10 min. The prepared compounds were first compression molded at temperature of 130°C and then the temperature was risen up to 210°C with a heating rate of 10°C/min and held for 10 min. Then, the mold pressure was released at the same temperature and foam was obtained.

Characterization of the foam samples

The state of crosslinking was evaluated using gel content measurement according to ASTM D2765-90.²⁶ A total of 300 ± 5 mg of material (initial weight) was extracted in 400 mL of boiling Xylene for 24 h. The remained material (gel) was dried for 3 h at 140°C in a vacuum oven and gel content was calculated using eq. (1):

$$\text{Gel Content (wt\%)} = \frac{\text{Gel weight}}{\text{Initial weight}} \times 100 \quad (1)$$

Density of the foam samples was determined according to ASTM D792.²⁷ Morphology of the foam samples, cell size and cell size distribution, were examined using SEM analysis (Hitachi S-2400 SEM with an electron potential of 25 kV). All the surfaces were gold sputtered for good conductivity of the electron beam, and microphotographs were taken within a magnification of 100×. SEM images were analyzed using Image Processing software to measure the cell size, cell size distribution, and cell density using following equations:

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left(-\frac{(x-\mu)^2}{2\sigma^2}\right) \quad (2)$$

$$N_0 = \left[\frac{NM^2}{A}\right]^{3/2} \left[\frac{\rho}{\rho_f}\right] \quad (3)$$

where μ and σ^2 are the average cell size and variance, respectively. N_0 , A , N , M , ρ , and ρ_f are cell

TABLE I
Compositions and the Corresponding Codes of Different Compounds

DCP (%)	ADCA (%)	PE	Sample
0.6	5	100	PEA5D0.6
0.8	5	100	PEA5D0.8
1	5	100	PEA5D1.0
0.6	10	100	PEA10D0.6
0.8	10	100	PEA10D0.8
1	10	100	PEA10D1.0
0.6	15	100	PEA15D0.6
0.8	15	100	PEA15D0.8
1	15	100	PEA15D1.0

density in unit volume, SEM micrograph area, the number of cells in area A , magnification factor of the SEM micrograph, polymer density, and foam density, respectively.²⁸

A Zwick/Roell tester (Z010) was used to carry out the compression tests that were performed according to ASTM D1621 with a compression speed of 10 mm/min.²⁹ The cross section of the specimens was 20 × 20 mm², and the thickness of the specimens was that of obtained thickness between 14 and 18 mm. Compressive creep recovery was recorded over a range of applied stresses at room temperature. The load program applied to the samples is shown in Figure 1(a). The creep stresses (σ_c) used were 0.07, 0.02, and 0.01 MPa for samples with 5, 10, and 15 wt % ADCA, respectively, and the time of the creep and recovery was 20 min. The recovery stress (σ_r) was 0.002 MPa.

A typical creep and creep-recovery curves of these materials and the parameters used to characterize their behavior are shown in Figure 1(b). If $\varepsilon(t)$ represents the strain as a function of the time (in min), the other parameters were defined as $\varepsilon_i = \varepsilon(t = 0.1 \text{ min})$, $\varepsilon_{max} = \varepsilon(t = 20 \text{ min})$, $\varepsilon_{ri} = \varepsilon(t = 20.1 \text{ min})$, and $\varepsilon_p = \varepsilon(t = 40 \text{ min})$.

The parameters used to compare the response of each sample were defined as follows²²:

Instantaneous strain:

$$E_i = \frac{\varepsilon_i}{\varepsilon_{max}} \quad (4)$$

Retarded strain:

$$E_r = 100 \times \frac{(\varepsilon_{max} - \varepsilon_i)}{\varepsilon_{max}} \quad (5)$$

Instantaneous recovery:

$$R_i = 100 \times \frac{(\varepsilon_{max} - \varepsilon_{ri})}{(\varepsilon_{max} - \varepsilon_p)} \quad (6)$$

Retarded recovery:

$$R_i = 100 \times \frac{(\varepsilon_{ri} - \varepsilon_p)}{(\varepsilon_{\max} - \varepsilon_p)} \quad (7)$$

Plastic strain:

$$E_p = 100 \times \frac{\varepsilon_p}{\varepsilon_{\max}} \quad (8)$$

RESULTS AND DISCUSSION

Physical properties

The processes in which foam expansion is accomplished by heating, cells need to be stabilized by crosslinking of the polymer. Crosslinking extends the rubbery plateau of the polymer melt and increases the temperature range in which stable foam can be produced.¹ Crosslinking is critical in PE foam processing, and it has an influential effect on foam properties. Figure 2 shows the effect of DCP and ADCA concentrations on gel content of different foam samples. The results show that gel content increases with increasing of DCP content. It can also be seen that increasing rate of gel content decreases at high DCP contents due to the gel saturation in these samples.³⁰

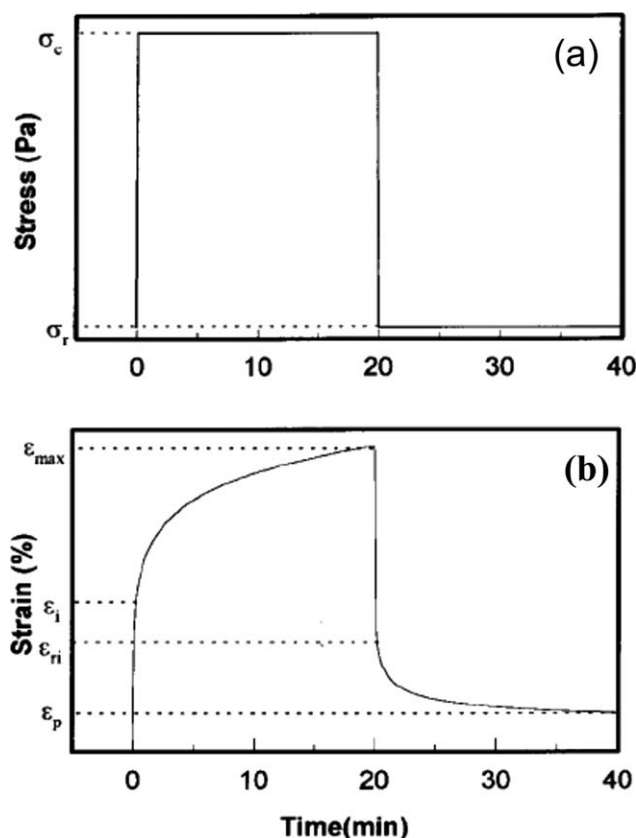


Figure 1 A typical creep and creep-recovery curve.²²

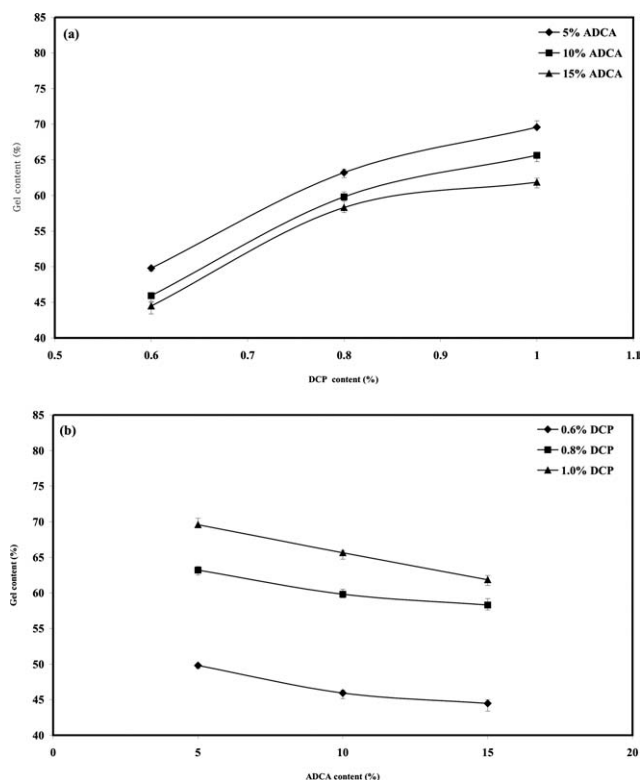


Figure 2 Effect of DCP and ADCA concentrations on gel content.

Increasing in ADCA concentration decreases the gel content may be due to its inhibition effect on crosslinking reactions. It was shown using DTA studies, not shown here, that the decomposition of DCP starts at lower temperatures (about 140°C) than that of ADCA decomposition (about 190°C) in PE compounds which underwent the heating rate of 10°C/min. Therefore, solid ADCA particles may inhibit PE crosslinking at least at lower temperatures than ADCA decomposition temperature. By comparing the effects of DCP and ADCA contents on gel content, it can be claimed that DCP content is the major controlling parameter of the gel content.

Figure 3 shows the effect of DCP and ADCA concentrations on the foam density. These results clearly show that increasing of ADCA concentration remarkably decreases the foam density at a fixed DCP concentration, whereas increasing of DCP concentration slightly increases the foam density. Increasing of the amount of the gas produced via decomposition of ADCA, increases the melt expansion during the foaming process leading to the foam density decrease. On the other hand, increasing of the degree of crosslinking increases the melt viscosity, which restricts the melt expansion, and therefore, increases the foam density. This effect is more obvious at low ADCA concentration (5 wt %) than that of other ADCA concentrations (10 and 15 wt %). These results indicate that ADCA concentration

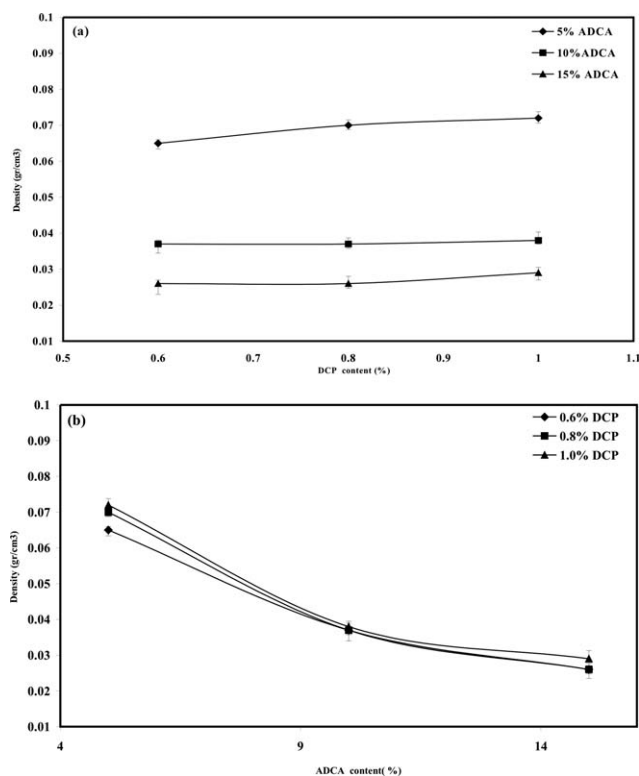


Figure 3 Effect of DCP and ADCA concentrations on foam density of the crosslinked LDPE foams.

is the main controlling parameter of the foam density. At high ADCA contents, because of the higher amount of produced gas, bubble rupture can occur at low DCP content (0.6 wt %), but at higher DCP contents (0.8 and 1.0 wt %), the bubble was more stable which decreased the rate of increasing density with DCP increase.

Morphology characterization

Figure 4 shows the SEM images of the foams prepared using different DCP and ADCA concentrations. It can be seen that most of the foams exhibit closed-cell structure, and the common shape of the cells are pentagonal, dodecahedrons, and/or tetracaidecahedra.

Figures 5 and 6 show the effect of DCP and ADCA on the cell size distribution of the foams, obtained using image analysis followed by Gaussian distribution assumption. The results clearly show that increasing of DCP concentration decreases both average cell size and cell size distribution particularly at low DCP concentrations, whereas ADCA has a negligible effect on the cell size and cell size distribution. Increasing of DCP concentration increases the degree of crosslinking which restricts and obstructs cell growth leading to lower expansion of the foam and, therefore, to formation of smaller cells with thicker cell walls. From these results, it can be concluded

that the morphology of the crosslinked PE foams is strongly affected by degree of crosslinking rather than the amount of gas and, therefore, in the way of controlling of the morphology more attention should be paid on the degree of crosslinking.

Figure 7 shows the average cell size and its variance as functions of DCP content at different ADCA contents. As previously stated and supported by these results, the effect of DCP on cell size and cell size distribution is more notable at low DCP contents, and at higher DCP contents, the decreasing trend of cell size and variance tend to level off. This can be due to the gel saturation at higher DCP contents as mentioned earlier.

Figure 8 shows the effect of DCP and ADCA on the cell density of the foams. The results indicate that increasing of both DCP and ADCA increases the cell density. It should be noted that the mechanisms of the increasing of cell density with DCP and ADCA are different. Increasing of DCP, as discussed earlier, decreases the cell size and cell size distribution leading to increase of cell number in unit volume, while increasing of ADCA increases the amount of produced gas and, therefore, increases the number of cells nucleated.

Mechanical properties

Figures 9 and 10 show the effect of DCP and ADCA on the compressive stress–strain behavior of the foams. The compressive stress–strain curves of all the samples show three different zones; linear elasticity at low stresses (zone 1) followed by a long collapse-plateau (zone 2) that truncated by a regime of densification in which the stress rises steeply (zone 3).¹⁶ Linear elasticity is controlled by cell wall bending and cell face stretching. The plateau is associated with collapse of the cells caused by elastic buckling. When the cells have almost completely collapsed, opposing cell walls touch and further strain compresses the solid polymer resulted in rapid stress increase.¹⁶ The results show that increasing of DCP increases Young's modulus which is clearer at low ADCA contents. By considering the results of the corresponding foam densities (Fig. 3), it can be concluded that the major controlling parameter of the mechanical properties is the foam density. It was shown that at low ADCA contents, increasing of DCP concentration increases the foam density, which could increase the modulus of elasticity. This finding is in a good agreement with the results obtained by Sims and Khunniteekool⁸ and Zakaria et al.⁷ In brief, with increasing foam density, Young's modulus increases which raises the plateau stress and reduces the strain at which densification starts.¹⁶ At higher ADCA contents, the variation of DCP concentration did not display a notable effect

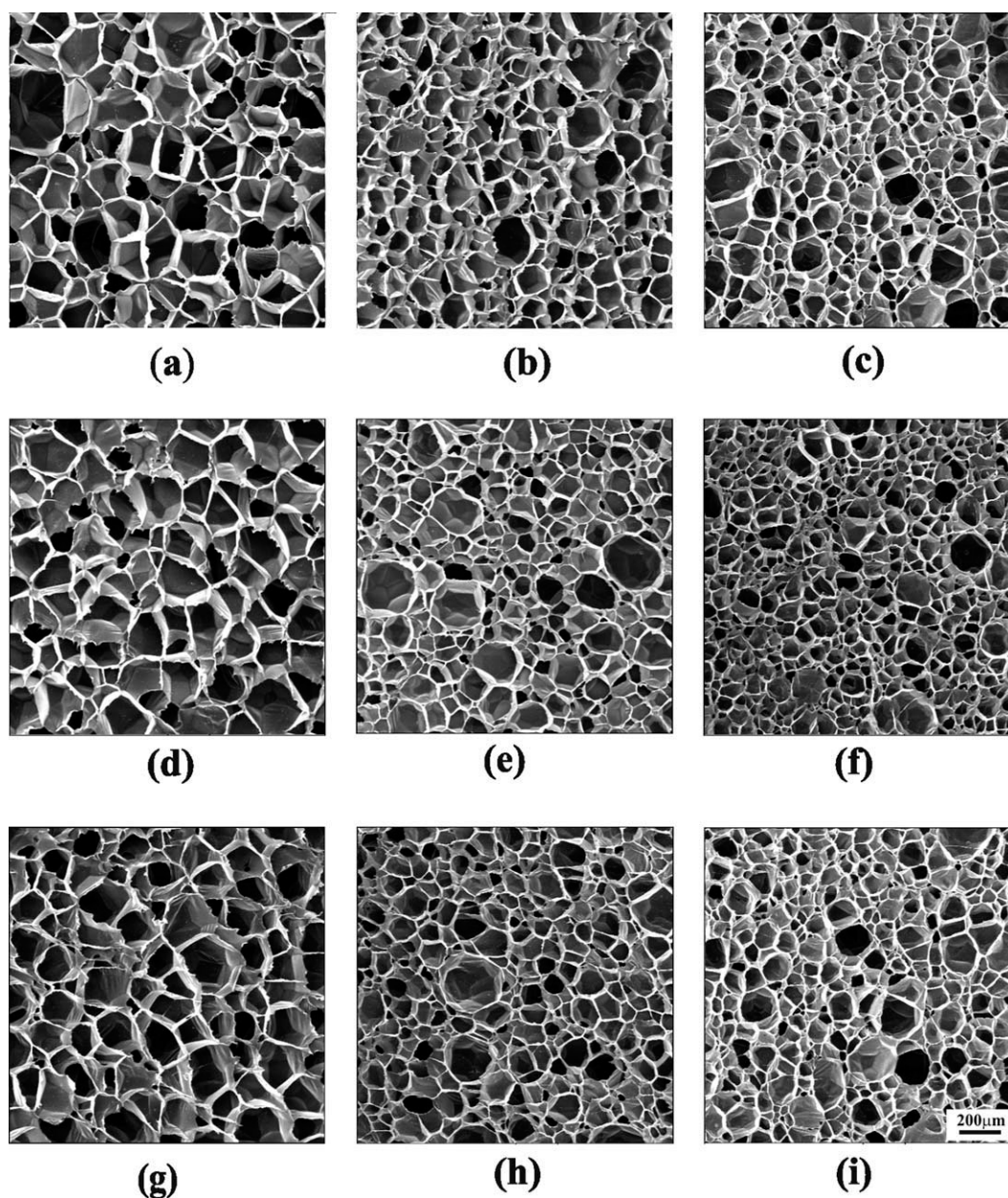


Figure 4 SEM images of the crosslinked LDPE foams (a) PEA5D0.6, (b) PEA5D0.8, (c) PEA5D1.0, (d) PEA10D0.6, (e) PEA10D0.8, (f) PEA10D1.0, (g) PEA15D0.6, (h) PEA15D0.8, and (i) PEA15D1.0.

on the foam density (Fig. 3). On the other hand, the results show that at high strain regions (zone 3) increasing of DCP increases the required stress of densification even for the foams with nearly the same density (ADCA 15 wt %). This observation can be related to the effect of cell size and cell size distribution. Increasing of DCP decreases the average cell size and cell size distribution as stated earlier (Fig. 7). For the foam with small cell size and narrow cell size distribution, the stress is uniformly distributed over the different cells and causes more resistance toward cell collapse during densification, whereas for foams with a broad cell size distribution, the stress is first transferred to larger cells and collapses

them followed by stress transfer to small cells. As the results show, increasing of ADCA decreases the mechanical properties of the foams, which is a consequence of decreasing of the foam density with increasing ADCA concentration. In this case, the effect of density on mechanical properties is predominant to the effect of cell size and cell size distribution.

Creep in polymeric foams such as PE is dominated by the base polymer viscoelasticity if the stress is less than the collapse stress, but at higher stresses, gas compression takes an increasing proportion of the load.³¹ Recovery after creep is a slow process. The deformation mechanism in creep and recovery

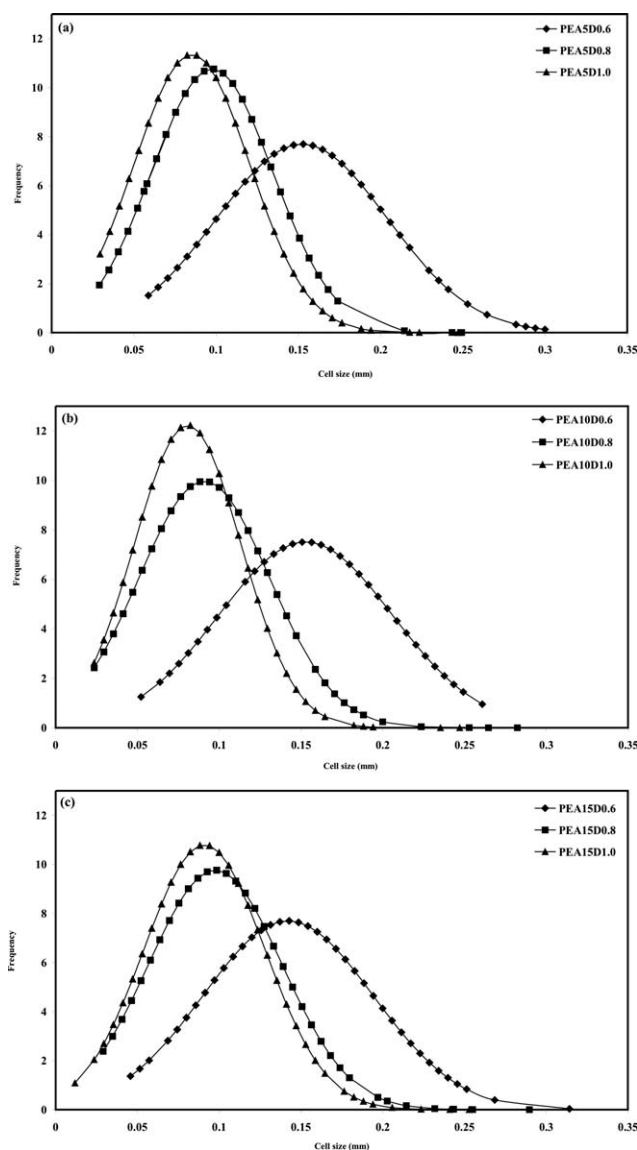


Figure 5 Effect of DCP concentration on the cell size distribution of the crosslinked LDPE foams.

occurs in different orders. If a significant percentage of air/gas within the foam cell has diffused out of the foam during creep test, the recovery will be slow, because there will be a weak viscoelastic recovery hindered by the slow reentry of the air to the foam.^{31,32} Bhatt et al.^{33,34} suggested that increasing of the crosslinking degree reduces the gas loss during mechanical testing, which is probably due to the reduction in polymer permeability. Because of predominant effect of ADCA on the foam density, which is the most controlling parameter of the mechanical properties, in this study only the effect of DCP content on the creep and creep recovery behaviors was studied at fixed ADCA contents. Figure 11 shows the results of creep and creep recovery tests for different foams. For 5 and 10 wt % ADCA, the results show that the strain rapidly increases with

time at 0.6 and 0.8 wt % DCP, whereas at 1.0 wt % DCP, it increases slower. This can be related to the modulus of elasticity of different foams as previously observed in Figure 9. Rodríguez-Pérez et al.,²³ Mills and Gilchrist,³¹ and Hilyard and Cunningham³⁵ stated that the creep behavior of polymeric foams at linear elastic region is controlled by the viscoelastic properties of the matrix polymer. The foam with 1.0 wt % DCP displays higher elasticity than foams with 0.6 and 0.8 wt % DCP, leading to lower strain at a constant stress during creep test. At 15 wt % ADCA concentration, the results show a different trend than that observed at 5 and 10 wt % ADCA. This is because of different stress–strain behavior of the foams with 15 wt % ADCA. For the foam with 15 wt % ADCA, stress–strain curves are nearly the same at zone 1 at different DCP concentrations. Therefore, the observed difference between the

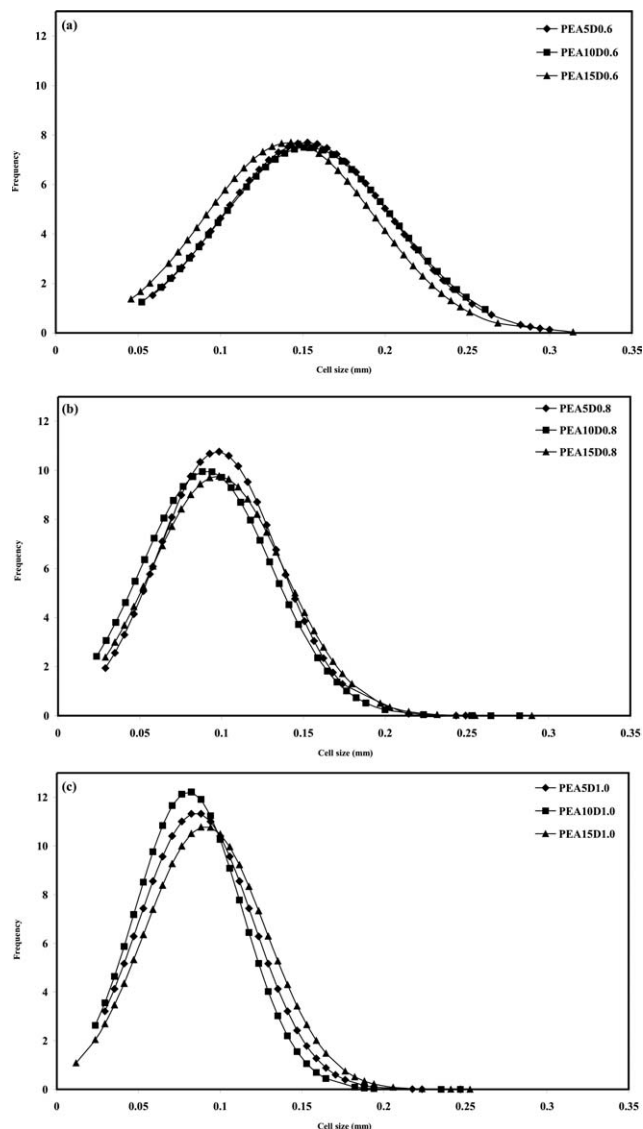


Figure 6 Effect of ADCA concentration on the cell size distribution of the crosslinked LDPE foams.

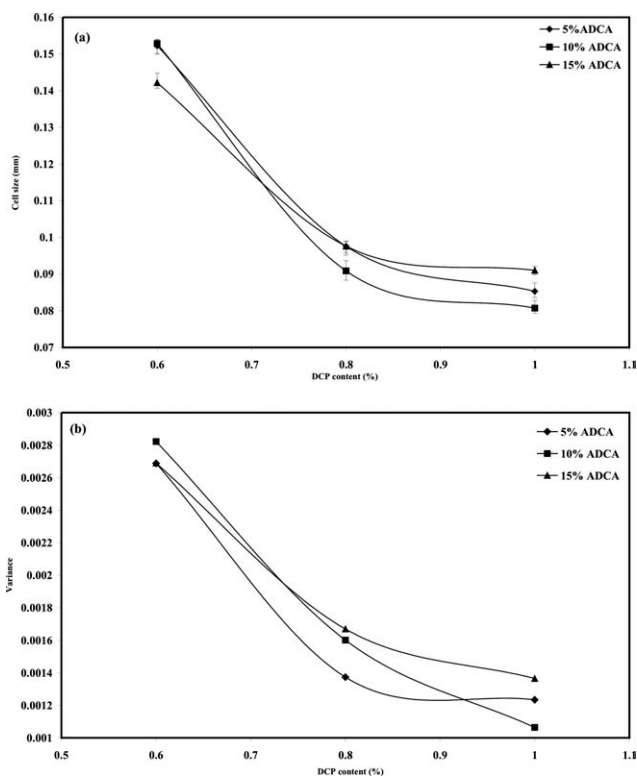


Figure 7 Effect of DCP concentration on average cell size and variance of the crosslinked LDPE foams.

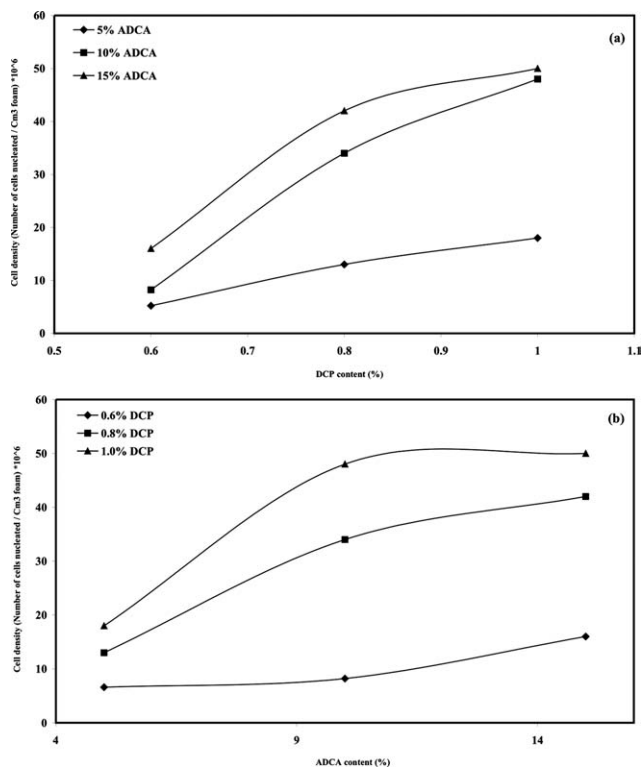


Figure 8 Effect of DCP and ADCA concentrations on the cell density of the crosslinked LDPE foams.

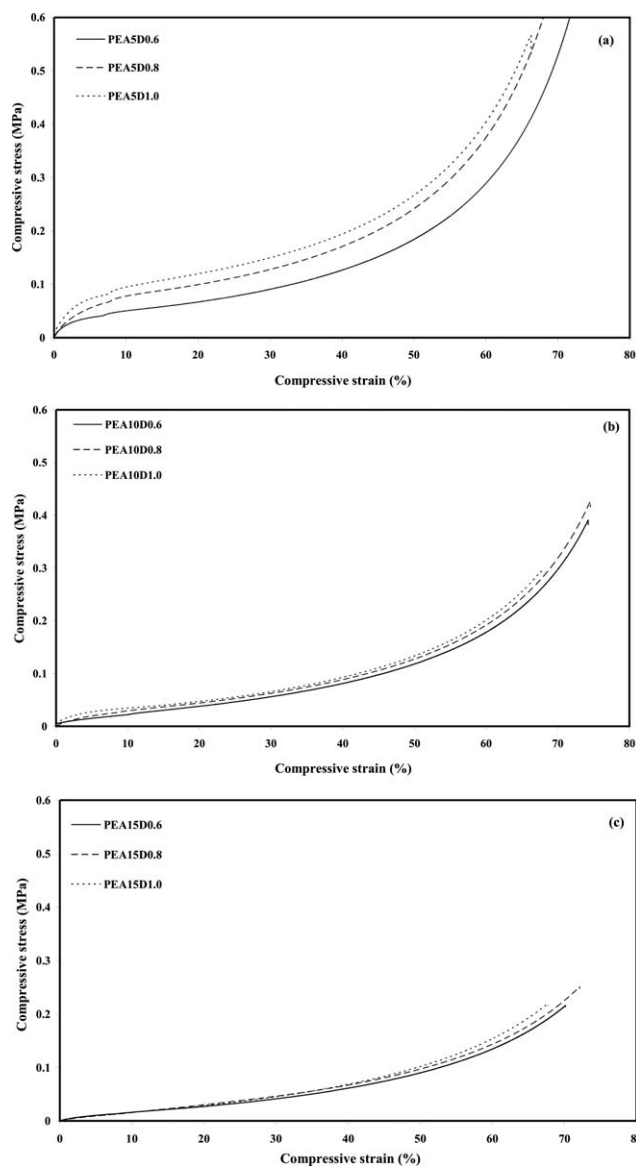


Figure 9 Effect of DCP concentration on the compressive stress-strain behavior of the crosslinked LDPE foams.

behaviors of three different samples can be related to the cell morphology of these foams. The foams with higher DCP contents display more closed-cell structure than that of lower DCP samples (Fig. 4). Therefore, in the creep test they exhibit lower gas diffusion than that of lower DCP samples, which in turn leads to lower creep rate. This effect is also presented for foams with 5 and 10 wt % ADCA, but the effect of elasticity at low strain range is predominant to the effect of the closed-cell percentage. The results also show that at any ADCA concentration, increasing of DCP decreases the plastic strain. This can be due to the higher elasticity of polymer with higher degree of crosslinking and also higher percentage of closed-cells at this state. Higher amount of closed-cell leads to lower amount of gas diffusion during creep test and, therefore, to higher recovery after

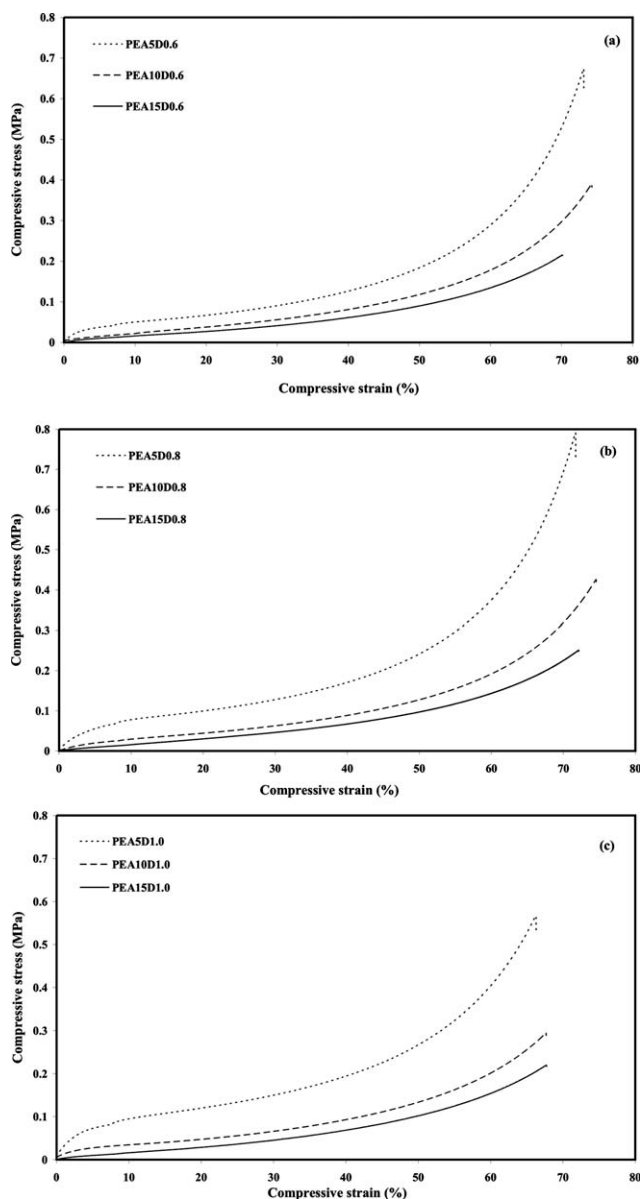


Figure 10 Effect of ADCA concentration on the compressive stress–strain behavior of the crosslinked LDPE foams.

removal of the stress. On the other hand, increase in DCP content would cause the cell walls become thicker and exhibited higher viscoelastic recovery of the bended cell walls. The numerical values of the creep and creep recovery parameters are calculated and presented in Table II. By considering the numerical value of E_i and E_r for each group of the foams, it can be seen that E_i and E_r have increasing and decreasing trends with foam density, respectively. Increasing of DCP concentration reduces the cell size and cell size distribution which increases cell wall thickness and, therefore, enhancing the viscoelastic properties of the foams leading to instantaneous strain increase and retarded strain decrease. The numerical values of R_i and R_r and E_p for each group of the foams show that R_i and R_r display independent

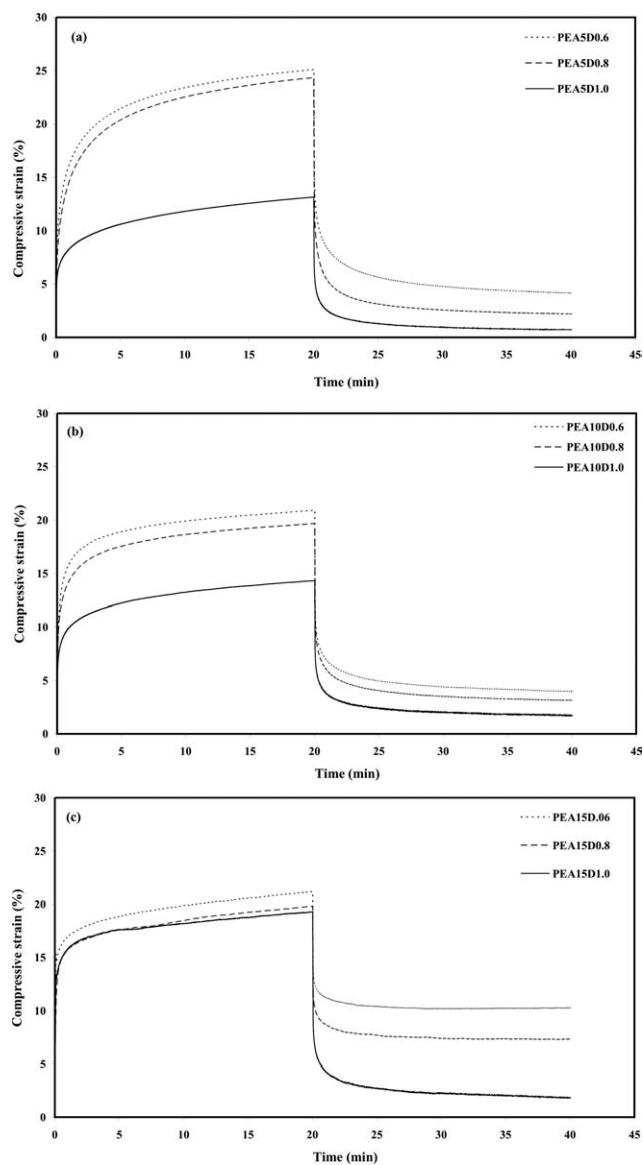


Figure 11 Creep and creep recovery behavior of cross-linked LDPE foams with different of DCP and ADCA concentration.

behavior of the foam density while E_p decreases with density increase. The independent response of R_i and R_r may be related to the fact that, when a

TABLE II
Numerical Values of the Creep and Creep Recovery Calculated Parameter

E_p	R_r	R_i	E_r	E_i	Sample
16.54	25.11	58.79	62.51	37.49	PEA5D0.6
9.04	25.55	62.37	67.71	32.29	PEA5D0.8
5.49	41.55	64.32	55.24	44.75	PEA5D1.0
19.12	19.12	65.5	52.79	47.21	PEA10D0.6
16.05	16.05	63.9	50.79	49.21	PEA10D0.8
11.97	11.97	64.46	49.65	50.35	PEA10D1.0
48.41	24.51	75.49	28.12	71.88	PEA15D0.6
37.32	27.21	72.79	44.61	55.39	PEA15D0.8
9.41	33.28	66.72	30.72	69.28	PEA15D1.0

constant stress is applied for each group, different foams may be stand in different region of stress-strain curve (zone 1 or zone 2). In such a case, different foams exhibit different creep mechanism and, therefore, different viscoelastic recovery response.

CONCLUSIONS

In this study, crosslinked closed-cell LDPE foams were prepared by varying in DCP and ADCA concentrations in their compound formulations using a one-step compression molding method. Their morphologies as well as their compressive and creep behavior were studied, and the obtained results are projecting the following conclusions.

1. The results showed while the density is mainly controlled by ADCA content, the DCP content is the major controlling parameter of the state of crosslinking.
2. The results of morphological studies showed that increasing of degree of crosslinking decreases both cell size and cell size distribution, whereas the ADCA had a negligible effect on the cellular structure. Increasing of DCP also increased the amount of the closed-cell structure. Increasing of both DCP and ADCA increased the cell density via increasing of the viscosity and amount of the gas, respectively.
3. The results of mechanical studies clearly showed that, although the density (which was mainly affected by the amount of ADCA) was the main controlling parameter of the mechanical properties, but at the same density of the foams, cell size and cell size distribution play a key role in determining of the mechanical properties. Smaller and uniform distributed cells showed higher resistance to deformation than that of large cells with a broad cell size distribution. Higher degree of crosslinking led to formation of higher extent of closed-cell structure which displayed low plastic strain during creep recovery tests.

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