Study on Cellular Structure and Mechanical Property of Foaming/Cross-linking Polyethylene System

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Abstract. The cellular structure and mechanical property of a sequential foaming/cross-linking polyethylene system were studied in this work. By adjusting the components, foaming starts before the cross-linking reaction initiated and the melt strength increases during the foaming process. Rubber Process Analyzer (RPA) was used for the in situ monitoring and measuring of the foaming and cross-linking process. The cellular structure and expansion ratio of polyethylene foam can be modulated by controlling the type and ratio of foaming agent and cross-linking agent, as well as the foaming/cross-linking conditions. The mechanical strength was tested by universal mechanical testing machine, the melt strength were also characterized and analyzed. Experimental results demonstrated that the cross-linking controlled the cellular size and improved mechanical strength.

1 Introduction

Because of the cellular structure and lightweight advantages, polyethylene foam materials are widely used in different and diverse applications, such as, building insulation, packaging, sealing, automotive trim, medical devices, water treatment[1-3]. The cellular structure is the most crucial and decisive property for polyethylene foam material application[4]. However, cellular structure is closely related with polyethylene matrix properties and foaming conditions, for example, the polyethylene resin with low viscosity usually produces large size cell and low cellular density foam material[5].

In order to realize controlling the factors of cellular growth, some improvement methods have been researched. Chen et al. took high-pressure rheometer to study Low Density Polyethylene (LDPE) crystallization, when the crystallinity was more than 50%, the crystal would affect the cellular size distribution and even constrain the cell growth, and the higher the crystallinity, the smaller the cell size[6]. Yang et al. used supercritical carbon dioxide method to prepare ultrahigh-cell-density polymer material in the presence of porous polytetrafluoroethylene (PTFE)

micro-powder. The voids of PTFE micro-particles were filled with supercritical CO₂, causing them to split into multiple granules due to the expansion force during the pressure release, resulting in the formation of a large number of nucleation sites, the prepared cellular size was around 10-30 μ m[7]. Because of the cross-linking reaction, the polyethylene molecules connected with each other and form three-dimensional network structure, this structure changing reduce realize controlling cell growth. In these above methods, cross-linking was the one of easiest and most widely used method[8,9] for improving cellular structure property, and there have already some papers been reported. Adem et al. took gamma ray from a cobalt-60 source to radiation crosslink LDPE, the obtaining material with smaller cells and high performance properties[10]. Chemical cross-linking method was using chemical agent as initiator to cross-link polyethylene molecules, just because the convenient and cheap of chemical initiator, chemical cross-linking was the more widely using method than radiation method[11], for example, it was widely used in rotation moulding field. However, up to now, few reports of cellular structure and mechanical property of cross-linking polyethylene foaming system which made by chemical cross-linking method were

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published[12], especially the sequential foaming/cross-linking reaction polyethylene system.

In this paper, a sequential foaming/cross-linking system was designed, in which foaming reaction was initiated before the cross-linking reaction started. The cellular structure and mechanical strength were controlled by changing the dilution ratio of a cross-linking master batch. Rubber Process Analyzer (RPA) was used for the in situ measurement of viscosity and pressure during the foaming/cross-linking process. The gel content and melt strength properties were tested and analyzed.

2 Materials and methods

2.1 Materials preparation

Cross-linking polyethylene master batch (XPE) was obtained from our laboratory, in which 2,5-dimethyl-2,5-double (tert-butyl peroxide) hexane (DBPH) from Arkema Co., Ltd was used as the cross-linking agent. 4, 4-oxybis benzenesulfonyl hydrazide (OBSh) from J&K Chemical Co., Ltd. was chosen as the foaming agent. Linear Low Density Polyethylene (LLDPE) DMDA7042 was purchased from Shenhua Baotou Co., Ltd., with melt index 2.6g/10min and density 0.918g/cm³. High density polyethylene (HDPE) DMDA8007 was supplied by Shenhua Baotou Co., Ltd, the melt index is 10.9g/10min and the density is 0.965g/cm³.

In this experiment, high density polyethylene (8007) was used as the XPE matrix resin, DBPH was used as the cross-linking agent. The XPE samples preparation process was as follows: firstly, take out some DBPH and 8007, make the quality ration was 1:20, then mix polyethylene resin and cross-linking agent, finally, put the obtained mixture into twin screw extruder, the temperature was set at 140°C.

2.2 Samples preparation

XPE with different dilution ratio and adding 2% OBSh to dilution XPE were investigated, for comparing, the control samples were also prepared. The sample compositions are shown in Table 1. The

HAAKE mixer temperature was preset at 130°C, 40 grams of the dry mixture of each composition were added to the mixer and mixed for 3min at a rotation speed of 60r/min. Then the mixtures were take out from HAAKE mixer and cooled to room temperature.

Table1 Sample code list

Serial number	Mixture formula	Sample code
1	7042:XPE=4	X4
2	7042:XPE=6	X6
3	7042:XPE=8	X8
4	7042:8007=4	B4
5	7042:8007=6	B6
6	7042:8007=8	B8
7	2%OBSh+(7042:XPE=4)	OX4
8	2%OBSh+(7042:XPE=6)	OX6
9	2%OBSh+(7042:XPE=8)	OX8
10	2%OBSh+(7042:8007=4)	OB4
11	2%OBSh+(7042:8007=6)	OB6
12	2%OBSh+(7042:8007=8)	OB8

2.3 Sheet samples preparation

Set the press machine temperature at 130°C, the obtained polyethylene mixtures were put in the mold with size of 16cm × 14cm × 1.8cm, then compression-molded under vacuumed condition for 3min at a pressure of 3MPa.

2.4 Oven foaming experiment

The pressed sheets were put into an oven and heated for 20min at 240°C for foaming and cross-linking. Then the samples were taken out and cooled at room temperature for 24h.

2.5 Melt flow index (MFI)

Samples were tested using a Ceast Modular Melt Flow plastometer (1B9580), at 190°C, 2.16kg load according to ASTM D1238-04C (2004).

2.6 Rubber process analyzer (RPA) test

Dynamic rheological and pressure measurements were

done. The change of viscosity, an index of cross-linking degree, and the pressure, an index of foaming level are simultaneously monitored and measured in situ using a *rubber process analyzer* RPA-2000P(Alpha Technologies Company). Set the temperatures at 230°C, 2g samples were put into the test wheel, preheating time for 1 min 230°C. The RPA testing panel was maintained at 0.5° amplitude and sample was subjected to the frequency sweep at 1.667Hz.

2.7 Elongation rheometer test

Take out 10 gram hake mixtures and put into pressure mold in 130°C, 3MPa for 1 min. The prepared sample size was 10cm×18cm×0.68cm. Take out the obtained film and cut into the 10cm×1cm test sample. Test sample was put into the elongation rheometer to investigate the melt strength during foaming and cross-linking process at different temperature period (150°C, 170°C, 190°C, 210°C and 230°C), test frequency was 0.1s⁻¹.

2.8 Expansion ratio test

The densities of foamed samples, ρ , were determined by deducing the Archimedes law involving weighing polymer foam in water with a sinker using an electronic analytical balance (HANG-PING FA2104), calculated using equation 1.

$$\rho = \frac{a}{a+b-c} \times \rho_w \quad (1)$$

where a, b, and c are the weights of the sample in air without sinker, the totally immersed sinker, and the specimen immersed in water with sinker, respectively, and ρ_w is the density of water. The volume expansion ratio of the cross-linking polyethylene foaming material, R_v , was the ratio of the bulk density of initial mixture density (ρ_0) to that of the cross-linking polyethylene foaming material (ρ), calculated as follows:

$$R_v = \frac{\rho_0}{\rho} \quad (2)$$

2.9 Gel content test

The gel content was determined by using xylene as the solvent according to ASTM-D2765, oil bath temperature was 170°C. A polyethylene sample of 0.400 ± 0.015 grams by weight was cut into small pieces and placed in a 100-mesh stainless steel wire cloth. The sample was dropped into xylene, boiled and reflux condensation for 8 hours. After the extraction cycle, the sample was dried in a vacuum oven at 160°C for 2 hours. The gel content (%) was calculated as follows:

$$X_{gel} = (m_3 - m_1) / (m_2 - m_1) \times 100\% \quad (3)$$

where m_1 is the weight of stainless steel wire cloth, m_2 is the weight of initial sample and stainless steel wire cloth, m_3 is the total weight of sample and stainless steel wire cloth after extraction.

2.10 Scanning electron microscope (SEM) test

Those foam samples were immersed in liquid nitrogen for 10min, and then fractured; the surface was vacuum coated with gold examined using a NOVA Nano SEM450 (oxford instruments). The size and size distribution of the cells were measured using Nano-measure software.

2.11 Flexural modulus test

Flexural experiments were performed at room temperature using a universal mechanical testing machine (5965, INSTRON Company, American). For evaluation of flexural modulus of cross-linking polyethylene foaming materials, three-point bend test is employed as per ASTM D790-032 standards. Length and width of each test sample is 80mm×10mm. Flexural test helps in evaluation of attributes of sample when it is subjected to a loading condition similar to simple beam loading[13].

3 Results and discussion

3.1 Interaction between cross-linking and foaming reactions

MFI is one of the most common parameters of polyethylene, it could affect the cross-linking reaction and the degree of cross-linking of polyethylene[14]. Similarly, it also has great influence on the dispersion of the foaming agent in the polyethylene, the gas diffusion speed, the interfacial tension between gas and liquid, and melt strength properties[15]. In this paper, 7042 was used as the dilution resin of XPE. And the MFI of B4, B6 and B8 mixtures was tested (table2), which represented the X4, X6 and X8's dispersion resin, respectively. Test results were showed in table 2: the higher content of 7042, the lower MFI; and vice, the more 8007, the higher MFI. That's because that 7042 has low MFI and 8007 has high MFI value.

Table 2 MFI of B4, B6, B8

Serial number	Sample code	MFI (g/10min)
1	B4	3.8
2	B6	3.4
3	B8	2.7

Density is another important parameter of polyethylene. For foaming polyethylene material, low density means that lightweight and high expansion ratio[16]. Table3 showed the density of B4, B6, B8, X4, X6 and X8 samples before foaming. Obviously, although polyethylene mixtures' density has little difference, they were all round about 0.92g/cm³.

Table 3 Density of B4, B6, B8, X4, X6 and X8

Serial number	Sample Code	Density (g/cm ³)
1	B4	0.927
2	B6	0.925
3	B8	0.923
4	X4	0.925
5	X6	0.927
6	X8	0.930

Previous studies have shown that cross-linking reaction and foaming reaction disturb each other if the two reactions occur simultaneously^[12]. Ideally, we want the foaming reaction starts first and keep the cross-linking degree at low level until the desired cellular structure is almost achieved. Thus OBSH was chosen as the foaming agent. Which decompose at a lower temperature than that DBPH starts to initiate the cross-linking reaction[17].

Figure 1 is a typical RPA test result of the sequential foaming/cross-linking polyethylene system. The measured pressure starts to increase after 30 seconds, which represents the starting of the foaming process. 70 seconds later, the viscosity starts to increase, which is in accordance with the initiation of cross-linking reaction. Once cross-linking reaction started, the increase of pressure slows down and the curve shows an inflection point. When the viscosity reaches the maximum value, the foaming and cross-linking reactions achieve a balance. Obviously, in this sequential foaming/cross-linking polyethylene system, the foaming and cross-linking reactions compete to each other and the foamed/cross-linked structure can be controlled by adjusting the composition and process conditions.

Figure 1. Viscosity and pressure curves of OX4

Figure 2a showed the viscosity curves of OX4 and X4, the maximum viscosity value of the X4 was about 30000Pa.s, where as the max viscosity value of OX4 was only about 16000Pa.s, indicating that the foaming agent interfered and inhibited the cross-linking reaction. It also can be seen that, the X4 viscosity increases rapidly due to the polyethylene chain-branching/cross-linking, and after reaching to

maximum, it decreased to a steady state level, this phenomenon maybe caused by the mechanical breakage of the branched/cross-linked chains formed during the reaction[18].

Figure 2b showed the pressure curves of OX4 and OB4, it can be seen that the foaming rate of OX4 decreased after 1min, obviously, but the OB4's pressure curve didn't have the decreasing inflection point, and foaming rate decreasing of OX4 resulted in the foaming reaction completion time was delayed when comparing with OB4. This difference was mainly explained by the cross-linking reaction

between polyethylene molecules and the formed three dimensional network structure reduced the foaming rate of polyethylene[19]. In addition, the pressure value of OB4 was same with OX4 when their foaming reaction completed, this appearance maybe explained by the foaming reaction earlier started than cross-linking reaction and pressure value was mainly determined by polyethylene matrix.

Figure 2 (a) Viscosity curves of X4 and OX4; (b) pressure curves of OX4 and OB4

In order to investigate the effect of foaming agent on cross-linking reaction furtherly, elongation rheometer was used to character the melt strength of X4, OX4 and OB4. The relationship between

temperature and melt strength were investigated with respect to the effects of temperature on the melt extensile stress and viscosity[20]. Test results were showed in Figure 3.

Figure 3 (a) Extensional rheology curves of X4 with different temperatures; (b) Extensional rheology curves of OX4 with different temperatures; (c) Extensional rheology curves of OB4 with different temperatures

In this experiment, the extensional strain rate was $0.1s^{-1}$, and testing temperature were 150°C, 170°C, 190°C, 210°C, 230°C, respectively. As shown in Figure 3a, at 150°C, the melt extensional stress and

viscosity of X4 has no obvious change, indicating that cross-linking reaction didn't start at this temperature; when the temperature raised to 170-190°C, the melt extensional stress and viscosity appeared enhancing

persistently with temperature increasing, this phenomenon showed that the XPE material was drawable and incomplete cross-linking during this temperature period. While, after the temperature $>200^{\circ}\text{C}$, the melt extensional stress was disappeared when test time $>15\text{s}$, this phenomenon indicated that the X4 specimen was broken and it was no longer drawable and the polyethylene molecules had been sufficient cross-linked.

Figure 3b was the melt extensional stress and viscosity curves of OX4 in 150°C – 230°C . It was obviously that the melt strengths also kept the raising trend with the reaction temperature increasing, this appearance indicated that cross-linking reaction promoted melt extensional and viscosity value. As a matter of fact, the melt strengths of OX4 were lower than X4 (Figure 3a), this phenomenon demonstrated that foaming agent OBSH interfered the cross-linking reaction and resulted in the melt extensional stress and viscosity decreased, and this result was consistent with Figure 2. The melt strength of OB4 was the lowest one when comparing with XPE and OX4, that's because OB4 didn't contain cross-linking agent. However, its melt strength was kept increasing with the temperature rising. This phenomenon maybe explained by the OBSH decomposition intermediates could interaction with polyethylene[21].

3.2 Effect of XPE on cellular structure

Gel content of polyethylene material means the cross-linking degree between molecule chains[22], at the same time, the cross-linking degree was interacted closely with the cellular structure and mechanical properties[23,24]. In this experiment, the gel content of X4, X6, X8, OX4, OX6 and OX8 were tested. As shown in Figure 4, the gel content of X4 as high as 76.26%, when the dilution ratio increases, the gel

content decreases significantly, the gel content of X8 was only 55.3%. This phenomenon indicating that less initiator causes lower cross-linking degree. After adding OBSH, the gel content decreased obviously compare to the counterpart without OBSH. The gel content of OX4 was only 24.19%, gel content of OX6 was 20.4% and the gel content of OX8 was even low to 13.7%, although the variation trend of gel content with dilution ratio was same with XPE system, but the huge reduction demonstrated that OBSH inhibited the cross-linking reaction, and this result was consisted with Figure 2 and Figure 3.

Figure 4 Gel content of X4, X6, X8, OX4, OX6 and OX8

XPE dilution ration not only affect gel content but also affect the viscosity and pressure during reaction process. Figure 5a showed the viscosity variation curves of OX4, OX6 and OX8. The viscosity value of OX4 was stay at $15000\text{Pa}\cdot\text{s}$, dilution ratio was 6, the viscosity value was about $12000\text{Pa}\cdot\text{s}$, and when dilution ratio added to 8, the viscosity value reduced to only $5000\text{Pa}\cdot\text{s}$. Test results indicated the higher the dilution ratio of OX, the lower the viscosity value, and it was similar to the gel content variation trend.

Figure 5(a) Viscosity curves and (b) Pressure curves of OX4, OX6 and OX8

Figure 5b was the pressure curves of OX4, OX6 and OX8, although their stable pressure values were all around 600–900kPa, but there have been subtle difference. During the period of foaming reaction started and cross-linking reaction was uninitiated (30 seconds–70 seconds), OX4 exhibited the fastest pressure growth rate, OX6's pressure growth rate was in the middle and OX8 presented the slowest pressure growth rate. This phenomenon was mainly caused by different MFI, the MFI of OX4 was 3.8, and it was higher than the MFI of OX8 (2.7). The higher MFI of polyethylene, the lower viscosity, and resulted in the higher gas diffusion rate[15]. After 70 seconds, cross-linking reaction started and the pressure rate slowed down, the pressure rate of OX4 slower than OX6, which was opposite with the initial pressure rate discipline. This appearance was mainly due to the cross-linking reaction, the more the dilution ratio, the less the cross-linking agent, the lower the viscosity, and the higher the pressure growth rate.

Cellular structure was the important property of polyethylene foam, which determined the properties of polyethylene foam. In this experiment, the cellular structure was tested by SEM. Figure 6a, Figure 6b and Figure 6c showed the SEM pictures of OX4, OX6 and OX8, respectively, these cells of the sequential foaming/cross-linking polyethylene system were all closed and circular structure, and the average diameters of OX4, OX6 and OX8 were about $320.9\mu\text{m}$, $353.5\mu\text{m}$ and $365.2\mu\text{m}$, respectively, cell sizes appeared normal distribution, their standard deviation of errors (σ) were $64.8\mu\text{m}$, $51.9\mu\text{m}$ and $98.4\mu\text{m}$, respectively, these phenomena indicated that cell size distribution was concentrated and mainly ranged in the period of $300\text{--}450\mu\text{m}$. In addition, the higher dilution ratio, the larger cellular size, this appearance indicated that the higher the dilution ratio of XPE, the lower the concentration cross-linking agent DBPH, the lower the cross-linking degree, and finally the larger the cellular size[23]. These phenomena demonstrated that the cellular size could be controlled by XPE dilution ratio.

Figure 6 SEM pictures and cell size distributions of (a)OX4, (b)OX6 and (c)OX8

For comparison, the foam structures of the samples without cross-linking agent were also studied. Figure 7a, Figure 7b and Figure 7c were the SEM pictures of OB4, OB6 and OB8, respectively, their corresponding diameters were $518.8\mu\text{m}$, $478.8\mu\text{m}$ and $466.1\mu\text{m}$ and these cellular sizes are much larger than OX samples. Although the cell were all closed structure, but their shape was not regular circular structure, some cellular size was large enough and some cellular size was small, their standard deviation of errors (σ) were

$256.8\mu\text{m}$, $194.4\mu\text{m}$ and $233.5\mu\text{m}$, respectively, the cell size distribution was scattered without cross-linking, which is possibly due to the lack of three-dimensional network structure restricting the random bubble growth during the foaming process. More interesting is that the higher the content of 7042, the smaller the size of cellular, which is consistent with the trend of MFI, and similar to that shown in Figure 5b. This comparing experiment demonstrated that the cross-linking agent not only control cellular size, but also control the cellular shape.

Figure 7 SEM pictures and cell size distributions of (a)OB4, (b)OB6 and (c)OB8

3.3 Effect of XPE on mechanical property

Due to closely relationship between cellular structure and mechanical property, the cross-linking polyethylene foaming materials' mechanical property will be transformed with the cellular size and structure changed which resulted by the different XPE dilution ratio[7,25]. In this experiment, flexural strength was taken as the typical mechanical property to test. Figure 8 showed that elasticity modulus of XPE polymers, OX polymers and OB polymer mixtures. The elasticity modulus of XPE4 was the highest (1547.9MPa), and as the dilution ratio increased, the elasticity modulus value decreased gradually (XPE8's elasticity modulus drop to 518.3MPa). This appearance was similarly with the gel content experiment (Figure 4). After adding the OBSH, elasticity modulus decreased obviously, the elasticity modulus of OX4 was 382.8MPa, similarly, OX8 was dropped to 183.5MPa. This experiment phenomenon was consistent with the melt strength of elongation rheometer test.

Figure 8 Elasticity modulus of XPE4, XPE6, XPE8, OX4, OX6, OX8, OB4, OB6 and OB8

In order to further clear the effect of XPE on cross-linking polyethylene foaming material's mechanical property, the flexural strengths of OB4, OB6 and OB8 were also measured. As shown in Figure 8, the elasticity modulus values of OB polymer mixtures were lower than OX polymers. The main reason for this phenomenon was that OB mixtures didn't contain cross-linking agent, OB polyethylene mixture molecular chains were independent with each other, and they didn't formed the effective and stability connection structure, finally resulted in the

weak mechanical property[26]. In addition to this, there was another phenomenon: the higher content of 7042, the lower elasticity modulus value. The appearance mainly caused by low elastic modulus of linear density polyethylene 7042[27].

4 Conclusions

In this paper, the sequential foaming/cross-linking system was developed, the cellular structure and mechanical property were researched. Test results showed that foaming reaction and cross-linking reaction interfered with each other. Additionally, the cross-linking reaction could control cellular structure during foaming process and finally improved mechanical strength.

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