# Photocrosslinking of Halogen-Free Flame-Retarded Ethylene-Vinyl Acetate Copolymer by Phosphorous-Nitrogen Compound NP28

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**ABSTRACT:** The photoinitiated crosslinking of halogen-free flame-retarded ethylene-vinyl acetate copolymer (EVA) by the phosphorous-nitrogen compound NP28 in the presence of photoinitiator and crosslinker and characterization of the related properties have been investigated by gel determination, heat extension test, thermogravimetric analysis (TGA), mechanical measurement, and thermal aging test. The photocrosslinking efficiency of EVA/NP28 blend and various factors affecting the crosslinking process, such as photoinitiator, crosslinker, NP28 content, and irradiation temperature, were studied in detail and optimized by comparison of gel content. The results show that the EVA/NP28 blend filled with 28.2 wt % NP28 with a thickness of 1.6 mm is homogeneously photocrosslinked to a gel content of above 80 wt % with 4.8 s UV-irradiation under optimum conditions. The data from TGA, mechanical measurement, and thermal aging test give evidence that the thermal stability and mechanical properties of photocrosslinked EVA/NP28 blend are much better than those of the unphotocrosslinked one. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 562–569, 2009

**Key words:** photocrosslinking; ethylene-vinyl acetate copolymer; phosphorous-nitrogen compound NP28; halogen-free flame-retardant

## INTRODUCTION

Ethylene-vinyl acetate copolymer (EVA) is extensively used in many fields, especially in the cable industry as excellent insulating materials with good physical and mechanical properties.[1] However, its usage is often limited because of its high flammability, low distortion temperature, and mechanical strength.

It is well known that the flame retardancy of EVA can be improved by the addition of flame retardants. At present, there is a trend to developing halogen-free flame-retarded polymeric materials with low emission of smoke and poisonous gas because of environmental and safety concerns. Thus, as main halogen-free flame retardants, metal hydroxides[2–4] and intumescent flame retardant (IFR) additive[5–7] are used. Very good efficiency in flame retardancy of polyolefin can be achieved with IFR systems. However, a higher loading of an IFR additive is needed in comparison with that of some halogen-containing flame retardants. This results in lowered mechanical properties of the flame-retarded compounds.[8]

To improve its mechanical strength and thermal resistance properties, the crosslinking of EVA is a useful method.[9] There are three methods for crosslinking of EVA at present: high-energy irradiation ([60]Co γ-rays or electron beam) crosslinking,[10,11] chemical crosslinking (peroxide and silane crosslinking),[12–14] and photoinitiated crosslinking.[15] Compared with the high-energy irradiation and chemical crosslinking, the major advantages of photocrosslinking are low cost of the equipment, low energy consumption, easy operation and maintenance, high production efficiency, and excellent electronic and mechanical properties. However, because the flame retardants in polyolefin resin reflect the UV light and reduce light penetration, the photocrosslinking of halogen-free flame-retarded polyolefin by IFR additives has not been studied at present, as we are aware.

Recently, the microwave excited UV lamp with UV light intensity higher than 1800 mW/cm$^2$ has been used as UV source in the photocrosslinking of polyolefin. It was found that the pure polyolefin sample with a thickness of 15 mm and colored

polyolefin sample can be homogenously photocrosslinked to satisfactory gel content for the industrial application as insulated materials of wire and cable under the irradiation of the microwave excited UV lamp.[16,17] In this work, by using the microwave excited UV lamp as UV source, the halogen-free flame-retarded EVA blends by a phosphorous-nitrogen compound, NP28, which is an excellent IFR additive for polyolefin according to the previous works in our laboratory,[18,19] was photocrosslinked in the presence of photoinitiator and crosslinker. The photocrosslinking reaction parameters, optimum conditions and the crosslink homogeneity of EVA/NP28 blend, and the corresponding thermal and mechanical properties of photocrosslinked EVA/NP28 blend were investigated by gel determination, heat extension test, thermogravimetric analysis (TGA), mechanical measurement, and thermal aging test.

## MATERIALS AND METHODS

### Materials

EVA copolymer containing 18 wt % vinyl acetate was supplied by Samsung Total Petrochemical (Korea). The phosphorus-nitrogen compound NP28, containing 15.6 wt % P and 27.5 wt % N, with a particle size of 400 mesh was obtained from Weizheng Fine Chemical Inc. (Zhuhai, China). Five kinds of photoinitiator were benzophenone (BP) from Shanghai Reagent Plant No. 1 (Shanghai, China); benzyl dimethyl ketal (BDK), 1-hydroxy-cyclohexyl-phenyl-ketone (Irgacure 184), 2-methyl-1-[4-(methylthio)-phenyl]-2-(4-morpholinyl)-1-propanone (Irgacure 907), and 2-hydroxy-1-[4-(2-hydroxyethoxy)phenyl]-2-methyl-1-propanone (Irgacure 2959) from Ciba (Basel, Switzerland). Four kinds of crosslinker were triallyl isocyanurate (TAIC) from the Anhui Institute of Chemical Engineering (Hefei, China); pentaerythritol triacrylate (PETA), neopentylglycol diacrylate (NPGDA), and trimethylolpropane triacrylate (TMPTA) from UCB (Brussels, Belgium). Antioxidant Irganox 1010 was obtained from Beijing Jiacheng Additives Research Institute (Beijing, China).

### Sample preparation

EVA resin with the desired amount of NP28, photoinitiator, crosslinker, and antioxidant was mixed for 10 min at 140°C with an XSS-300 rheomixer (Shanghai, China). After mixing, the samples were hot-pressed into sheets under 10 MPa for 5 min at 140°C with a hot press. The thickness of samples was 1 mm, except where mentioned differently.

### Sample irradiation

The samples were preheated and then UV-irradiated in LC6B Benchtop Conveyer equipped with F300SQ lamp system ("D" bulb, the light intensity 1800 mW/cm$^2$) constructed by Fusion UV system (USA). The distance between the F300SQ lamp and the surface of sample was 5.3 cm. The irradiation time was controlled by the speed of conveyer belt. The preheated temperature was usually 120°C unless stated otherwise.

### Measurement

#### Limiting oxygen index

The limiting oxygen index (LOI) values were measured by using a HC-2-type instrument (Nanjing Analytical Instrument Factory, China) on the specimens measuring $120 \times 6.5 \times 3$ mm$^3$, according to ASTM D2863.

#### UL-94 test

The UL-94 vertical burning test was carried out by using a CZF-1 type instrument (Nanjing Analytical Instrument Factory) on the $127 \times 12.7 \times 3$ mm$^3$ specimens, according to ASTM D635.

#### Gel content

The gel content was determined by extracting the UV-irradiated sample ($m_1$) in boiling xylene for 48 h. The solvent was renewed after the first 24 h. After being dried, the insoluble residue ($m_2$) was weighted. The gel content was calculated as $m_2/m_1 \times 100\%$. Three samples were analyzed to determine the average gel content for a given set of irradiation conditions. This method of measuring gel content has been proved to give reproducible results[10,20]

#### Heat extension test

The heat extension test was carried out according to the specifications of IEC 60811. The dumbbell-shaped specimens were strained with a stress of 0.2 MPa in an oven at $200 \pm 1$°C for 15 min, after which the elongation under load was measured. Then, the stress was removed, and the specimens were kept in the oven for another 5 min before being cooled at the ambient temperature. On the cooled samples, the permanent elongation was then measured.

#### Thermogravimetric analysis

The thermogravimetric analysis (TGA) was performed on a Shimadzu TGA-50H thermoanalyzer at a scan rate of 10°C/min under the air flow rate of $2 \times 10^{-5}$ m$^3$/min.

#### Combustion analysis for char-forming rate and char yield

The combustion analysis was conducted with a HC-2-type instrument used to measure the LOI at the

oxygen concentration of 3% above LOI, according to Refs. 21 and 22. The specimen and char were carefully weighed before and after burning. The time ($t_1$) from the ignition to the completion of burning or extinguishing was recorded. The char yield was calculated as $W_3/(W_1-W_2) \times 100\%$, where $W_1$ was the total weight of the specimen, $W_2$ was the weight of unburned specimen, and $W_3$ was the weight of char. The char-forming rate was calculated as $W_3/t_1$.

## Thermal aging test

The thermal aging test was carried out according to ASTM D794 by suspending the dumbbell-shaped specimens in an air circulating oven at 145°C for 168 h.

## Mechanical properties

The mechanical properties were measured with a universal testing machine (Instron 1185) at temperatures 25°C ± 2°C. The crosshead speed was 25 mm/min. Dumbbell-shaped specimens were prepared according to ASTM D412. The tensile strength and elongation at break were recorded.

## RESULTS AND DISCUSSION

### Flammability of EVA/NP28

The LOI and UL-94 tests are widely used to evaluate the flammability of flame-retarded materials. Table I lists the related LOI and UL-94 data of EVA/NP28 blends with different NP28 content. It can be seen from Table I that the LOI value of EVA/NP28 rapidly increased from 18 to 28% when the NP28 content increased from 0 to 16.4 wt % and then slowly increased to 33% with further increasing the NP28 content to 30.6 wt %. The UL-94 test result shows that the EVA/NP28 blend passed UL-94 V-0 rating test when the NP28 content was higher than 28.2 wt %. These results indicate that the NP28 is a good flame-retardant additive for EVA, and 28.2 wt % is an adequate amount to achieve the satisfactory flame-retardant properties for the EVA/NP28 blend. In the following work, the photoinitiated crosslinking of EVA/NP28 blend filled with 28.2 wt % NP28 was studied.

**TABLE I**
**Effect of NP28 Content on the Flammability of EVA/NP28 Blend**

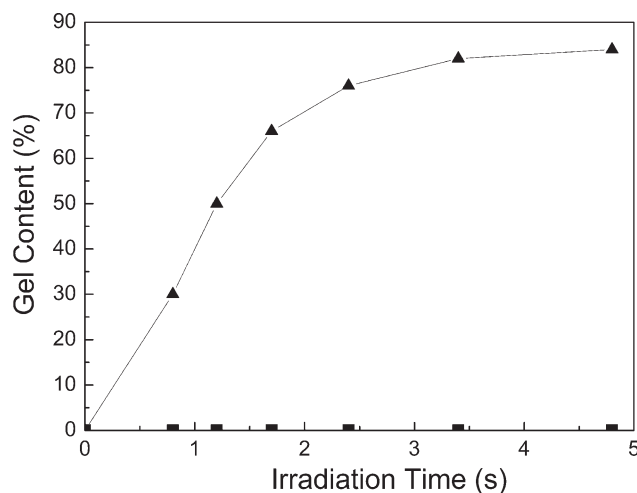| NP28 content (wt %) | 0 | 16.4 | 22.7 | 25.5 | 28.2 | 30.6 |
|---|---|---|---|---|---|---|
| LOI (%) | 18 | 28 | 30 | 31 | 32 | 33 |
| UL-94 test | Fail | V-2 | V-1 | V-1 | V-0 | V-0 |

**Figure 1** Kinetics of gel formation during the photocrosslinking of EVA/NP28. (■) No additives, (▲) BP: 0.7 wt %, TAIC: 0.7 wt %.

## Characteristics of photocrosslinking of EVA/NP28

Gel content is shown in Figure 1 as a function of UV-irradiation time for the EVA/NP28 blend. It is evident that no crosslinking occurred for the EVA/NP28 sample without photoinitiator and crosslinker because no measurable gel was formed after UV irradiation. However, in the presence of photoinitiator BP and crosslinker TAIC (0.7 wt % each), the EVA/NP28 sample was quickly photocrosslinked by UV-irradiation, which is seen from the rapid increase of gel content with increasing irradiation time. The gel content of about 84 wt % was obtained for the EVA/NP28 sample when the UV irradiation time increased to 4.8 s. The kinetic characteristics of photocrosslinking for the EVA/NP28 blend has the following features: (1) the initial rate of crosslinking during the first 2.4 s is very fast; (2) after 2.4 s, the rate decreases to a certain level; and (3) the final gel content is almost constant.

The heat extension test can provide a quick and accurate determination on the crosslinking of polymeric samples and can be correlated with gel content. The results of heat extension test for the EVA/NP28 blend UV-irradiated for different times are listed in Table II. It shows that the unirradiated EVA/NP28 failed the test immediately. EVA/NP28 irradiated for 1.2–2.4 s still failed the test but maintained longer. However, the sample UV-irradiated for 3.4 s passed the test, which indicates that the adequate crosslinking network was formed in the EVA/NP28 blend. The value of heat extension gradually decreased with the further increase of UV irradiation time. The results of the heat extension test show that the EVA/NP28 blend can be photocrosslinked to satisfactory gel content for the industrial application as insulated materials of wire and cable.

| Irradiation time (s) | 0 | 1.2 | 1.7 | 2.4 | 3.4 | 4.8 |
|---|---|---|---|---|---|---|
| Heat extension (%) | Failed immediately | Failed in 50 s | Failed in 2 min 40 s | Failed in 5 min 30 s | 90 | 60 |
| Permanent extension (%) | – | – | – | – | 7.5 | 5 |

## Reaction condition

The effects of the reaction condition such as photo-initiator, crosslinker, NP28 content, and irradiation temperature on the photoinitiated crosslinking of EVA/NP28 have been studied.

### Photoinitiator

Five different photoinitiators (BP, BDK, Irgacure 184, Irgacure 907, and Irgacure 2959) were selected and used in this work. Figure 2 compares their photoinitiating efficiencies for EVA/NP28 blend with 0.7 wt % photoinitiator and no added crosslinker. Apparently, it is evident that all the five photoinitiators can initiate the photocrosslinking of EVA/NP28 because the gel contents of all the five EVA/NP28 samples increased with increasing the UV-irradiation time. However, the initial rates and final gel contents were different and decreased in the following order:

$$BP > BDK > Irgacure\ 2959 > Irgacure\ 907 > Irgacure\ 184$$

The results show that BP is the most efficient one among the five photoinitiators.

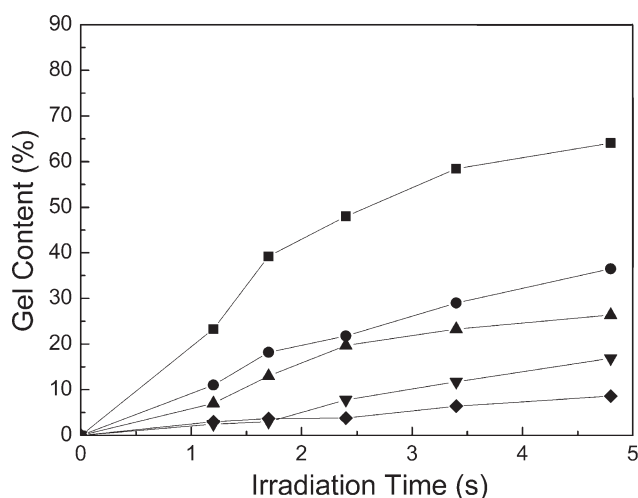Figure 3 shows the dependence of gel content on the concentration of BP for the EVA/NP28 sample.

It can be seen that the optimum concentration of BP is about 0.7 wt %. A further increase in the amount of photoinitiator does not increase the gel content. The sample with 1.4 wt % BP shows an even lower gel content. This is interpreted as being due to two effects. The BP in the ground state and triplet state absorbs UV light in the region of n-π transition, which will screen the UV light and act as an "autoretardant." In addition, some ketyl radicals formed by photoinitiators will combine with polymer radicals and prevent the crosslinking of EVA. Both effects are expected to increase at higher BP concentration.

### Crosslinker

The effect of four different crosslinkers on the gel content of EVA/NP28 blend is compared in Figure 4. Apparently, the addition of a multifunctional crosslinker accelerates the crosslinking process. The initial rate and final gel content of EVA/NP28 sample with crosslinker were higher than those of EVA/NP28 sample without crosslinker. TAIC is the most efficient among the four crosslinkers, as shown in Figure 4. This is because TAIC with a cyanurate group has better solubility and is more homogeneously distributed in polyolefine than PETA or TMPTA.[23] In addition, TAIC promotes deeper penetration of crosslinking in polyolefin and thus enhances the efficiency of photocrosslinking.[23,24]
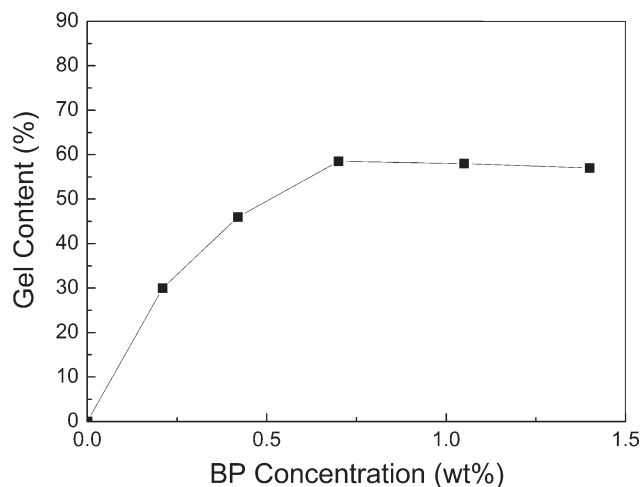
**Figure 2** Comparison of the photocrosslinking of EVA/NP28 blends with 0.7 wt % initiators and no crosslinker added. (■) BP, (●) BDK, (▲) Irgacure 2929, (▼) Irgacure 907, (◆) Irgacure 184.

**Figure 3** Effects of BP concentration on the gel contents of EVA/NP28 blend. Irradiation time: 3.4 s.
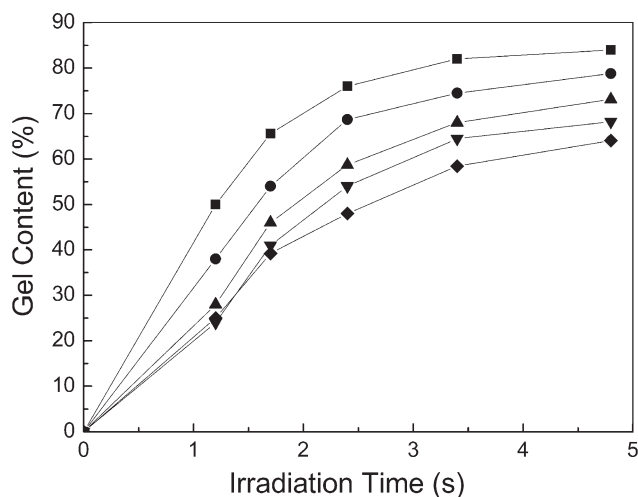
**Figure 4**  Effects of crosslinker on the photocrosslinking of EVA/NP28 blend. BP: 0.7 wt %, crosslinker: 0.7 wt %. (■) TAIC, (●) TMPTA, (▲) PETA, (▼) DPGDA, (◆) No crosslinker.

The effect of TAIC concentration on the gel content of the EVA/NP28 blend is shown in Figure 5. The results demonstrate that 0.7 wt % TAIC is an optimal amount for gel formation. The further addition of TAIC has only a minor effect. Even at 1.4 wt % TAIC, the gel contents of EVA/NP28 decrease. This situation suggests that excessive TAIC can form a low-molecular-weight TAIC homopolymer,[25] which is easily extracted by xylene. Therefore, the TAIC homopolymer reduced the crosslinking between TAIC and EVA and thus caused a reduction of the gel content.

## NP28 content

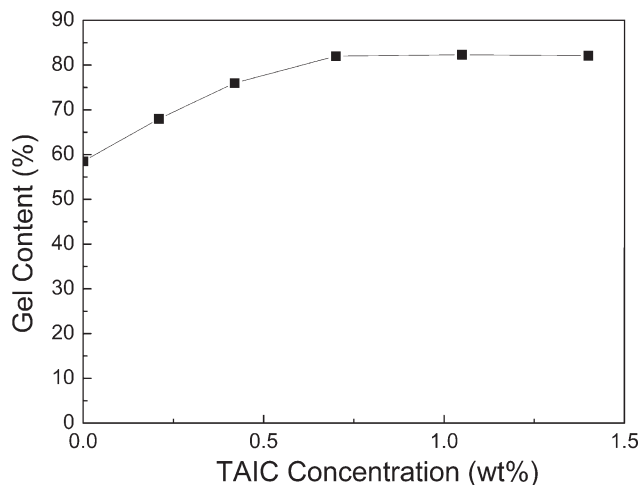The effect of NP28 content on the gel formation of EVA/NP28 blend is shown in Figure 6. It can be



**Figure 5**  Effect of TAIC concentration on the gel contents of EVA/NP28 blend. BP: 0.7 wt %, irradiation time 3.4 s.
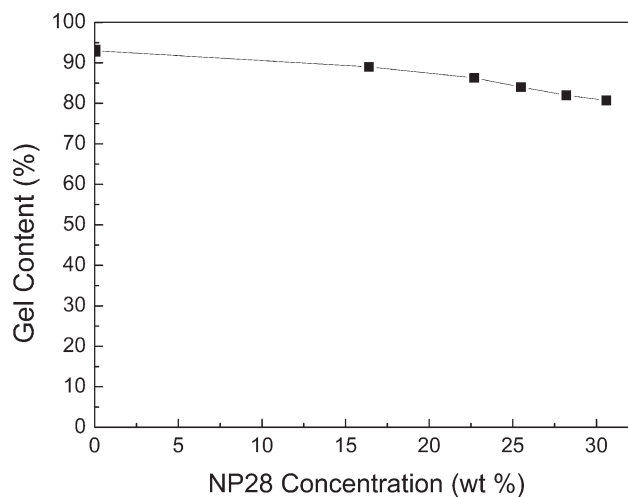


**Figure 6**  Effect of NP28 content on the gel contents of EVA/NP28 blend. BP: 0.7 wt %; TAIC: 0.7 wt %; irradiation time: 3.4 s.

seen that the NP28 had a negative effect on the photocrosslinking of EVA. The gel content of photocrosslinked EVA/NP28 decreased from 93 wt % to 82 wt % when the NP28 content increased from zero to 28.2 wt %. The decrease of gel content is probable due to the reflection of NP28 in the EVA/NP28 blend decreasing the UV energy absorption of photoinitiator and the amount of radicals formed by the photoinitiator.

## Irradiation temperature

The gel contents of photocrosslinked EVA/NP28 blend irradiated at different temperatures were listed in Table III. It can be seen that the gel content increased with the increase of irradiation temperature. The increase of gel content can be interpreted as being due to the free volume in EVA and the motion rate of EVA segments increased with increasing the irradiation temperature. These two factors accelerate the molecular motions of polymer chains and thus speed the combination of macroradicals to form the crosslinks. The results of the heat extension test for the photocrosslinked EVA/NP28 blend irradiated at different temperatures were also listed in Table III. It shows that the photocrosslinked EVA/NP28 blend irradiated at temperatures higher than 120°C passed the heat extension test, which indicates the formation of adequate crosslinking network for the industrial application.

## Homogeneity of crosslink

The homogeneity of crosslink is of great importance for the industrial application of photocrosslinked EVA/NP28 material. It was studied by cutting from the top surface of the thick sample (3 mm) into

**TABLE III**
**Gel Contents and Heat Extension of EVA/NP28 Blend UV-Irradiated for 3.4 s at Different Temperature**

| Irradiation temperature (°C) | 50 | 80 | 100 | 110 | 120 | 130 | 140 |
|---|---|---|---|---|---|---|---|
| Gel content (%) | 25 | 50 | 64 | 75 | 82 | 85 | 87 |
| Heat extension (%) | Failed immediately | Failed in 30 s | Failed in 2 min 10 s | Failed in 5 min | 90 | 70 | 60 |
| Permanent extension (%) | – | – | – | – | 7.5 | 6 | 5 |

layers, and the gel content of each layer was measured. The results for the photocrosslinked EVA/NP28 blend are shown in Figure 7. It can be seen that satisfactory homogeneity of photocrosslinking for EVA/NP28 blend exists within the range of 1.6 mm thickness. The gel content at 1.6 mm from the surface was 78 wt %, which is only 6 wt % lower than the gel content (84 wt %) at the surface. However, when the depth of penetration was further increased, the crosslinks of photocrosslinked EVA/NP28 became inhomogenous rapidly. The gel content decreased to 70 and 18 wt %, respectively, when the depth of penetration increased to 2.0 and 3.0 mm. The results indicated that the method of photocrosslinking is suitable for the thin sheets of EVA/NP28 blend with a thickness of less than 1.6 mm.

### Thermal stability

Figure 8 shows the TG(A) and DTG(B) curves of EVA/NP28 blend UV irradiated for different time. It can be seen from Figure 8(A) that the weight loss of unphotocrosslinked and photocrosslinked EVA/NP28 samples all occur in four steps. The first step is considered to involve the initial stage of the decomposition of NP28 in the range of 200–270°C.
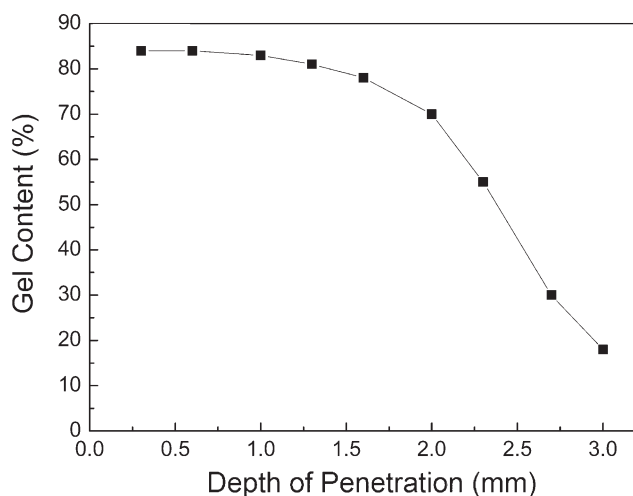
The second step is due to the loss of acetic acid from EVA at temperatures between 290 and 390°C.[26,27] The third step is attributed to the scission of the polymeric main chain. The temporary carbonaceous residue (or char) formed during this step increased with increasing the irradiation time. The EVA/NP28 samples irradiated for 2.4 and 4.8 s have 30.6 and
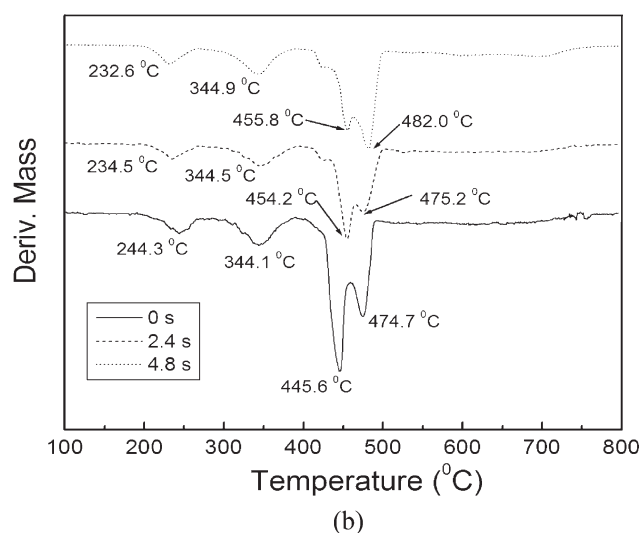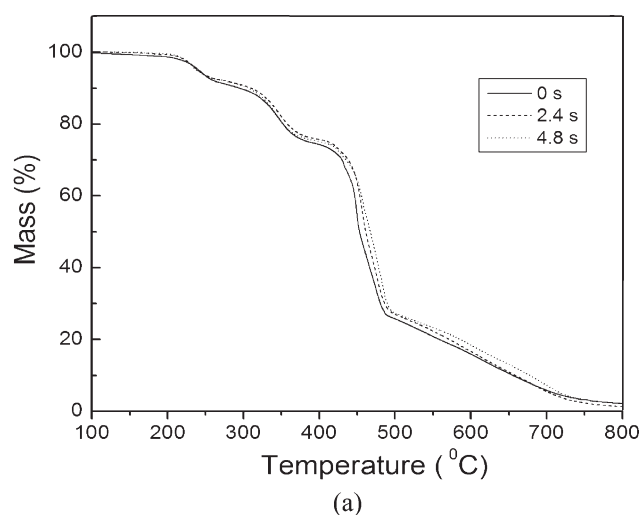


(a)



(b)

**Figure 8** (a) TGA and (b) corresponding DTG curves of EVA/NP28 blend irradiated for different time. BP: 0.7 wt %; TAIC: 0.7 wt %.



**Figure 7** Photocrosslinking homogeneity of EVA/NP28 blend.

**TABLE IV**
**Char-Forming Rate and Char Yield of EVA/NP28 Blend UV-Irradiated for Different Time**

| Irradiation time (s) | Char-forming rate (mg/min) | Char yield (%) | LOI (%) |
|---|---|---|---|
| 0 | 90 | 23.9 | 32 |
| 2.4 | 93 | 24.7 | 33 |
| 4.8 | 95 | 25.1 | 34 |

**TABLE V**
**Tensile and Thermal Aging Properties of Photocrosslinked EVA/NP28 Blend**

| Irradiation time (s) | 0 | 1.7 | 3.4 | 4.8 |
|---|---|---|---|---|
| Tensile strength (MPa) | 7.1 | 8.2 | 9.9 | 12.2 |
| Elongation at break (%) | 860 | 860 | 850 | 770 |
| Retention of tensile strength (%) | – | 98.3 | 96.0 | 93.4 |
| Retention of elongation at break (%) | – | 95.9 | 94.1 | 90.9 |

33.7 wt % residues, respectively, while the unirradiated EVA/NP28 sample has 28.0 wt % residues. The fourth step is owing to the volatilization of the carbonaceous residue formed during the degradation of the polymeric main chain.[27]

From the DTG curves of Figure 8(B), it is observed that the pyrolysis temperature corresponding to the first DTG peak of EVA/NP28 blend decreased 9.8 and 11.7°C, respectively, when it was irradiated for 2.4 and 4.8 s, which indicates that the thermal stability of NP28 decreased under UV irradiation. However, the thermal stability of the main chain in the EVA/NP28 blend increased after photocrosslinking, which is seen from the increase of the pyrolysis temperatures the third and fourth DTG peaks.

### Char-forming rate and char yield

The concept of char-forming rate was proved to be able to explain the variation of flame retardancy of intumescent flame-retardant polymer materials.[21,22] Table IV lists the char-forming rate and char yield of the EVA/NP28 blend UV-irradiated for different times. As it can be seen, the char-forming rate and char yield increased from 90 mg/min and 23.9% for the unphotocrosslinked EVA/NP28 to 95 mg/min and 25.1% for the sample irradiated for 4.8 s, respectively. The faster the char is formed on the burning surface of a polymer sample during its combustion, the quicker the fire propagation will be stopped and the better flame retardancy thus obtained. It is why the LOI value of photocrosslinked EVA/NP28 increased with the increase of irradiation time (Table IV).

### Mechanical and thermal aging properties

Table V lists the tensile properties of the EVA/NP28 blend UV-irradiated for different times. As it can be seen, the tensile strength increased sharply from 7.1 MPa for the unphotocrosslinked EVA/NP28 to 12.2 MPa for the sample irradiated for 4.8 s. However, the elongation at break slightly decreased from the original 860 to 770% after 4.8 s irradiation. Both are attributed to the increase of crosslinking density in the crosslinked sample.

The retention of tensile properties of the EVA/NP28 blend thermal aged at 145°C for 168 h are also listed in Table V. The unirradiated EVA/NP28 sample underwent fatal degradation during thermal aging test. However, the photocrosslinked EVA/NP28 samples exhibited very good thermal aging properties. The retention of tensile strength and elongation at break for the photocrosslinked EVA/NP28 samples were above 90%.

### CONCLUSIONS

1. The results of gel content and heat extension test demonstrate that the halogen-free flame-retarded EVA/NP28 blend can be photoinitiated crosslinked by UV-irradiation in the presence of photoinitiator and crosslinker.
2. By optimizing reaction parameters such as photoinitiator, crosslinker, and irradiation time, the EVA/NP28 blend with a thickness of 1.6 mm can be homogeneously photocrosslinked in short irradiation times of 3–5 s with a satisfactory crosslinking degree of above 80% gel content.
3. The TGA results demonstrate that the thermal stability of main chain in EVA/NP28 can be increased by the photocrosslinking.
4. The photocrosslinked EVA/NP28 samples have higher tensile strength and better thermal aging properties than the unphotocrosslinked one.

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