Effect of co-monomer on the adhesive performance of PP-g-MAH between cast polypropylene film and aluminum foil

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Abstract

Three functionalized polyolefinic (PPs) were successfully prepared for use as the inner adhesive layer of aluminum/polymer laminated film by melt grafting of maleic anhydride (MAH) on PP with styrene (St), diallyl phthalate (DAP) and triallyl isocyanurate (TAIC) as co-monomers, respectively. The effects of these co-monomers (St, DAP and TAIC) on the grafting degree of MAH, gel content, thermal property and adhesive performance of MAH grafted PP (PP-g-MAH) were investigated. The results show that the T-peeling and heat-sealing strengths of TAIC-assisted PP-g-MAH (PP-g-MAH/TAIC) are 28.6 and 70.4 N / 15 mm, which are increased by 33.6% and 20.8% compared to DAP-assisted PP-g-MAH (PP-g-MAH/DAP), and by 142.4% and 81.0% compared to St-assisted PP-g-MAH (PP-g-MAH/St), respectively. The grafting degree of MAH reaches a maximum of 2.3 wt% in PP-g-MAH/TAIC, which leads to a higher crystallization temperature than PP-g-MAH/DAP and PP-g-MAH/St. PP-g-MAH/TAIC also has better thermal stability than the other two functionalized PPs, as TAIC can effectively reduce PP chain scission and promote the formation of a cross-linked structure during the melt grafting.

1. Introduction

Aluminum (Al) and its alloys have a wide range of applications in aerospace, automobile, building and packaging materials owing to their high strength ratio, good formability and plasticity [1–3]. Despite its wide use in the packaging industry due to the excellent barrier performance to moisture and air [4], Al foil is easy to tear and undergo galvanic corrosion when contacting with other metals [5, 6]. Thus, Al/polymer laminated (Al/P) film has been developed to combine the advantages of Al foil and polymers, especially resistance to tearing and galvanic corrosion, which makes it an appealing soft packaging material of lithium-ion battery. The packaging material is often composed of the nylon, adhesive, Al foil, conversion coating, adhesive and cast polypropylene (CPP) layers from the outside to the inside, as figure 1 shows. Previous studies have shown that the T-peeling and heat-sealing strengths of Al/P film used in lithium-ion battery should be further improved, as interfacial delamination may occur during the contact with electrolyte [2, 7]. It is accepted that both modification of the adhesive and surface treatment of Al foil are effective methods to improve the adhesive properties of Al/P film.

There is an ongoing interest in the adhesive between Al foil and CPP layers such as epoxy resin [5], polyurethane [8] and modified polypropylene (PP) [2], the last of which is known to be the most appropriate adhesive among them. Many modified PPs such as PP-g-MAH, glycidyl methacrylate grafted PP and acrylic grafted PP are used in Al/P film, and PP-g-MAH is particularly effective in improving the adhesive properties of Al/P film and corrosion resistance to electrolyte [7]. PP-g-MAH is commonly prepared by melt free-radical grafting due to the relatively low toxicity. However, chain scission of PP usually occurs during the grafting, which may result in a decrease of grafting degree and molecular weight. Therefore, many attempts have been made to increase the grafting degree of MAH and reduce PP chain scission by introducing a co-monomer [9, 10].
Previous studies have shown that the presence of styrene (St) during the grafting can effectively reduce PP chain scission and increase the grafting degree of MAH [11, 12]. Compared with St, the co-monomer with multifunctional groups shows higher reactivity towards PP macroradicals and MAH, which may result in a higher grafting degree of MAH and less chain scission. In addition, numerous functional groups are introduced on PP with the assistance of these co-monomers, leading to stronger interaction and higher adhesive strength between the grafted PP and Al foil.

The surface treatment of Al foil can also improve the adhesive properties and corrosion resistance of Al/P film by the physical and chemical reactions between adhesives and the treated Al foil. However, chemical conversion treatments appear to be better than physical methods such as mechanical grinding and grit blasting to improve the adhesion between the adhesive and Al foil, which may be ascribed to the formation of strong and stable bonds between the conversion coating and the adhesive layer [7, 13]. Chromate conversion coating has been widely used in the surface treatment of Al foil for preparing Al/P film with high corrosion resistance, T-peeling and heat-sealing strengths [5, 14, 15]. However, hexavalent chromium is highly toxic and may cause severe environmental pollution, and thus many environmentally friendly alternatives have been developed, such as titanium (Ti) [7], molybdate [16, 17], zirconium [18] and cerium conversion coatings [19, 20]. Xia et al [7] prepared an environmentally friendly Ti conversion coating on the Al foil by a simple method, and the resulted Al/P film showed high peeling strength and corrosion resistance. Thus, Ti conversion coating may be a promising alternative to chromate treatment.

Although functionalized PPs are often used as adhesives to improve the adhesive performance between Al foil and PP [2, 21, 22], there are few studies on the effects of co-monomer on the properties of functionalized PP and the adhesive properties of functionalized PP with the Ti treated Al foil. Thus, the main objective of this study is to prepare PP-g-MAH for use as the adhesive layer in Al/P film by melt grafting reaction with the assistance of styrene (St), diallyl phthalate (DAP) and triallyl isocyanurate (TAIC), respectively. Purified functionalized PPs were characterized by Fourier transform infrared (FTIR) spectroscopy, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA), and Ti treated Al foil was characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and contact angle measurement. In addition, the adhesive property of functionalized PPs with the Ti treated Al foil was measured by T-peeling test.

2. Experimental

2.1. Materials

Al foil (AA 8021) used in experiments was supplied by Dongding Industrial Co., Ltd (Henan, China). CPP film was supplied by Lianyungang Zhongjin Medical Co., Ltd (Jiangsu, China) and used as the sealant layer. PP (K8003) with a melt flow index of 2.5 g /10 min at 230 °C /2.16 kg was supplied by Dushanzi Petrochemical Co., Ltd (Xinjiang, China). DAP, TAIC, MAH and dicumyl peroxide (DCP) were purchased from Titan Scientific Co., Ltd (Shanghai, China). St and Hexafluorotitanic acid (H2TiF6) were purchased from Aladdin Reagent Co., Ltd (Shanghai, China). All reagents are analytically pure grade (AR).

2.2. Preparation of adhesives

St-assisted PP-g-MAH (PP-g-MAH/St), DAP-assisted PP-g-MAH (PP-g-MAH/DAP) and TAIC-assisted PP-g-MAH (PP-g-MAH/TAIC) were prepared at 180 °C and a screw speed of 60 rpm for 8 min in a torque rheometer (Rheocord 300 P, Haake, Karlsruhe, Germany). The mass ratio of PP/DCP/MAH and the molar ratio of MAH/co-monomer (St, DAP or TAIC) were 100/0.4/6 and 1/1, respectively.
Functionalized PPs were dissolved in xylene and then precipitated in acetone, and the obtained product was purified with acetone at 80 °C for 48 h by Soxhlet extraction and then dried at 120 °C for 24 h.

2.3. Treatment of Al surface
Before Ti treatment, Al foil was degreased in an alkaline solution composed of anhydrous sodium carbonate (Na₂CO₃, 10 g L⁻¹), sodium metasilicate (Na₂SiO₃, 10 g L⁻¹), sodium fluoride (NaF, 10 g L⁻¹) and sodium phosphate (Na₃PO₄, 20 g L⁻¹) at 60 °C for 5 min, and then washed with distilled water. The alkali treated Al foil was immersed in 2.3 g L⁻¹ H₂TiF₆ solution with a pH of 1.9 for 3 min at 25 °C, and then washed with distilled water and dried at 80 °C for 5 h.

2.4. Adhesive film on Al foil and lamination with CPP
Al/PP-g-MAH composite for T-peeling test was prepared at 180 °C and 10 MPa for 1 min using a BL-6170-A-25J plate vulcanizing press (Bolon Precision Testing Machines, Dongguan, China), and then cooled to room temperature. Al/PP-g-MAH/CPP composite for heat-sealing test was prepared by laminating CPP film with the Al/PP-g-MAH composite, and then sealed at 180 °C and 300 kPa for 3 s using a HFT-H3 heat-sealing machine (Bogoo Experimental Apparatus, Jinan, China). The preparation and structures of samples for T-peeling and heat-sealing tests are shown in figure 2.

2.5. Characterization of adhesives
2.5.1. Grafting degree of MAH and gel content
Purified functionalized PPs were dissolved in xylene, and then excessive potassium hydroxide (KOH)-ethanol solution was added into the mixture for 1 h. Then, the mixture was titrated using hydrochloric acid (HCl)-isopropanol solution with 1% phenolphthalein as the indicator. The grafting degree (G) was calculated from equation (1):

\[ G = \left( \frac{C_1 V_1 - C_2 V_2}{2 M} \right) \times \frac{98.06 \times 10^{-3}}{} \times 100 \% \]  

where \( C_1 \) and \( C_2 \) represent the concentrations (mol/l) of the KOH-ethanol and HCl-isopropanol solutions, \( V_1 \) and \( V_2 \) are their volumes (mL), and \( M \) (g) is the mass of purified samples, respectively.

Purified functionalized PPs were wrapped with a piece of filter paper, and then packed in a copper net. The net was extracted in excessive xylene at 140 °C for 24 h, and then taken out and rinsed several times with hot xylene. After that, the net was dried at 120 °C for 24 h. The gel content (Gel) was calculated from equation (2):

\[ \text{Gel} = \left( \frac{W_1 - W_6}{W} \right) \times 100 \% \]  

where \( W \) is the weight of the functionalized PPs, \( W_6 \) represents the total weight of filter paper and copper net, and \( W_1 \) represents the weight of filter paper and copper net with the sample after extraction.
2.5.2. FTIR analysis
The FTIR spectra of purified functionalized PPs were recorded at a scan number of 32 and a resolution of 4 cm\(^{-1}\) on a Nicolet 6700 FTIR spectrometer (Thermo Fisher Scientific, New York, USA). The sample used in FTIR analysis was prepared by hot pressing.

2.5.3. Thermal analysis
DSC measurements were performed using a differential scanning calorimeter (Q2000, TA, USA) under a nitrogen atmosphere. The temperature was calibrated with indium. Samples (8–12 mg) were heated from 30 to 200 °C at 20 °C min\(^{-1}\) to eliminate the former thermal history, maintained at that temperature for 2 min, and then cooled to 30 °C at 10 °C min\(^{-1}\). After that, they were re-heated to 200 °C at the same rate.

TGA was carried out on a thermogravimetric analyzer (STA 409PC, Netzsch, Germany) at 10 °C min\(^{-1}\) from room temperature to 600 °C under a nitrogen atmosphere.

2.6. Characterization of Ti conversion coating
2.6.1. Coating analysis
The surface morphologies and compositions of Al foils before and after treatment were characterized by SEM (S4800, Hitachi, Tokyo, Japan) and EDS (QUANTAX 400–30, Falcon, Germany) at an acceleration voltage of 15 kV, respectively.

2.6.2. Contact angle measurement
The contact angle on the untreated and treated Al foil surfaces was evaluated using a JC2000D2 contact angle measurement apparatus (Zhongchen Digital Technic Apparatus, Shanghai, China).

2.7. Measurement of T-peeling and heat-sealing strengths
The T-peeling strength between adhesive and Al foil and the heat-sealing strength between two CPP layers were measured by a universal testing machine (E43, MTS, Shenzhen, China) at a stretching rate of 300 mm min\(^{-1}\) according to GB/T 8808–1988 and QB/T 2358–1998 standards, respectively. The width of samples for T-peeling and heat-sealing tests was 15 mm. In the tests, force-displacement curves were recorded. The average force was determined as T-peeling strength (Unit: N/15 mm), and the maximum force was determined as the heat-sealing strength (Unit: N/15 mm). At least five replicates were conducted and the average values were reported.

3. Results and discussion
3.1. The grafting degree of MAH and gel content
The effects of co-monomers (St, DAP and TAIC) on the grafting degrees of MAH and the gel content at a MAH/co-monomer molar ratio of 1.0 with a MAH content of 6 wt% are shown in figure 3, and it shows that the grafting degrees of MAH with the assistance of St, DAP and TAIC are 1.3 wt%, 2.0 wt% and 2.3 wt%, respectively. These three co-monomers (St, DAP and TAIC) can improve the reactivity of MAH towards the initiator (DCP) or PP macroradicals to form copolymers, which may increase the grafting degree of MAH [10].
The co-monomers with high reactivity can be activated by the initiator at high temperatures, indicating that they can react with macroradicals to form stabilized radicals, and thus reduce chain scission and promote the grafting of MAH \[9\]. Compared with St, the introduction of DAP and TAIC results in a higher grafting degree of MAH, which can be attributed to the presence of more functional groups with high reactivity towards PP macroradicals and MAH. Many functional groups are introduced on PP by the reaction of these co-monomers with PP macroradicals, and thus the grafting degree reaches a maximum in PP-\(g\)-MAH/TAIC. PP-\(g\)-MAH/TAIC also has the highest gel content among the three functionalized PPs as shown in figure 3, since crosslinked structures are easily formed by TAIC that acts as the bridge between PP chains, which should be responsible for the excellent thermal stability and resistance to damage in T-peeling and heat-sealing testing. The results show that the addition of multi-functional co-monomer TAIC is more effective in increasing the grafting degree of MAH and the gel content than St and bi-functional co-monomer DAP.

3.2. FTIR analysis
Figure 4 shows the FTIR spectra of pure PP, PP-\(g\)-MAH/St, PP-\(g\)-MAH/DAP and PP-\(g\)-MAH/TAIC. The characteristic absorption peaks at 2949 and 2867 cm\(^{-1}\), 2919 and 2838 cm\(^{-1}\) are related to the stretching vibrations of C–H from –CH\(_3\) and –CH\(_2\)- groups, respectively; whereas those peaks at 1454 and 1375 cm\(^{-1}\) are related to C–H bending vibration \[23\]. New absorption peaks observed at 1856 and 1780 cm\(^{-1}\) in the FTIR spectra of the three functionalized PPs are attributed to the symmetric and asymmetric stretching vibration of the carbonyl groups (C=O) of MAH \[9, 11, 24\]. The absorption peaks observed at 1722 cm\(^{-1}\) in PP-\(g\)-MAH/DAP and at 1690 cm\(^{-1}\) in PP-\(g\)-MAH/TAIC are assigned to the C=O of DAP and TAIC, respectively \[25, 26\]. These results show that MAH has been successfully grafted on PP with the assistance of different co-monomers.

3.3. DSC analysis
The crystallization and melt curves of pure PP, PP-\(g\)-MAH/St, PP-\(g\)-MAH/DAP and PP-\(g\)-MAH/TAIC are shown in figure 5, and the crystallization temperature \(T_c\), melt temperature \(T_m\), crystallization enthalpy \(\Delta H_c\) and melt enthalpy \(\Delta H_m\) obtained from figure 5 are listed in table 1. The \(T_c\) values of the three functionalized PPs shift to higher temperatures compared with that of pure PP, which is attributed to the heterogeneous nucleation of the grafted MAH \[24, 27\]. The \(T_c\) values of functionalized PPs follow the order of PP-\(g\)-MAH/TAIC > PP-\(g\)-MAH/DAP > PP-\(g\)-MAH/St due to higher grafting degree of MAH. The \(T_m\) values of functionalized PPs are lower than that of pure PP, as many imperfections are present in PP crystals due to the introduction of the grafted groups \[28\]. In addition, PP-\(g\)-MAH/TAIC has a higher \(T_m\) than the other two functionalized PPs, probably because TAIC can reduce the chain scission during the grafting and result in a higher molecular weight by forming a cross-linked structure. Both \(\Delta H_c\) and \(\Delta H_m\) values of functionalized PPs are lower than that of pure PP, which is attributed to the introduction of MAH with a rigid ring \[29\]. Table 1 shows that the \(\Delta H_c\) and \(\Delta H_m\) values of PP-\(g\)-MAH/TAIC are lower than that of PP-\(g\)-MAH/St and PP-\(g\)-MAH/DAP, because the addition of multi-functional TAIC can increase the grafting degree of MAH and form a cross-linked structure.
3.4. Thermal stability

The thermal decomposition of pure PP, PP-g-MAH/St, PP-g-MAH/DAP and PP-g-MAH/TAIC was evaluated using TGA and digital thermogravimetry (DTG). The TGA and DTG curves are shown in figures 6 and 7, respectively. It is interesting to find that the thermal performance of PP-g-MAH differs substantially depending on the co-monomer incorporated. Figure 6 shows that the onset degradation temperature is lower in PP-g-MAH/St and PP-g-MAH/DAP than in pure PP due to the limitation of St and DAP in reducing chain scission of PP molecules in the presence of the initiator during the grafting [26, 28]. However, PP-g-MAH/TAIC has a higher onset degradation temperature, as TAIC is effective in reducing chain scission of PP molecules and promoting the formation of a cross-linked structure that has higher thermal stability than pure PP. For PP-g-MAH/St, the temperature of weight loss step shifts to a lower temperature and the peak of DTG curve becomes wider, implying that St has a limited ability to hinder the chain scission [10]. The temperatures of 5% weight loss ($T_{5\%}$), 50% weight loss ($T_{50\%}$) and rapidest decomposition ($T_{\text{rpd}}$) are shown in table 2. The $T_{5\%}$, $T_{50\%}$ and $T_{\text{rpd}}$ values of PP-g-MAH/St

![Figure 5. DSC crystallization (A) and melt (B) curves of pure PP and three functionalized PPs.](image)

![Figure 6. TGA curves of pure PP and three functionalized PPs.](image)

| Sample                  | $T_c$ (°C) | $T_m$ (°C) | Δ$H_c$ (J g$^{-1}$) | Δ$H_m$ (J g$^{-1}$) |
|-------------------------|------------|------------|---------------------|---------------------|
| pure PP                 | 117.9      | 166.1      | 74.1                | −73.2               |
| PP-g-MAH/St             | 122.0      | 164.7      | 72.6                | −71.2               |
| PP-g-MAH/DAP            | 126.6      | 164.4      | 69.6                | −68.7               |
| PP-g-MAH/TAIC           | 126.9      | 165.1      | 65.5                | −64.9               |

Table 1. DSC data for pure PP and three functionalized PPs.
Table 2. Thermal decomposition temperatures of pure PP and three functionalized PPs.

| Sample                  | Temperature (°C) |
|------------------------|------------------|
|                        | \( T_{-5} \)  | \( T_{-50} \) | \( T_{rpd} \) |
| pure PP                | 391.3           | 447.7         | 453.4         |
| PP-g-MAH/St            | 281.1           | 390.2         | 401.3         |
| PP-g-MAH/DAP           | 395.6           | 463.0         | 466.5         |
| PP-g-MAH/TAIC          | 417.6           | 463.3         | 466.7         |

3.5. Surface analysis of Ti treated Al foil

Figure 8 shows the SEM images of untreated, alkali treated and Ti treated Al foils. Some holes and rolling lines are observed in untreated Al foil, as shown in figure 8(A), and the foil is mainly composed of Al matrix and iron granules which are marked by red circles. Compared with untreated Al foil, the iron granules are more apparent in alkali treated Al foil (figure 8(B)), which is attributed to the removal of the greases and Al oxides on the Al foil by alkali treatment. Figure 8(C) shows that a rougher Ti conversion coating is deposited on Ti treated Al foil, and thus granules become more apparent [7]. The EDS results of the granules marked by red circles are shown in table 3. The removal of the greases and Al oxides by alkali treatment makes Al foil have a higher Fe content compared to untreated Al foil. Ti element is detected on the Ti treated Al foil surface, implying the formation of Ti conversion coating [31, 32].

The contact angles of untreated, alkali treated and Ti treated Al foils are shown in figure 9. The untreated Al foil shows low hydrophilicity with a contact angle of 81.3 ± 2.7°, which is attributed to the presence of greases on the Al foil surface. Compared with untreated Al foil, the contact angle of alkali treated Al foil is decreased to 52.8 ± 2.2°, as greases are removed by alkali treatment and more hydroxyl groups with high polarity are introduced onto Al foil surface, which leads to better wetting performance [33]. Figure 9(C) shows that the contact angle of Ti treated Al foil is 73.3 ± 1.9°, which is much higher than that of alkali treated Al foil owing to the reaction of hydroxyl groups on the Al foil surface with Ti conversion solution at a pH of 1.9.
3.6. Adhesive performance

In order to explore the effects of co-monomers (St, DAP and TAIC) on the adhesive properties of PP-g-MAH, the T-peeling and heat-sealing strengths of Al/P films were measured using Ti treated Al foil. Figure 10 shows the T-peeling and heat-sealing strengths of Al/P films with different adhesives. Pure PP shows almost no adhesion with the Al foil. The lowest T-peeling and heat-sealing strengths are achieved with the assistance of St, which is primarily due to the low grafting degree of MAH that can undergo chemical reactions and hydrogen bonding with hydroxyl groups on Al foil surface and chemical reactions with Ti conversion coating [4, 34, 35]. However, St has a limited ability to reduce chain scission of PP molecules, resulting in poor mechanical performance and thus the tearing of PP-g-MAH/St during the testing process. PP-g-MAH/DAP shows better T-peeling and heat-sealing strengths than PP-g-MAH/St, as shown in figure 10, which is due to the higher grafting degree of MAH and the restriction of DAP on chain scission [25]. The coordination between DAP and Ti treated Al foil surface might also contribute to the improvement of T-peeling and heat-sealing strengths [36]. Both T-peeling and heat-sealing strengths reach a maximum with the assistance of TAIC, which is ascribed to its highest grafting degree among the three functional PPs and excellent mechanical properties that are related to the formation of cross-linked structures during the grafting [9, 26]. The results show that TAIC as a co-monomer is effective to improve the adhesive properties of PP-g-MAH.

4. Conclusions

In this study, three functionalized PPs (PP-g-MAH/St, PP-g-MAH/DAP and PP-g-MAH/TAIC) were successfully prepared by melt grafting with the assistance of St, DAP and TAIC, respectively. The grafting degrees
of MAH and the gel content follow the order of PP-g-MAH/TAIC > PP-g-MAH/DAP > PP-g-MAH/St, and PP-g-MAH/TAIC has higher T-peeling and heat-sealing strengths due to the chemical reaction and hydrogen bonding between MAH and Al foil surface and excellent resistance to damage in testing. The T-peeling and heat-sealing strengths of PP-g-MAH/TAIC are 28.6 and 70.4 N/15 mm, which are increased by 33.6% and 20.8% compared to PP-g-MAH/DAP, and by 142.4% and 81.0% compared to PP-g-MAH/St, respectively. The addition of TAIC with multi-functional groups can also effectively reduce chain scission of PP molecules and promote the formation of cross-linked structures during the melt grafting process, thus resulting in better thermal stability of PP-g-MAH/TAIC. The $T_{5\%}$ value of PP-g-MAH/TAIC is 417.6 °C, with an increase of 26.3, 136.5 and 22.0 °C compared to pure PP, PP-g-MAH/St and PP-g-MAH/DAP, respectively.

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Figure 10. T-peeling and heat-sealing strengths of Al/P films.
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