

CHAPTER IV

RESULT AND DISCUSSION

4.1 Characterization of Grafting Material, HDPE-g-MAH and PP-g-MAH

4.1.1 % Grafting

Table 4.1 shows content of grafting degree of four types of grafting materials that prepared in the laboratory. All of the grafting materials had high of grafting being approximately 2.0 %- 2.5 %, HDPE-g-MAH and PP-g-MAH were produced from condition which added the lowest amount of dicumyl peroxide (DCP) during melt grafting processing step. In case of HDPE-g-MAH types produced from 4 phr MAH and 0.125 phr of DCP was degree of grafting approximately 2.41 %, while PP-g-MAH types used the different amount of MAH and DCP from HDPE-g-MAH at 5 phr and 0.6 phr respectively and result of grafting showed to be around 2.17 %. The difference amount of MAH and DCP used was due to the different grafting mechanism between HDPE with MAH and PP with MAH.

Table 4.1 Grafting degree of four grafting materials

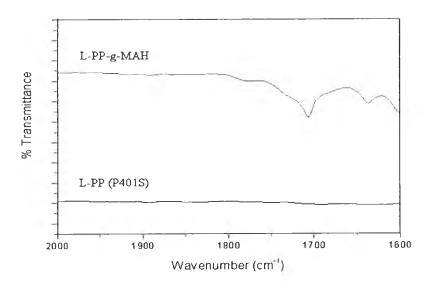
| No | Grafting Material | DCP (phr) | MAH (phr) | Grafting degree (%) |
|----|-------------------|--------------|--------------|------------------------|
| 1 | L-HDPE-g-MAH | 0.125 | 4 | 2.38 |
| 2 | H-HDPE-g-MAH | 0.125 | 4 | 2.44 |
| 3 | L-PP-g-MAH | 0.6 | 5 | 2.15 |
| 4 | H-PP-g-MAH | 0.6 | 5 | 2.20 |

All of these 4 grafting material were used as compatibilizer in HDPE/PP blend in blending step.

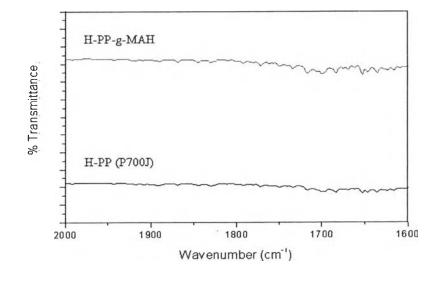
พยสมุดแลาง สำนักงานว่ายกร่พยากร พาลงกรณ์มหาวิทหาลัย

4.1.2 Fourier Transform Infrared Spectroscopy (FT-IR)

The confirmation of grafting was studied using Fourier Transform Infrared Spectroscopy (FT-IR)

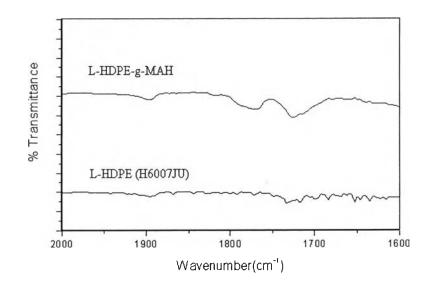






(B)

Figure 4.1 FT-IR spectrum of (A) L-PP-g-MAH and (B) H-PP-g-MAH.



(A)

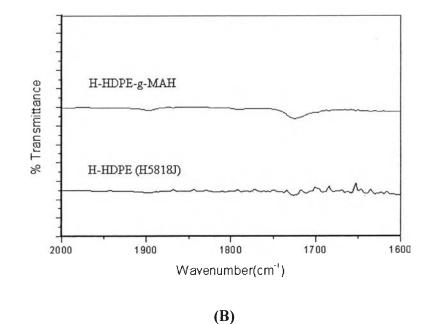


Figure 4.2 FTIR spectrum of (A) L-HDPE-g-MAH and (B) H-HDPE-g-MAH.

Figure 4.1 and 4.2 show the FT-IR spectra of all 4 grafting materials compared with neat polymer in order to confirm that the maleic anhydride functional group was introduced into these polymers, characterized peak of cyclic anhydride at

around 1725-1705 cm⁻¹ on FTIR spectra of the grafted polymers indicated that the grafting was achieved on the two grafted polymer.

4.1.3 Flow properties of Grafting Material : Melt Flow Index (MFI)

Melt flow index of pure materials obtained from the supplier was rechecked and compared with MFI of the grafted material using the same condition as suggested by the supplier at the condition of 190 $^{\circ}$ C/2.16 Kg as shown in the figure 4.3

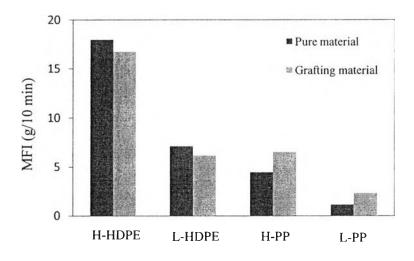


Figure 4.3 Melt flow index of pure material and grafting material.

In the case of HDPE types, MFI of grafting material H-HDPE-g-MAH and L-HDPE-g-MAH were decreased a little from the original. This could be due to side effect called cross-linking reaction occurred during the melt grafting processing step and result in the increasing of viscosity of outcome material and affected by decreasing of MFI. PP-g-MAH types, the MFI of grafting materials were increase for both low and high initial MFI indicated that chain scission occurred during grafting.

4.2 Characterization and Testing Properties of HDPE/PP Blend with and without Compatibilizer

4.2.1 Flow properties of the blends : Melt Flow Index (MFI)

The effect of compatibilizer content on the melt flow index of the blend at fixed blend ratio of 75/25 and 25/75 HDPE/PP are shown in Figure 4.4 and 4.5 respectively. The results shown that MFI of all four sets of the blend were not affected by adding compatibilizer which can be observed from constant of MFI when compatibilizer content was increased, this was due to small amount of compatibilizer added into the blend, so it could not interrupt the MFI of main polymer. Furthermore the value of MFI was relatively similar with each other phases between minor and major phase, however the MFI of the blend was still depended on type of major phase.

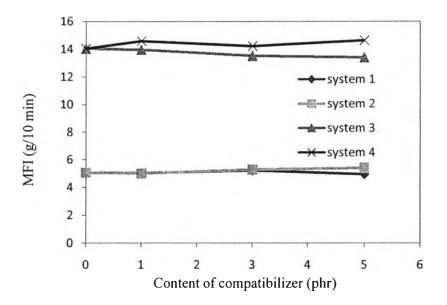


Figure 4.4 Melt flow index (MFI) VS content of compatibilizer of different system of the blend at fixed blend ratio 75/25 HDPE/PP.

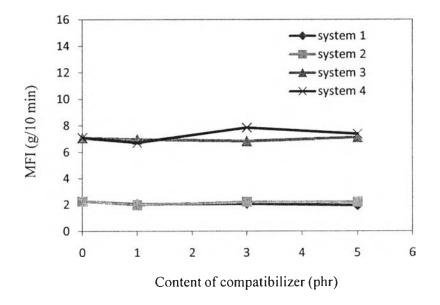


Figure 4.5 Melt flow index VS content of compatibilizer of different System of the blend at fixed blend ratio 25/75 HDPE/PP.

4.2.1 Mechanical Properties of the Blends

4.2.1.1 The Blend of System 1. L-HDPE/L-PP/L-HDPE-g-MAH/ L-PP-g-MAH

4.2.1.1 a) Tensile Properties

Tensile properties of the system 1 blend which was the blend of L-HDPE/L-PP/L-HDPE-g-MAH/L-PP-g-MAH, Figure 4.6 show the tensile strength at yield of the blend. Increasing of compatibilizer content did not significant change the tensile strength of compatibilized blend compared with uncompatibilized blend. In case of 25/75 L-HDPE/L-PP base blend, shown higher value of tensile strength than that of 75/25 L-HDPE/L-PP base blend indicated that increasing content of L-PP could increase the tensile strength of the blend. Figure 4.7 shows the % strain at break of the blend system 1, the unchange % strain at break could be observed at 25/75 L-HDPE/L-PP base blend as compatibilizer was added into the blend around 5 times of uncompatibilized blend. The optimum content of vompatibilizer was at 5 phr of L-HDPE/L-PP-g-MAH compatibilizer.

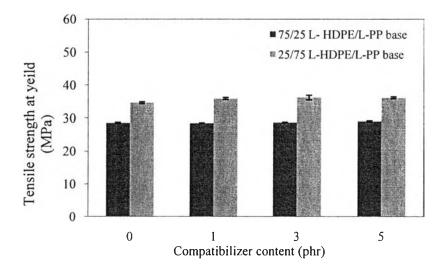


Figure 4.6. Tensile strength of the blend system 1. L-HDPE/L-PP/L-HDPE-g-MAH/L-PP-g-MAH at varied compatibilizer content.

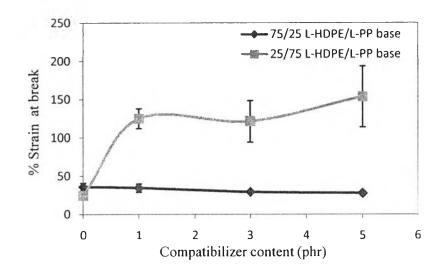


Figure 4.7 % Strain at break of the blend system 1.L-HDPE/L-PP/L-HDPE-g-MAH at varied compatibilizer content.

4.2.1.1 b) Impact Properties

The impact properties of the system 1 blend was shown in Figure 4.8 which indicated that no remarkably change of impact strength at every content of the compatibilizer in both of two blend ratio of 75/25 and 25/75 L-HDPE-L-PP base blend, due to close impact strength of the neat L-HDPE and neat L-PP.

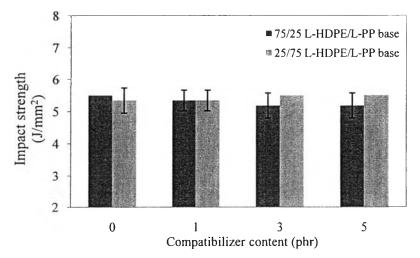


Figure 4.8 Impact strength of the blend system 1. L-HDPE/L-PP/L-HDPE-g-MAH/L-PP-g-MAH at varied compatibilizer content.

4.2.1.2 The Blend of System 2. L-HDPE/L-PP/H-HDPE-g-MAH/ H-PP-g-MAH

4.2.1.2 a) Tensile Properties

Tensile strength of the system 2 blend was presented in Figure 4.9, the same result shown as in the blend system 1 because using the L-HDPE/L-PP as base material same as system 1, but used different type of compatibilizer. Thus the result of tensile strength could indicate that using H-HDPEg-MAH/H-PP-g-MAH as the compatibilizer in this blend was not different from using L-HDPE-g-MAH/L-PP-g-MAH. Figure 4.10, at 25/75 L-HDPE/L-PP base blend, the % strain at break of the blend system 2 was increased when the compatibilizer content increased and the effective content began at 3 phr and the optimum at 5 phr, however the % strain of the blend system 2 was lower than the value in system 1 at every compatibilizer content.

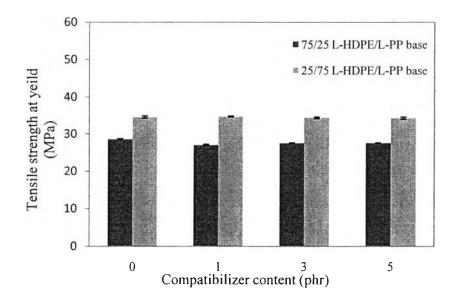


Figure 4.9 Tensile strength of the blend system 2. L-HDPE/L-PP/H-HDPE-g-MAH/ H-PP-g-MAH at varied compatibilizer content.

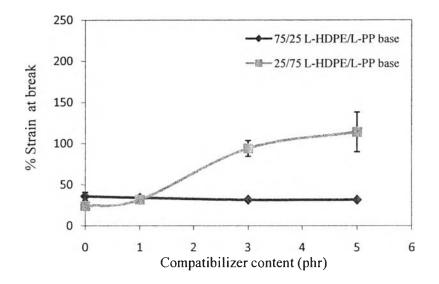


Figure 4.10 % Strain at break of the blend system 2. L-HDPE/L-PP/H-HDPE-g-MAH/H-PP-g-MAH at varied compatibilizer content.

4.2.1.2 b) Impact Properties

The impact strength of the blend system 2 was determined as function of compatibilizer content and shown in Figure 4.11. There were no significant different of impact strength between uncompatibilized and compatibilized blend when compatibilizer content increased in both of the two blend ratio.

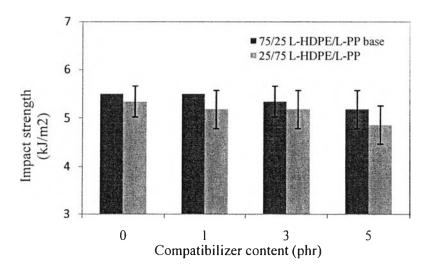


Figure 4.11 Impact strength of the blend system 2. L-HDPE/L-PP/H-HDPE-g-MAH at varied compatibilizer content

4.2.1.3 The Blend of System 3. H-HDPE/H-PP/L-HDPE-g-MAH/ L-PP-g-MAH

4.2.1.3 a) Tensile Properties

Tensile strength of the blend system 3 which was the blend of H-HDPE/H-PP/L-HDPE-g-MAH/L-PP-g-MAH. The continuous stable of tensile strength between uncompatibilized and compatibilized blend were observed. While the % strain at break of the blend at 25/75 H-HDPE/H-PP base blend which had the H-PP as major phase was higher than that at 75/25 H-HDPE/H-PP base blend. However adding compatibilizer L-HDPE-g-MAH/L-PP-g-MAH did not effected to the % strain of this system of blending.

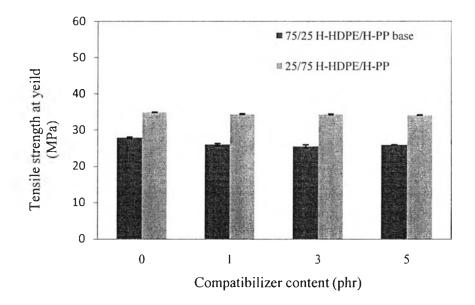


Figure 4.12 Tensile strength of the blend system 3. H-HDPE/H-PP/L-HDPE-g-MAH/ L-PP-g-MAH at varied compatibilizer content.

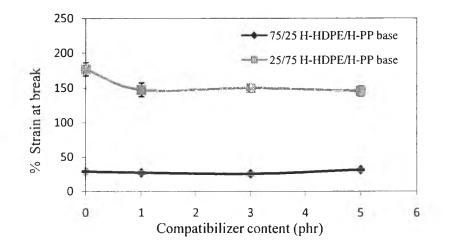


Figure 4.13 % Strain at break of the blend system 3. H-HDPE/H-PP/L-HDPE-g-MAH at varied compatibilizer content.

4.2.1.3 b) Impact Properties

The result of impact strength of the blend system 3 was illustrated in Figure 4.14 adding the compatibilizer L-HDPE-g-MAH/L-PP-g-MAH into the blend of H-HDPE/H-PP caused the decreased of impact strength compared with the uncompatibilized blend because of the large different of MFI between L-HDPE-g-MAH/L-PP-g-MAH compatibilizer and H-HDPE/H-PP base material, so the added compatibilizer acted as the stress concentration point of the blend.

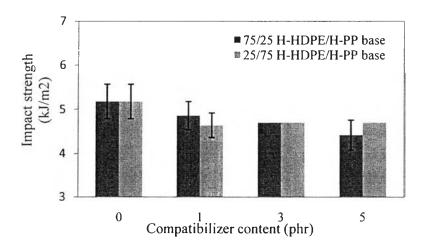


Figure 4.14 Impact strength of the blend system 3. H-HDPE/H-PP/L-HDPE-g-MAH/L-PP-g-MAH at varied compatibilizer content.

4.2.1.4 The Blend System 4. H-HDPE/H-PP/H-HDPE-g-MAH/H-PPg-MAH

4.2.1.4 a) Tensile Properties

The tensile properties of the blend system 4 was not different from system 3 which used the same base material H-HDPE/H-PP indicated that using H-HDPE-g-MAH/H-PP-g-MAH as compatibilizer did not affect the tensile properties of the blend. At 25/75 H-HDPE/H-PP base blend shown higher tensile strength and % strain in both case of uncompatibilized and compatibilized blends.

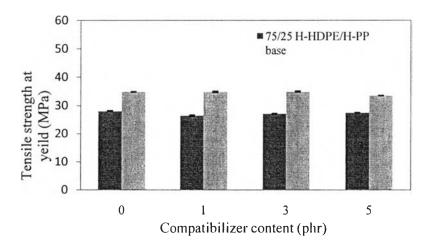


Figure 4.15 Tensile strength of the blend system 4. H-HDPE/H-PP/H-HDPE-g-MAH/H-PP-g-MAH at varied compatibilizer content.

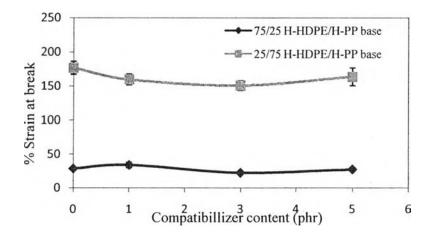


Figure 4.16 % Strain at break of the blend system 4. H-HDPE/H-PP/H-HDPE-g-MAH at varied compatibilizer content.

Figure 4.17 shows the impact strength of the blend system 4 H-HDPE/H-PP/H-HDPE-g-MAH/H-PP-g-MAH as function of compatibilizer content. The decreased impact strength of these blends was still observed when the compatibilizer H-HDPE-g-MAH/H-PP-g-MAH was added into the blends. The key reason of decreasing in impact strength in this blend was the compatibilizer acting as stress concentration point in the system of high MFI.

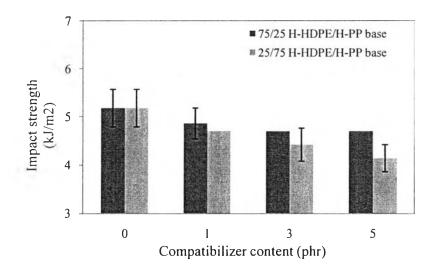


Figure 4.17 Impact strength of the blend system 4. H-HDPE/H-PP/H-HDPE-g-MAH/H-PP-g-MAH at varied compatibilizer content.

4.2.1.5 Effect of Type of HDPE/PP base and Type of Compatibilizer on the Impact Strength

Figure 4.18 shows the impact strength of each system of the uncompatibilized blend. Litle change was observed from using L-HDPE/L-PP as base material in system 1, system 2 and using H-HDPE/H-PP base material. The different result occurred in the 5 phr compatibilized blend was illustrated in Figure 4.19, impact strength of the blend having L-HDPE/L-PP as base material in system 1 and system 2 was higher than the impact strength of the blend system 3 and system 4 which having H-HDPE/H-PP base material in the blend. These result indicated that adding 5 phr compatibilizer was more effective in the L-HDPE/L-PP blend, but poor in case of high MFI base material H-HDPE/H-PP because it flow easily.

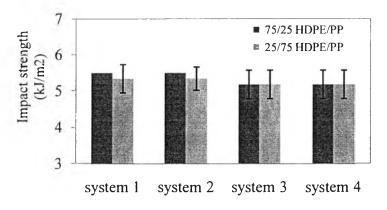


Figure 4.18 Impact strength of 4 different Systems of uncompatibilized blend.

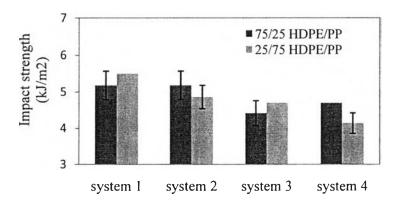


Figure 4.19 Impact strength of 4 different Systems of HDPE/PP blend at 5 phr compatibilizer.

4.2.2 <u>Thermal Properties of the Blend (Differential Scanning Calorimeter)</u>

4.2.2.1 The Pure Material and Grafting Material

The thermal properties and crystalinity of the pure and grafted materials were characterized by DSC technique and the result shown in Table 4.2 ,DSC result of four grafting materials, used as the compatibilizer in the blend, were not changed from pure material. The enthalpy of melting of HDPE was higher than that of PP. The difference was related with the 60 % Crystalinity of HDPE higher than that of PP. In addition the crystallization temperature (Tc) and melting temperature (Tm) of grafting material were also not much difference from pure material.

| | | Enthalpy of melting (J/g) | | Tm (°C) | | Crystalinity | |
|--------------|-------|------------------------------|-------|------------|-------|--------------|-------|
| System 1. | Tc | | | | | (%) | |
| | | PE | PP | PE | PP | PE | РР |
| | (°C) | phase | phase | phase | phase | phase | phase |
| L-HDPE | 119.9 | 180.3 | - | 132.3 | - | 61.54 | - |
| H-HDPE | 119.3 | 181.4 | - | 130.7 | - | 61.91 | - |
| L-PP | 115.4 | - | 86.4 | - | 161.2 | - | 41.74 |
| H-PP | 114.0 | - | 82.8 | - | 156 | - | 40.00 |
| L-HDPE-g-MAH | 120.5 | 187.9 | - | 131.5 | - | 64.13 | - |
| H-HDPE-g-MAH | 119.5 | 180.3 | - | 129.4 | - | 61.54 | - |
| L-PP-g-MAH | 119.2 | - | 90.2 | - | 161.5 | - | 43.57 |
| H-PP-g-MAH | 120.4 | - | 86.6 | - | 157.2 | - | 41.84 |

Table 4.2 DSC results of pure and grafted material

4.2.2.1 The Blend of System 1. L-HDPE/L-PP/L-HDPE-g-MAH/ L-PP-g-MAH

From DSC thermogram of the blend system 1. The observed melting peak of HDPE phase was found at 130 $^{\circ}$ C and melting peak of PP was at 162 $^{\circ}$ C. The separation of 2 melting peak indicated that these blend were not compatible or phase separated. The crystalinity of these blend were not different from pure material and added compatibilizer did not affect the crystalinity of the blends as shown in table 4.3

Table 4.3 DSC result of the blend System 1

| System 1. | Com ^a | Тс | Enthalpy of melting | | Tr (°C | | Crysta (% | |
|------------|------------------|-------|------------------------|-------|-----------|-------|--------------|-------|
| | | | PE | PP | PE | PP | PE | PP |
| | (phr) | (°C) | phase | phase | phase | phase | phase | phase |
| System 1/1 | 0 | 120.0 | 135.9 | 21.3 | 131.6 | 161.0 | 61.84 | 41.16 |
| System 1/2 | 1 | 120.1 | 138.9 | 19.4 | 131.4 | 161.4 | 63.21 | 37.49 |
| System 1/3 | 3 | 120.4 | 133.8 | 20.5 | 131.9 | 161.6 | 60.89 | 39.61 |
| System 1/4 | 5 | 120.4 | 137.5 | 21.6 | 131.3 | 161.3 | 62.57 | 41.74 |
| System 1/5 | 0 | 119.2 | 58.2 | 56.4 | 130.9 | 161.5 | 79.45 | 36.33 |
| System 1/6 | 1 | 120.0 | 50.2 | 61.1 | 130.9 | 161.8 | 68.53 | 39.36 |
| System 1/7 | 3 | 120.1 | 50.4 | 60.9 | 130.6 | 161.8 | 68.81 | 39.23 |
| System 1/8 | 5 | 120.4 | 50.0 | 60.7 | 131.0 | 162.3 | 68.26 | 39.10 |

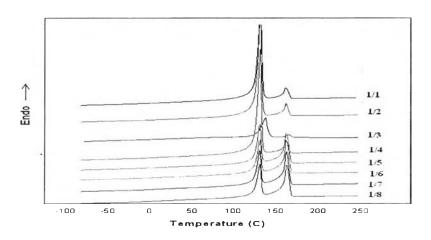


Figure 4.20 DSC thermogram of 2nd heating: Melting peak of the blend system 1.

4.2.2.2 The Blend of System 2. L-HDPE/L-PP/H-HDPE-g-MAH/ H-PP-g-MAH

Table 4.4 and Figure 4.21 show DSC thermogram of the blend system 2. The crystalinity of the compatibilized blends were not significance difference from uncompatibilized blend because of phase separation of HDPE and PP with confirm to occurrence of 2 melting peak in DSC thermogram.

| System 2. | Com ^a | Tc | mel | lpy of ting (g) | Tm (C) | | Crystalinity (%) | |
|-----------------------|------------------|----------------|-------|-----------------------|-----------|-------|---------------------|-------|
| | 建設 為 | L. B. S. China | PE | PP | PE | PP | PE | рр |
| and the second second | (phr) | (C) | phase | phase | phase | phase | phase | phase |
| System 2/1 | 0 | 120.0 | 135.9 | 21.3 | 131.6 | 161.0 | 61.84 | 41.16 |
| System 2/2 | 1 | 120.1 | 138.6 | 20.7 | 131.4 | 161.2 | 63.07 | 40.00 |
| System 2/3 | 3 | 120.2 | 136.4 | 21.3 | 131.4 | 161.3 | 62.07 | 41.16 |
| System 2/4 | 5 | 120.3 | 135.3 | 21.5 | 131.2 | 160.9 | 61.57 | 41.55 |
| System 2/5 | 0 | 119.2 | 58.2 | 56.4 | 130.9 | 161.5 | 79.45 | 36.33 |
| System 2/6 | 1 | 119.4 | 50.5 | 60.6 | 131.0 | 161.9 | 68.94 | 39.03 |
| System 2/7 | 3 | 119.7 | 53.1 | 57.8 | 131.0 | 162.0 | 72.49 | 37.23 |
| System 2/8 | 5 | 120.4 | 50.7 | 61.9 | 130.5 | 161.5 | 69.22 | 39.87 |

 Table 4.4 DSC results of the blend system 2

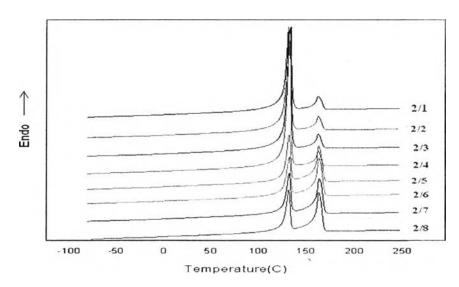


Figure 4.21 DSC thermogram 2nd heating : Melting peak of the blend System 2.

4.2.2.3 The Blend of System 3. H-HDPE/H-PP/L-HDPE-g-MAH/ L-PP-g-MAH

The crystalinity of the blends system 3 were reported in table 4.5, no significant change from uncompatibilized and compatibilized blend at every content of compatibilizer and 2 individual peak of melting temperature were confirmed that this blend system was phase separated.

| | Com | | Enthalpy of melting | | T | Tm | | Crystalinity | |
|------------|-------|-------|---------------------|-------|-------|-------|-------|---------------------|--|
| System 3. | 8 | Tc | (J | /g) | ((| (C) | | (%) | |
| | 建合理 | 1.13 | PE | PP | PE | PP | PE | PP | |
| | (phr) | (C) | phase | phase | phase | phase | phase | phase | |
| System 3/1 | 0 | 119.0 | 128.3 | 19.7 | 129.4 | 154.6 | 58.38 | 38.07 | |
| System 3/2 | 1 | 119.4 | 134.9 | 18.3 | 129.5 | 156.5 | 61.39 | 35.36 | |
| System 3/3 | 3 | 119.8 | 135.4 | 19.2 | 129.5 | 157.8 | 61.61 | 37.10 | |
| System 3/4 | 5 | 119.5 | 128.9 | 18.3 | 130.1 | 157.2 | 58.66 | 35.36 | |
| System 3/5 | 0 | 117.3 | 50.1 | 55.1 | 129.1 | 156.5 | 68.40 | 35.49 | |
| System 3/6 | 1 | 118.5 | 49.7 | 57.1 | 129.1 | 156.3 | 67.85 | 36.78 | |
| System 3/7 | 3 | 118.6 | 53.4 | 56.3 | 129.1 | 157.4 | 72.90 | 36.26 | |
| System 3/8 | 5 | 118.5 | 52.5 | 55.6 | 129.3 | 157.1 | 71.67 | 35.81 | |

 Table 4.5
 DSC results of the blend system 3

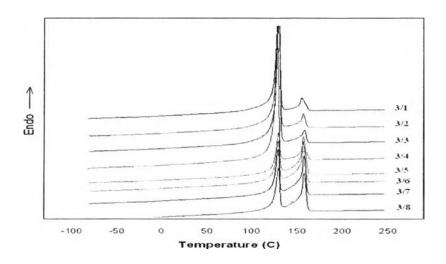


Figure 4.22 DSC thermogram 2nd heating: Melting peak of the blend system 3.

4.2.2.4 The Blend of System 4. H-HDPE/H-PP/H-HDPE-g-MAH/ H-PP-g-MAH

Table 4.6 shows the DSC result of the blend system 4, the crystalinity of compatibilized blend were not significant different from uncompatibilized blend. Furthermore the crystalinity were still depended on pure material of each phase. At the same time the DSC thermogram confirmed that the phase separated blend of system 4 were also produced because of clearly occurrence of 2 melting peak which still base on the melting temperature of pure material.

| Table 4.6 | DSC | result of | the b | lend | system 4 |
|-----------|-----|-----------|-------|------|----------|
|-----------|-----|-----------|-------|------|----------|

| System 4. | Com | Тс | Enthalpy of melting (J/g) | | Tm (°C) | | Crystalinity (%) | |
|------------|-------|------------|---------------------------------|-------|------------|-------|---------------------|-------|
| a.112 | No. C | 1. 10 5.10 | PE | PP | PE | PP | PE | PP |
| | (phr) | (°C) | phase | phase | phase | phase | phase | phase |
| System 4/1 | 0 | 119.0 | 128.8 | 19.7 | 129.4 | 154.6 | 58.61 | 38.07 |
| System 4/2 | 1 | 119.5 | 138.2 | 16.5 | 129.3 | 155.2 | 62.89 | 31.88 |
| System 4/3 | 3 | 119.7 | 136.6 | 17.9 | 129.3 | 156 | 62.16 | 34.56 |
| System 4/4 | 5 | 119.7 | 136.6 | 18.5 | 129.5 | 156.2 | 62.16 | 35.75 |
| System 4/5 | 0 | 117.3 | 50.1 | 55.1 | 129.1 | 156.5 | 68.40 | 35.49 |
| System 4/6 | 1 | 117.6 | 48.3 | 56.9 | 128.1 | 155.9 | 65.94 | 36.65 |
| System 4/7 | 3 | 118 | 50.5 | 54.7 | 129.1 | 156.2 | 68.94 | 35.23 |
| System 4/8 | 5 | 118.4 | 38.6 | 43.6 | 129.1 | 156.5 | 52.70 | 28.08 |

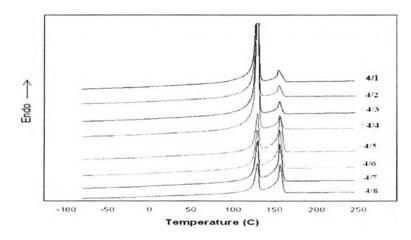


Figure 4.23 DSC thermogram 2nd heating: Melting peak of the blend system 4.