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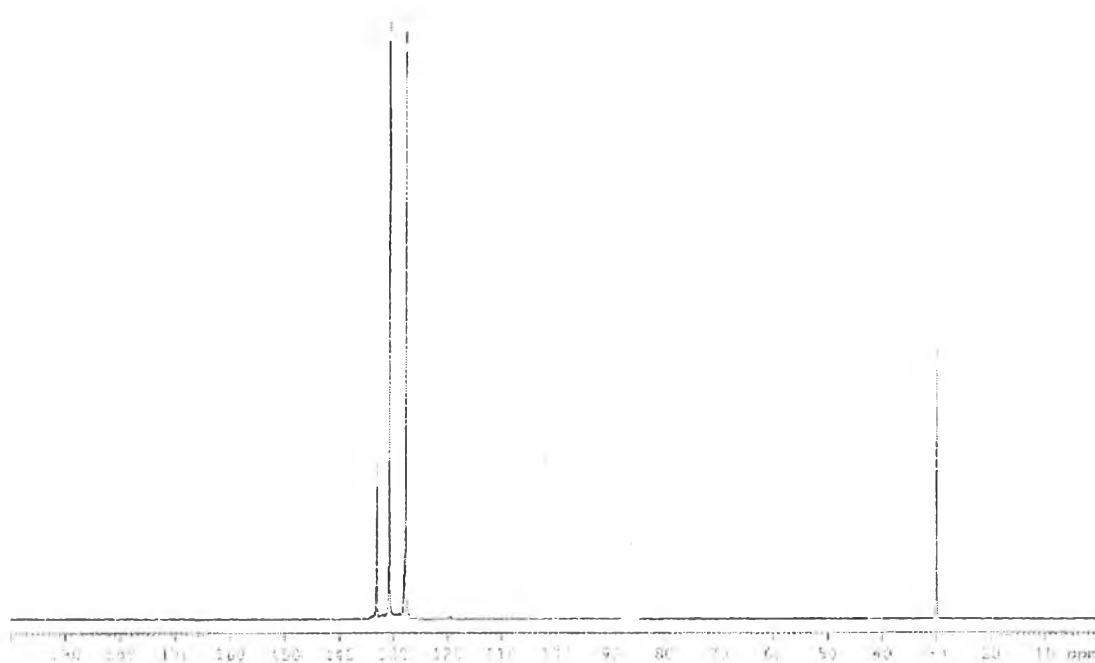
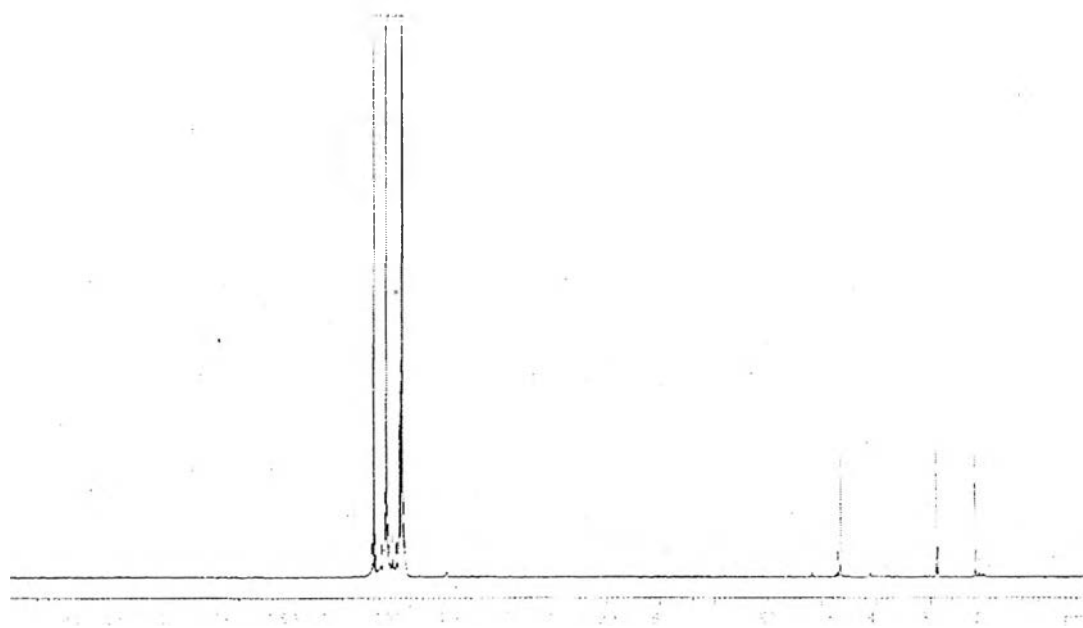
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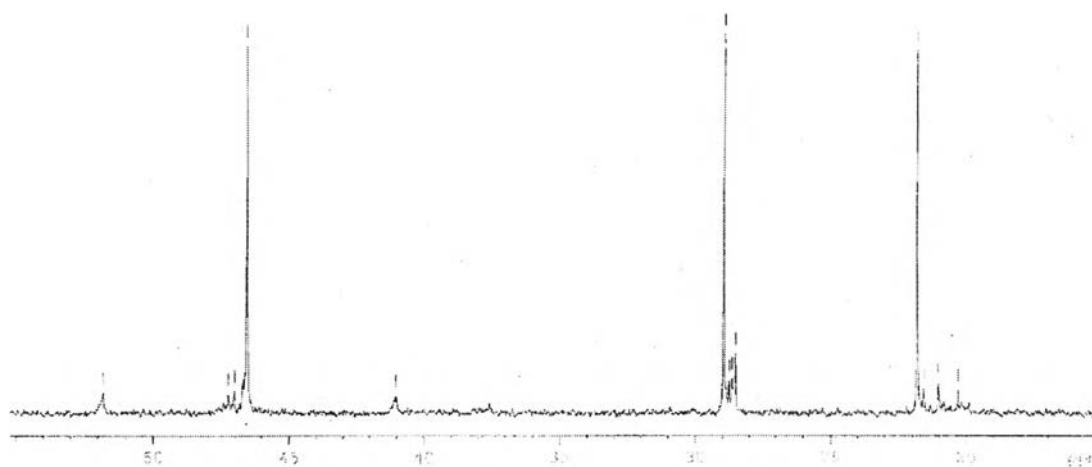


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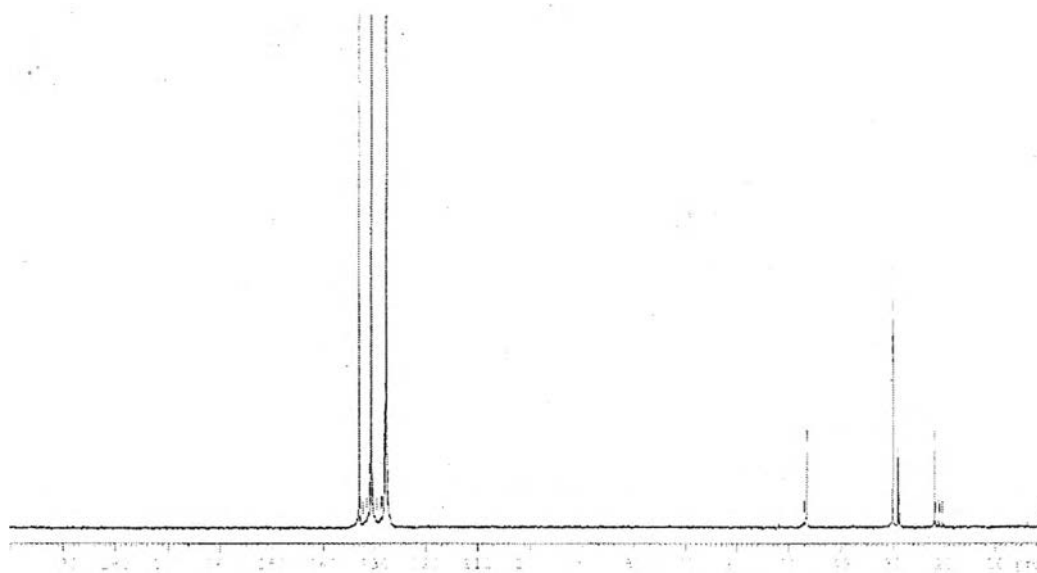
## **APPENDICES**

## Appendix A

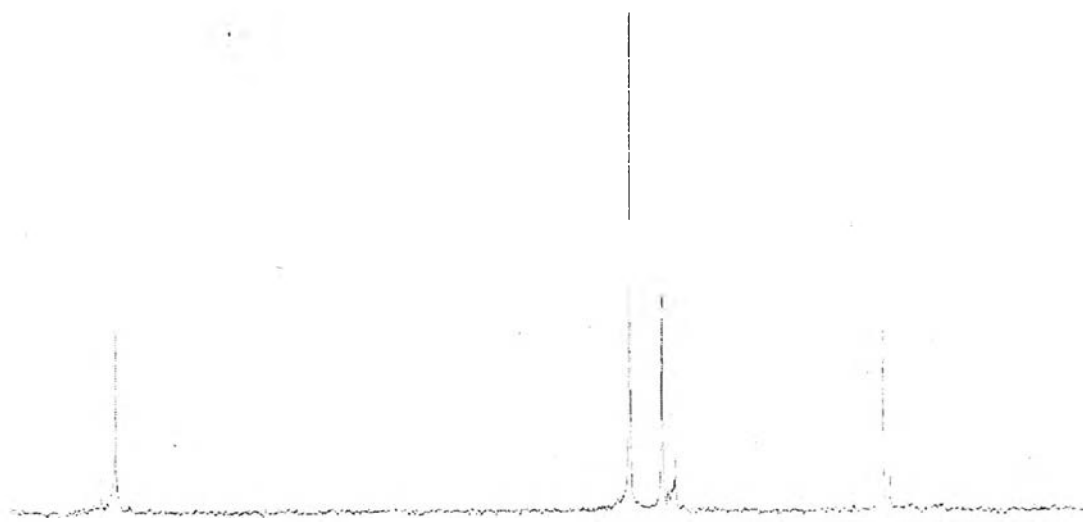
 $C^{13}$  NMR CharacterizationFigure A.1  $C^{13}$  NMR spectrum of PE-b-PEFigure A.2  $C^{13}$  NMR spectrum of PP-b-PP



**Figure A.3**  $^{13}\text{C}$  NMR spectrum of PP-b-PP



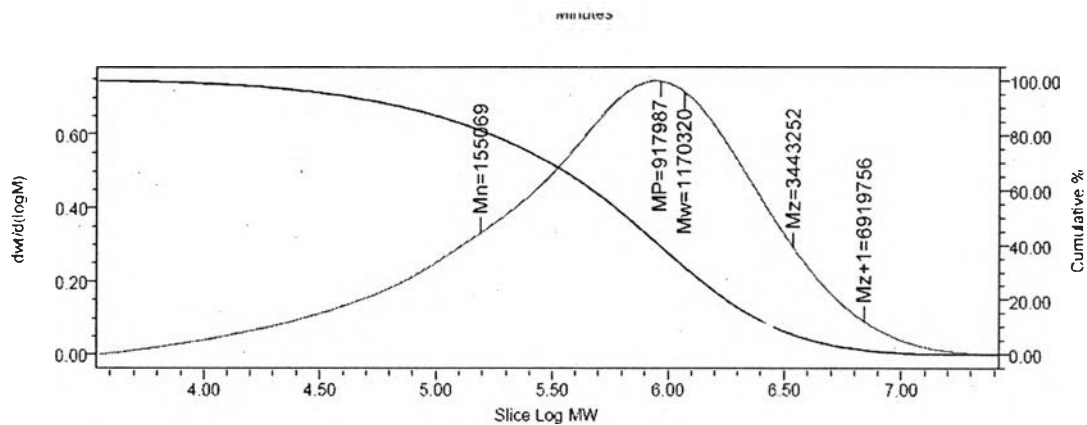
**Figure A.4**  $^{13}\text{C}$  NMR spectrum of PE-b-PP



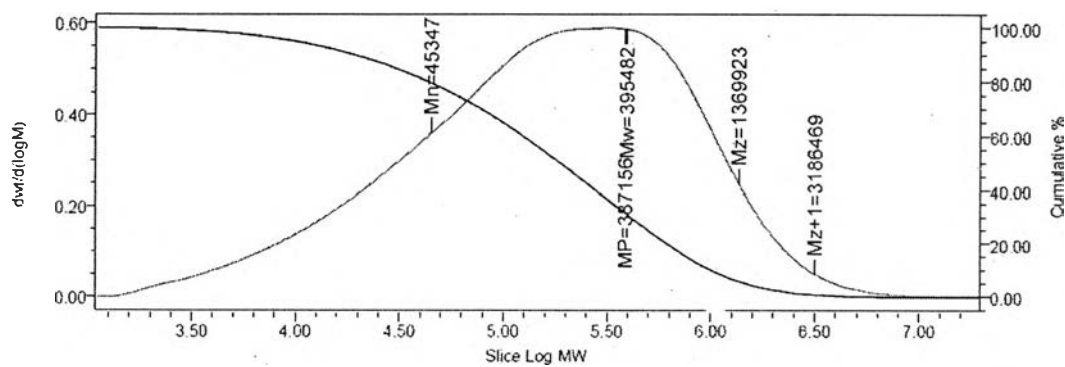
**Figure A.5**  $^{13}\text{C}$  NMR spectrum of PE-b-PP

## Appendix B

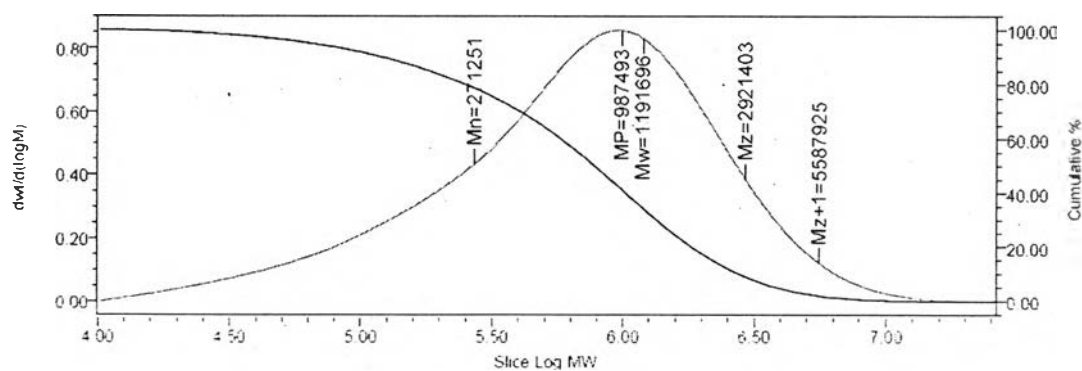
### The Data of GPC Characterization



**Figure B.1** GPC curve of PEOH



**Figure B.2** GPC curve of PPOH



**Figure B.3** GPC curve of PE-b-PE01

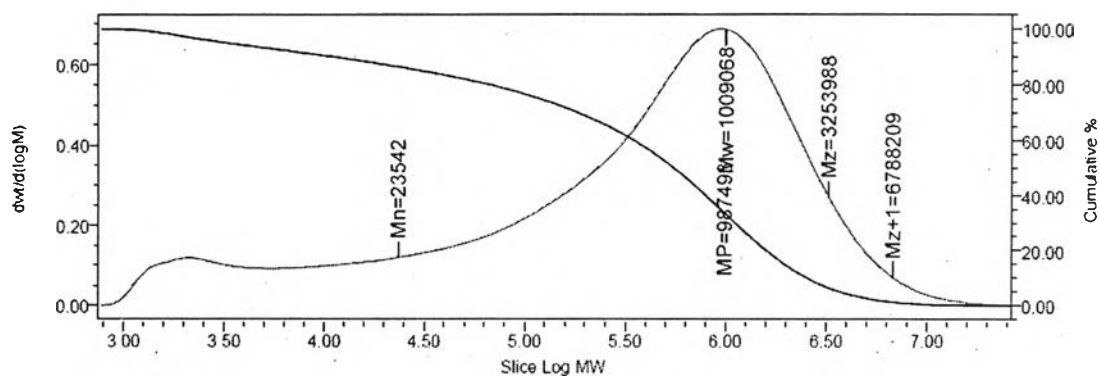


Figure B.4 GPC curve of PE-b-PE02

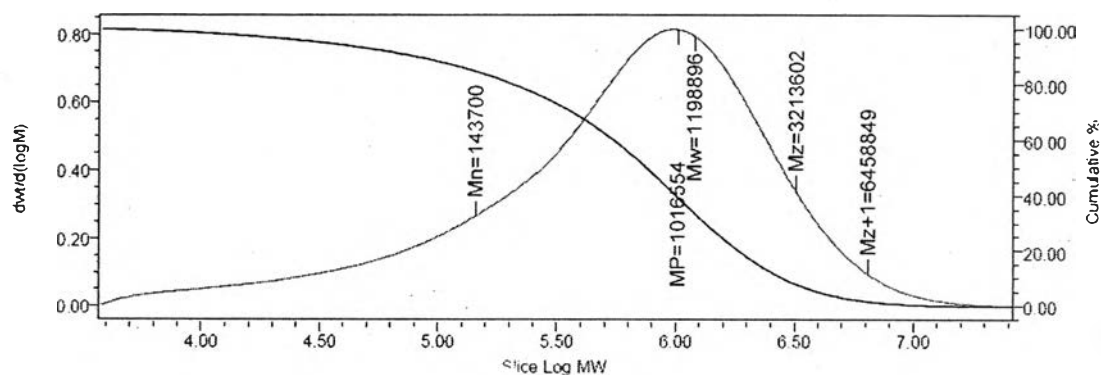


Figure B.5 GPC curve of PE-b-PE03

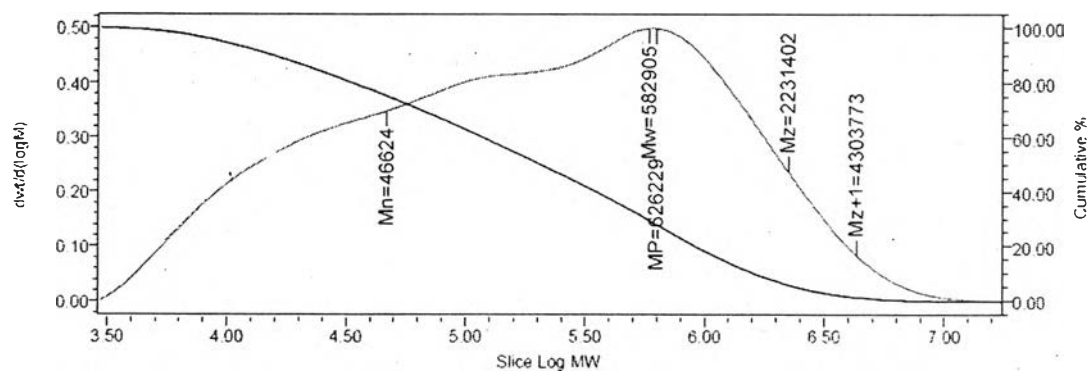


Figure B.6 GPC curve of PE-b-PP01



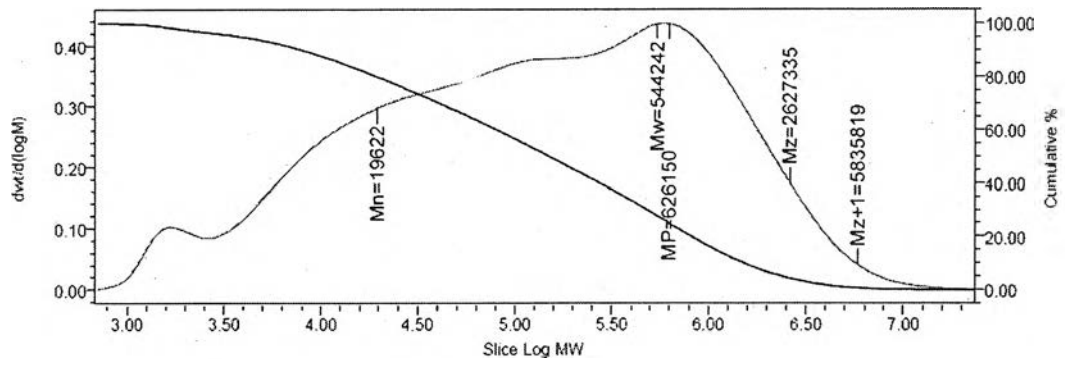


Figure B.7 GPC curve of PE-b-PP02

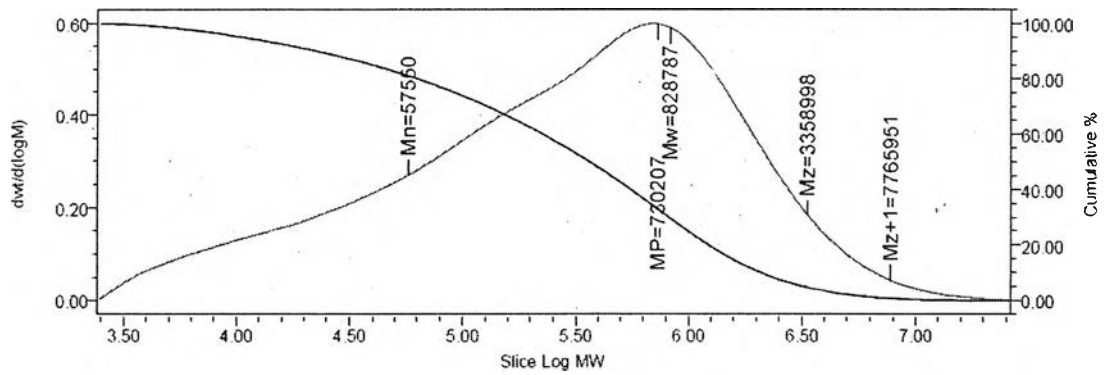


Figure B.8 GPC curve of PE-b-PP03

## Appendix C

### The Data of DSC Characterization

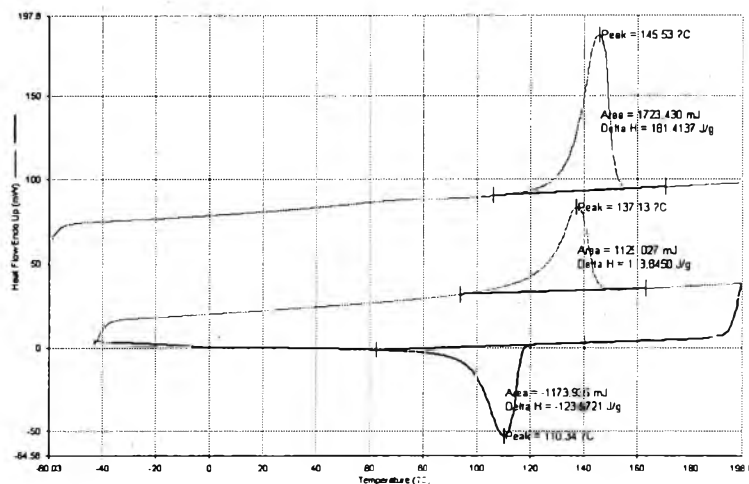


Figure C.1 DSC curve of PEOH

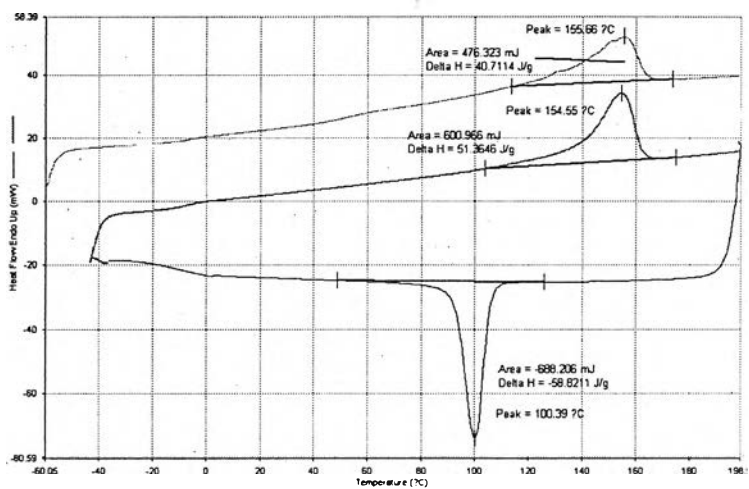


Figure C.2 DSC curve of PPOH

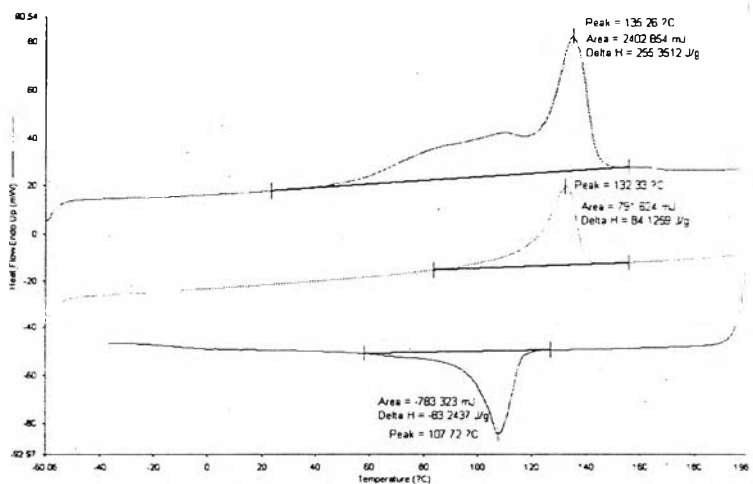


Figure C.3 DSC curve of PE-b-PE

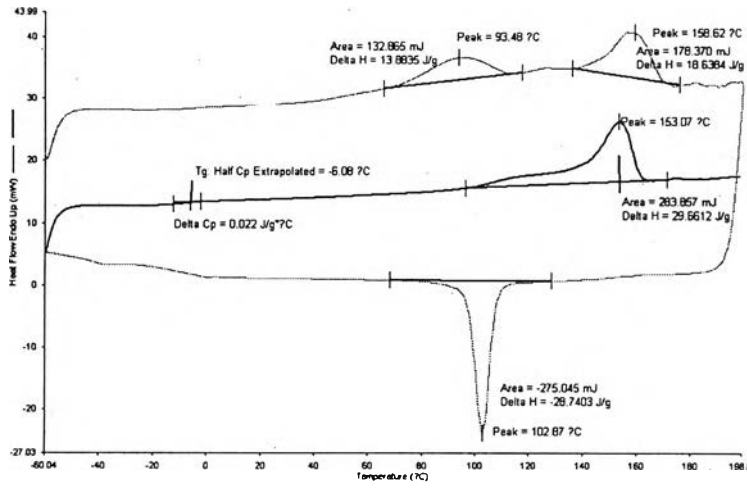


Figure C.4 DSC curve of PP-b-PP

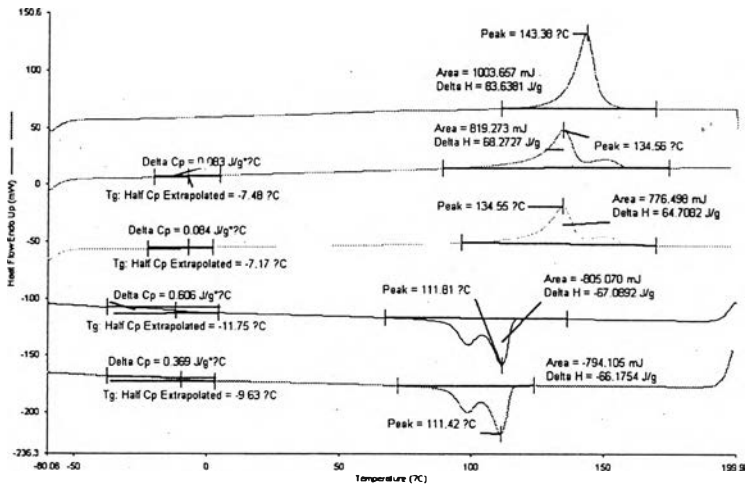


Figure C.5 DSC curve of PE/PP blend

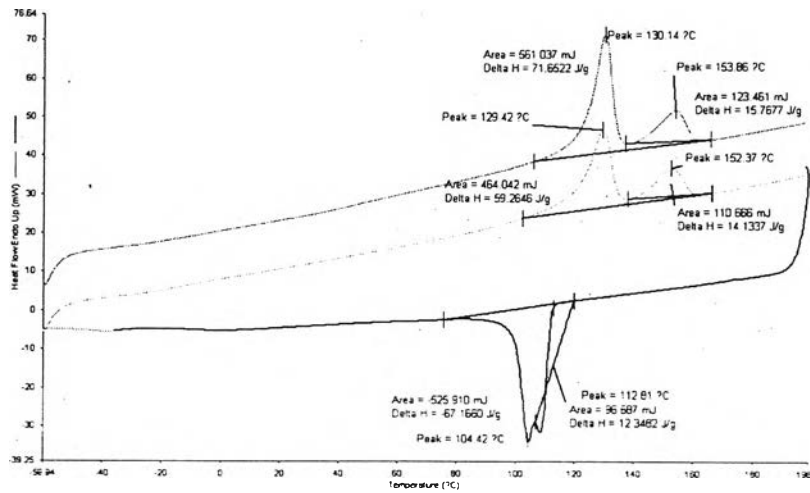


Figure C.6 DSC curve of PE-b-PP

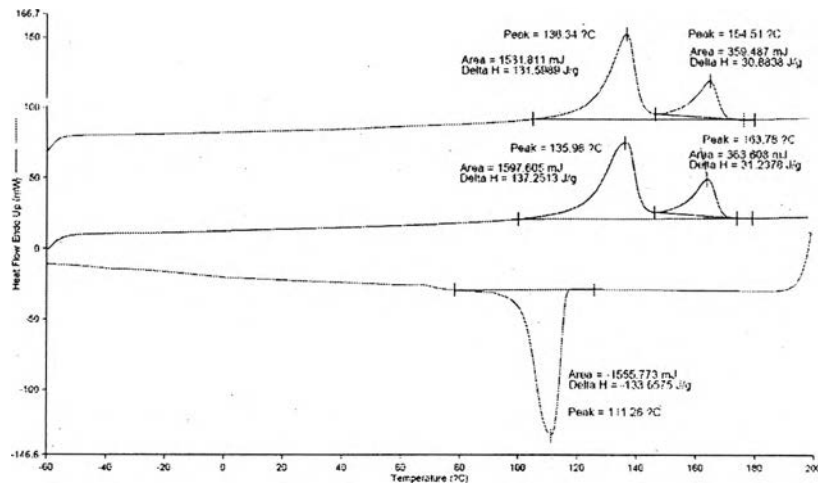


Figure C.7 DSC curve of PE/PP blended with 3%PE-b-PP

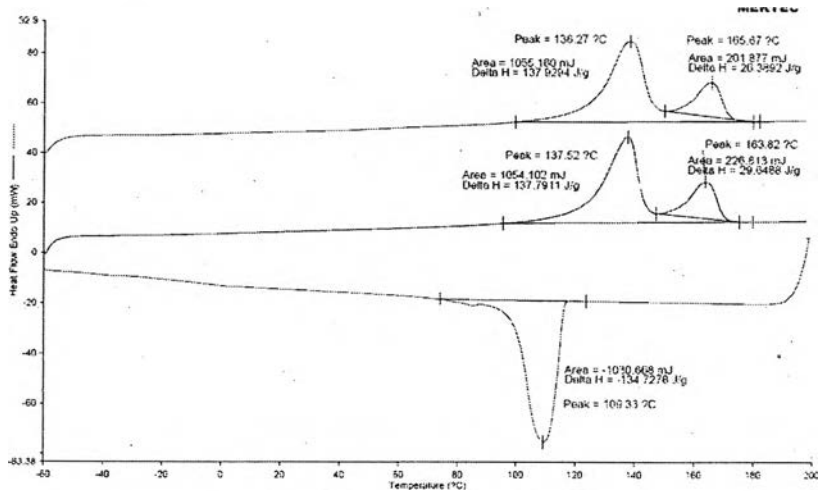


Figure C.8 DSC curve of PE/PP blended with 6%PE-b-PP

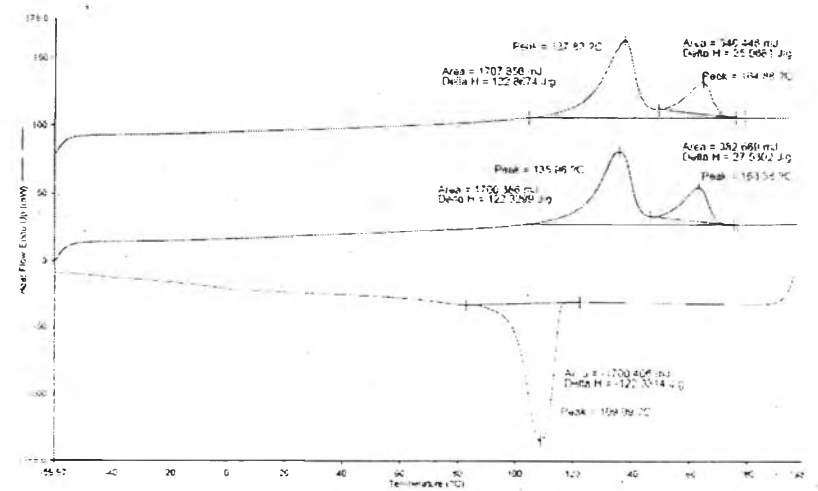


Figure C.9 DSC curve of PE/PP blended with 12%PE-b-PP

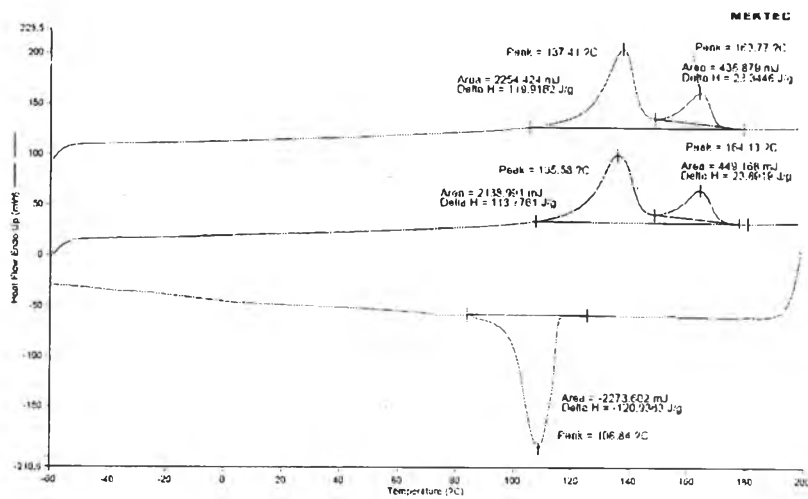


Figure C.10 DSC curve of PE/PP blended with 20%PE-b-PP

## Appendix D

### The Data of DMA Characterization

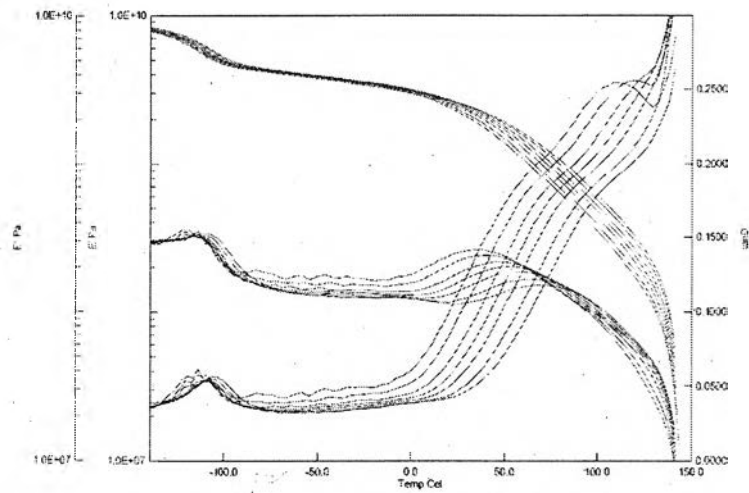


Figure D.1 DMA curve of PE

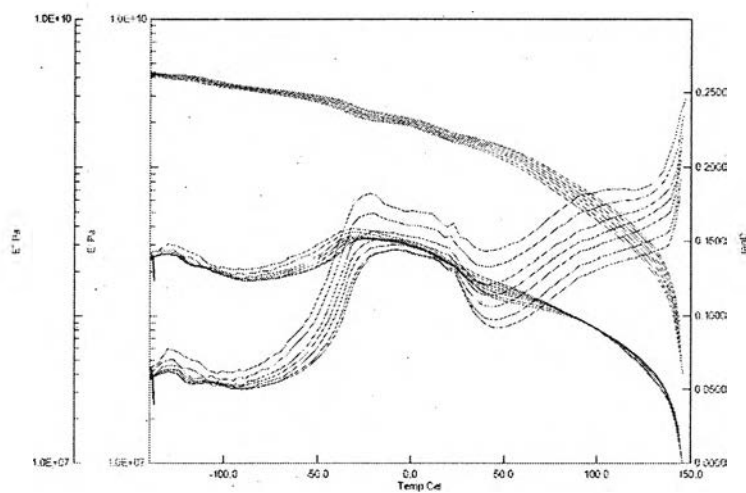


Figure D.2 DMA curve of PE/PP blend

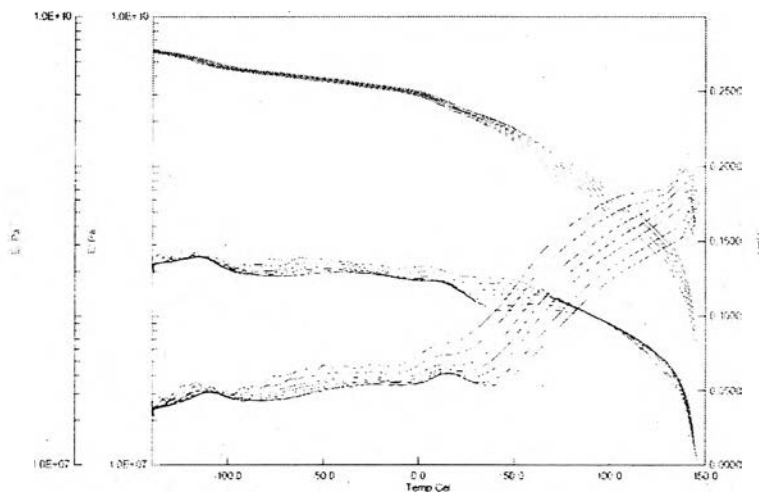
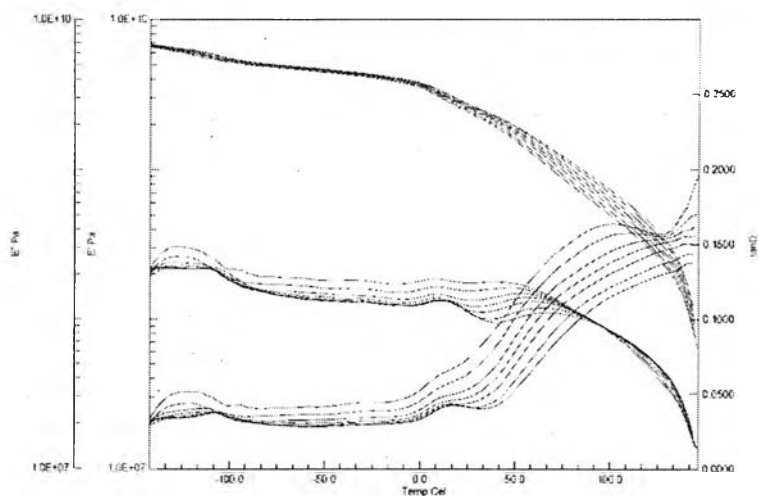
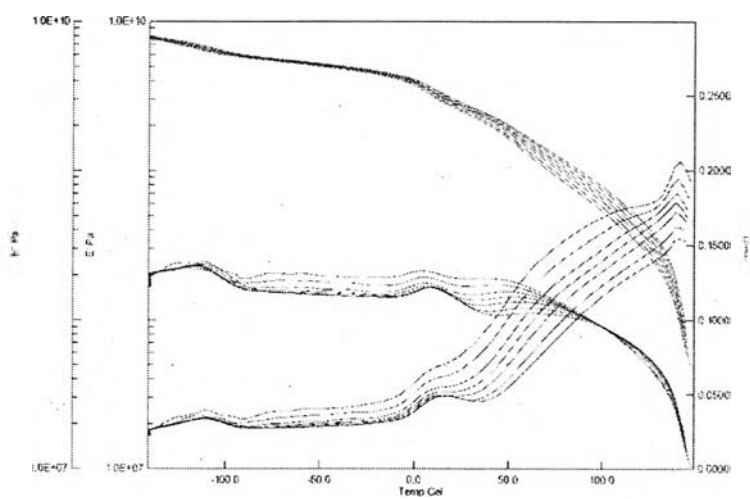


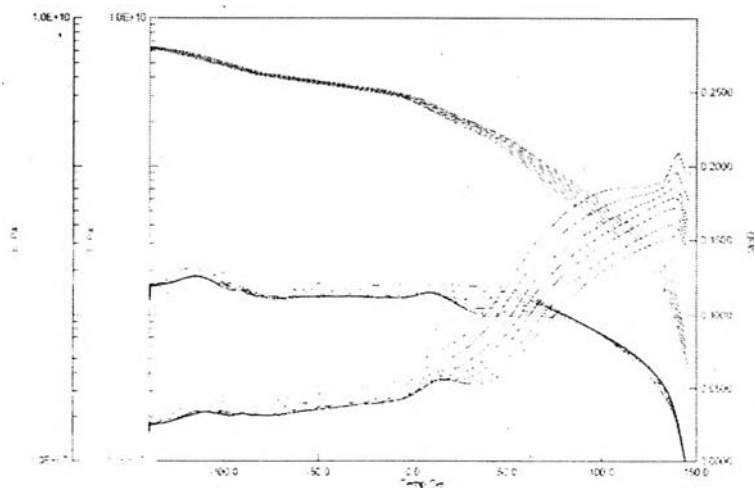
Figure D.3 DMA curve of PE/PP blended with 3% PE-b-PP



**Figure D.4** DMA curve of PE/PP blended with 6% PE-b-PP



**Figure D.5** DMA curve of PE/PP blended with 12% PE-b-PP



**Figure D.6** DMA curve of PE/PP blended with 20% PE-b-PP

**Appendix E****The Submitted Paper To Journal of Applied Polymer Science**



## Reinforcement of Polyethylene-Polypropylene mixtures by adding synthesized diisocyanate compatibilizers

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### Abstract

Immiscible and incompatible binary blends of commercial polypropylene(PP)/polyethylene(PE) display poor mechanical properties. The addition of compatibilizer to reinforce and enhance an adhesion at the interfaces between polyethylene-polypropylene mixtures has been conducted. The compatibilizer chosen was in the group of Ziegler-Natta's polyethylene-polypropylene block copolymer with diisocyanate linkage. The effects of adding the compatibilizers were assessed by morphology studies, thermal analysis and mechanical testing. DSC curves of crystallization and FTIR provided evidences to support the formation of PP/PE block copolymer. Significant improvements in the mechanical properties of 50/ 50 PE/PP blends containing compatibilizer have been noted.

**Keywords:** PE/PP blend, Polyethylene-polypropylene block copolymer, reinforce

### 1. Introduction

Polyethylene (PE) and Polypropylene(PP) are among the most common plastic wastes, because they are among the most frequently used commercial plastics in our daily lives as well as in industries.[1] It's impossible and not appropriate to identify and totally separated waste mixtures of this two polymers. Usually their waste mixture can recycle as mixed waste plastics in the form of blends. This reuse approach is attractive, because it avoids the difficult task of separation. As a consequence, academic and industrial interest in virgin and recycled polymer blends is rapidly expanding.

It is well known that homopolymer of polypropylene predominately used for high modulus applications but shows unsatisfactory impact strength at subzero temperatures, owing to its relatively higher glass transition temperature and its large dimension of spherulite. In contrast, PE has a ductile-to-brittle transition temperature well below that of PP. Combining the low temperature ductility of PE with the high stiffness of PP has the potential to significantly expand the applications base of these two polyolefins.

Unfortunately, the incompatibility between PE and PP has already been reported by various authors [2]. The strong phase separation leading to a coarse phase structure and the low interfacial adhesion between the phases is responsible for a decrease in mechanical properties especially related to its morphology, including impact strength, strain at break and ductile to brittle transition. According to Shanks [3] the immiscibility between the phases makes the rule of mixtures ineffective in predicting some properties of interest.

To overcome this difficulty, the usages of various coupling agents have been reported. Incorporating a compatibilizer into a multiphase system generally leads to a fine phase structure and results in the enhanced interfacial adhesion. Amongst others

[4-6], Yang [7] showed that the addition of a commercial ethylene/propylene block copolymer improved the ductility of LDPE/PP blends, particularly for PP rich blends. Bertin, J.J. et al. [8] studied and characterized virgin and recycled LDPE/PP blends and the usage of compatibilizing agents, such as ethylene-propylene-diene monomer copolymer (EPDM) or PE-g (2-methyl-1,3-butadiene) graft copolymer, to enhance their impact strength and elongation at break. Claudia M.C. et al. showed the partial compatibility of the PP/HDPE reflected in the improvement of tensile strength and elongation by the addition of extracted recycled PP.[9] Although this may solve the compatibility problem, the use of compatibilizers adds cost to the recycled product, usually resulting in loss of interest from the recycling sector.

In this present work, we try to synthesize the cost effective compatibilizer in simple system that can improve the mechanical properties of PE/PP mixtures. Thus we applied the rapid reaction between a polyfunctional isocyanate and a hydroxyl terminated oligomer leads to urethane linkage. Consequently, the addition of Ziegler-Natta PE/PP block copolymer synthesized by diisocyanate has the ability to reinforce the polyethylene-polypropylene mixtures as it is expected. Thermodynamically the PP-b-PE will sit at the interface between the two components. In this work, the morphology thermal properties and tensile properties of PP/PE blends were evaluated.

## 2. Experimental

### 2.1 Chemicals

Commercial grade polypropylene and polyethylene were donated from Thai Polyplastic Industry. Polymerization-grade propylene, and ethylene were donated from National Petrochemical Corporation Co., Ltd., Thailand. The  $\text{AlEt}_3$  (TEA) and MDI were donated from Bangkok Polyethylene Co., Ltd., Thailand. The  $\text{TiCl}_4$  were purchased from Merck Ltd. Anhydrous  $\text{MgCl}_2$  was supplied from Sigma-Aldrich Inc., Phthalic anhydride, Diethylphthalate (DEP, used as an internal donor) and n-Decane were purchased from Fluka Chemie A.G. Switzerland. Hexane was donated from Exxon Chemical Thailand Ltd. It was purified by refluxing over sodium/benzophenone under argon atmosphere prior to use. Ultra high purity (UHP) argon (99.999%) and oxygen (UHP) was obtained from Thai Industrial Gas Co., Ltd. and was further purified by passing through molecular sieves  $3\text{\AA}$ , BASF catalyst R3-11G, NaOH and phosphorus pentoxide ( $\text{P}_2\text{O}_5$ ) in order to remove traces of oxygen and moisture.

All operations were carried out under an inert atmosphere of argon using a vacuum atmosphere glove box and/or standard Schlenk techniques.

### 2.2 Catalyst preparation

A catalyst of type  $\text{TiCl}_4/\text{DEP}/\text{MgCl}_2$  was synthesized in the following manner. 0.476 g of anhydrous  $\text{MgCl}_2$  was added to 2.5 ml of n-decane. This suspension was treated with 2.34 ml of 2-ethylhexanol and 0.1089 g of phthalic anhydride at  $130^\circ\text{C}$ . It was stirred until the  $\text{MgCl}_2$  was dissolved. 20 ml of  $\text{TiCl}_4$  was added dropwise at  $-20^\circ\text{C}$ , subsequent treatment of the solution in the presence of 0.26 ml of diethylphthalate (DEP) at  $110^\circ\text{C}$  for 2 h. The resulting solid product was separated by filtration and the addition of 20 ml of  $\text{TiCl}_4$  was repeated at room temperature. After keeping the solution at  $120^\circ\text{C}$  for 2 h, this slurry was siphoned off and washed twice

with 10 ml of n-decane and three times with 10 ml of hexane, respectively. The catalyst was dried under vacuum for 30 min at 40°C and contained 3% Ti.

## 2.2 Polymerization reaction

### 2.1.1 Polyethylene and polypropylene terminal hydroxyl group

The propylene polymerization and ethylene polymerization reactions were carried out in a 100 ml semi-batch stainless steel reactor equipped with magnetic stirrer. 26.55ml Hexane, 0.01 g catalyst (Al/Ti molar ratio = 167) and 3.45 ml TEA were introduced into the reactor and stirred for 5 minutes at room temperature in the atmospheric glove boxes. Followed by immediately put the reactor in liquid nitrogen to stop reaction between the catalyst and cocatalyst. After the solution was frozen for 15 minutes, the reactor was evacuated for 3 minutes to remove argon. The reactions were conducted at 60°C. Polymerization was started by continuous feeding ethylene (propylene) at constant pressure of 50 psi for 1 hr. Then stopped the polymerization by directly brought into contact with oxygen gas at room temperature followed by precipitation in hydrochloric acid solution in methanol and dried at room temperature.

### 2.1.2 Polyethylene block polypropylene copolymerization

Copolymerization was carried out in a glass reactor equipped with magnetic stirrer. Polyethylene and polypropylene containing hydroxyl group 50/50 %wt were added and dissolved in *o*-dichlorobenzene at 120 °C. Follow by dropped of excess MDI in the solution that remained stir for 1 hr. The solution was washed with excess methanol. Polymer was filtered and dried.

## 2.2 Blend and molding preparation

The melt mixing method was performed in digital hot plate stirrer at 220 °C. All blends were prepared with 50 wt% of PE and 50 wt% of PP (both polymers are commercial grade). When the block copolymer was used, 3 wt% of the block copolymer was added base on the total weight of the 50/50 blend Added the block copolymer in PE/PP blend for 3, 6 ,12 , 20 %wt. All polymer blends was molded with The LAB TECH hydraulic hotpress LP-50 M/C 9701.

## 2.3 Measurements and characterization of block copolymers and blends

Infrared survey spectra were recorded with FTIR (IFS28) The scanning ranged from 400 to 4000  $\text{cm}^{-1}$  on the pallet sample of KBr powder with scanning 12 times.

The molecular weight and its distribution were determined by using GPC Model: Waters 2000 Column: Styragel HT6E with 1,2,4-Trichlorobenzene as a solvent at 135 °C.

The melting temperatures of the block copolymer added polymer blends were determined with a Perkin-Elmer DSC-Pyris Diamond over the range -60 to 200 °C of scanning rate 40 °C/min under nitrogen atmosphere.

Tensile properties were characterized using an Instron universal testing machine with a test speed of 12.5 mm/min. The tests were conducted according ASTM D 882-02.

Dynamic mechanical properties of blending polymers were determined by using Dynamic Mechanical Analysis (DMA) with a Perkin-Elmer DMA-Pyris Diamond operated at a fixed frequency of 10 Hz. All the experiments were carried out in a bending mode over the temperature range of -130 to 150 °C at a temperature

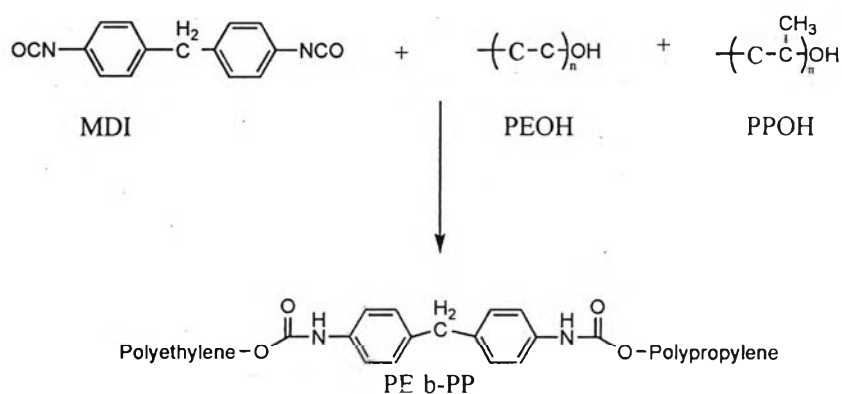
ramp of 1.5 °C/min, using liquid nitrogen as a cryogenic medium. The dimensions of the test specimens, which were cut out from compression moulded plaques, were 10x50x0.5 mm. The data were processed using proprietary software.

The morphologies of all block copolymers were investigated by JSM-5410LV Scanning Microscope. The samples for SEM analysis were coated with gold particles by ion sputtering device to provide electrical contact to the specimens.

## Results and Discussion

### 3.1 Chain structure of PE/PP block copolymer diisocyanate linkage

On the basis of this result, a plausible products of the block copolymerization is proposed as shown in (Scheme 1)



Scheme 1.

In addition, beside PE-b-PP, there have others 2 byproducts of the reaction which are PE-b-PE and PP-b-PP. Moreover there are others two products that end chain with hydrogen (not hydroxyl, OH) that are PE, PP and the small amount of remaining reactant, that are PEOH, PPOH. The fractions of these byproducts are hardly to be quantified and it can not be completely fractionated by solvent extraction. Thus unidentified fraction distribution of the block copolymers will be along with what is identified as PE-b-PP throughout this study. However, the mixture identify as PE-b-PP had very good phase binding with the melted blend of PP/PE that will be later discuss. Consequently the mixture of PE-b-PP copolymer in this study (PE-b-PP, and various size of PP, PE) comprised the phases that will dissolve in the melted blend of PP/PE without any difficulty.

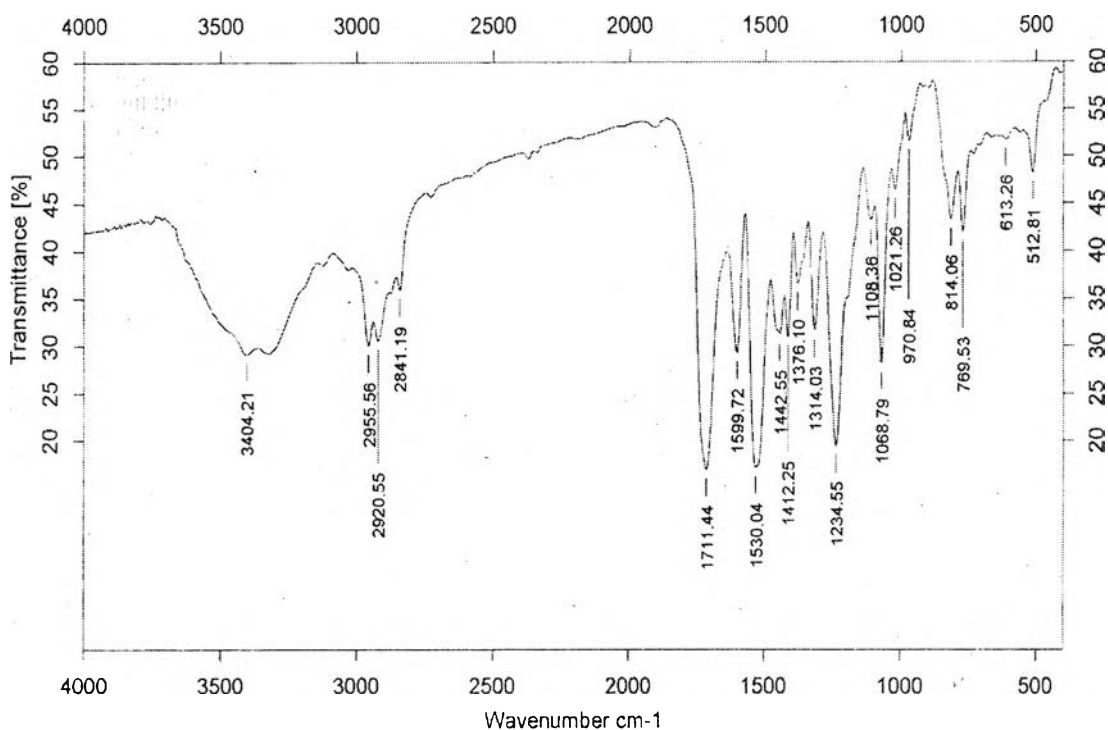


Fig. 1. IR-spectra of PE-b-PP at 25 °C

From GPC results, the PE-b-PP has very wide molecular weight distribution as a result from the reaction of wide MWD of PPOH and PEOH with diisocyanate. In order to confirm the reaction that contribute to the block copolymer of PE-b-PP in this system, the IR spectrum of block copolymer obtained at 25 °C is illustrated in Figure 1. The peak of isocyanate (NCO) transmittance is  $1530\text{ cm}^{-1}$ ,  $\nu_{\text{C=O MDI}} = 1711\text{ cm}^{-1}$ ,  $\nu_{\text{NH}} = 3404, 1599$  and  $814\text{ cm}^{-1}$ . Thus IR spectrum has identified the diisocyanate linkage in PE-b-PP copolymer structure. More over the binding properties of PE-b-PP can be confirmed in SEM figure followed.

Table 1. Characterization of polymer

Polymer	Mw <sup>a</sup> $\times 10^{-4}$	MWD <sup>a</sup>	Heating 1(°C) <sup>b</sup>				Cooling (°C) <sup>b</sup>				Heating 2(°C) <sup>b</sup>		
			Tm <sub>1</sub>	Tm <sub>2</sub>	Tg	$\Delta H_1$	Tc <sub>1</sub>	Tc <sub>2</sub>	$\Delta H_2$	Tm <sub>1</sub>	Tm <sub>2</sub>	Tg	$\Delta H_3$
PEOH	117.3	7.5	145	Nd	Nd	181.4	110	Nd	123.6	137	Nd	Nd	118.9
PPOH	39.5	8.7	Nd	156	-5	40.7	100	Nd	58.8	Nd	154	-5	51.4
PE-b-PE	Na	Na	135	Nd	Nd	255.4	108	Nd	83.2	132	Nd	Nd	84.1
PP-b-PP	Na	Na	93	158	-7	32.5	102	Nd	28.7	Nd	153	-8	29.7
PE-b-PP	58.3	12.5	130	153	-6	71.7	113	120	96.7	129	152	-7	59.3
PE/PP	32.4	6.1	135	151	-6	68.3	98	110	67.1	135	151	-6	64.7
3%PEbPP	Na	Na	136	163	Nd	131.6	111	Nd	133.7	134	164	Nd	137.3
6%PEbPP	Na	Na	138	166	Nd	137.9	109	Nd	134.7	138	164	Nd	137.8
12%PEbPP	Na	Na	138	165	Nd	122.9	109	Nd	122.3	136	163	Nd	122.3
20%PEbPP	Na	Na	137	164	Nd	119.9	109	Nd	120.9	136	164	Nd	113.9

a Determined by gel permeation chromatography, PS standard

b Determined by DSC,  $\Delta H = (\text{J/g})$

Na not available

Nd not detected

As shown in Table 1. The PEOH has larger molecular weight compared to PPOH. Consequently, the molecular weights of PE-b-PP are higher than PPOH. The  $\Delta H$  of the melted blend of pure PE/PP lower than all of the PE-b-PP addition samples, this implied that the crystallinity of melted blend of pure PE/PP are increased when added with PE-b-PP. In other words, the copolymer enhanced the crystallization of both PE and PP in the melted blend of PE/PP. From the highest  $\Delta H$ , the largest percent of crystallinity is at 6% PE-b-PP in PE/PP. This may be the results in the highest reinforcement because of the formed crystal and contribute to the highest tensile properties. In addition, portion of  $T_m$  that represented the PP crystal in PE/PP were increased from pure PE/PP in all the composition of added PE-b-PP. Usually  $T_m$  describing the quality of crystallinity in polymer blend (form crystalline and decrease entanglement polymer blend). This can be concluded that the addition of PE-b-PP alter both the quality and quantity of the crystallinity of PE/PP.

The chain structure of polymer blend and block copolymer was studied by DSC analysis of crystalline segregated samples. After stepwise annealing of the samples at different temperatures, the long PP and PE segments can form crystalline lamellae of different thickness according to their sequential lengths, and these lamellae will melt at different temperatures [10]. By recording the endothermic curves of the polymer blend and block copolymer in a DSC scan, we are able to judge the sequential contribution of PE/PP blend and role of PE-b-PP in crystallinity. As shown in figure 2.

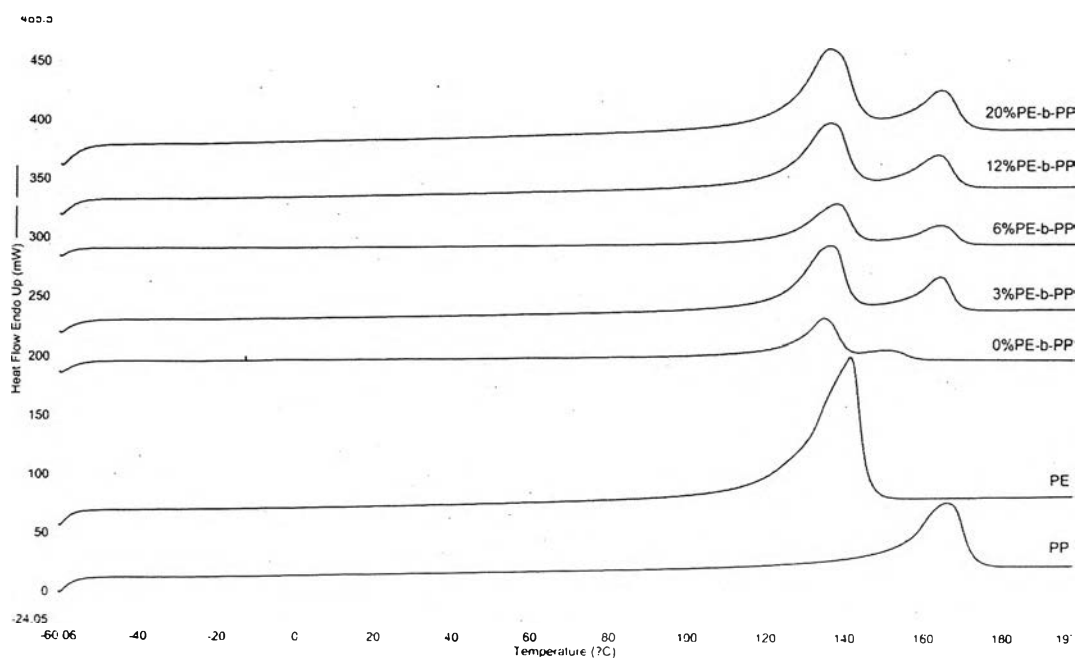


Fig . 2. DSC endotherms of PP/PE blend and addition of PE-b-PP to PE/PP blend after thermal segregation treatment.

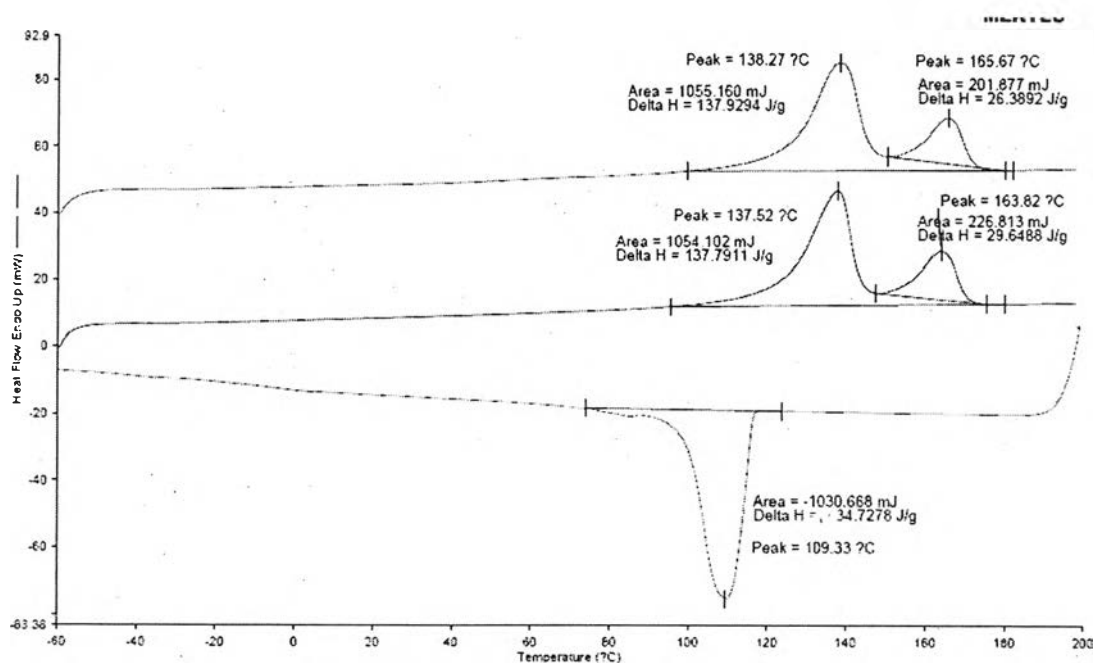


Fig. 3. DSC curve of the addition of 6% PE-b-PP to PE/PP blend .

The melting peak at about 130-140 °C correspond to the melting temperature of PE crystal and the peak above 140 °C correspond to the melting temperature originate from PP crystal. However the appearance of the curves of PE/PP blend and PE-b-PP are similar. In the cases of adding PE-b-PP to PE/PP blend, the melting temperatures of PP increase (as shown in Table 1.)

### 3.2 Mechanical properties

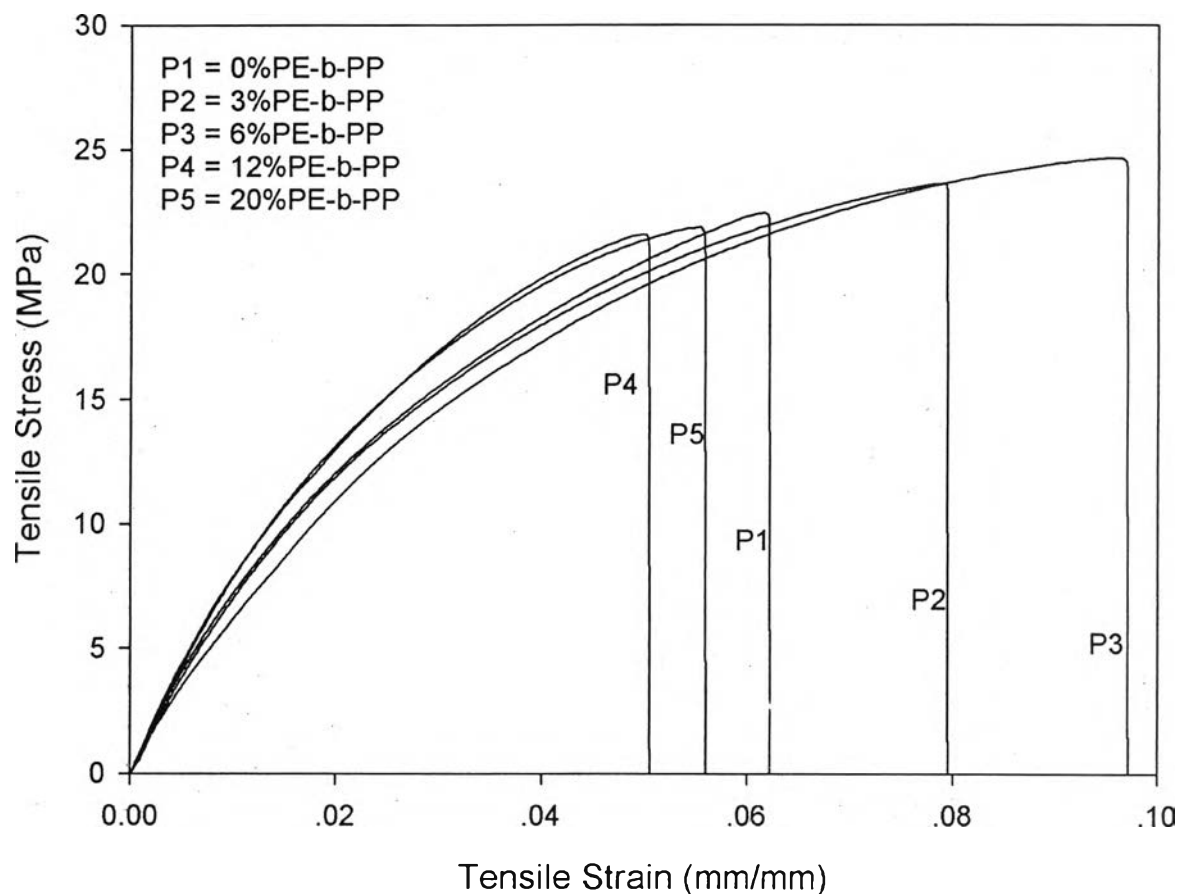


Fig. 4. Additive effect of PE-b-PP to PE/PP blend on stress -strain behavior at 25 °C

The results of tensile stress tests are shown in Fig. 4. As the concentration of PE-b-PP in melted blend of pure PE/PP increased, both the tensile strength and the elongation at break increased. These show that the addition of PE-b-PP to PE/PP can improve the reinforcement of polymer by increasing the interfacial adhesion between PE and PP. At 6% PE-b-PP in PE/PP, the highest tensile stress of the blend occurred, which resulted from more stiffness and toughness of the samples. Thus the tensile properties of PE-b-PP in PE/PP were in agreement with DSC results as we discussed above that the 6%wt PE-b-PP has the most reinforcement characteristics. The reasons of decreasing tensile strength and elongation at break at 12% and 20% PE-b-PP contents might be because of the lower percent crystal in the samples together with the increase of PE, PP pure from PE-b-PP that have the higher molecular weight than the commercial PE/PP. The amount of high molecular weight might result in the more incompatible of PE/PP.



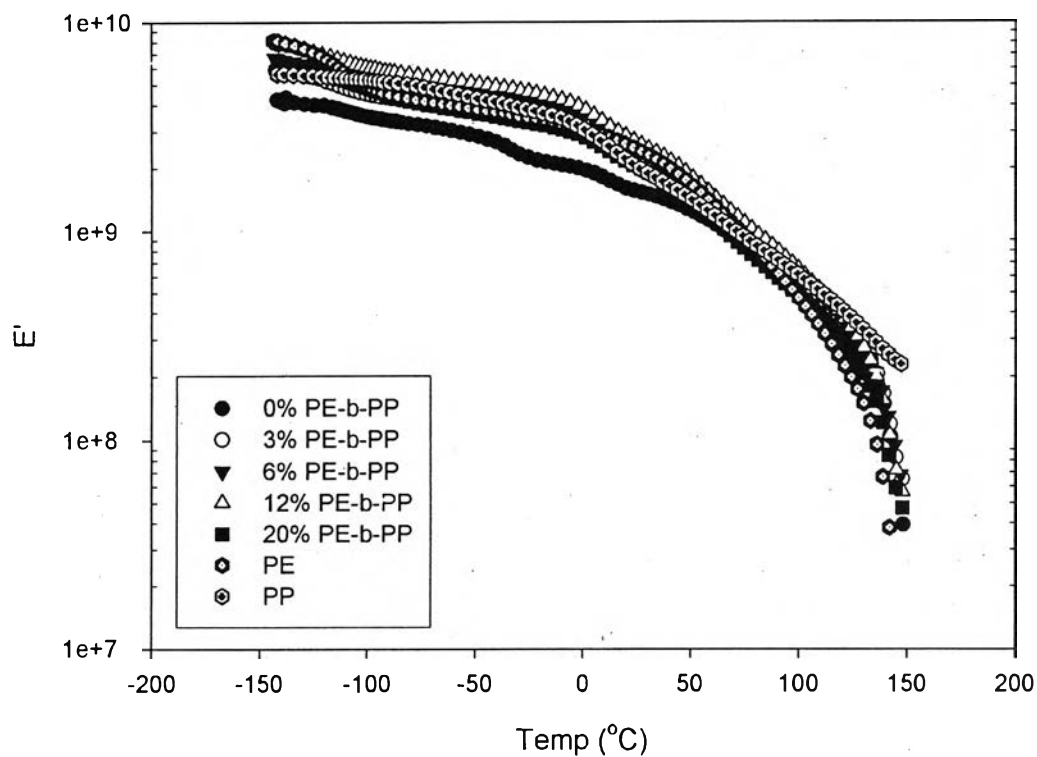


Fig.5 DMA curves ( storage modulus ( $E'$ ) and temperature) for PE/PP and compatibilised PE/PP at 1 Hz, heating rate = 1.5 °C/min , tensile holder, size (W\*L\*T) = 10\*50\*0.5 mm.

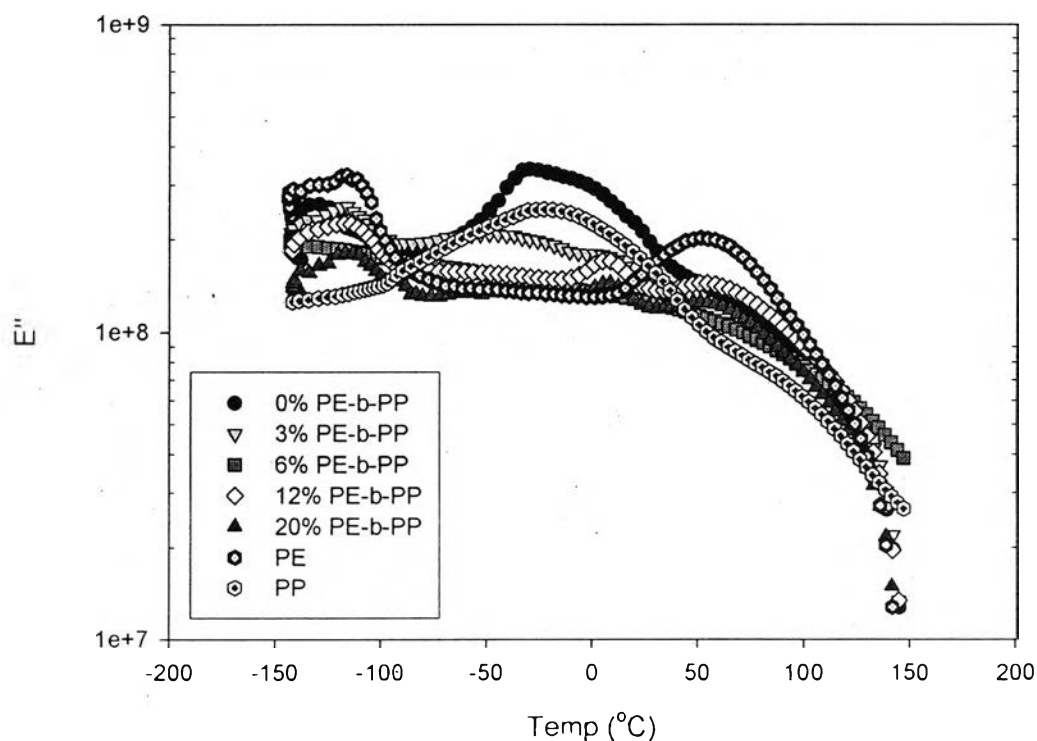


Fig.6 DMA curves ( loss modulus ( $E''$ ) and temperature) for PE/PP and compatibilised PE/PP at 1 Hz, 1.5 °C/min , tensile holder, size (W\*L\*T) = 10\*50\*0.5 mm.

Figure 5, 6, 7 compared the effects of temperature on the dynamic mechanical properties of compatibilized polymer blend. The DMA properties were measured for both pure components and melted mix of the pure components (PP, PE, PE/PP blend) and their physical blends (of PE/PP) with various PE-b-PP contents at 1 Hz. From Figure 5, the compatibilized PE/PP blends show that the storage modulus ( $E'$ ) that decreased with increasing temperature as usual in thermoplastics. At low temperature DMA, PE has more stiffness and strength properties than PP especially below  $0^{\circ}\text{C}$ . In the melted blend of PE with PP, the moduli are lower than both pure components because PE&PP are immiscible and strong phase separation occurred. As already proof that PE and PP were immiscible because of structural consideration, though both exhibit the same spherulitic morphology, the two polymers may not be compatible in bulk since polyethylene crystallizes in the more stable orthorhombic form whereas isotactic polypropylene crystallizes in the monoclinic  $\alpha$ -form thus the decrease of interfacial adhesion follows which causes loss of the mechanical properties of the blend.[11] After adding PE-b-PP to PE/PP, the storage modulus of various compositions of PE-b-PP all increased from the pure PE/PP at all temperature. The maximum of the average storage modulus was at 12%wt PE-b-PP in PE/PP blend. At 20%wt PE-b-PP in PE/PP, the samples are more ductile and less stiff than at 12% PE-b-PP content because the blend has too high portions of high molecular weight of PE, PP that contaminated in PE-b-PP. As usual, the portions of high molecular weight in the blend will result in phase separation of the high molecular weight species easier than low molecular weight portions [12].

From the loss modulus ( $E''$ ) as shown in Figure 6, a  $\alpha$  relaxation (indicating strong mobility of polymer molecules) can be seen for all compatibilized polymers and virgin PE, PP. These  $\alpha$  relaxations, indicated by the peak in the  $E''$  curves, occur at temperatures ranging from  $-115$  to  $-100^{\circ}\text{C}$  and from  $-20$  to  $20^{\circ}\text{C}$  for compatibilized polymers. For virgin PE/PP, relaxation temperatures occur at temperature ranging from  $-130$  to  $-120^{\circ}\text{C}$  and from  $-50$  to  $20^{\circ}\text{C}$ , which are lower than when consisted of PE-b-PP. This showed that the a relaxation temperature of PE/PP blends composed of PE-b-PP increased due to increased in percent of crystallinity and less flexible amorphous part remained.

Figure 7 shows the effects of temperature on the tan delta responses of various blend and virgin PE/PP. It's well known that block copolymers should exhibit the glass transition of each of the respective homopolymer component as same as polymer blends[13,14]. The  $T_m$  above  $30^{\circ}\text{C}$  has contribution for both PP and PE crystalline segments[15,16]. The  $Tg_2$  relaxation between  $-20^{\circ}\text{C}$  and  $+20^{\circ}\text{C}$  is the glass transition of polypropylene [15]. The  $Tg_1$  peak at about  $-125^{\circ}\text{C}$  represents the relaxation of the methylene group [16]. Both  $Tg$  of PE and PP components in compatibilised PE/PP (with PE-b-PP) increase when compare with virgin PE/PP. For pure PE/PP blend, value of  $\tan\delta$  is high according to high amorphous phase, as has been concluded from Table 1. The lower prediction of  $Tg$  and the high magnitude of  $\tan\delta$  might result from the increase in the free volumes of the immiscible virgin PE/PP blend that directly affected the increase flexibility of the amorphous part when higher free volume obtained. The addition of PE-b-PP in PE/PP increase the  $Tg, T_m$  to the value of the pure component PE and PP.

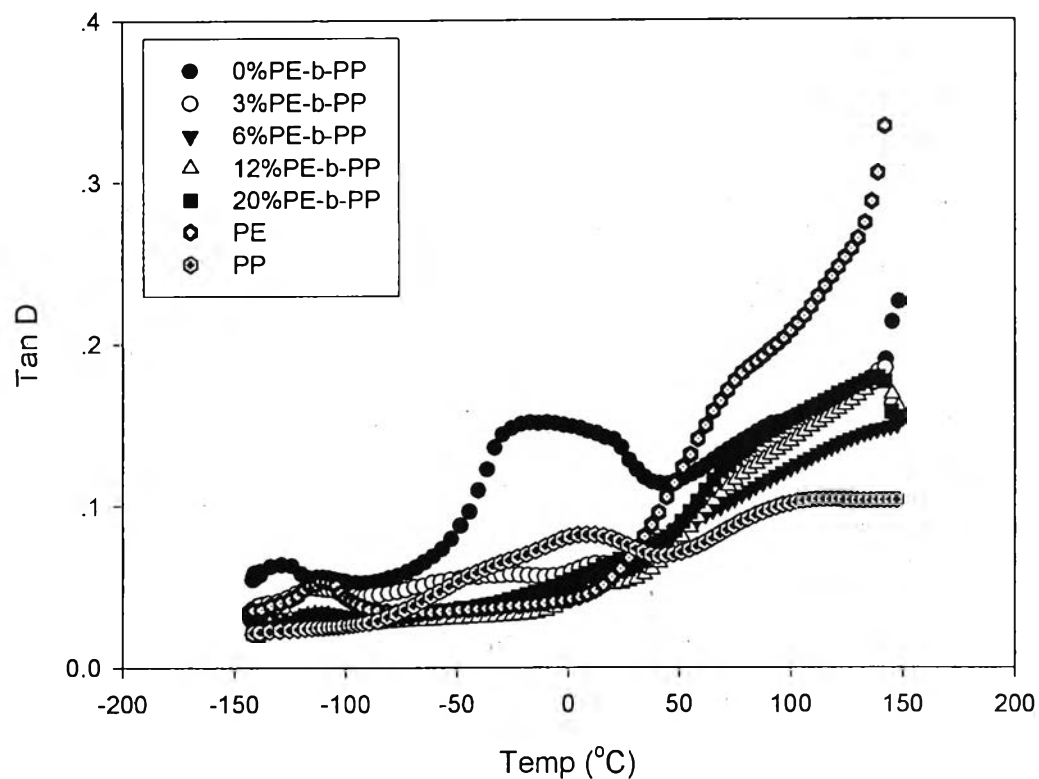


Fig.7 DMA curves ( $\tan\delta$  and temperature) for PE/PP and compatibilised PE/PP at 1 Hz, heating rate = 1.5 C/min, tensile holder, size (W\*L\*T) = 10\*50\*0.5 mm.

Table 2 : Prediction of  $T_g$ ,  $T_m$  from DMA experiment from Figure 7

polymer	$T_{g1}$	$T_{g2}$	$T_m$
PE	-110	nd	nd
PP	nd	12	nd
0%PE-b-PP	-128	-2	Nd
3%PE-b-PP	-110	15	138
6%PE-b-PP	-110	12	141
12%PE-b-PP	-110	12	138
20%PE-b-PP	-110	16	141

## 3.3 Morphology

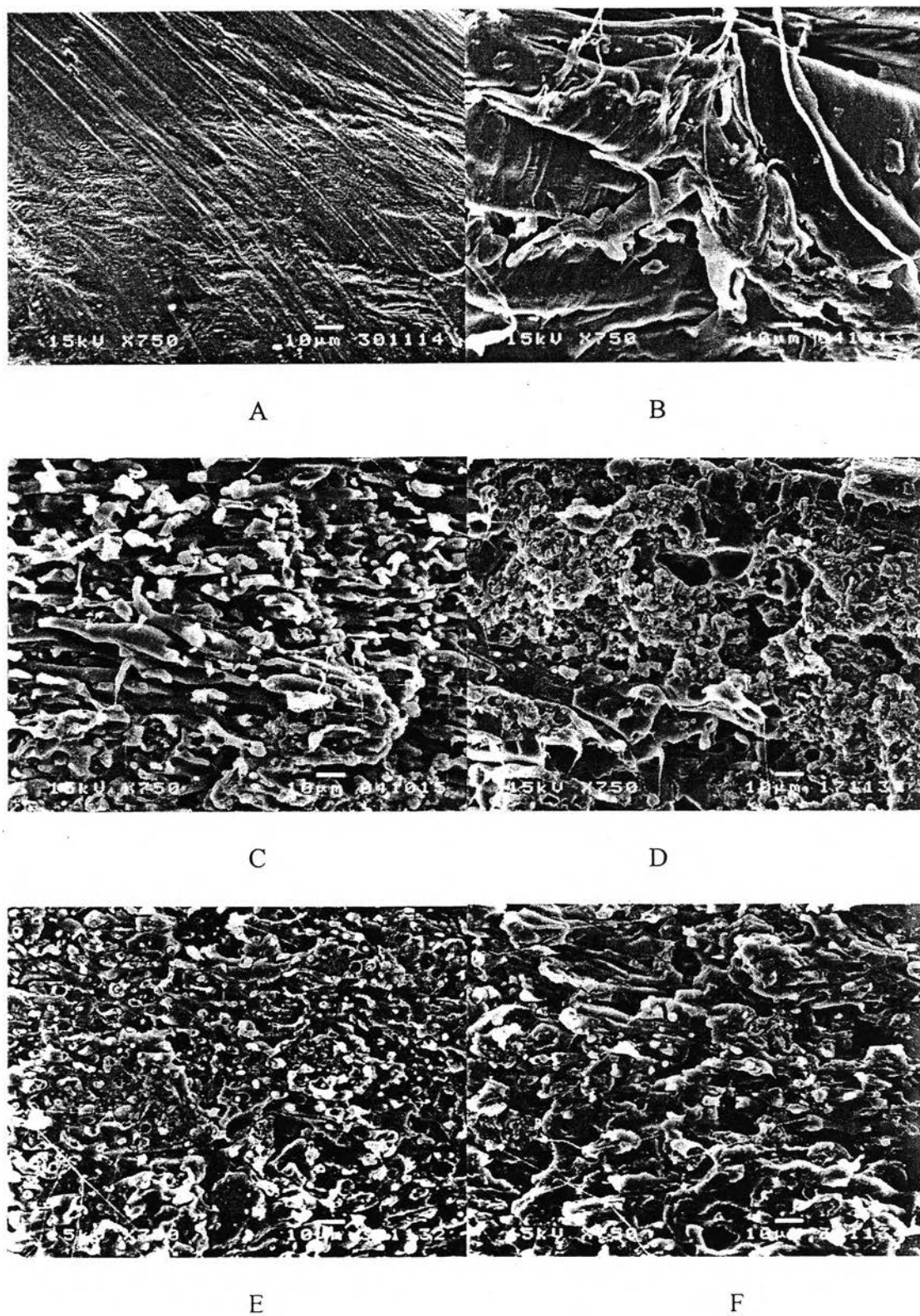


Fig. 8. SEM of tensile fracture surface of a) PP b) PE/PP blend c) PE/PP+ PEbPP3% d) PE/PP+ PEbPP6% e) PE/PP+ PEbPP12% f) PE/PP+ PEbPP20%

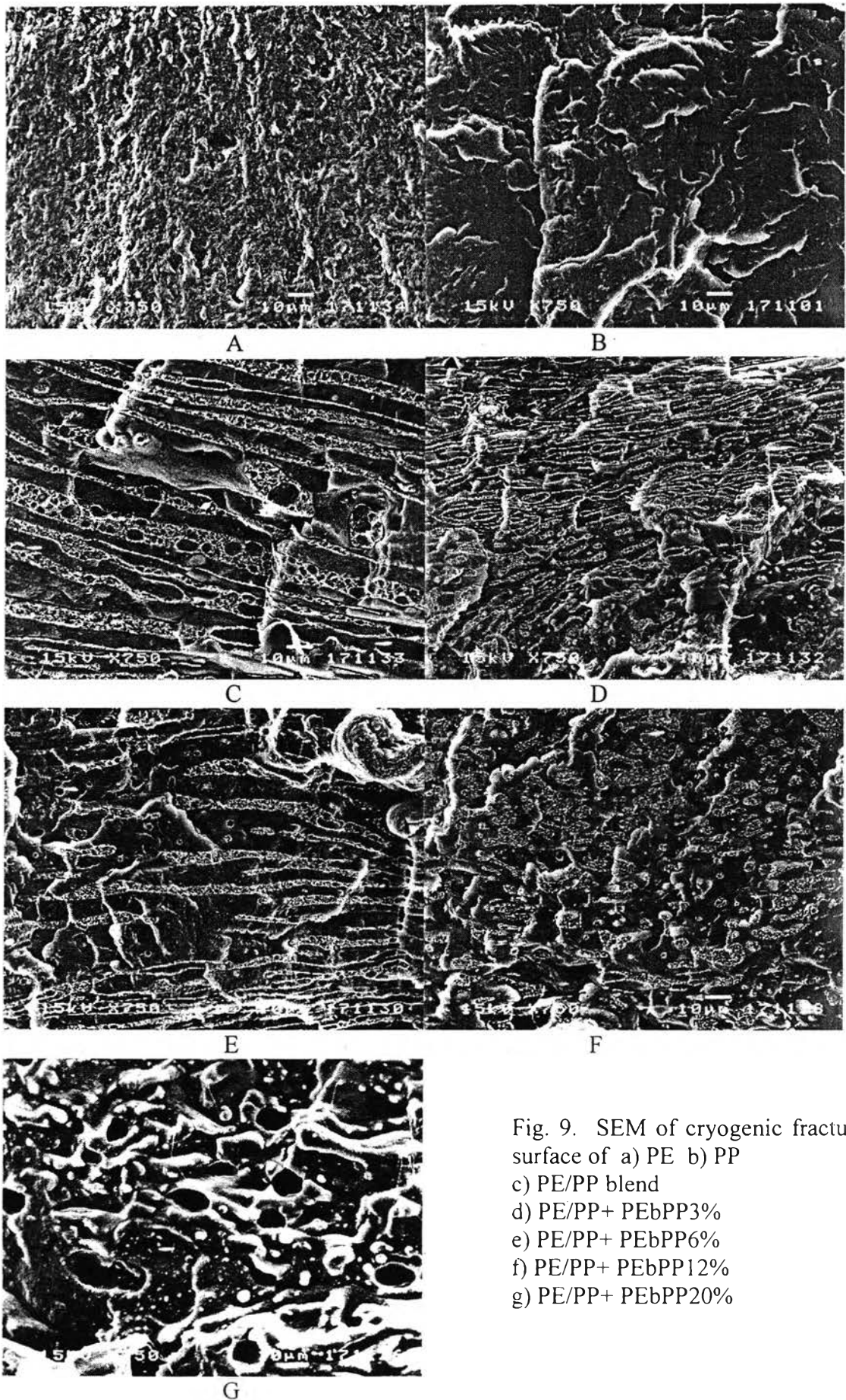


Fig. 9. SEM of cryogenic fracture surface of a) PE b) PP c) PE/PP blend d) PE/PP+ PEbPP3% e) PE/PP+ PEbPP6% f) PE/PP+ PEbPP12% g) PE/PP+ PEbPP20%

According to SEM picture, it clearly shows the differences of the rough surface particles and the bridge formation with PP matrix of PE/PP blend which continuously changed according to the concentration of PE-b-PP. The addition of PE-b-PP to PE/PP blend vividly shows the smaller phase particles size as increased concentrations. Many studies [17-20] on polymer alloys have shown that for multiphase polymer systems, the toughening effect is determined by two factors. First, the smaller the particles and the narrower the particles size distributions are, the better impact strength is. Secondly, the stronger is the adhesion between particle and the matrix; the better is the impact properties.

The SEM micrographs of compatibilized PE/PP blends (3%, 6%, 12%, 20% PE-b-PP) can be shown in Figure 8,9. Figure 8 shows the tensile fracture of PE/PP and compatibilized PE/PP, while indicated that the interfacial adhesions, and therefore the compatibility of the PE and PP phases, are better than uncompatibilized PE/PP. In room temperature fracture experiments, the PE are in the form of tough rubbery polymer compare to PP. These might be shown as the stretch rubbery structure in the blends. The cryogenic fracture of the similar blends will result in the clear domain size because at the cryogenic temperature both PE/PP are in the glassy states and the fractures cut directly to the cross-sections of the segregation size in the blends.

Fig.9 shows the cryogenic fracture of PE/PP and compatibilized PE/PP, while indicated that the clearly decrease domain sizes (dispersion of PE in PP) and finer particle size distributions. As might be confirmed by the SEM, the phase segregation decrease deliberately but the clear second dots of PE/PP are captured in the 12%, and 20%wt PE-b-PP which might be the reason for the weaker interfacial ability than the 6%wt PE-b-PP. Those samples have lower tensile properties than the 6%wt PE-b-PP. These phenomena can be confirmed by mechanical properties. However, the DMA properties of 12%wt PE-b-PP showed the superior properties than all the blends. This might be because of the amorphous part of 12%wt PE-b-PP are larger than the 6%wt PE-b-PP, but the existing of the small segregations according to the high molecular weight PE could lower down the tensile properties of the 12%wt PE-b-PP. As can be observed from the GPC data, the amount of PE large molecular weight portions from PEOH and PE-b-PE can give the separated phases of segregations in the blend especially with the high content of PE-b-PP in the blend.

#### 4. Conclusion

A new synthesis method of PE-b-PP copolymer by converting the H-terminated chain-ends to hydroxyl-terminated ones and blocking with diisocyanate is performed the effective compatibilizer for immiscible blend of PE/PP. The diisocyanate linkages of PE-b-PP copolymers were confirmed by IR, which indicated that PE-b-PP copolymers occurred in the blocking reaction. The effects of PE-b-PP copolymers on the morphology of PE/PP blends were investigated by SEM with image analysis. The presence of the block copolymer dramatically reduced the phase size. Furthermore the mechanical properties, such as tensile strength, elongation at break and crystallinity have been improved due to PE-b-PP copolymers. The phase binding phenomena happened because the PE-b-PP contained PE and PP segments which attached and bound the PE/PP segregate phase blend, leading to superior properties via changing morphology than the normal mixing blend without PE-b-PP. As confirmed by DMA and DSC, the compatibilized blends showed the increase of crystallinity percentage. The optimum content of compatibilizer is 6%wt PE-b-PP which shows the best optimal values from DSC, DMA and SEM results.

**Appendix F**  
**The Proceeding of Technology and Innovation for sustainable Development**  
**Conference (TISD2006)**



## Block copolymer of Polypropylene and Polyethylene by way of diisocyanate

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### Abstract

Immiscible and incompatible binary blends of commercial polypropylene/ polyethylene display poor mechanical properties. The addition of compatibilizer to reinforce and enhance an adhesion at the interfaces between polyethylene-polypropylene mixtures has been conducted. The compatibilizer chosen was in the group of Ziegler-Natta's polyethylene-polypropylene block copolymer with diisocyanate linkage. The effects of adding the compatibilizers were assessed by morphology studies, thermal analysis and mechanical testing. DSC curves of crystallization and IR provided evidences to support the formation of PP/PE block copolymer. Significant improvements in the mechanical properties of PE/PP blends containing compatibilizer have been noted.

**Keywords:** PE/PP blend, Polyethylene-polypropylene block copolymer, reinforce

### 1. Introduction

It is well known that homopolymer of polypropylene predominately used for high modulus applications but shows unsatisfactory impact strength at subzero temperatures, owing to its relatively high glass transition temperature and its large dimension of spherulite. In contrast, PE has a ductile-to-brittle transition temperature well below that of PP. Combining the low temperature ductility of PE with the high stiffness of PP has the potential to significantly expand the applications base of these two polyolefins. Unfortunately, the incompatibility between PE and PP has already been reported by various authors [1]. The strong phase separation leading to a coarse phase structure and the low interfacial adhesion between the phases is responsible for a decrease in mechanical properties especially related to its morphology, including impact strength, strain at break and ductile to brittle transition. According to Shanks [2] the immiscibility between the phases makes the rule of mixtures ineffective in predicting some properties of interest.

To overcome this difficulty, the usages of various coupling agents have been reported. Incorporating a compatibilizer into a multiphase system generally leads to a fine phase structure and

results in the enhanced interfacial adhesion. Amongst others [3-5], Yang [6] showed that the addition of a commercial ethylene/propylene block copolymer improved the ductility of LDPE/PP blends, particularly for PP rich blends. Bertin, J.J et.al [7] studied and characterized virgin and recycled LDPE/PP blends and the usage of compatilizing agents, such as ethylene-propylene-diene monomer copolymer (EPDM) or PE-g (2-methyl-1,3-butadiene) graft copolymer, to enhance their impact strength and elongation at break. Although this may solve the compatibility problem, the use of compatibilizers adds cost to the recycled product, usually resulting in loss of interest from the recycling sector.

In this present work, we try to synthesized the cost effective compatibilizer in simple system that can improve the mechanical properties of PE/PP mixtures. Thus we applied the rapid reaction between a polyfunctional isocyanate and a hydroxyl terminated oligomer leads to urethane linkage. Consequently, the addition of Ziegler-Natta PE/PP block copolymer synthesized by diisocyanate has the ability to reinforce the polyethylene-polypropylene mixtures as it is expected. Thermodynamically the PP-b-PE will sit at the interface between the two components. In this work, the morphology, thermal properties and tensile properties of PP/PE blends were evaluated.

### 2. Experimental

#### 2.1 Chemicals

Commercial grade polypropylene and polyethylene were donated from Thai Polyplastic Industry. Polymerization-grade propylene, ethylene and AlEt<sub>3</sub> (TEA) and MDI were donated from Bangkok Polyethylene Co., Ltd., Thailand. TiCl<sub>4</sub> were purchased from Merck Ltd. Anhydrous MgCl<sub>2</sub> was supplied from Sigma-Aldrich Inc., Phthalic anhydride, Diethylphthalate (DEP, used as an internal donor) and n-Decane were purchased from Fluka Chemie A.G. Switzerland. Hexane was donated from Exxon Chemical Thailand Ltd. It was purified by refluxing over sodium/benzophenone under argon atmosphere prior to use. Ultra high purity (UHP) argon (99.999%) and oxygen (UHP) was obtained from Thai Industrial Gas Co., Ltd. and was further purified by passing



through molecular sieves 3Å, BASF catalyst R3-11G, NaOH and phosphorus pentoxide (P<sub>2</sub>O<sub>5</sub>) in order to remove traces of oxygen and moisture.

All operations were carried out under an inert atmosphere of argon using a vacuum atmosphere glove box and/or standard Schlenk techniques.

## 2.2 Catalyst preparation

A catalyst of type TiCl<sub>4</sub>/DEP/MgCl<sub>2</sub> was synthesized in the following manner. 0.476 g of anhydrous MgCl<sub>2</sub> was added to 2.5 ml of n-decane. This suspension was treated with 2.34 ml of 2-ethylhexanol and 0.1089 g of phthalic anhydride at 130°C. It was stirred until the MgCl<sub>2</sub> was dissolved. 20 ml of TiCl<sub>4</sub> was added dropwise at -20°C, subsequent treatment of the solution in the presence of 0.26 ml of diethylphthalate (DEP) at 110°C for 2 h. The resulting solid product was separated by filtration and the addition of 20 ml of TiCl<sub>4</sub> was repeated at room temperature. After keeping the solution at 120°C for 2 h, this slurry was siphoned off and washed twice with 10 ml of n-decane and three times with 10 ml of hexane, respectively. The catalyst was dried under vacuum for 30 min at 40°C and contained 3% Ti.

## 2.2 Polymerization reaction

### 2.1.1 Polyethylene and polypropylene terminal hydroxyl group

The propylene polymerization and ethylene polymerization reactions were carried out in a 100 ml semi-batch stainless steel reactor equipped with magnetic stirrer. 26.55 ml Hexane, 0.01 g catalyst (Al/Ti molar ratio = 167) and 3.45 ml TEA were introduced into the reactor and stirred for 5 minutes at room temperature in the atmospheric glove boxes. Followed by immediately put the reactor in liquid nitrogen to stop reaction between the catalyst and cocatalyst. After the solution was frozen for 15 minutes, the reactor was evacuated for 3 minutes to remove argon. The reactions were conducted at 60°C. Polymerization was started by continuous feeding ethylene (propylene) at constant pressure of 50 psi for 1 hr. Then stopped the polymerization by directly brought into contact with oxygen gas at room temperature followed by addition of hydrochloric acid solution in methanol and dried at room temperature.

### 2.1.2 Polyethylene block polypropylene copolymerization

Copolymerization was carried out in a glass reactor equipped with magnetic stirrer. Polyethylene and polypropylene containing hydroxyl group 50/50 %wt were added and dissolved in *o*-dichlorobenzene at 120 °C. Follow by dropped of excess MDI in the solution that remained stir for 1 hr. The solution was washed with excess methanol. Polymer was filtered and dried.

## 2.2 Blend and molding preparation

The melt mixing method was performed in digital hot plate stirrer at 220 °C. All blends were prepared with 50 wt% of PE and 50 wt% of PP (both polymers are commercial grade). When the block copolymer was used, 3 wt% of the block copolymer was added base on the total weight of the 50/50 blend. Added the block copolymer in PE/PP blend for 3, 6 %wt. All polymer blends was molded with The LAB TECH hydraulic hotpress LP-50 M/C 9701.

## 2.3 Measurements and characterization of block copolymers and blends

Infrared survey spectra were recorded with FTIR (IFS28) The scanning ranged from 400 to 4000 cm<sup>-1</sup> on the pallet sample of KBr powder with scanning 12 times.

The molecular weight and its distribution were determined by using GPC Model: Waters 2000 Column: Styragel HT6E with 1,2,4-Trichlorobenzene as a solvent at 135 °C.

The melting temperatures of the block copolymer added polymer blends were determined with a Perkin-Elmer DSC-Pyris Diamond over the range -60 to 200 °C of scanning rate 40 °C/min under nitrogen atmosphere.

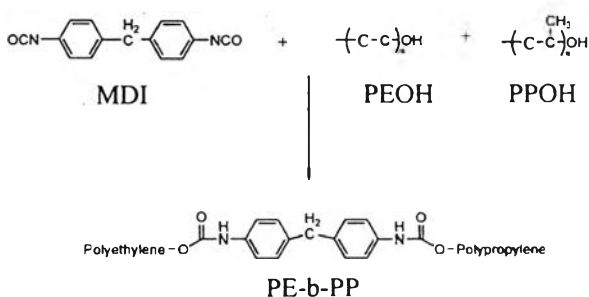
Tensile properties were characterized using an Instron universal testing machine with a test speed of 12.5 mm/min. The test were conducted according ASTM D 882-02.

The morphology of all block copolymers were investigated by JSM-5410LV Scanning Microscope. The samples for SEM analysis were coated with gold particles by ion sputtering device to provide electrical contact to the specimens.

## Results and Discussion

### 3.1 Chain structure of PE/PP block copolymer diisocyanate linkage

On the basis of this result, a plausible products of the block copolymerization is proposed as shown in (Scheme 1)



Scheme 1.

Table 1. Characterization of polymer

Polymer	Mw x 10 <sup>-4a</sup>	MWD <sup>a</sup>	Heating <sup>b</sup> 1 (°C)		Cooling <sup>b</sup> (°C)		Heating <sup>b</sup> 2 (°C)	
			T <sub>m1.1</sub>	T <sub>m1.2</sub>	T <sub>c1</sub>	T <sub>c2</sub>	T <sub>m2.1</sub>	T <sub>m2.2</sub>
PEOH	117.3	7.5	145	-	110	-	137	-
PPOH	39.5	8.7	-	156	100	-	-	154
PEbPE	n/a	n/a	135	-	108	-	132	-
PPbPP	n/a	n/a	93	158	102	-	-	153
PEbPP	58.3	12.5	130	153	113	120	129	152
PE/PP	32.4	6.1	135	151	98	111	135	151
PE/PP+PEbPP3%	n/a	n/a	136	163	111	-	134	164
PE/PP+PEbPP6%	n/a	n/a	138	166	109	-	138	164

<sup>a</sup> Determined by gel permeation chromatography, PS standard

<sup>b</sup> Determined by DSC

In addition, beside PE-b-PP, there have others 6 byproducts of the reaction which are PE, PP, PEOH, PPOH, PE-b-PE and PP-b-PP. The fractions of these byproducts are hardly to be identified and it can not be fractionated by solvent extraction. Thus unidentified fraction distribution of the block copolymers will be everywhere in the product of PE-b-PP. From GPC results, the molecular weight distribution of PP-b-PE is rather broad because of the MWD of PE and PP. Consequently we can not certainly obtain only the PE-b-PP copolymer in this study.

In order to confirm the reaction that contribute to the block copolymer of PE-b-PP in this system, the IR spectrum of block copolymer obtained at 25 °C is illustrated in Figure 1. The peak of isocyanate (NCO) transmittance is 1530 cm<sup>-1</sup>,  $\nu_{C=O}$  MDI = 1711 cm<sup>-1</sup>,  $\nu_{NH}$  = 3404, 1599 and 814 cm<sup>-1</sup>. Thus IR spectrum has identified the diisocyanate linkage in PE-b-PP copolymer structure.

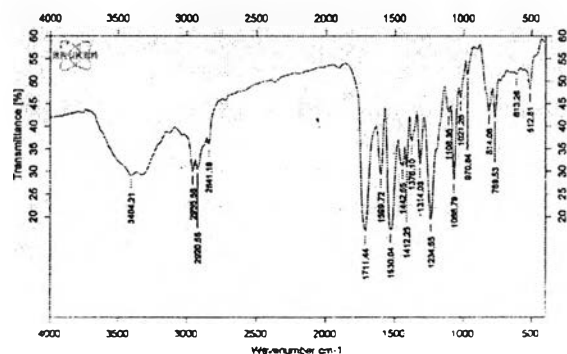


Fig. 1. IR-spectra of PE-b-PP at 25 °C

The chain structure of polymer blend and block copolymer was studied by DSC analysis of crystalline segregated samples. After stepwise annealing of the samples at different temperatures, the long PP and PE segments can form crystalline lamellae of different thickness according to their sequence lengths, and

these lamellae will melt at different temperatures [8]. By recording the endothermic curves of the polymer blend and block copolymer in a DSC scan, we will able to judge the sequence distribution of PE/PP blend and PE-b-PP. As shown in figure 2.

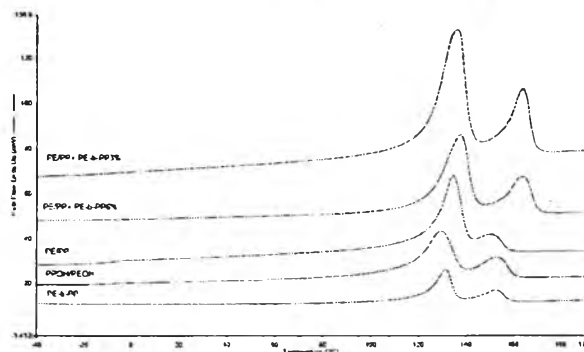


Fig. 2. DSC endotherms of PP/PE blend, PE-b-PE and addition of PE-b-PP to PE/PP blend after thermal segregation treatment.

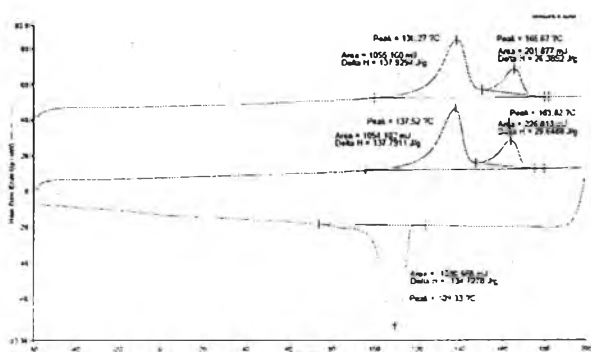
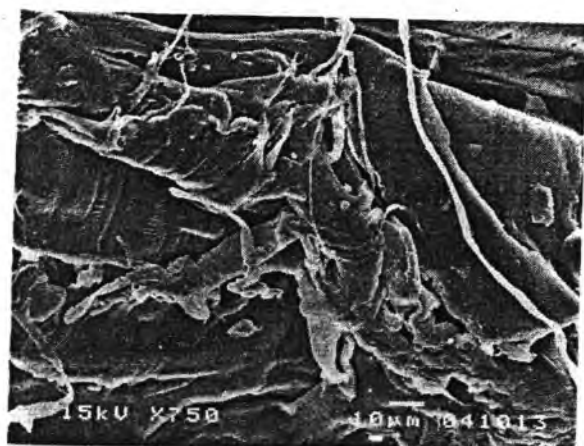
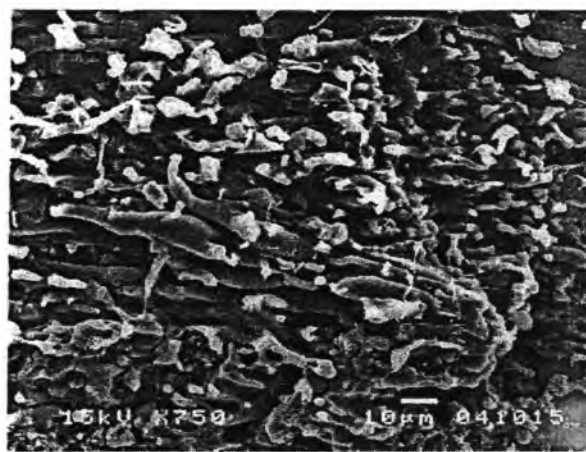


Fig. 3. DSC curve of the addition of PE-b-PP 6% to PE/PP blend.

The peak about 130-140 °C is PE melting temperature and the peak above 140 °C is originate from PP. However the appearance of the curves of PE/PP blend and PE-b-PP are similar. In the cases of adding PE-b-PP to PE/PP blend, the melting temperatures of PP increase (as shown in Table 1.)



(A)



(B)

Fig. 5. SEM of tensile fracture surface of a) PE/PP blend b) PE/PP+ PEbPP3%

### 3.2 Tensile mechanical properties

The addition of PE-b-PP to PE/PP(50/50) blends have modified the overall stress-strain behavior as exemplified in Fig. 4. It was found that the stress or yield stress increases with increasing PE-b-PP content. The largest value of elongation at break is found for the blend with 6 wt% PE-b-PP. It can be shown that PE-b-PP effects the compatibility and impact strength of PE/PP blends. The phase morphology of PE/PP blend and addition of PE-b-PP to PE/PP has shown in Fig. 5.

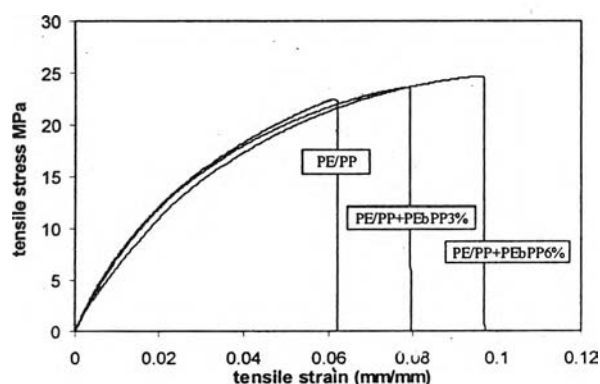


Fig. 4. Additive effect of PE-b-PP to PE/PP blend on stress -strain behavior at 25 °C

Due to SEM picture, it clearly shows the different of the rough surface particles and the bridge formation with PP matrix of PE/PP blend, the addition of PE-b-PP to PE/PP blend shows the smaller phase particles size. Many studies [9-12] on polymer alloys have shown that for multiphase polymer systems, the toughening effect is determined by two factors. First, the smaller the particles and the narrower the particles size distributions are, the better impact strength is. Secondly, the stronger is the adhesion between particle and the matrix; the better is the impact properties. Thus, it can be conclude that the synthesized block copolymer via diisocyanate is

be able to decrease particle size distributions as seen in SEM.

### 4. Conclusion

A new synthesis method of PE-b-PP copolymer by convert the H-terminated chain-ends to hydroxyl-terminated ones and blocking with diisocyanate is performed the effective compatibilizer for immiscible blend of PE/PP. The diisocyanate linkage of PE-b-PP copolymers were investigated by IR, which indicates that PE-b-PP copolymers occurred in the blocking reaction. The effects of PE-b-PP copolymers on the morphology of PE/PP blends were investigated by SEM with image analysis. The presence of 3,6 %wt block copolymer dramatically reduced the phase size. Furthermore the mechanical properties, such as tensile strength, elongation at break have been improve due to PE-b-PP copolymers contain PE and PP segments which attached to PE/PP blend, leading to superior properties via changing morphology than the normal mixing blend without PE-b-PP.

### Acknowledgement

The author would like to thank Bangkok Polyethylene Co. for supplying the ethylene and propylene gas and GPC characterization. Furthermore we would also like to thank MEKTEC manufacturing corporation (Thailand) Ltd. for supporting the characterize equipments and Miss Nongnaphat for IR analysis.

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