CHAPTER V

RESULTS & DISCUSSION

The purpose of this work is to study the method to synthesis graft copolymer between EPDM and polystyrene and the properties of polymer blends between polystyrene and synthesis graft copolymer. Therefore, this chapter provided information about some preliminary results concerning the reaction conditions such as, polymerization time, polymerization temperature, concentration of initiator, concentration of styrene monomer, effect of oxygen for condition of synthesis, effect of solvent as an synthesis medium and effect of aging for initialize the synthesis. The characterization of graft copolymer were produced by NMR, DSC. Moreover, the tensile strength tests was used for investigated the mechanical properties of polymer blends between graft copolymer and polystyrene.

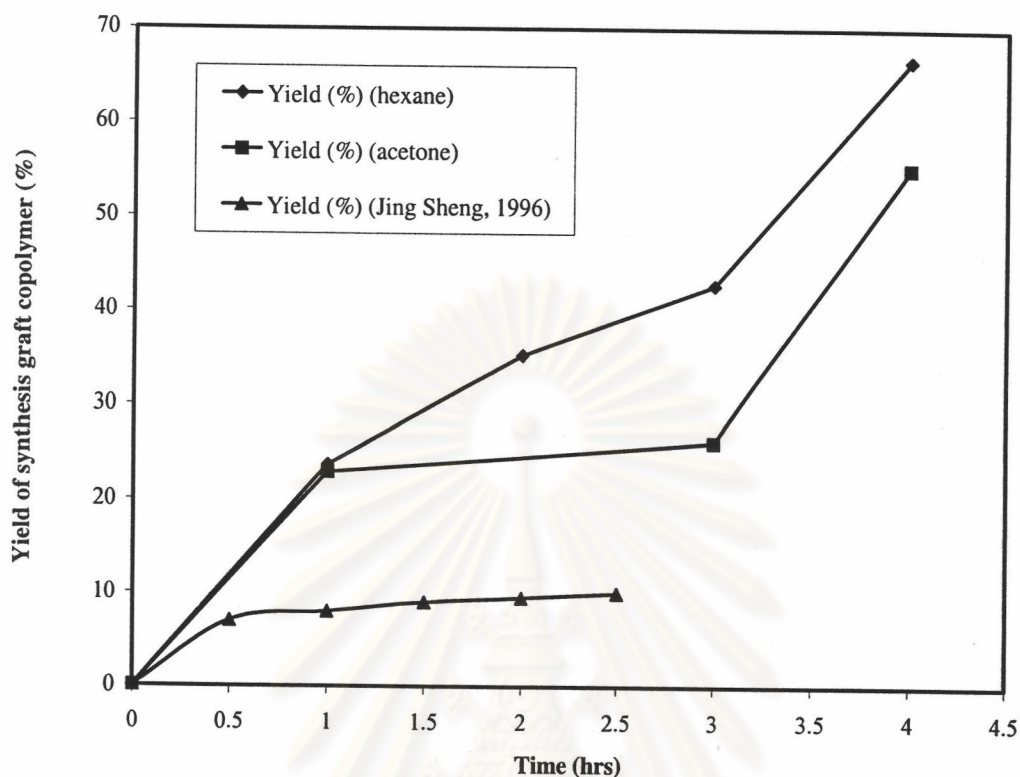## 5.1. Condition for synthesis graft copolymer between EPDM and Styrene

5.1.1 Effect of Time for Synthesis Graft copolymer between EPDM and Styrene

The effect of time was investigated by using BPO as the initiator. The time for polymerization of EPDM and styrene was varied in the range of 1 to 4 hrs. The copolymerization was performed in toluene at 90°C using EPDM approximately 3-5 g, BPO concentration in the range of 1.50×10^{-3} - 2.08×10^{-3} mol/L with total styrene concentration 2.49 mol/L. The results of the influence of time for the synthesis graft copolymer shown in Table 5.1.1 and Figure 5.1.1

Table 5.1.1 Yield of synthesis graft copolymer at different time

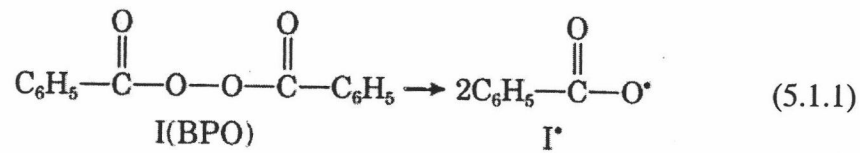| Time (hrs)	Yield (%) (hexane)	Yield (%) (acetone)	Activity (g of polymer/mol(BPO).hrs)
1	23.80	22.98	3335
2	35.22	n.d.	n.d.
3	42.76	26.19	1020.8
4	66.65	55.18	1613.1

Polymerization conditions : EPDM = 3-5 g , [St] = 2.49 mol/L (20 ml), BPO = 1.50×10^{-3} - 2.08×10^{-3} mol/L , Toluene solvent , polymerization temperature = 90 °C,
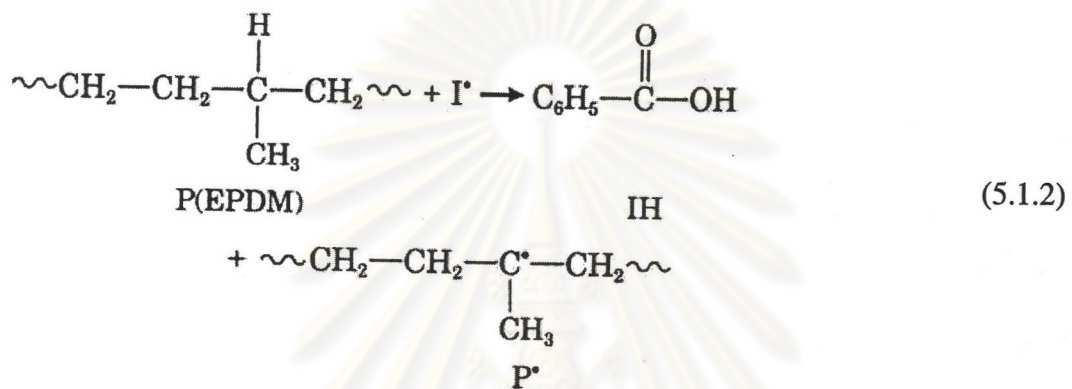
Figure 5.1.1 Yield of synthesis graft copolymer at different time

Polymerization conditions EPDM = 3-5 g , [St] = 2.49 mol/L (20 ml), BPO = 1.50 x10^{-3} - 2.08 x10^{-3} mol/L , Toluene solvent , polymerization temperature = 90 ^{O}C,
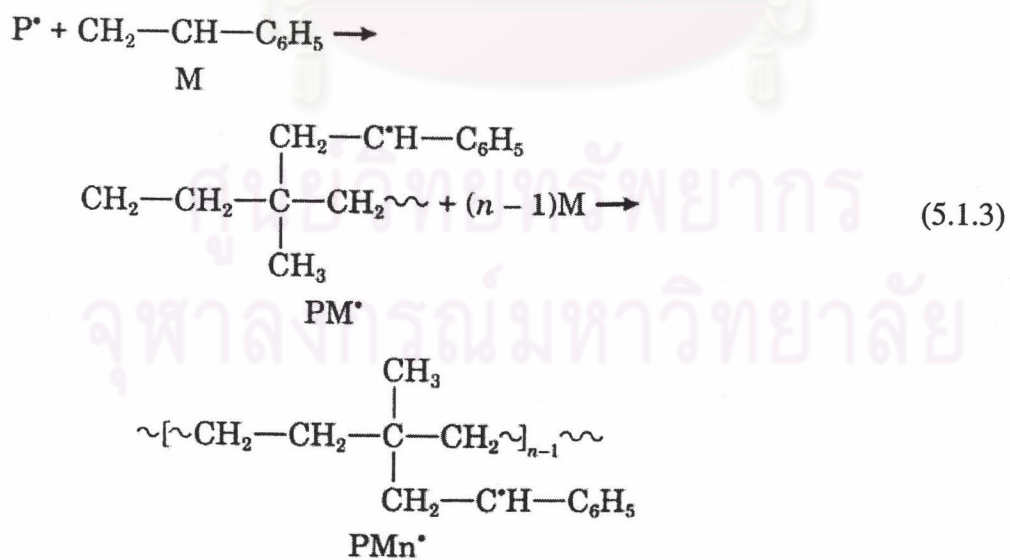
As shown in Figure 5.1.1 , yield of synthesis graft copolymer rapidly increases at the polymerization time between 3.0 and 4.0 hours. The polymerization time between 1.0 and 3.0 hours slightly increase the yield. Because the initiator have more time to persue the free radical polymerization of styrene by decompose to free radical and increasing the chain length of PS-g-EPDM so that the copolymer can not be dissolved by hexane. The free radical has the enough time to form graft copolymer. The mechanism of decomposition initiator is shown in equation 5.1.1 [Jing Sheng et al,1996]. BPO initiator decompose to the free radical form and transfer the radical into the main chain of EPDM. The structure of main chain radical (P^{o}) shown in equation 5.1.2. After that the main chain radical reacted with styrene monomer to initiate graft copolymer between EPDM and styrene shown in equation 5.1.3. All above mechanism give the increased grafting of styrene on EPDM.

$$C_6H_5-\overset{\overset{O}{\|}}{C}-O-O-\overset{\overset{O}{\|}}{C}-C_6H_5 \longrightarrow 2C_6H_5-\overset{\overset{O}{\|}}{C}-O^\bullet \qquad (5.1.1)$$

I(BPO)   I$^\bullet$

Main-chain polymer radical formation:

$$\sim\sim CH_2-CH_2-\overset{\overset{H}{|}}{\underset{\underset{CH_3}{|}}{C}}-CH_2\sim\sim \ + I^\bullet \longrightarrow C_6H_5-\overset{\overset{O}{\|}}{C}-OH$$

P(EPDM)   IH   (5.1.2)

$$+ \ \sim\sim CH_2-CH_2-\overset{\underset{\underset{CH_3}{|}}{C^\bullet}}{}-CH_2\sim\sim$$

P$^\bullet$

Formation of graft polymer radical:

$$P^\bullet + CH_2-CH-C_6H_5 \longrightarrow$$

M

$$CH_2-CH_2-\overset{\overset{CH_2-C^\bullet H-C_6H_5}{|}}{\underset{\underset{CH_3}{|}}{C}}-CH_2\sim\sim \ + (n-1)M \longrightarrow \qquad (5.1.3)$$

PM$^\bullet$

$$\sim[\sim CH_2-CH_2-\overset{\overset{CH_3}{|}}{\underset{\underset{CH_2-C^\bullet H-C_6H_5}{|}}{C}}-CH_2\sim]_{n-1}\sim\sim$$

PMn$^\bullet$

5.1.2 Effect of BPO concentration initiator for Synthesis Graft copolymer between EPDM and Styrene

The effect of concentration initiator was investigated by using 3 hrs polymerization time. The concentration BPO initiator was varied in the range of 2.08×10^{-3} to 6.25×10^{-3} mol/L. The copolymerization were performed in toluene at 90°C using EPDM approximately 5 g by weight with styrene concentration 2.49 mol/L. The results yields of the influence BPO concentration for synthesis graft copolymer are shown in Table 5.1.2 and Figure 5.1.2

Table 5.1.2 Yield of synthesis graft copolymer at different amount of BPO

$BPOx10^3$ (mol/L)	Yield (%) (hexane)	Yield (%) (acetone)	Activity (g of polymer/mol(BPO).hrs)
2.08	42.76	16.62	647.67
4.67	70.19	32.18	576.59
6.25	64.55	28.17	358.91

Polymerization conditions : EPDM = 5.687 g , [St] = 2.49 mol/L (20 ml), time = 3 hrs
Toluene solvent , polymerization temperature = 90 °C,
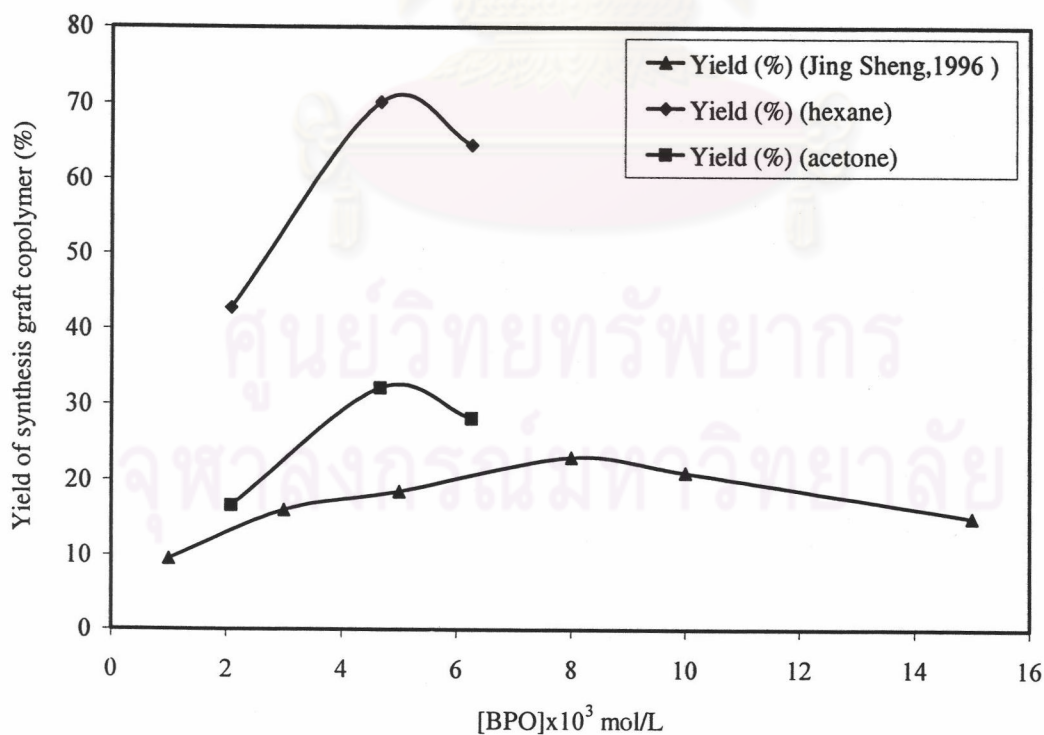
Figure 5.1.2 Yield of synthesis graft copolymer at different BPO

Polymerization conditions : EPDM = 5.687 g , [St] = 2.49 mol/L (20 ml), time = 3 hrs,
Toluene solvent , polymerization temperature = 90 °C,

As shown in Figure 5.1.2 , yield of synthesis graft copolymer increased with increasing concentrations of BPO leading to a maximum value at about 4.67 x 10^{-3} mol/L and then decreased. When the percent of BPO was under maximum value, the amounts of monomer radical, polymer radical, and graft copolymer radical increased with increasing concentrations of BPO, which increased the probability of interaction of radicals. When the percent of BPO was excess, the grafting percent of styrene onto EPDM was decreased because of the reaction of BPO by itself gradually increase. The viscosity of polymerization was increased when synthesis time proceed, and because of the increasing concentration of BPO that was evident, the movement of radicals was more difficult [Jing Sheng et al, 1996].

5.1.3 Effect of Oxygen for Synthesis Graft copolymer between EPDM and Styrene

The effect of oxygen was investigated by controlling the polymerization atmosphere. The polymerization times of EPDM and styrene in the experiments was fixed at 1 hr. The copolymerization were performed in toluene at 90°C using EPDM 4.0313 g weight approximately, BPO concentration 1.507 x10^{-3} mol/L with total styrene concentration 1.24 mol/L. The results of the influences of the oxygen in synthesis graft copolymer shown in Table 5.1.3 and Figure 5.1.3

Table 5.1.3 Yield of synthesis graft copolymer at different condition

Condition	Yield (%) (hexane)	Activity (g of polymer/mol(BPO).hrs)
no oxygen	6.02	539.54
Oxygen or air	1.21	108.43

Polymerization conditions : EPDM = 4.0313 g, [St] = 1.24 mol/L (10 ml), BPO = 1.507 x10^{-3} mol/L , Toluene solvent , polymerization temperature = 90 °C,
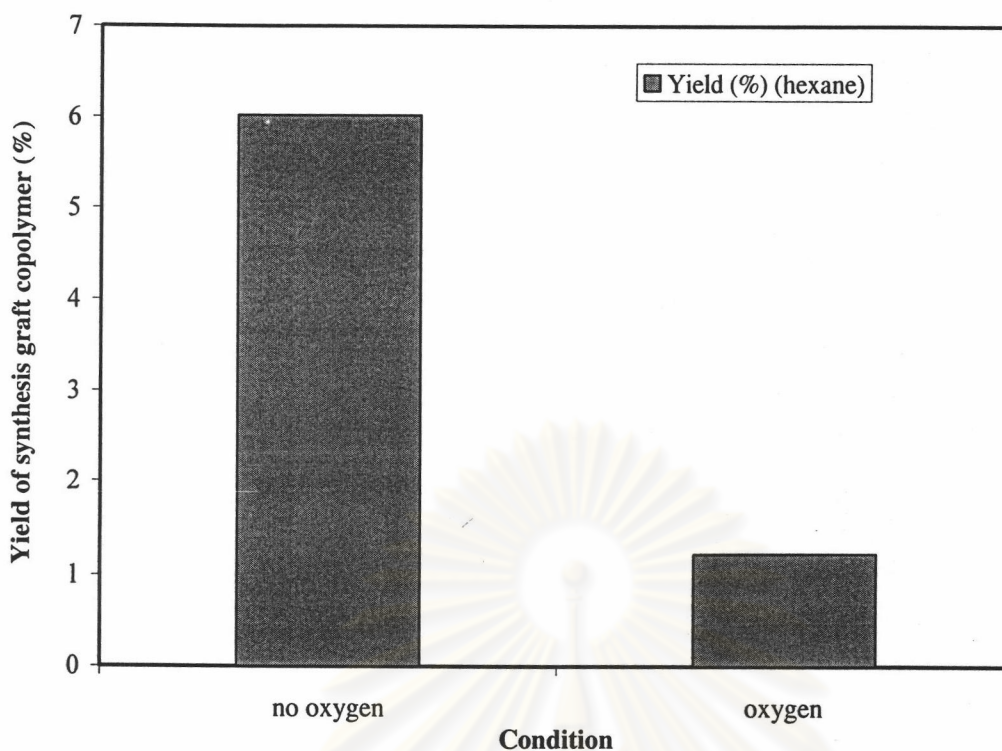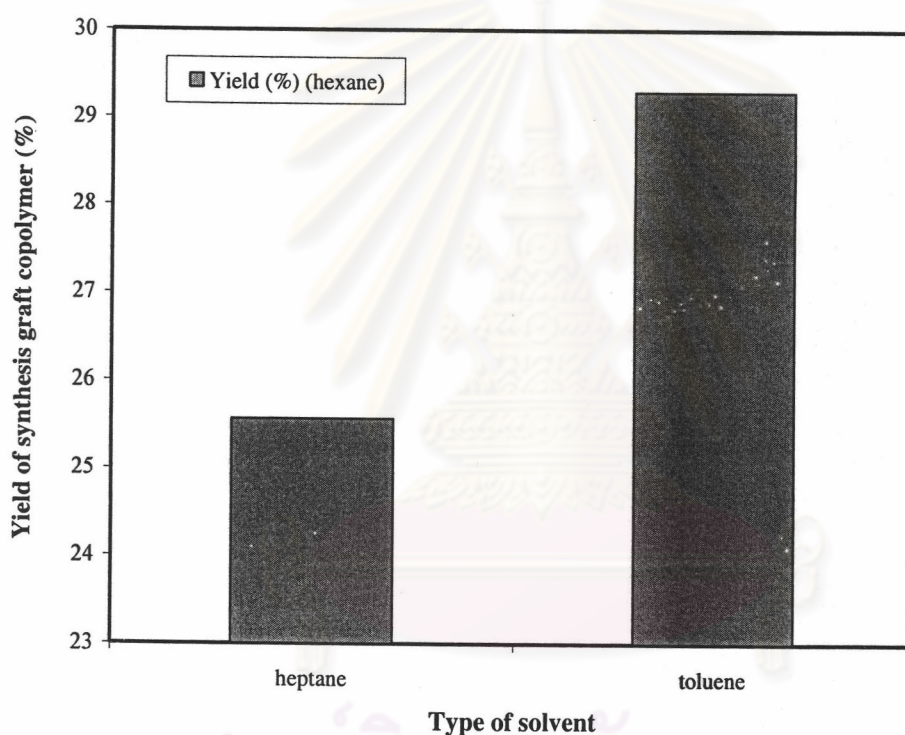
Figure 5.1.3 Yield of synthesis graft copolymer at different condition

Polymerization conditions : EPDM = 4.0313 g, [St] = 1.24 mol/L (10 ml), BPO = 1.507 x10^{-3} mol/L , Toluene solvent , polymerization temperature = 90 $^{\circ}$C,

As shown in Figure 5.1.3, yield of synthesis graft copolymer was higher in polymerization condition without oxygen and the yield of polymerization in air was dramatically lower. Because oxygen can destroyed the radical initiator and, as a results, the concentration of initiator was depleted. So, the yield of synthesis graft copolymer was decreased.

5.1.4 Effect of Solvents for Synthesis Graft copolymer between EPDM and Styrene

The effects of solvents were investigated by using polymerization time at 1 hr. The temperature for polymerization of EPDM and styrene was performed at 60 $^{\circ}$C and 90 $^{\circ}$C. The copolymerization were using EPDM 4-5 g weight approximately, BPO concentration 1.507 x10^{-3} – 2.039 x10^{-3} mol/L with total styrene concentration 1.24 mol/L. The results of the influences of solvent for synthesis graft copolymer are shown in Tables 5.1.4.1 – 5.1.4.2 and Figures 5.1.4.1 -5.1.4.2

Table 5.1.4.1 Yield of synthesis graft copolymer of each solvent at 60 $^{\circ}$C

Solvent	Yield (%) (hexane)	Activity (g of polymer/mol(BPO).hrs)
toluene	1.65	147.83
hexane	0.24	21.357
heptane	2.71	203.25
THF	0.03	2.72

Polymerization conditions : EPDM = 4-5 g, [St] = 1.24 mol/L (10 ml), BPO = 1.507 x10^{-3} - 2.039 x10^{-3} mol/L , polymerization temperature = 60 $^{\circ}$C,
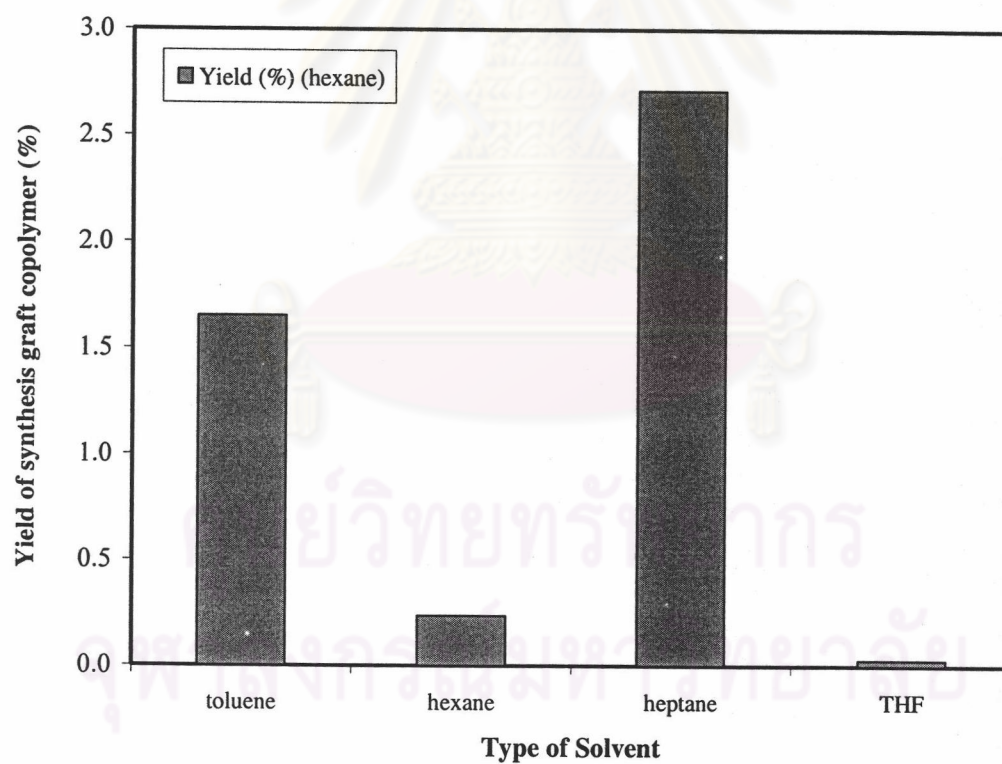
Figure 5.1.4.1 Yield of synthesis graft copolymer at different solvents at 60°C

Polymerization conditions : EPDM = 4-5 g, [St] = 1.24 mol/L (10 ml), BPO = 1.507 x10^{-3} - 2.039 x10^{-3} mol/L , polymerization temperature = 60 $^{\circ}$C,

Table 5.1.4.2 Yield of synthesis graft copolymer of each solvent at 90 ^{o}C

Solvent	Yield (%) (hexane)	Activity (g of polymer/mol(BPO).hrs)
heptane	25.57	1915.76
toluene	29.30	2192.96

Polymerization conditions : EPDM = 4-5 g, [St] = 1.24 mol/L (10 ml), BPO = 1.507 x10^{-3} - 2.039 x10^{-3} mol/L , polymerization temperature = 90 ^{O}C,

Figure 5.1.4.2 Yield of synthesis graft copolymer at different solvents at 90°C

Polymerization conditions : EPDM = 4-5 g, [St] = 1.24 mol/L (10 ml), BPO = 1.507 x10^{-3} - 2.039 x10^{-3} mol/L , polymerization temperature = 90 ^{O}C,

As shown in Figure 5.1.4.1 at 60 °C, yields of synthesis graft copolymer were increasing from THF, hexane, toluene and heptane respectively. However, at the temperature of 90 °C, toluene gave more yield than heptane and 8 time higher yield from the same solvent at 60 °C. Because of the nature of solvent may also affect k_d (rate of thermal decomposition) [Krzysztof et al,1998]. Rate of thermal decomposition of BPO to give free radical depend on various solvent and temperature ,as can be seen Therefore, at temperature of 90 °C, cyclic solvent give a higher initiator radical (5.1.1) than linear solvent to give a high grafting polymer.

5.1.5 Effect of Temperatures for Synthesis Graft copolymer between EPDM and Styrene

The effect of temperature was investigated by using BPO concentration 1.507×10^{-3} and 2.039×10^{-3} mol/L at 1 hr for polymerization of EPDM and styrene. The copolymerization were performed in toluene and using EPDM 4-5 g weight approximately, styrene concentration was 1.24 mol/L. The results of the influence of temperature for synthesis graft copolymer shown in Table 5.1.5 and Figure 5.1.5

Table 5.1.5 Yield of synthesis graft copolymer at different Temperature

Temp (°C)	Yield (%) (hexane)	Activity (g of polymer/mol(BPO).hrs)
60	1.65	147.83
90	29.30	2192.95

Polymerization conditions : EPDM = 4-5 g , [St] = 1.24 mol/L (10 ml), BPO = 1.507×10^{-3} – 2.039×10^{-3} mol/L , Toluene solvent
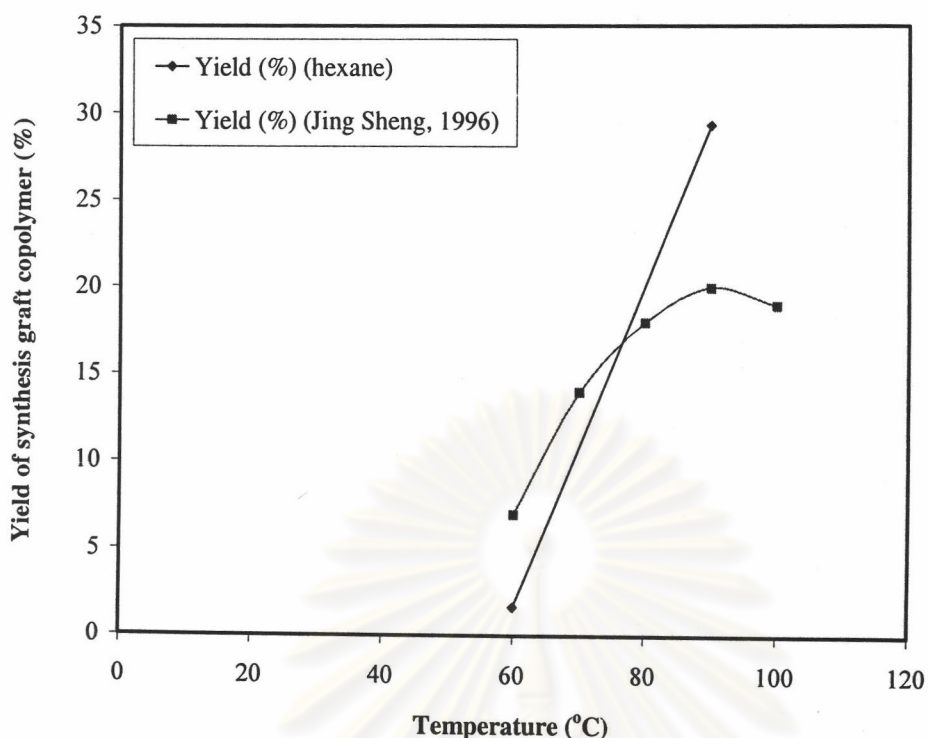
Figure 5.1.5 Yield of synthesis graft copolymer at different Temperature

Polymerization conditions : EPDM = 4-5 g , [St] = 1.24 mol/L (10 ml), BPO = 1.507 x10^{-3} – 2.039 x10^{-3} mol/L , Toluene solvent

As shown in Figure 5.1.5 , yield of synthesis graft copolymer increased with increasing temperature lead to a maximum value at 90 °C and then decreased at the same time, presumably because of the reduction of the half-life of BPO with rising temperature [Malcolm,1999], which would increase the number of BPO radicals [reaction 5.1.2]. When the temperature was over 90°C, the extent of grafting decreased with rising temperature because the number of radicals was deceased with increasing velocity of decomposition of BPO [Jing Sheng et al,1996].

5.1.6 Effect of Styrene concentrations for Synthesis Graft copolymer between EPDM and Styrene

The effect of styrene concentration was investigated by using polymerization time for 2 hrs. The concentration of BPO initiator was 2.08x10^{-3} mol/L. The copolymerization were performed in toluene at 90°C using EPDM 5.68 g weight approximately. The results of the influence styrene concentration for synthesis graft copolymer shown in Table 5.1.6 and Figure 5.1.6

Table 5.1.6 Yield of synthesis graft copolymer at different Styrene concentration

Styrene (mol/L)	Yield (%) (hexane)	Activity (g of polymer/mol(BPO).hrs)
1.24	29.30	1096.47
2.49	35.22	2064.03

Polymerization conditions : EPDM = 5.68 g , BPO = 2.08 x10^{-3} mol/L , Toluene solvent , Polymerization temperature 90 $^{\circ}$C and polymerization time 2 hrs
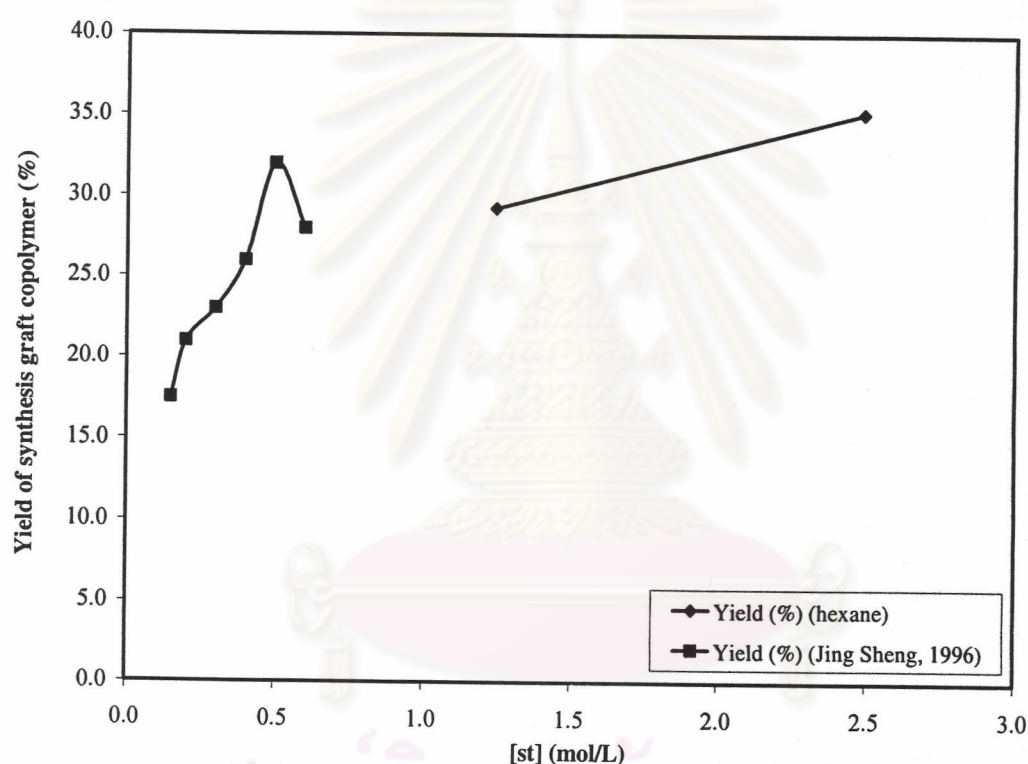
Figure 5.1.6 Yield of synthesis graft copolymer at different styrene concentration

Polymerization conditions : EPDM = 5.68 g , BPO = 2.08 x10^{-3} mol/L , Toluene solvent , Polymerization temperature 90 $^{\circ}$C and polymerization time 2 hrs

Figure 5.1.6 indicates the extent of yield increased with increasing concentration of Styrene up to a maximum value and then decreased. Before the maximum value, the extent of yield increased with increasing number of monomer [reaction 5.1.3]. When the yield percent was over maximum value, the number of Styrene homopolymer increased with increasing amount of monomer compete with the graft polymerization, so the grafting percent decreased [Jing Sheng et al,1996].

5.1.7 Effect of Ageing Time for Synthesis Graft copolymer between
EPDM and Styrene

The effect of aging time was investigated by using BPO as the initiator. The time for polymerization of EPDM and styrene was set at 2 hrs. The copolymerization were performed in toluene at 90°C using EPDM 3-5 g weight approximately, BPO concentration was about 1.50×10^{-3} - 2.08×10^{-3} mol/L with total styrene concentration was 2.49 mol/L. The results of the influence of time for synthesis graft copolymer shown in Table 5.1.7 and Figure 5.1.7

Table 5.1.7 Yield of synthesis graft copolymer at different Ageing Time

Ageing Time (mins)	Yield (%) (hexane)	Yield (%) (acetone)	Activity (g of polymer/mol(BPO).hrs)
0	23.80	22.98	1667.77
5	35.22	n.d.	n.d.
10	45.90	38.13	2229.51
30	49.32	22.49	1314.77
60	15.57	11.21	655.46

Polymerization conditions : EPDM = 3-5 g , BPO = 1.5×10^{-3} -2.08×10^{-3} mol/L , Toluene solvent , Polymerization temperature 90 °C and polymerization time 2 hrs
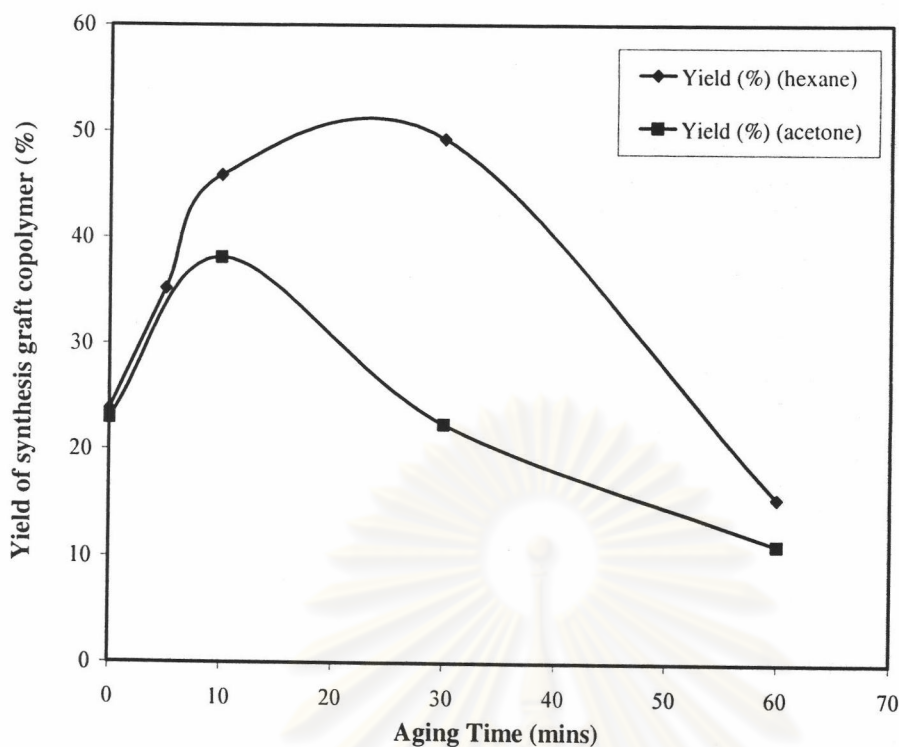
Figure 5.1.7 Yield of synthesis graft copolymer at different ageing times

Polymerization conditions : EPDM = 3-5 g , BPO = 1.5 x10^{-3} -2.08 x10^{-3} mol/L , Toluene solvent , Polymerization temperature 90 $^{\circ}$C and polymerization time 2 hrs

As shown in Figure 5.1.7 , yield of synthesis graft copolymer increased with increasing aging time lead to a maximum value at 10 mins (acetone) and 30 mins (hexane) and then decreased at the same temperature. The yield of synthesis graft copolymer increased because the ageing time increased the free radical initiator have enough time to form P^{o} with the double bound of EPDM as in the equation (5.1.2) and higher probability to formation of graft copolymer follow the equation (5.1.3) was affected. The yield of synthesis graft copolymer decreased after the maximum value because the free radical initiator has a reversible reaction of equation (5.1.1) and its can decompose as in the equation (5.1.4) and (5.1.5)

$$Ph-\overset{\overset{\displaystyle O}{\|}}{C}-O-O-\overset{\overset{\displaystyle O}{\|}}{C}-Ph \longrightarrow 2\ Ph-\overset{\overset{\displaystyle O}{\|}}{C}-O\cdot$$

$$Ph-\overset{\overset{\displaystyle O}{\|}}{C}-O\cdot \longrightarrow Ph\cdot + CO_2 \tag{5.1.4}$$

$$2\,Ph\cdot \longrightarrow Ph\!-\!\!-Ph$$

$$\underset{Ph-\overset{\displaystyle O}{\overset{\|}{C}}-O\cdot\ +\ Ph\cdot\ \longrightarrow\ Ph-\overset{\displaystyle O}{\overset{\|}{C}}-O\cdots Ph}{} \qquad (5.1.5)$$
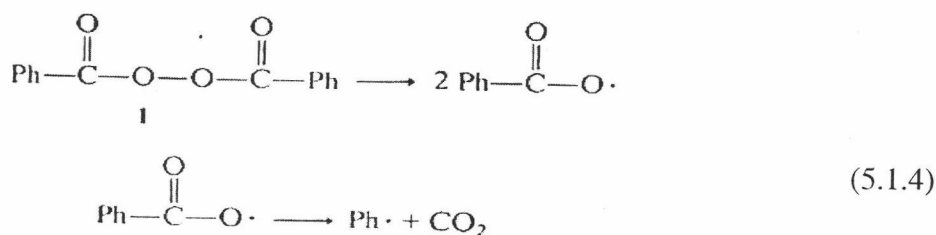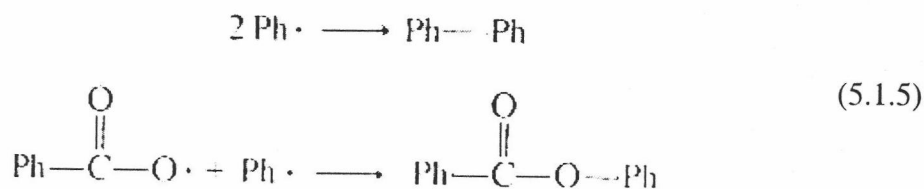
5.2. Morphologies of polymer and polymer blend

The morphologies of polymer blend between synthesis graft copolymer (EPDM-g-PS) and polystyrene obtained with BPO initiator at low and high styrene concentrations were observed by scanning electron microscopy technique and transmission electron microscopy technique. The results compared the morphology of the blend with polystyrene (PS) and High Impact Polystyrene (HIPS) as shown in Figure 5.2.1.1-5.2.2.3

5.2.1 Scanning Electron Microscopy (SEM)

Scanning electron microscopy technique was used to investigated the morphology of polymer and polymer blend such as PS, HIPS and blend between synthesis graft copolymer (EPDM-g-PS) and polystyrene (5 % w/w) at the magnification of 750 and 3500 times.
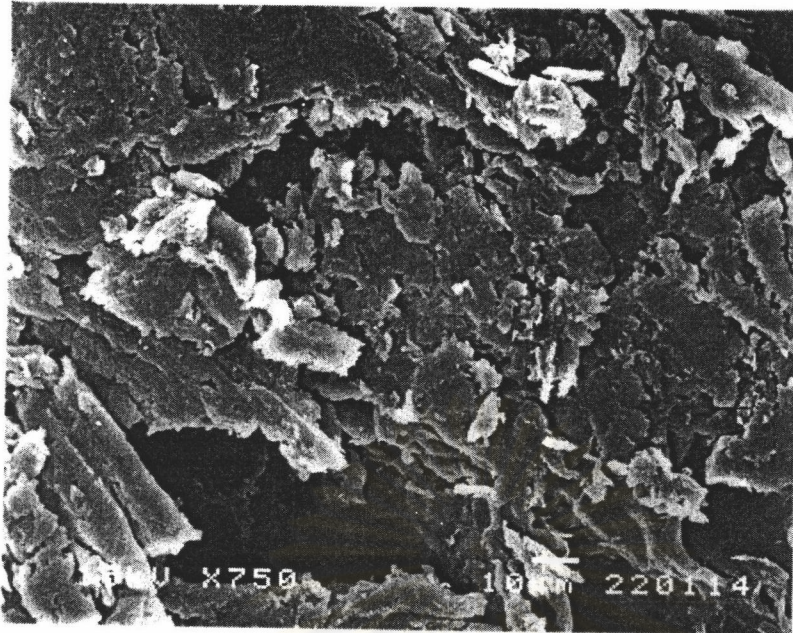
(a)
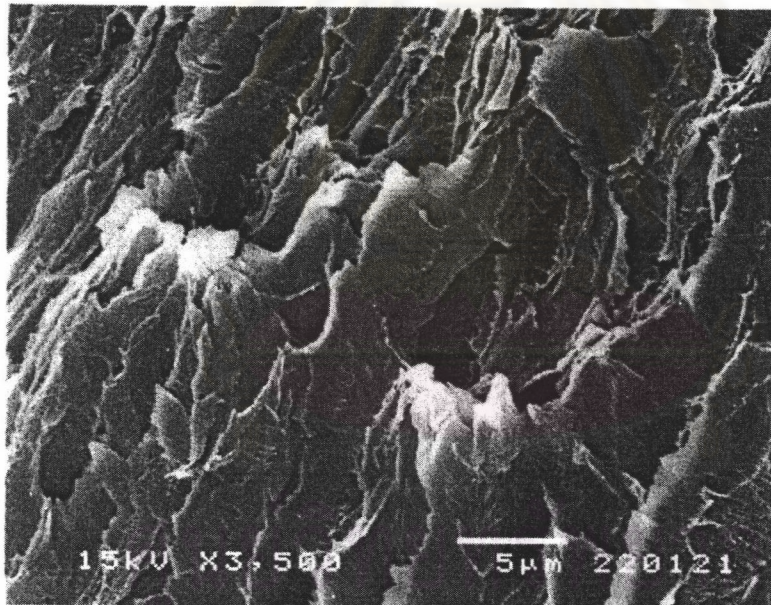
(b)

Figure 5.2.1.1 Scanning electron microscope Photograph of polystyrene

(a) ×500 (b) ×3500
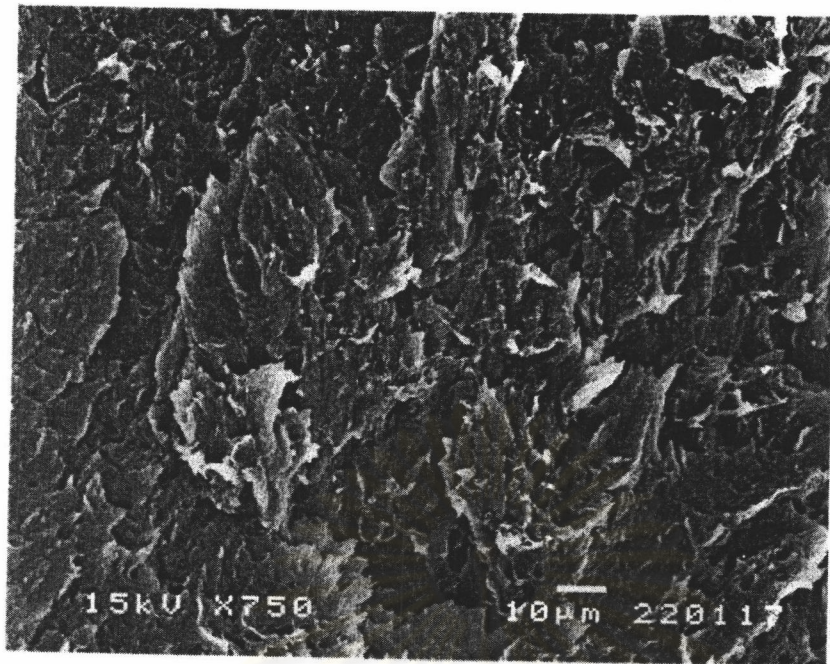
(a)
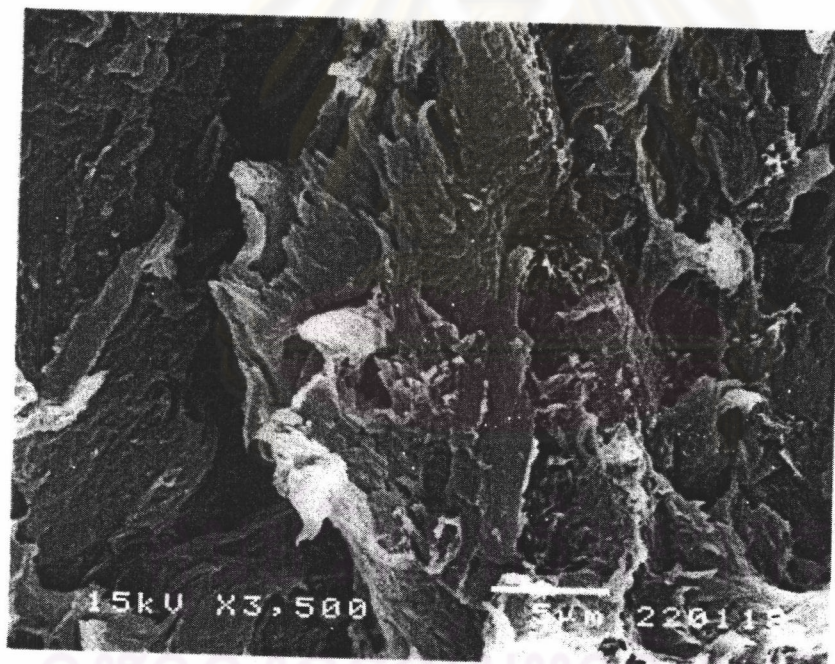
(b)

Figure 5.2.1.2 Scanning electron microscope Photograph of

High Impact Polystyrene (a) ×500 (b) ×3500
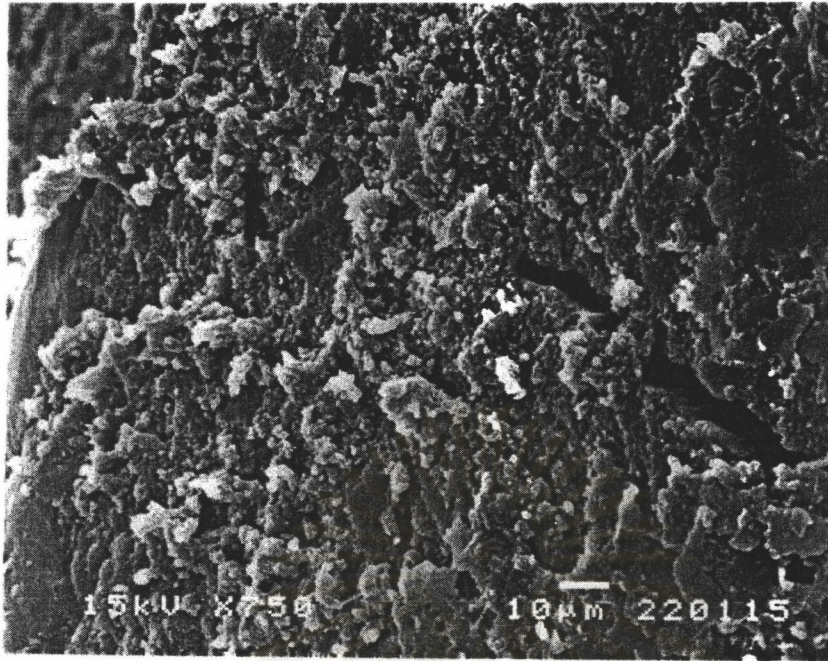
(a)
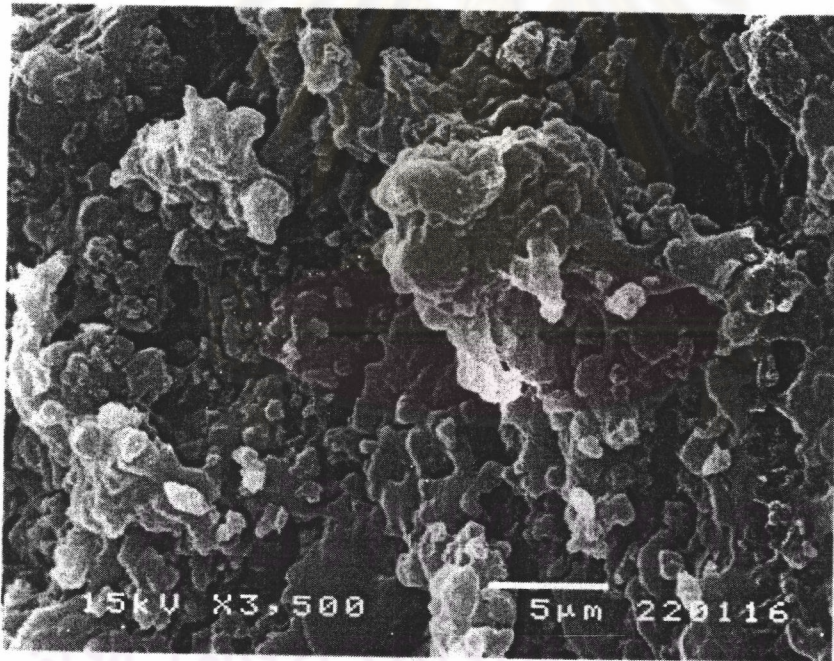
(b)
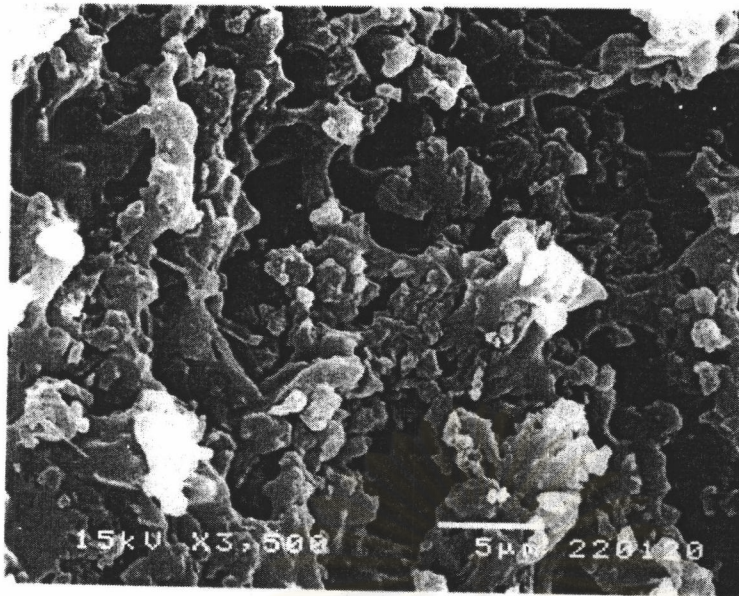
(c)
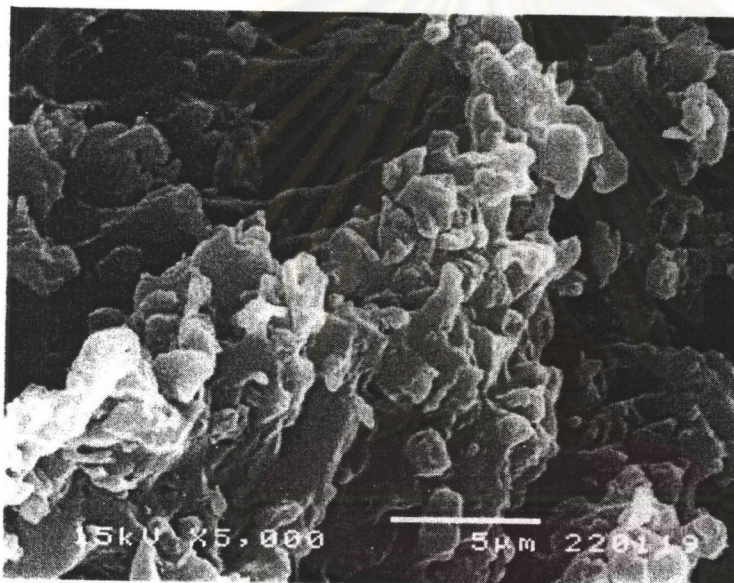
(d)

Figure 5.2.1.3 Scanning electron microscope Photograph of Polymer blend between
synthesis graft copolymer(EPDM-g-PS) and Polystyrene
(a) ×750 (b) ×3500 (c) ×3500 (d) ×5000

The morphologies of polystyrene and high impact polystyrene were shown in Figures 5.2.1.1 and 5.2.1.2. The photographs show the morphology of the fracture surface of PS were sharp and tight like a rubber. However, the morphology of polymer blend between synthesis graft copolymer (EPDM-g-PS) and Polystyrene were different. The morphologies have the laminar shape due to spherical particles agglomerate at the matrix of polystyrene shown in Figure 5.2.1.3. Therefore, the scanning electron photograph indicated presumably polymer blend between synthesis graft copolymer (EPDM-g-PS) and polystyrene have a good fracture morphology than the one of pure polystyrene.

5.2.2 Transmission Electron Microscopy (TEM)

Transmission electron microscopy technique was used to investigated the morphology of polymer and polymer blend such as PS, HIPS and blend between synthesis graft copolymer (EPDM-g-PS) and polystyrene (5 % w/w) at the magnification of 4000 to 6000 times.
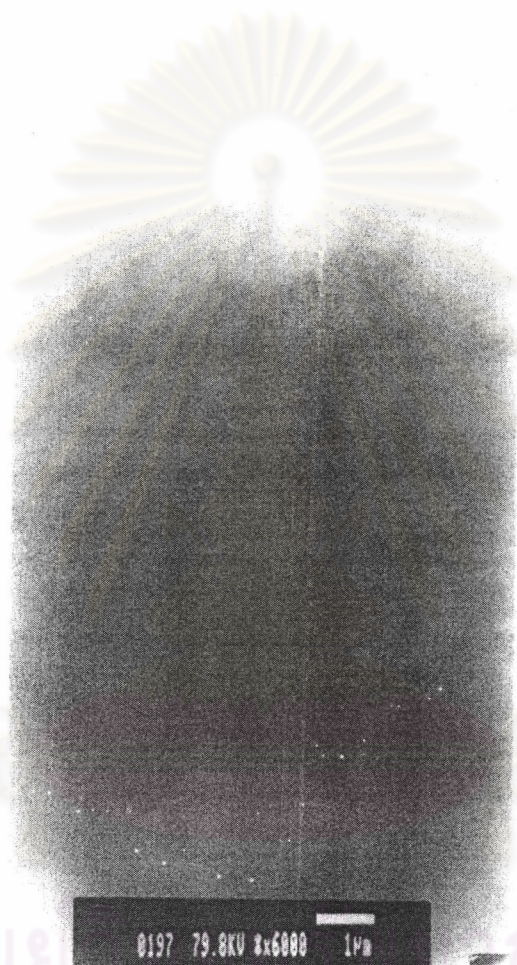
Figure 5.2.2.1 Transmission electron microscope Photograph of
polystyrene ×6000

(a)

(b)
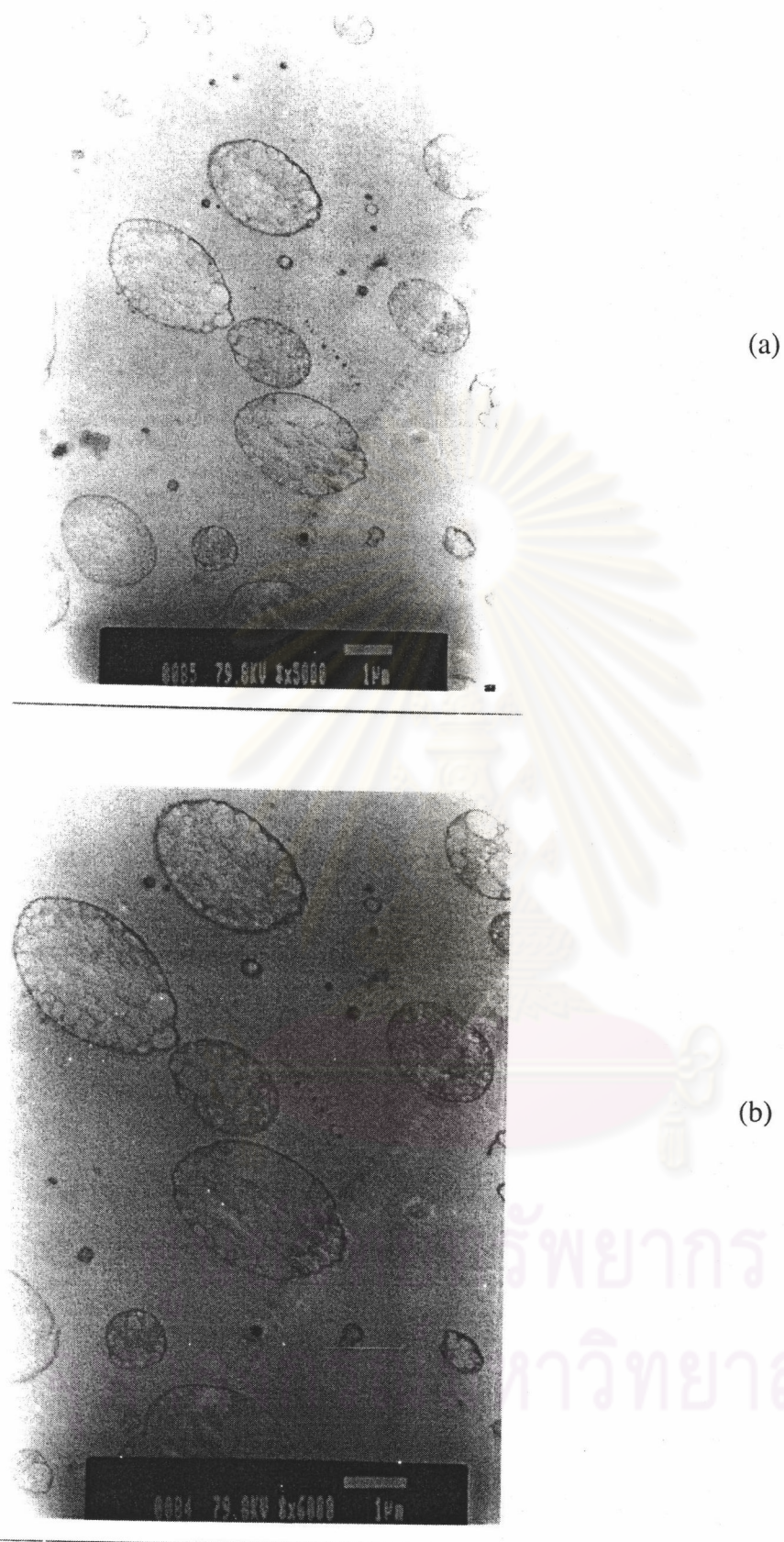
Figure 5.2.2.2 Transmission electron microscope Photograph of
High Impact Polystyrene (a) ×5000 (b) ×6000

(a)

(b)

(c)

(d)
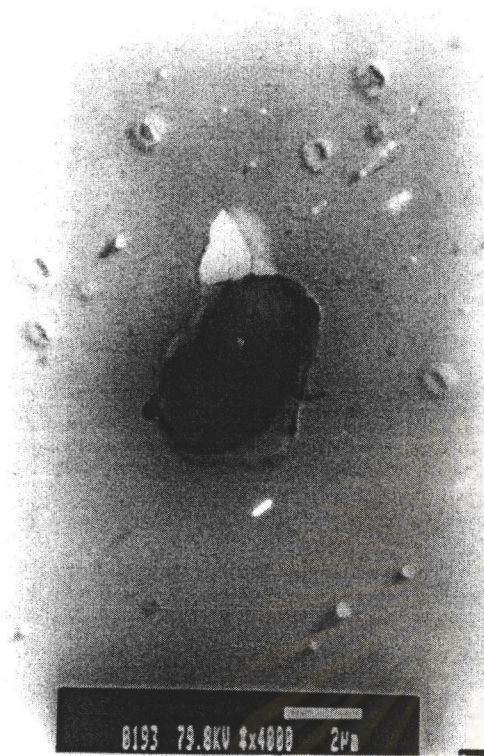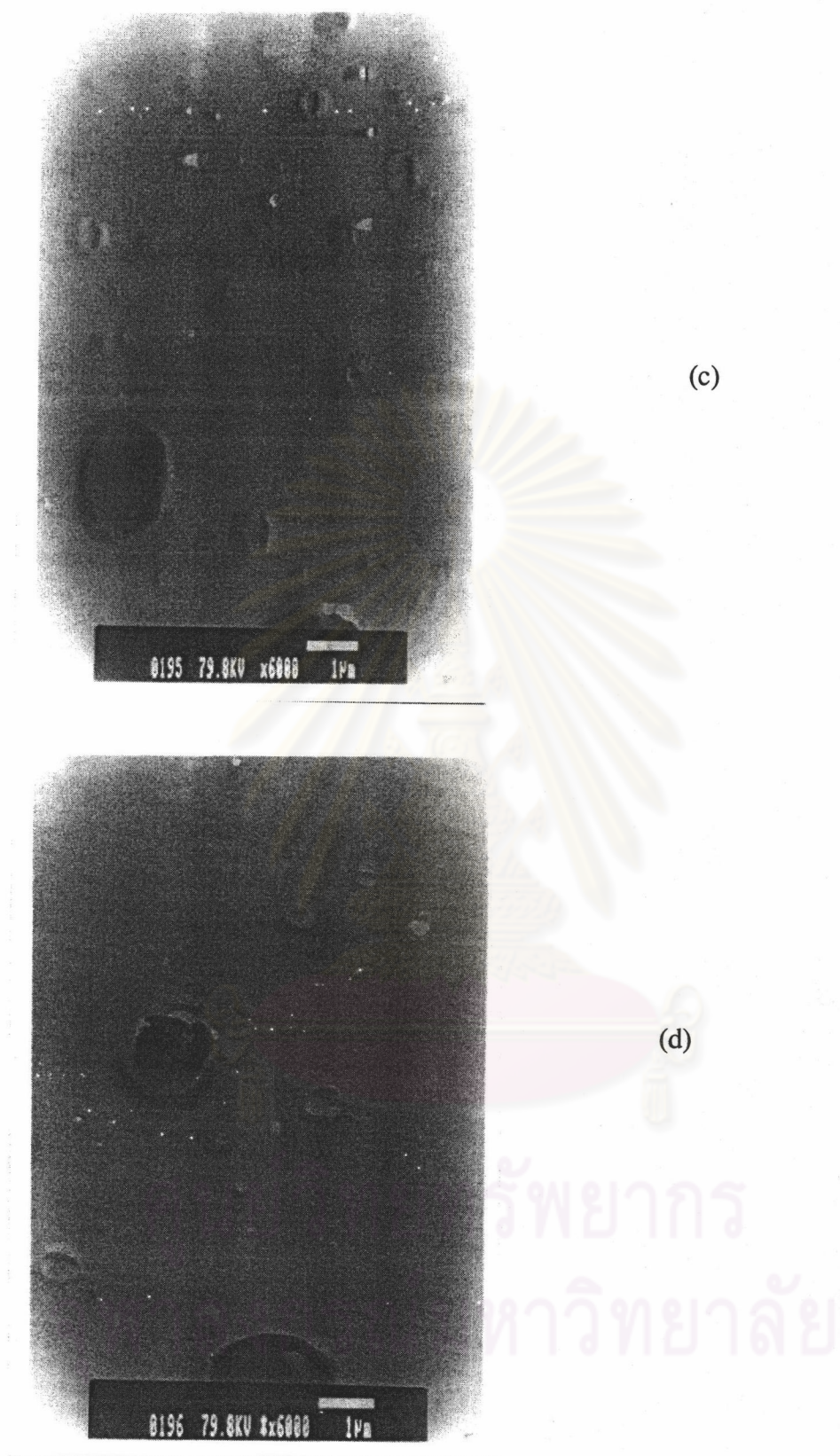
Figure 5.2.2.3 Transmission Electron Microscope Photograph of Polymer blend
between synthesis graft copolymer (EPDM-g-PS) and Polystyrene
(a) ×4000 (b) ×6000 (c) ×6000 (d) ×6000

The morphology of polystyrene was shown in Figure 5.2.2.1. From the photograph, the morphology of polystyrene can not be seen by transmission electron microscopy. Figure 5.2.2.2 shows the morphologies of high impact polystyrene in salami structure. The white part in salami structure is a polystyrene part and the black part is a butadiene part which similar to many observations [Fisher et al, 1995]. Figure 5.2.2.3 show the morphology of Polymer blend between synthesis graft copolymer (EPDM-g-PS) and Polystyrene at composition of 5% graft copolymer. The morphologies were seen the copolymer as spherical particles (EPDM-g-PS) which were the particle size were about 1-5 μm and it was different from HIPS morphology salami structure.

5.3 Tensile Strength Test

Tensile tests were performed using LLOYD tensile testing machine model 2000R. Obtained results are the stress-strain behaviors of polymers and polymer blend such as polystyrene, high impact polystyrene, polymer blend between EPDM (5 % w/w)/PS and synthesis graft copolymer (5 % w/w)/PS .The specimen dimension are of 4 cm × 0.8 mm × 10 cm for tensile strength test. The load cell of tensile strength is 10 kN.

Table 5.3.1 Young's Modulus and Stiffness for difference Polymer

Polymer	Young's modulus (MPa)	Stiffness (N/m)
Polystyrene (PS)	453.3	156,938.7
High Impact Poly styrene (HIPS)	371.4	236,270.0
EPDM blends with PS	400.0	212,035.0
Synthesis graft copolymer blends with PS	226.2	152,404.8

Young's Modulus and Stiffness shown in table 5.3.1. Young's Modulus is related to the bonding strength between atom of polymer. Polymer with high Young's Modulus are relatively stiff and do not defect easily [William F. Smith, 1996]. The order of maximum Young's Modulus was PS (453.3 MPa) > EPDM blends with PS (400 MPa) > HIPS (371.4 MPa) > Synthesis graft copolymer blends with PS (226.2 MPa) respectively. The order of maximum stiffness was HIPS (236,270 N/m) > EPDM blends with PS (212,035 N/m) >PS (156,938.7 N/m) > Synthesis graft copolymer blends with PS (152,404.8 N/m) respectively.
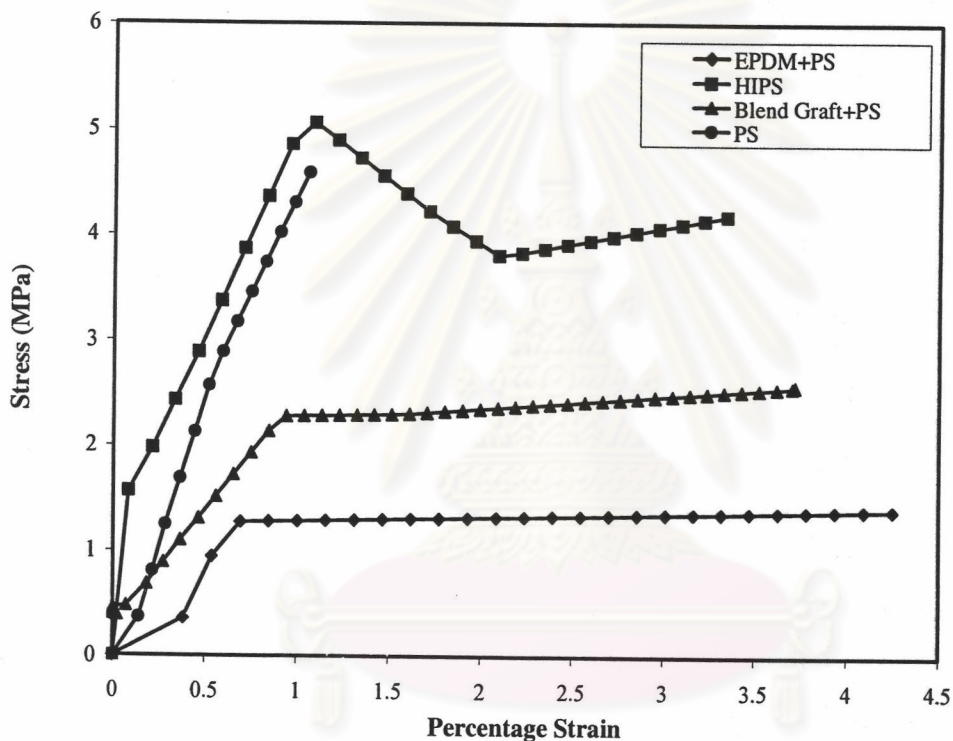
Figure 5.3.1 Stress – Strain Diagram for difference Polymer

Table 5.3.2 Toughness of each Polymer

Polymer	Toughness (area under curve)(MPa)
Polystyrene (PS)	0.03450
High Impact Poly styrene (HIPS)	0.12920
EPDM blends with PS	0.05252
Synthesis graft copolymer blends with PS	0.07904

Polymer were shown in Figure 5.3.1. Stress-strain diagram curve for a difference polymer. The character of each curve indicated the mechanical properties of each polymers. For the polystyrene curve show the character of the brittle material because it is brittle (not plastic behavior). The other polymers (not PS) show the character of ductile material. The area under curve of stress-strain curve indicated the toughness of polymer show in table 5.3.1. The order of maximum toughness was HIPS (0.12920 MPa) > Synthesis graft copolymer blends with PS (0.07904 MPa) >EPDM blends with PS (0.05252 MPa) > PS (0.03450MPa) respectively. Therefore, polymer blend between synthesis graft copolymer (EPDM-g-PS) (5 % w/w) and polystyrene and polymer blend between EPDM(5 % w/w) and polystyrene can improve the toughness of polystyrene twice in approximately. However, the toughness of polymer blend is still lower than HIPS.

5.4 Scanning Electron Microscopy (SEM) of fracture after tensile strength test

Scanning electron microscopy technique was used for investigation of the morphology of polymer and polymer blend such as PS, HIPS, polymer blend between synthesis graft copolymer (EPDM-g-PS) (5 % w/w) and polystyrene and polymer blend between EPDM (5 % w/w) and polystyrene at magnification of 500 times.
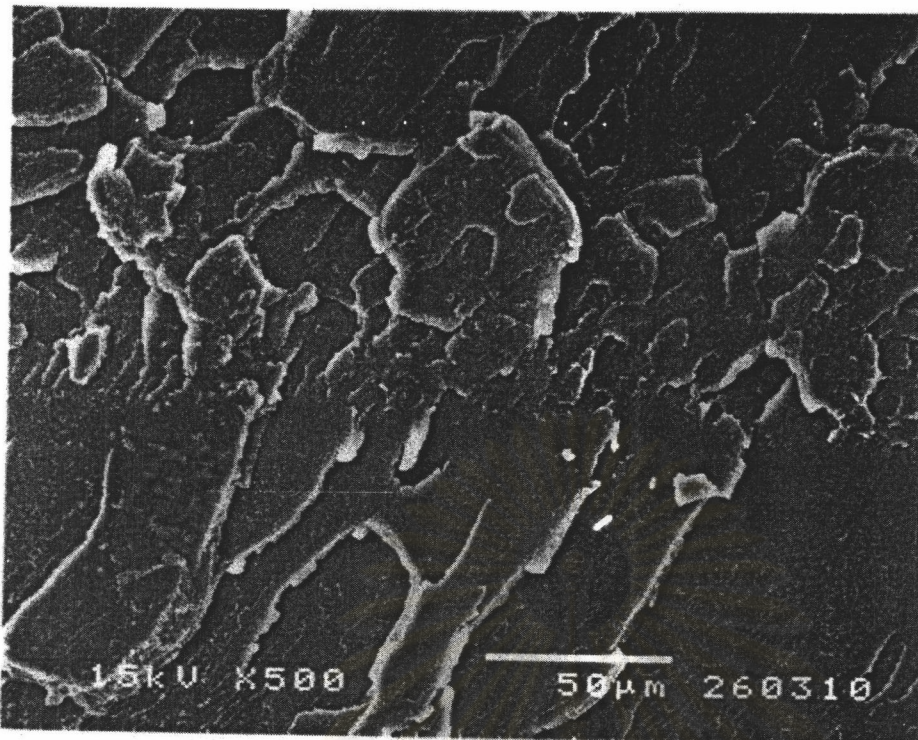
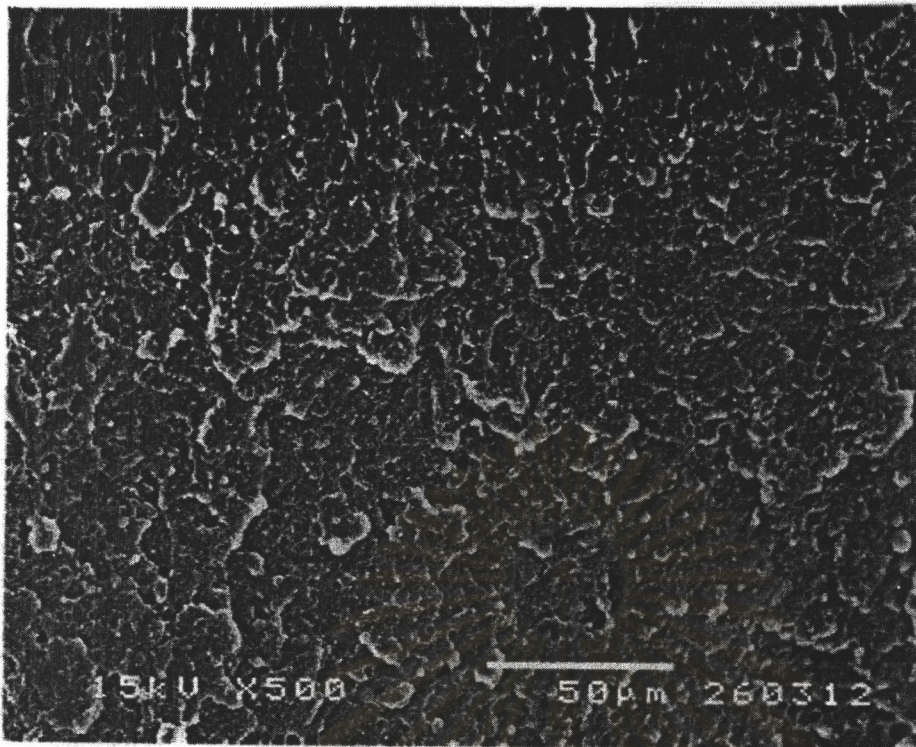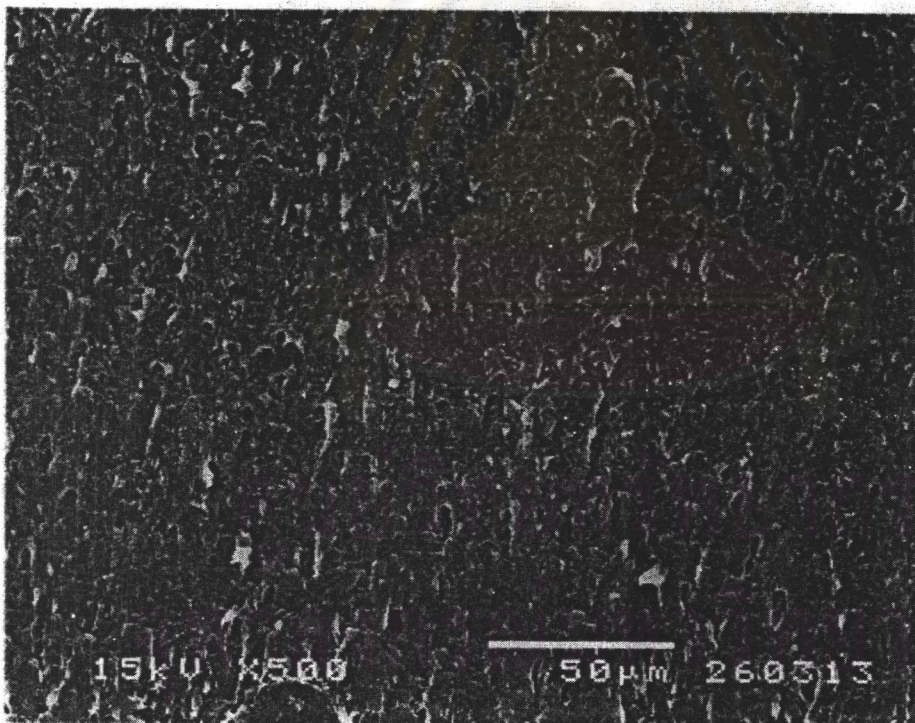(a)
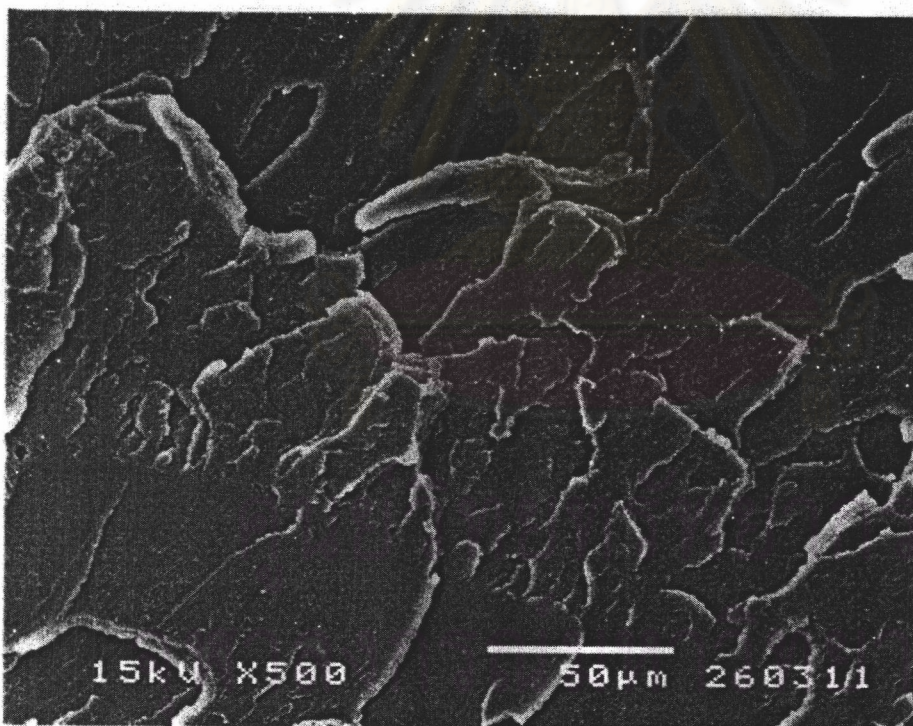
(b)

Figure 5.4.1. Scanning electron microscope Photograph of fracture surface after
yielding from tensile strength test of polystyrene
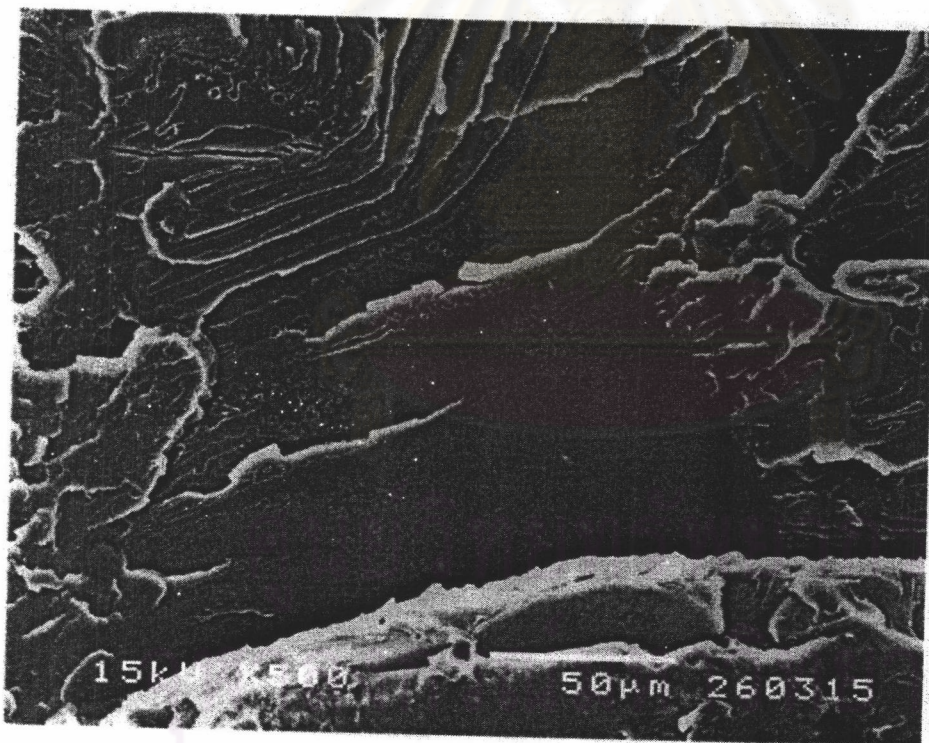(a) ×500 (b) ×500

(a)

(b)

Figure 5.4.2 Scanning electron microscope Photograph of fracture surface after yielding from tensile strength test of High Impact Polystyrene
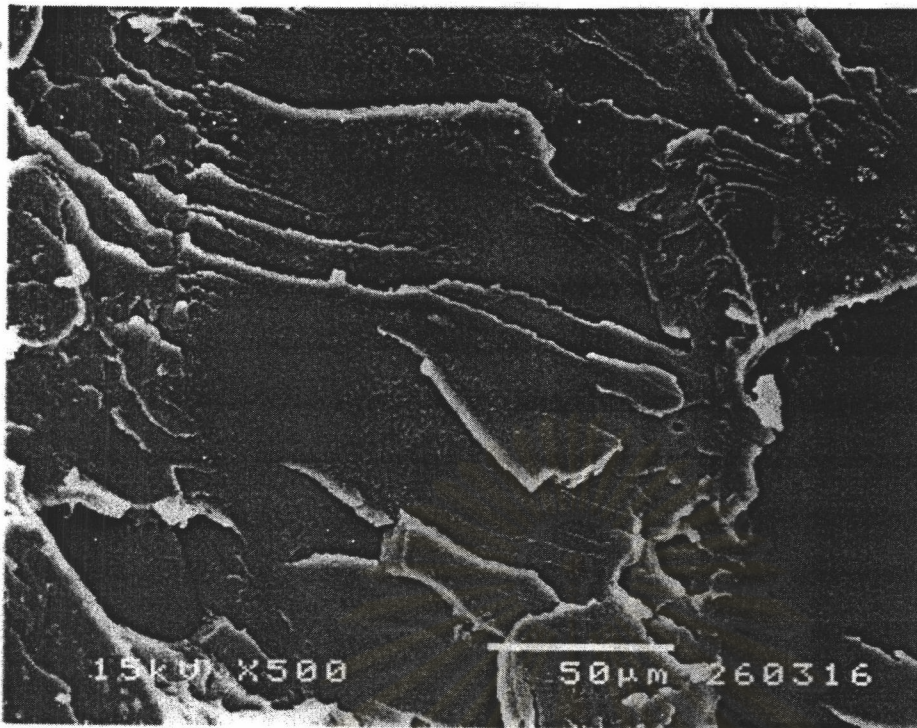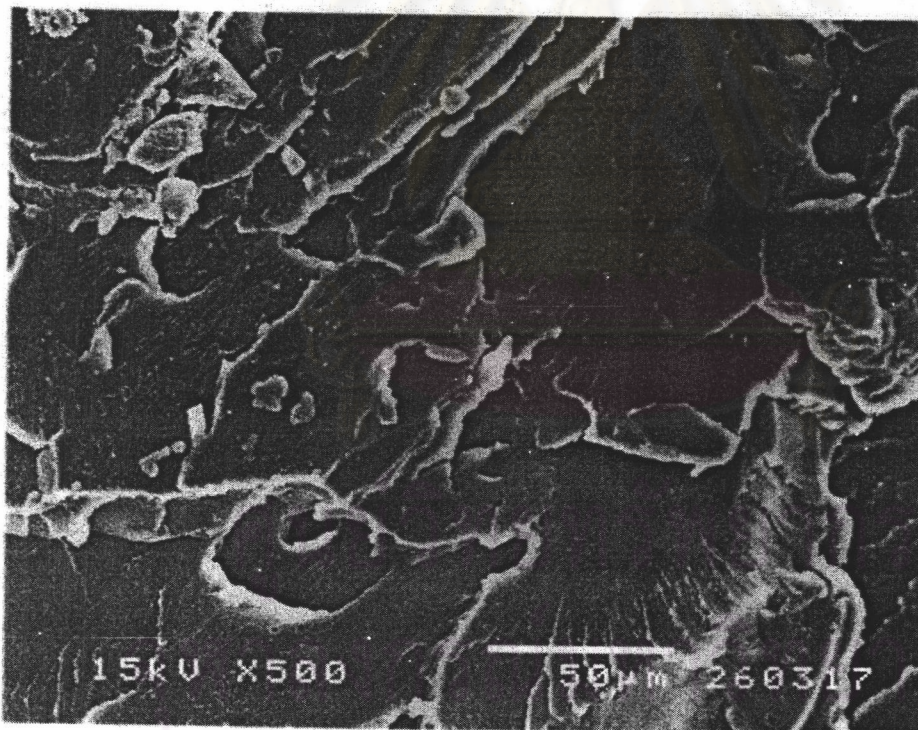(a) ×500 (b) ×500

(a)

(b)

Figure 5.4.3 Scanning electron microscope Photograph of fracture surface after
yielding from tensile strength test of Polymer blend between EPDM
(5 % w/w) and Polystyrene (a) ×500 (b) ×500

(a)

(b)

Figure 5.4.4 Scanning electron microscope Photograph of fracture surface after
yielding from tensile strength test of Polymer blend between synthesis
graft copolymer (EPDM-g-PS) (5 % w/w)   and Polystyrene
(a) ×500 (b) ×500

Figure 5.4.1 showed Scanning electron microscope Photograph of fracture surface after yielding from tensile strength test of polystyrene. The result of PS is different from other polymer (Figure 5.4.2-5.4.4) because the fracture surface of polymer are rough and peel. Moreover, the polymer matrix have a stripe. Figure 5.4.2 showed Scanning electron microscope Photograph of fracture surface after yielding from tensile strength test of HIPS. The fracture surface of the polymer has order and fine. Figure 5.4.3 and 5.4.4 are similar. However, polymer blend between synthesis graft copolymer (EPDM-g-PS) and polystyrene have better morphology than polymer blend between pure EPDM and polystyrene. Because of, synthesis graft copolymer (EPDM-g-PS) and polystyrene have a good miscibility in polystyrene than polymer blend between pure EPDM and polystyrene. Moreover, the polymer matrix is not stripe and peel.

5.5 Differential Scanning Calorimeter (DSC)

The melting temperatures and glass transition temperatures of the polymers were measured by a Perkin-Elmer DSC 7. The heating rate of 20° C/min in the temperature range -150 to 120 $^\circ$C was employed. The heating cycle were twice operated. In the first scan, samples were heated up and then cooled down to room temperature. In the second scan, samples were reheated at the same rate. Only the results of the second scan were reported because the DSC data of the first scan were influenced by the mechanical and thermal history of the samples during the polymerization. Moreover, DSC can be used for investigation of the synthesis graft copolymer between EPDM and Polystyrene. DSC of synthesis polymer show in Fig 5.5.1- 5.5.5. Figure 5.5.1 is the DSC of pure polystyrene, Figure 5.5.2 – 5.5.3 is the DSC of synthesis graft copolymer after soxhlet hexane and acetone and Figure 5.5.4- 5.5.5 is the DSC of the residue in solvent after soxhlet hexane and acetone respectively. The residue can be obtained by precipitate the solvent by methanol and used residual polymer solid after drying to run DSC.
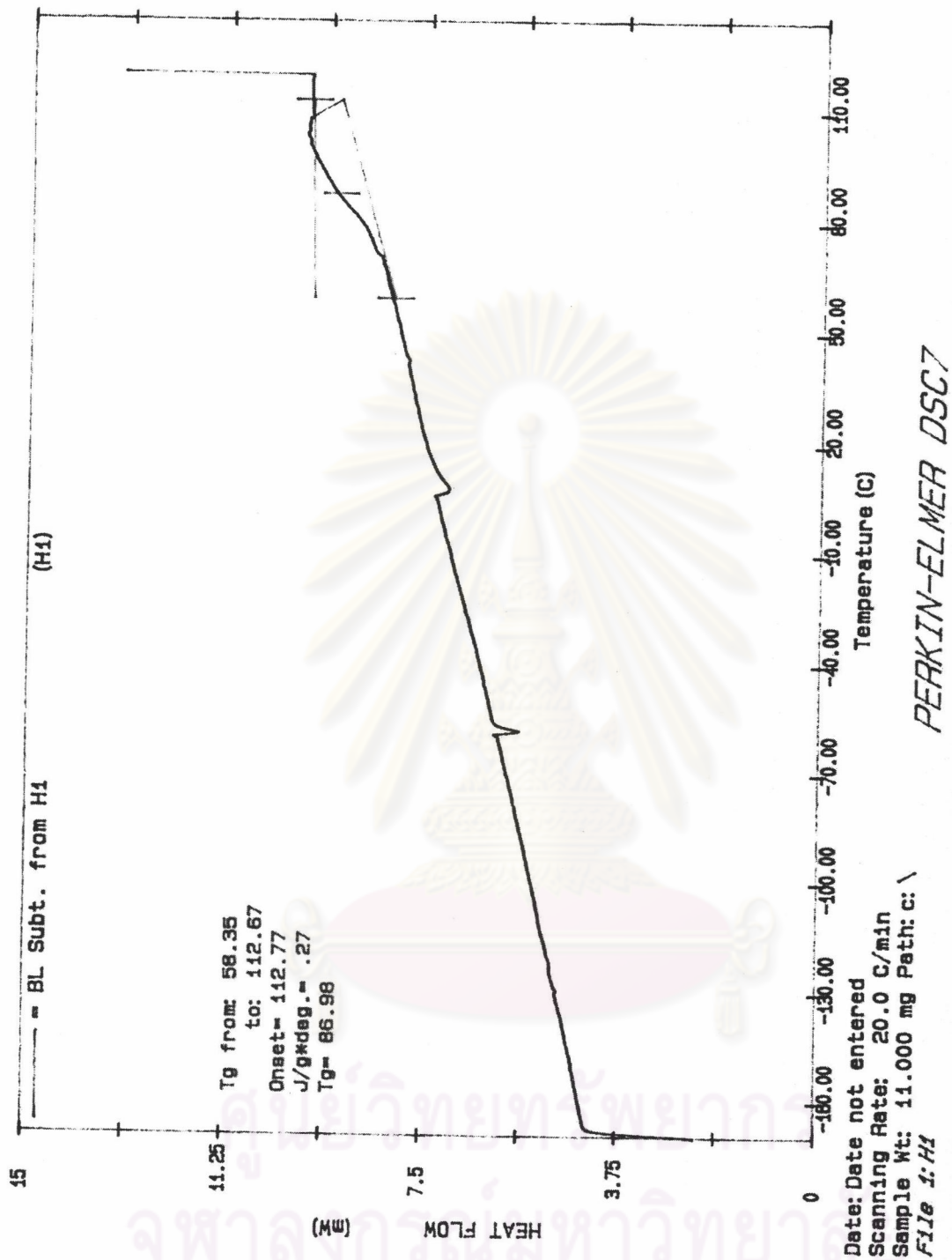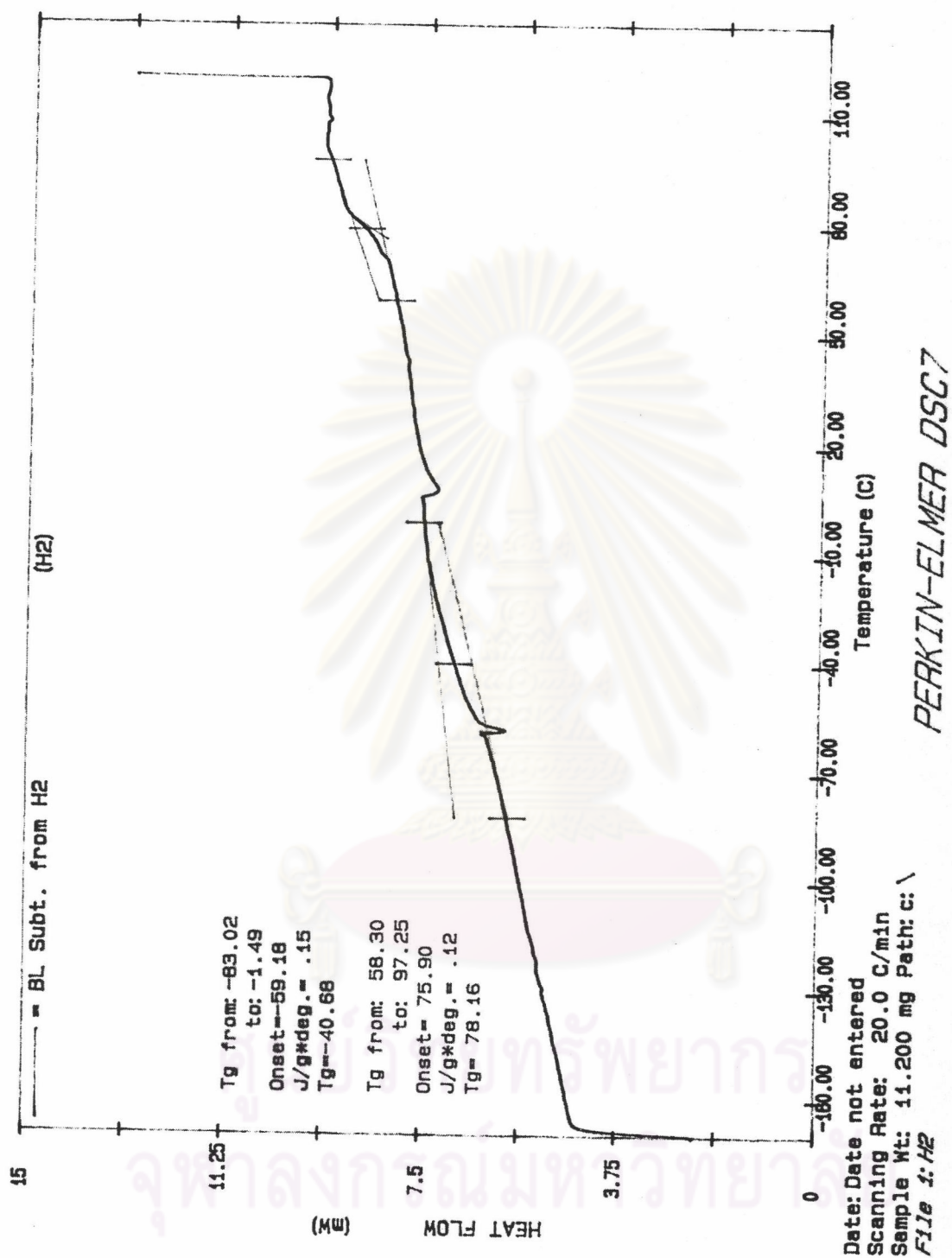
Figure 5.5.1 DSC of pure polystyrene

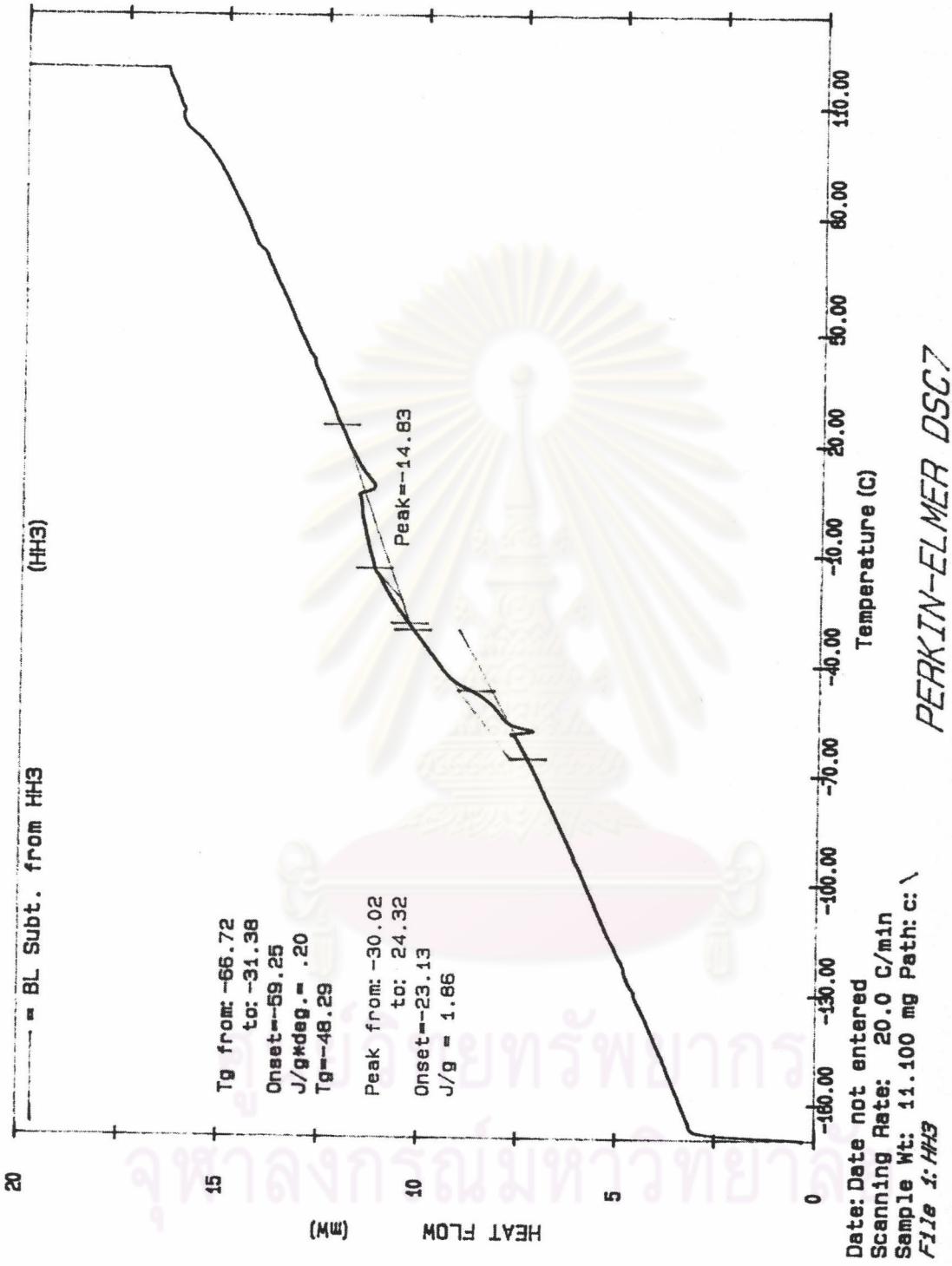Figure 5.5.2 DSC of synthesis polymer after soxhlet hexane

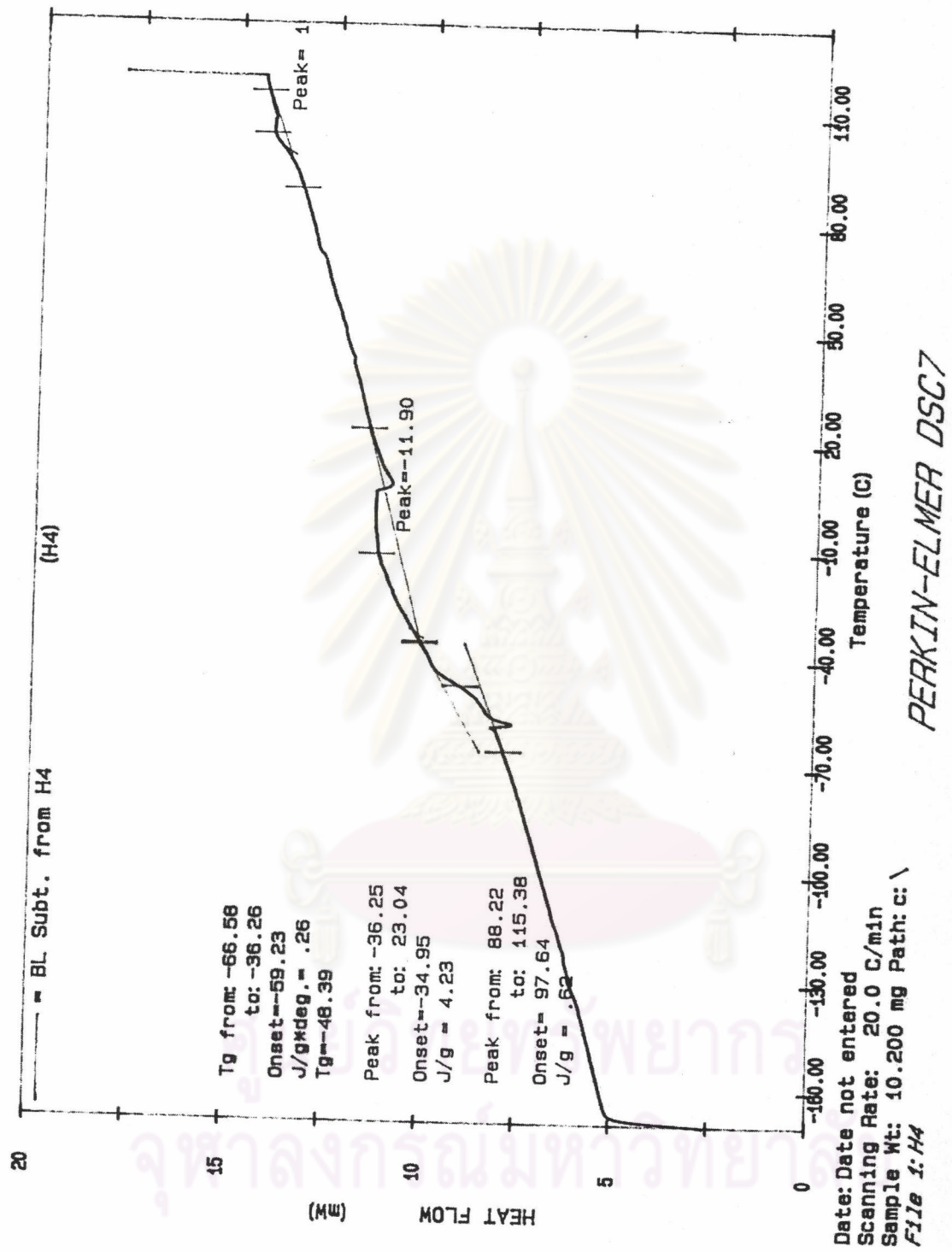Figure 5.5.3 DSC of synthesis polymer after soxhlet acetone

PERKIN-ELMER DSC7

(HH3)

= BL Subt. from HH3

Tg from: -66.72
to: -31.38
Onset=-59.25
J/g*deg.= .20
Tg=-48.29

Peak from: -30.02
to: 24.32
Onset=-23.13
J/g = 1.86

Peak=-14.83

Temperature (C)

HEAT FLOW (mW)

Date: Date not entered
Scanning Rate: 20.0 C/min
Sample Wt: 11.100 mg Path: c: \
File 1: HH3

Figure 5.5.4 DSC of solvent hexane after soxhlet hexane
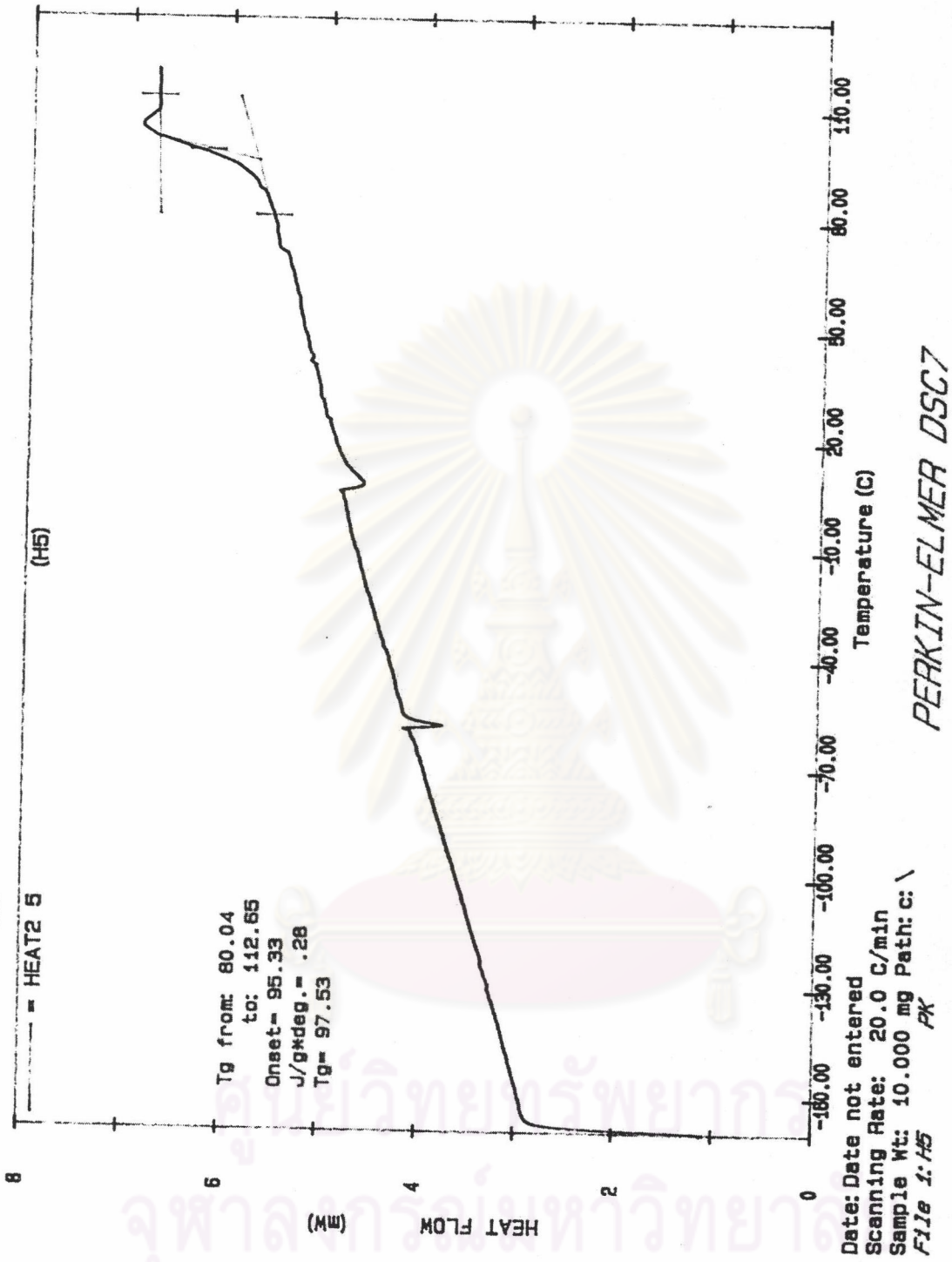
Figure 5.5.5 DSC of solvent acetone after soxhlet acetone

Figure 5.5.1 show the character of pure polystyrene. Peak of DSC appear in temperature range 80 to110 °C. Figure 5.5.2 and 5.5.3 show the character of synthesis graft copolymer. Peak of both figures are similar indicated that the Synthesis graft copolymers are in both product. Two peaks appear in both figures. The first peak appear in temperature range -50 to -15 °C which is the character of EPDM and the second peak appear in temperature range 80 to 110 °C is the character of polystyrene. Figure 5.5.4 show the DSC curve of residue in hexane solvent after soxhlet process with hexane. Peak appear in temperature range -50 to -15 °C and 80 to 110 °C imply that the extraction by the hexane solvent can eliminate unreacted EPDM and small branch of PS onto EPDM. Figure 5.5.5 show DSC curve of residue in acetone solvent after soxhlet process with acetone. The only one peak appear in this figure in temperature range 80 to 110 °C which is similar to the figure 5.5.1 and is indicated pure polystyrene that formed in the extract by the acetone solvent.

5.6 ^{13}C-Nuclear Magnetic Resonance (^{13}C-NMR)

NMR is one of the most powerful tools available to chemists and biochemists for elucidating the structure of both organic and inorganic species. NMR is based upon the measurement of absorption of electromagnetic radiation in the ratio-frequency region of roughly 4 to 600 MHz. Spectra were referenced to 0 ppm for trimethylsilane-d_6 and 7.26 ppm for deuterochloroform. Chemical shifts were given in parts per million (ppm) and coupling constant (J) are in Hertz (Hz).

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* CHULALONGKORN UNIVERSITY *
* JNM-A500 *
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SFILE : 1.DI EPDM-4640-BCM
COMNT : EPDM-4640-13C

EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQU : 18115.94 HZ
SCANS : 600
DUMMY : 4
ACQTM : 1.9088 sec
PD : 11.3500 sec
RGAIN : 23
PW1 : 9.50 usec
OBNUC : 13C
OBFRQ : 125.65 MHZ
OBSET : 124104.51 HZ

IRNUC : 1H
IRFRQ : 500.00 MHZ
IRSET : 152410.00 HZ
IRATN : 150
IRAPW : 55.0 usec
IRBP1 : 30
IRBP2 : 6
IRANS : 0

ADBIT : 16
CTEMP : 130.0 c
CSPED : 11 HZ
SLVNT : C6D6
RESOL : 0.55 HZ
BF : 2.00 HZ
REFVL : 128.00 ppm
XE : 18115.94 HZ
XS : 0.00 HZ

OPERATOR :

Figure 5.6.1 NMR (Carbon 13) of EPDM
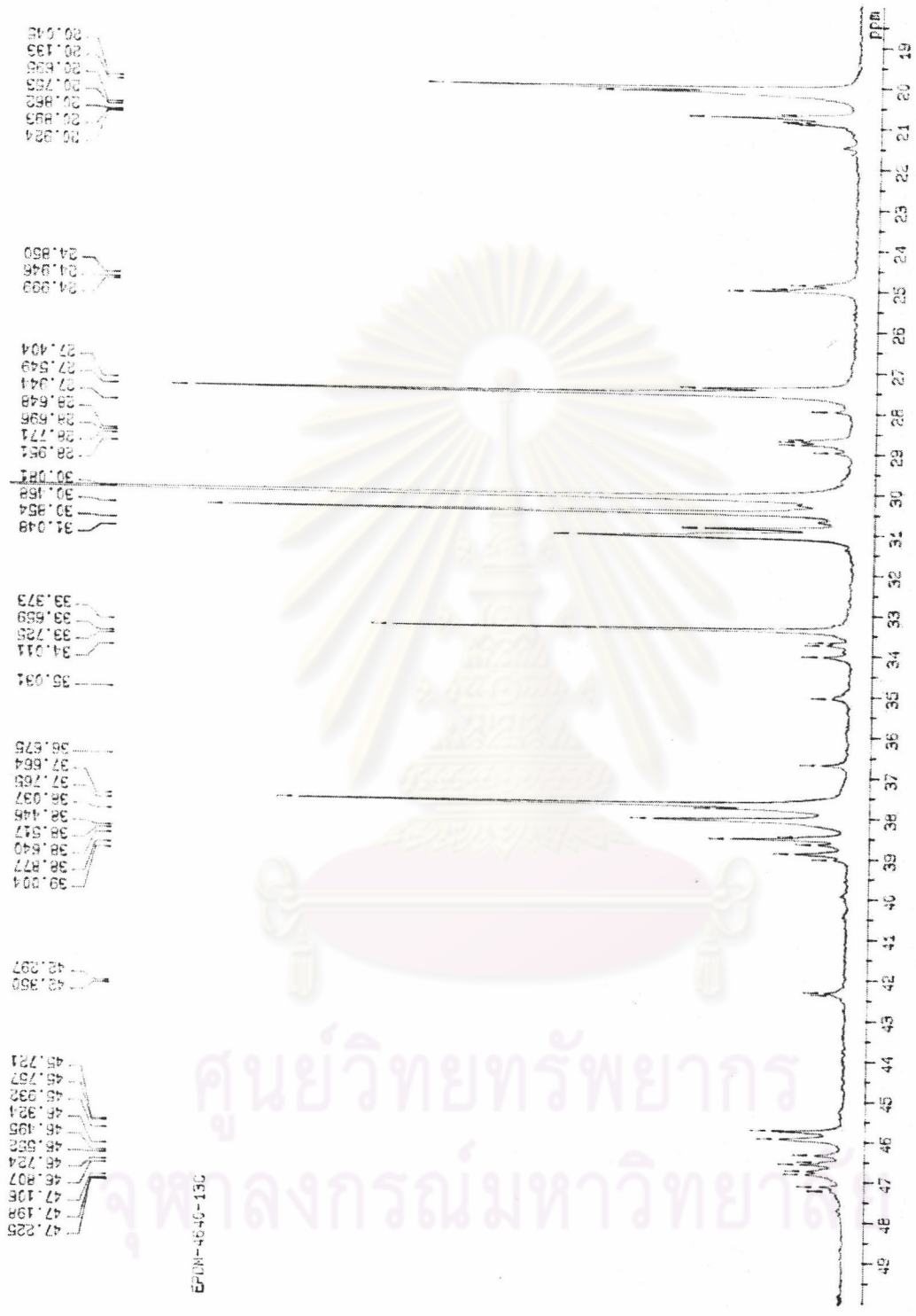
Figure 5.6.2 NMR (Carbon 13) of EPDM
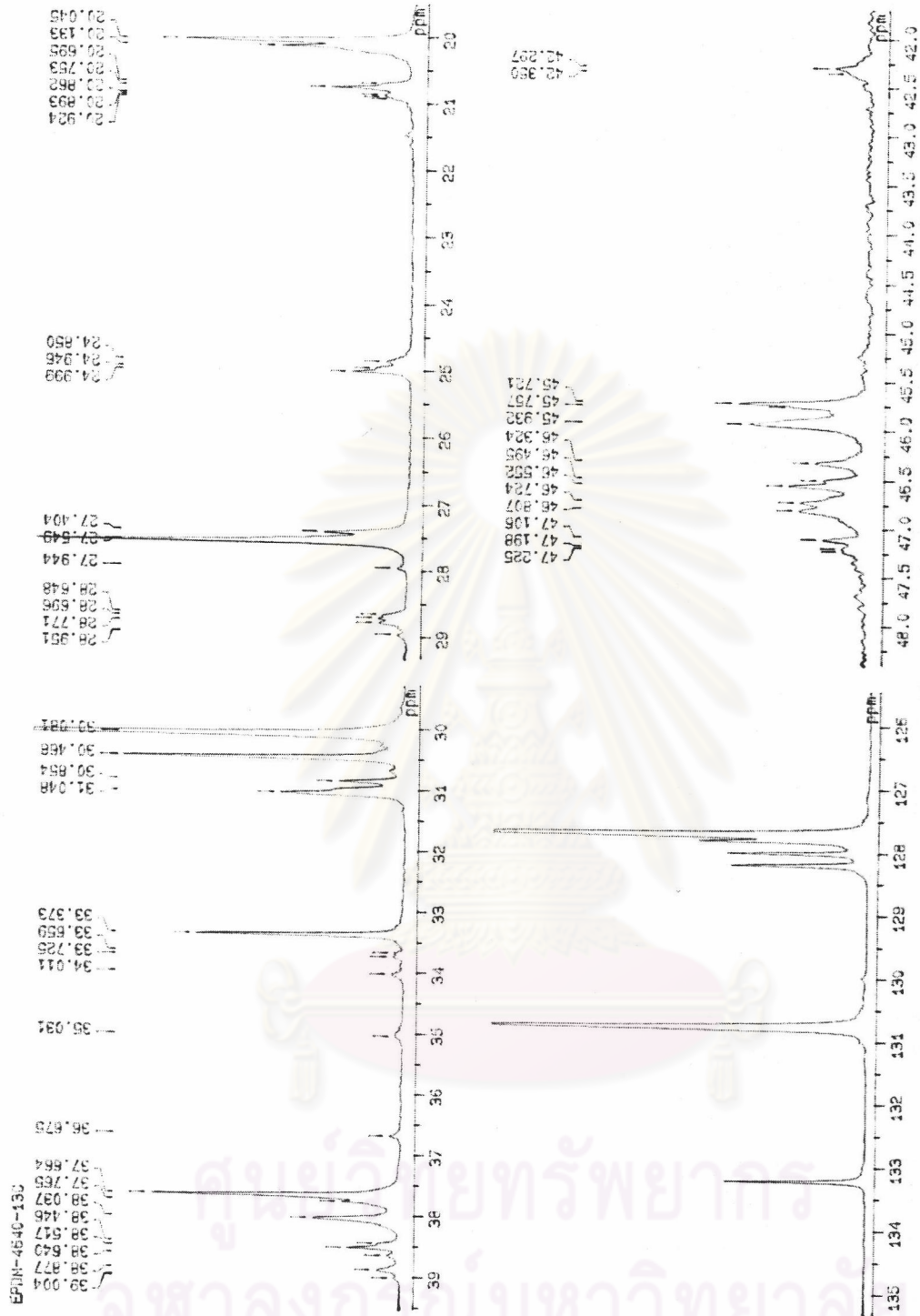
Figure 5.6.3 NMR (Carbon 13) of EPDM
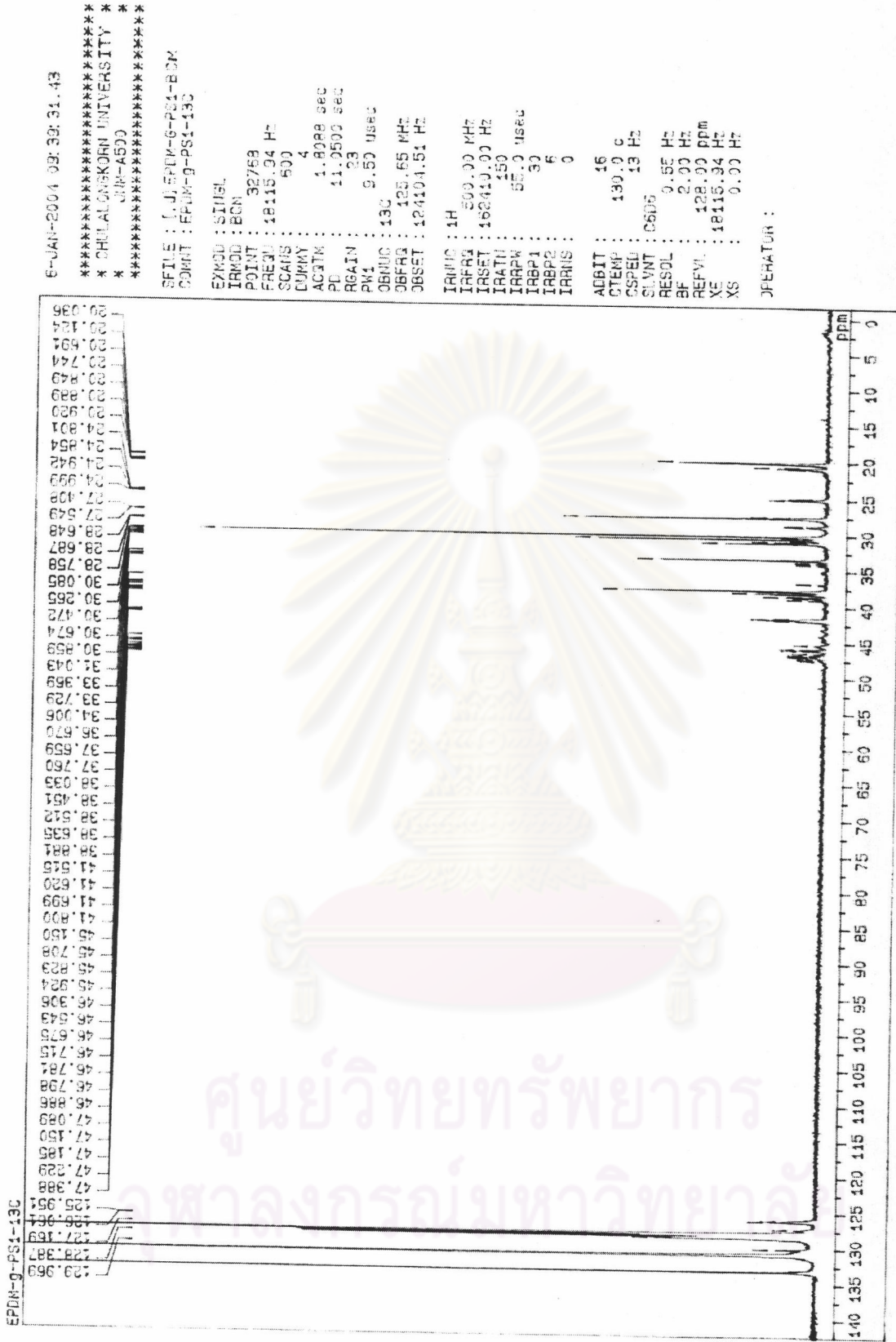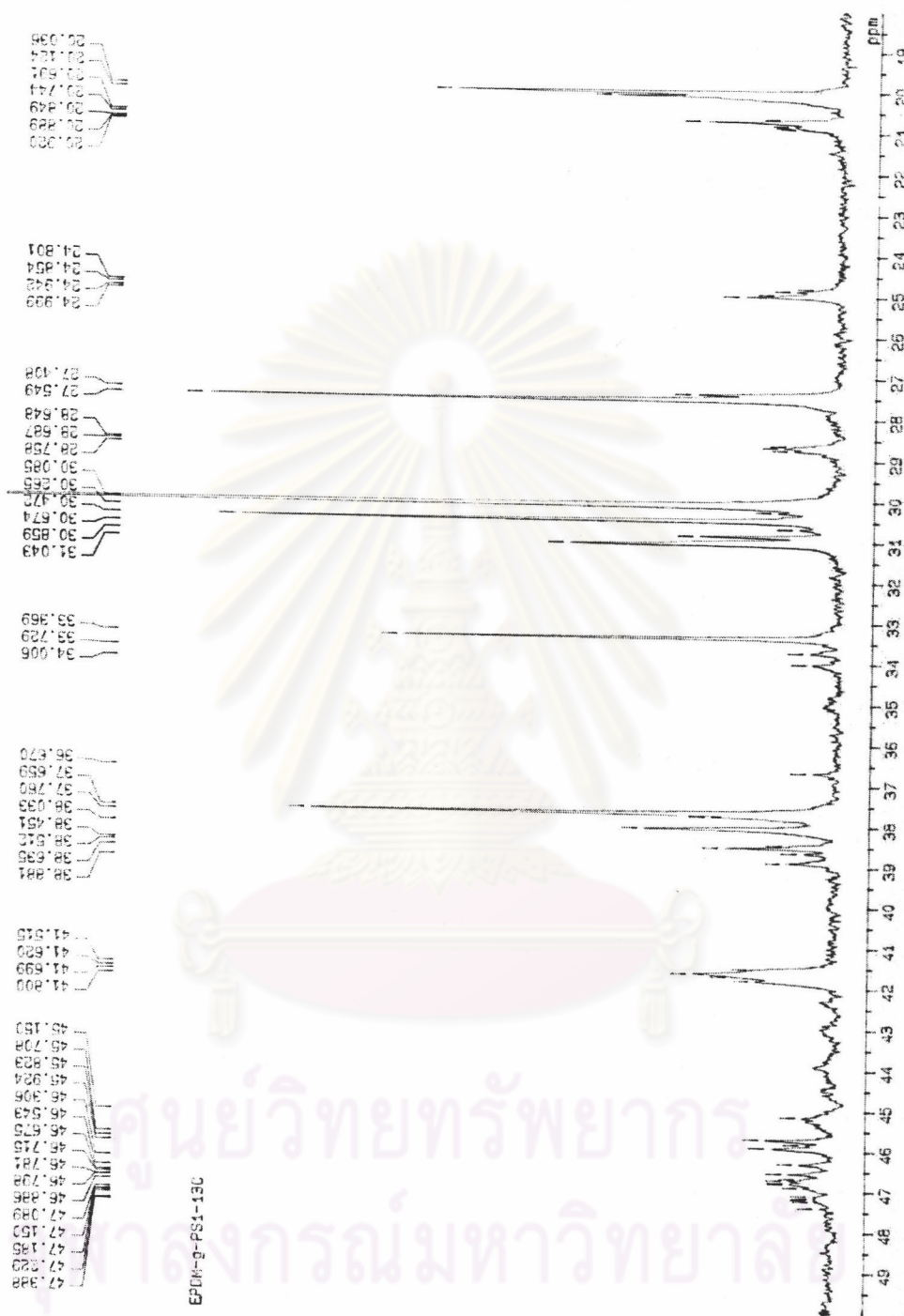
Figure 5.6.4 NMR (Carbon 13) of EPDM-g-PS1

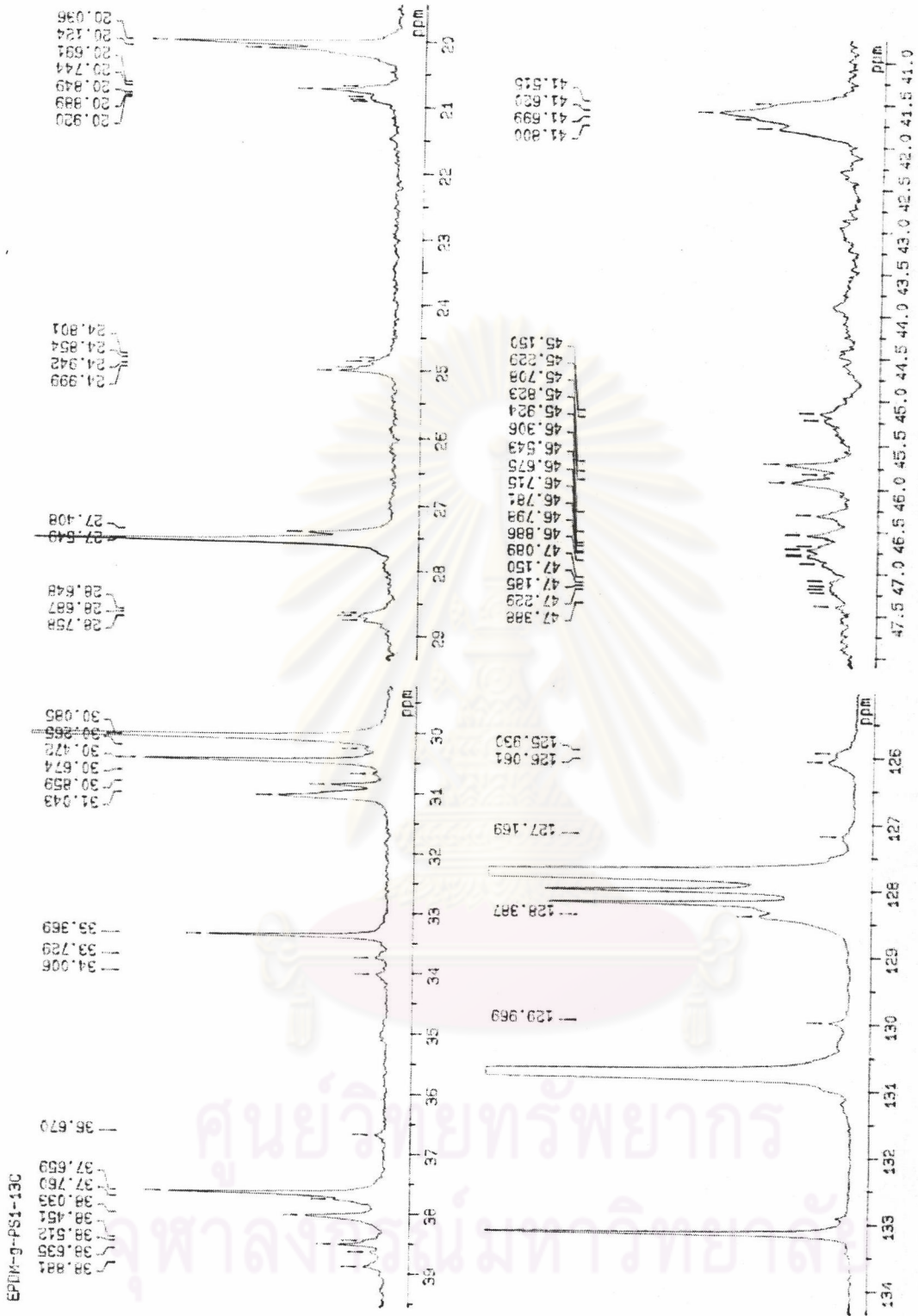Figure 5.6.5 NMR (Carbon 13) of EPDM-g-PS1
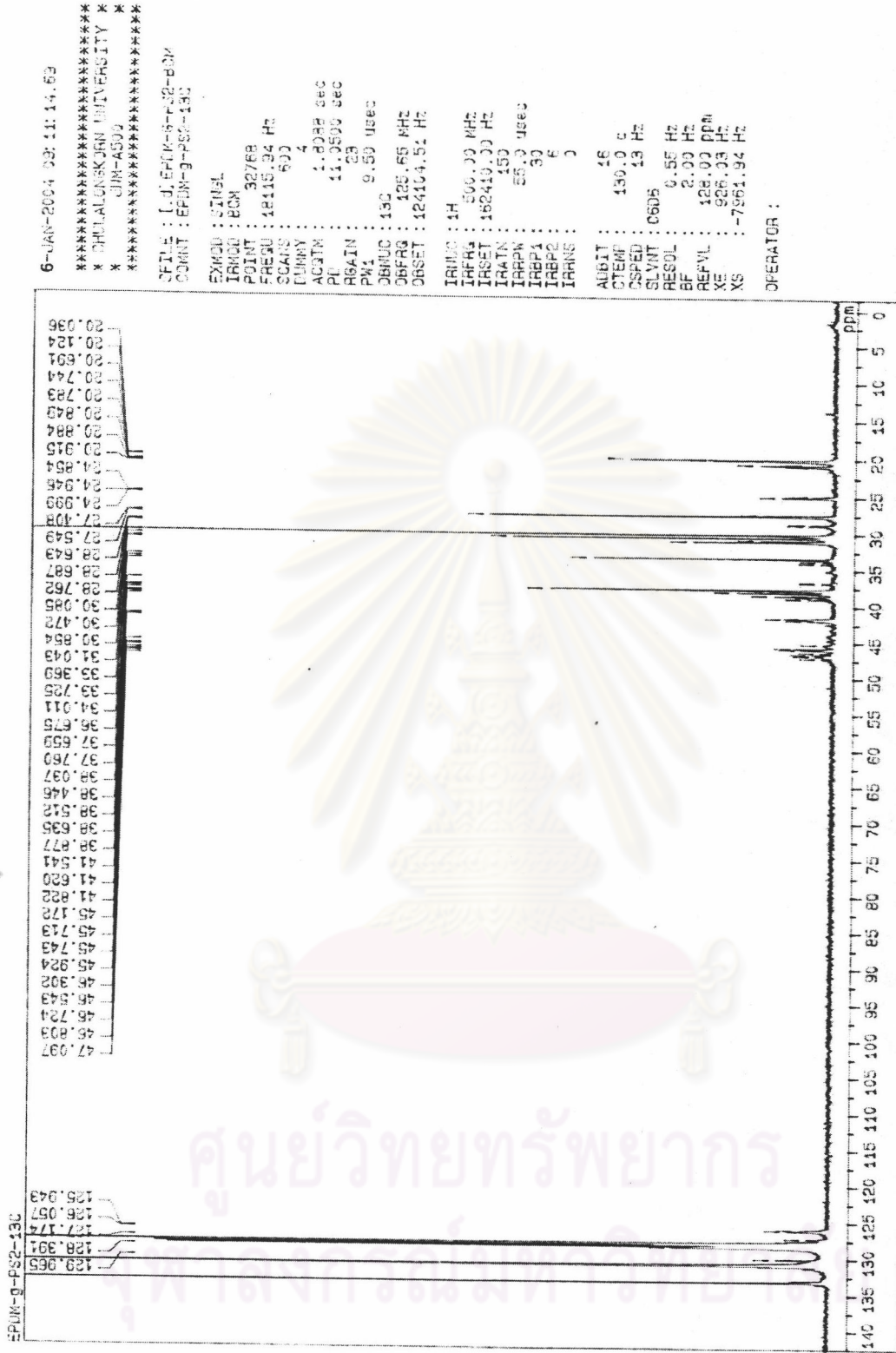
Figure 5.6.6 NMR (Carbon 13) of EPDM-g-PS1
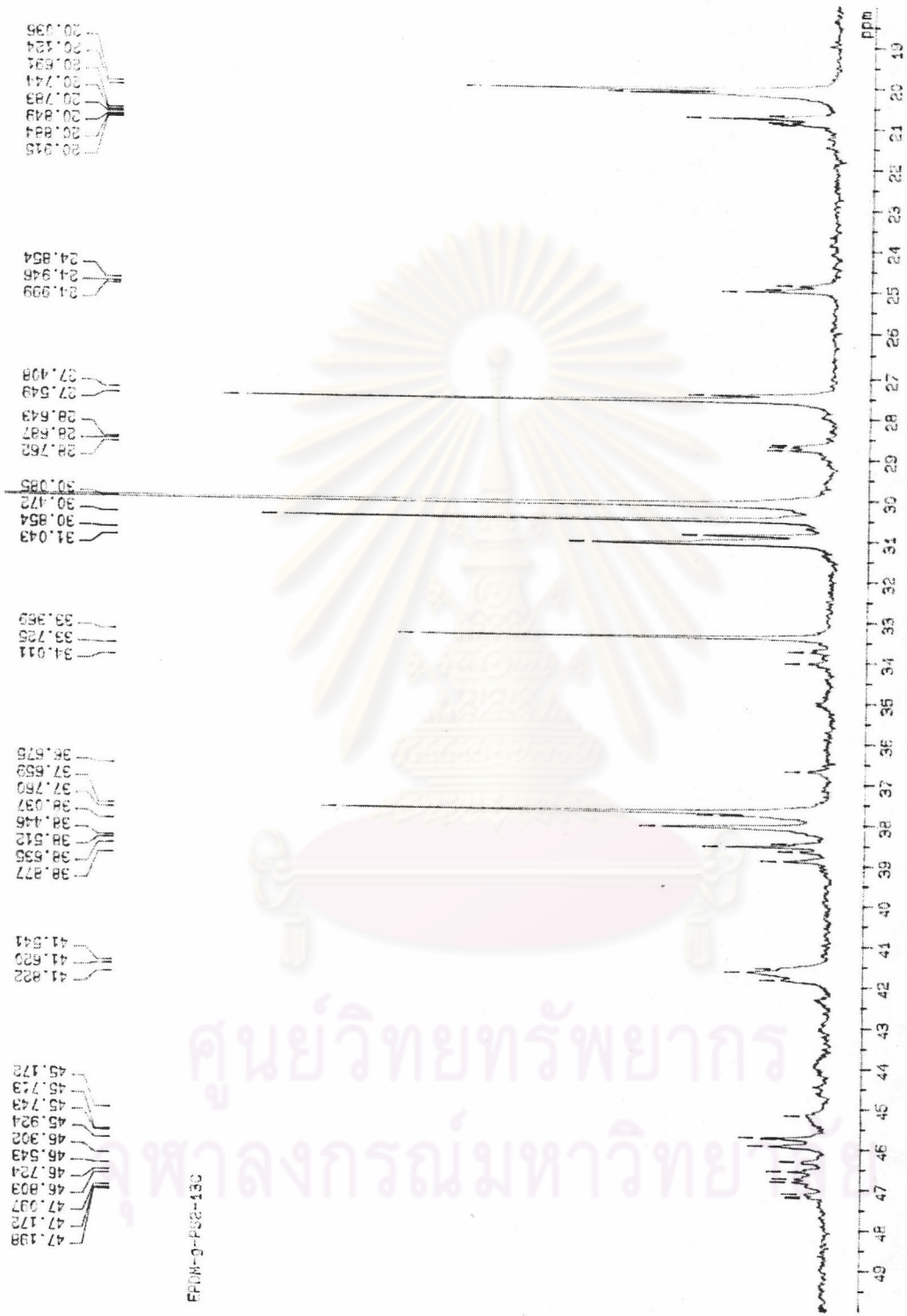
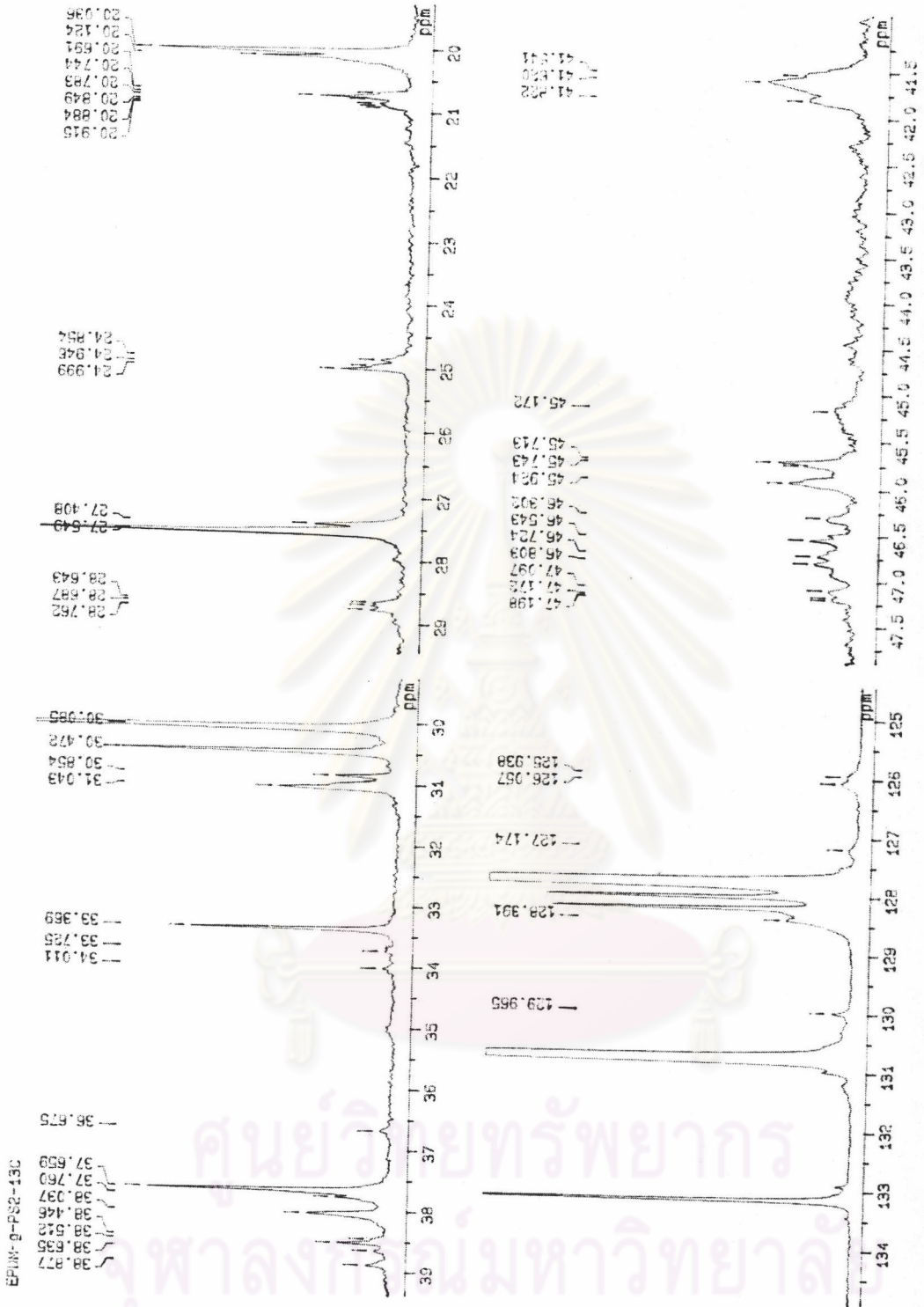Figure 5.6.7 NMR (Carbon 13) of EPDM-g-PS2

Figure 5.6.8 NMR (Carbon 13) of EPDM-g-PS2

Figure 5.6.9 NMR (Carbon 13) of EPDM-g-PS2

Figure 5.6.1- 5.6.3 show the ^{13}C NMR of pure EPDM, Figure 5.6.4-5.6.9 show the ^{13}C NMR of synthesis graft copolymer between EPDM and Polystyrene. From the difference in Figure 5.6.1 -5.6.3 from 5.6.4-5.6.9, they all have a two difference in chemical shift at 41.9 and 127.5 ppm. However, pure EPDM haven't the two peak at 41.9 and 127.5 ppm. Therefore, the both of synthesis graft copolymer can prove it is different structure from pure EPDM by ^{13}C-NMR analysis and can be implied the cooperate of styrene molecules in the main chain EPDM after extract off the pure EPDM by hexane.