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## APPENDIX A

### Characterization Data of Molecular Weight

#### Calibration Methods for Gel Permeable Chromatography

##### Determination of Molecular Weight of Standard Polystyrene (Calibration Curve).

Natural rubber can be dissolved in many organic solvents, for example benzene, chloroform, tetrahydrofuran (THF), and toluene. The molecular weight of natural rubber can be measured rather easily due to its soluble characteristic. Gel permeable chromatography (GPC) is a method to determine molecular weight of polymers on the basis of different size for different molecular weight molecules. The bigger molecules have bigger size than the smaller ones. The solvent is needed to be a mobile phase and it must be very clean, without any fine impurities, to prevent any clog in the pore of porous beads. The porous beads are made of crosslink materials, e.g. polystyrene, and used as a separation units for different molecular sizes. The beads are packed tightly in a separation column. The pores of the beads have certain size range to be capable to trap molecules with certain sizes matching to its pore sizes. Thus the bigger molecules can not diffuse into the pores and hence they move down and exit the column first. The smallest molecules come out the last. This technique is quite practical but it is a relative method and hence it needs a calibration curve. It was know that molecular weights of polymers having various shapes (linear or branch polymers), various chemistry and chemical structure can be fitted on a “universal calibration



curve”. This curve needs more information of intrinsic viscosity to construct and use it properly. Hence to use this curve, it is quite complicated.

However, the typical way to make calibration curve of GPC is by using a standard polymer (usually linear shape), for example polystyrene with narrow molecular weight distribution. This may be due to the simplicity and well-known polymerization method of styrene. However, there is a controversial on using standard material that is different from the sample material. The different chemical structure may leads to different results. So, this weak point of GPC should be awared.

#### Testing at Room Temperature

Test condition: Packed column used was Styragel HT4 with effective molecular weight of 5,000-600,000. It could be used from ambient temperature to 150 °C. The efficiency was 10,000 count plate. The solvent used was THF (HPLC grade) and was filtered by 0.45 µm membrane and purged with helium at 30 ml/min. Solvent flow rate was set at 1.0 ml/min. Baseline was checked at 0.1 and 1.0 ml/min 3 times.

Test Procedure: Standard polystyrene 0.012 g was dissolved in THF 5 g and filtered with 0.45 µm membrane. The 0.3%wt/wt solution of 60 µl was injected into the GPC.

Temperature of pump, injection loop, packed column, detectors (refractive index and ultraviolet) were kept at 35.2 °C.

Sensitivity of detector: For standard polystyrene, it was set at -32 but for sample (LLDPE/NR blends) it was set at +32.

The molecular weight and retention time of standard polystyrene passing through column styragel HT4 are shown in Table A-1 and Figure A-1, respectively. The backpressure during run increased to 246 psi (maximum

500 psi). This plot is the calibration curve for the samples (natural rubber only).

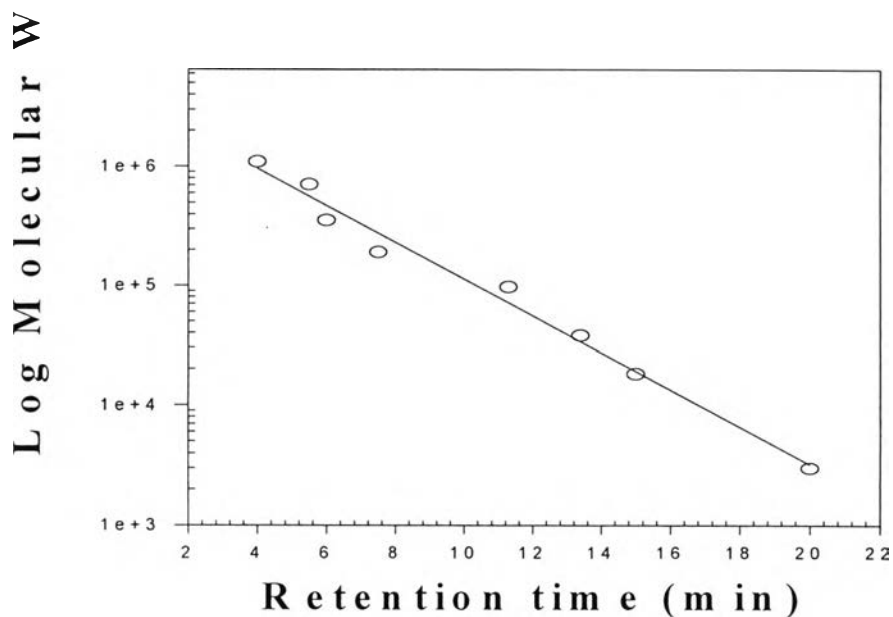
**Table A1** Retention time of standard polystyrene with known molecular weight at 35.2 °C.

Molecular Weight of Standard Polystyrene	Retention Time (min)	Calculated Molecular Weight
2,980	20.00	3,087
18,100	15.00	19,406
37,900	13.40	34,581
96,400	11.30	73,246
190,000	7.50	278,547
355,000	6.00	468,219
706,000	5.50	556,160
1,090,000	4.00	929,295

Equation of curve (from the instrument software):

$$\text{Log } M_w = 6.55 - (1.45 \times 10^{-1})R - (4.33 \times 10^{-4})R^2$$

Where R = Retention time (min)



**Figure A1** The calibration curve of standard polystyrene in THF at 35.2 °C and flow rate of 1.0 ml/min.

#### Testing at High Temperature

Test condition: Packed column used was Styragel HT4 with effective molecular weight of 5,000-600,000. It could be used from ambient temperature to 150 °C. The efficiency was 10,000 count plate. The solvent used was o-dichlorobenzene (HPLC grade) and purged with helium at 30 ml/min. Solvent flow rate was set at 1.0 ml/min. Baseline was checked at 0.1 and 1.0 ml/min..

Test Procedure: Standard polystyrene 0.012 g was dissolved in o-dichlorobenzene 5 g and filtered with 0.45 µm membrane. The 0.3 %wt/wt solution of 100 µl was injected into the GPC Water 150-c. Temperature of pump/solvent compartment were kept at 58 °C, injector compartment at

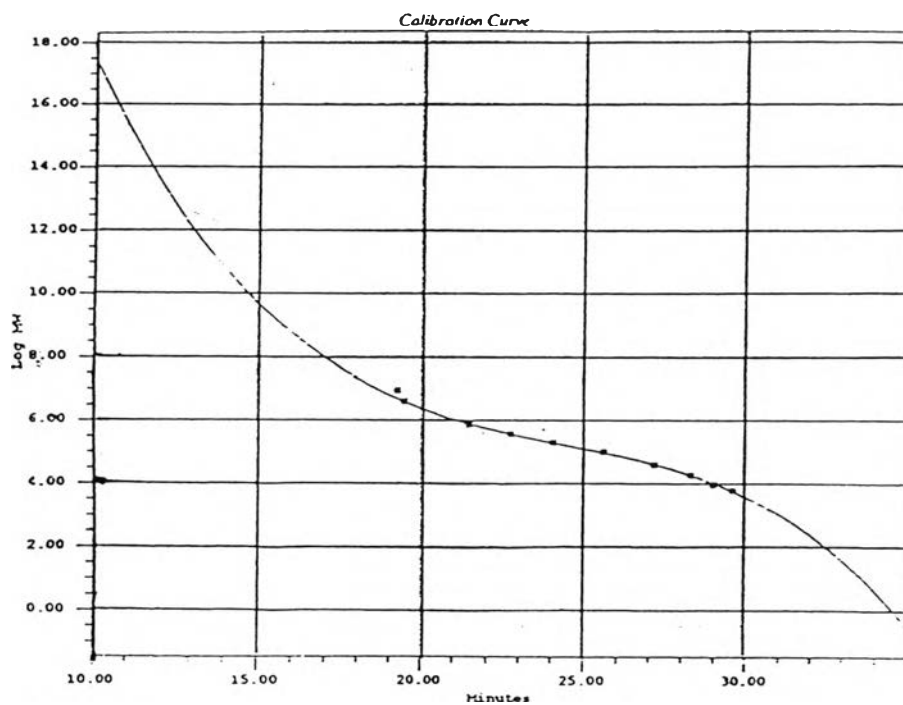
140 °C, column compartments at 140 °C, and detectors (refractive index model RI410) at 140 °C. Melennium version 2.10 was software used to operate and calculate molecular weight.

Sensitivity of detector: For standard polystyrene, it was set at -32 but for sample (LLDPE/NR blends) it was set at +32.

Results: Table below shows the molecular weight and retention time of standard polystyrene passing through series of Styragel columns. The backpressure during run did raised beyond maximum pressure of 38 bars. Figure A2 is the plot of molecular weight of polystyrene versus retention time. This plot is the calibration curve for the samples (LLDPE/NR blends) and pure LLDPEs.

**Table A2** Retention time of standard polystyrene with known molecular weight at 140 °C.

Molecular Weight of Standard Polystyrene	Retention Time (min)
5,970	29.633
9,100	29.017
18,100	28.317
37,900	27.200
96,400	25.617
190,000	24.083
355,000	22.750
706,000	21.467
3,840,000	19.450
8,420,000	19.250



**Figure A2** The calibration curve of standard polystyrene in o-dichlorobenzene at 140 °C and flow rate 1.0 ml/min.

## APPENDIX B

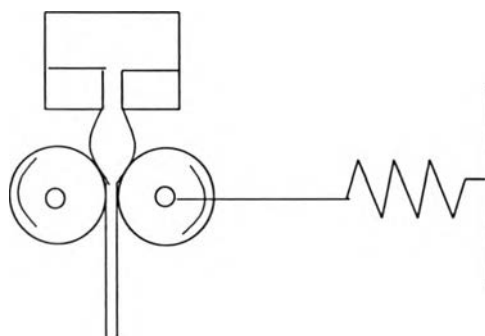
### Determination of Melt Strength

Melt strength refers to the elongation deformation of polymer melt during process, for example drawing, blowing, and stretching. It was stated by Micic *et al.* (1996) as following "... the melt must have sufficient strength to withstand the high strain arising from the drawing device, and if the tension within the melt exceeds the melt rupture stress, then the melt will tear. Melt strength is defined as the force at the break point and is indicative of the relative extensibility of the melt, i.e. the ability of the melt to be drawn down to thin gauges." Hence, the measurement of melt strength is to determine breaking force for elongational polymer melts. On the other words, it is the ability of polymer melts to be elongated or stretched before failure or flow instability occurs. The flow field during shaping process usually involves both shear and elongational deformations but the latter is often dominated. Micic *et al.* (1996) suggested a method to measure melt strength by using an instrument called "Gottfert Rheotens" melt strength tester as shown in Figure B1. The principle of this device was described that the melt extrudate from die was passed through the nip between two counter rotation rollers at certain acceleration or constant speed. The melt was driven until it was broken or instability and thus the load used was determined by the sensitive beam connected to one roller. The unit of the load was cN ( $10^{-2}$  Newton). It was also mentioned that melt strength was depended on process parameters, e.g. operation and ambient temperatures shear rate, distance between die exit to the two rollers, velocity, and acceleration of the tester.

Another work by Endo *et al.* (1986) reported that they measured draw down tension of the melt extrudate by a device called "Rothschild

tensiometer”. They described the device was used to sense the draw down stress of the melt when passing through a rotation take-up roll for spinning process of tapes. Hence this device is capable to measure melt strength as well by determining the load or stress used to drive the melt until it failed. However, the picture of the device was not shown.

In our experiment, both devices were not used because they were not found in the our laboratory. Thus the melt strength of LLDPE/NR blends were described comparatively by the draw ability (maximum draw ratio or thickness) between pure LLDPE and the blends. This means if the blends have better melt strength than pure LLDPE and the blends. This means if the blends have better melt strength than pure LLDPE, they must be drawn or stretched to higher draw ratio or become thinner than pure LLDPE.



**Figure B1** The picture of melt strength tester as shown by Micic *et al.* (1996).

## APPENDIX C

### Thermal Properties Data

The thermal properties of samples were studied on a NETZSCH DSC-200 at the temperature rate of 10 °C/min. The sample was heated from 30 °C to 150 °C and cooled down to 30 °C to determined melting temperature ( $T_m$ ), crystallization temperature ( $T_c$ ) and area under the melting temperature which is enthalpy of fusion ( $\Delta H_f$ ).  $T_g$  was determined by cooled down the sample from 30 °C to -120 °C and heated up to 30 °C (see Table C1-C2). The  $T_g$  of the sample was taken as the temperature corresponding to 50 % of the transition. The degree of crystallinity ( $\%X_c$ ) is calculated using as following equation, see Table C3-C7.

$$\%X_c = \frac{\Delta H_f}{\Delta H_{100}} \times 100 \quad A(1)$$

$$\Delta H_{100} = 289.1 \text{ J/g}$$

**Table C1** Glass transition temperature data of LLDPE.

Polymer	$T_{g1}$ (°C)	$T_{g2}$ (°C)
LLDPE	-27	-



**Table C2** Glass transition temperature data of LLDPE/NR<sub>10</sub> blends.

Blend Ratio LLDPE/NR <sub>10</sub>	MA (%wt)	T <sub>g1</sub> (°C)	T <sub>g2</sub> (°C)
90/10	0	-38.0	-25.1
	1	-40.0	-26.0
	3	-60	
	5	-60	
	7	-60	
80/20	0	-21.0	-48.0
	1	-25.0	-45.0
	3	-37.0	-45.0
	5	-44.0	
	7	-44.8	
70/30	0	-43.0	-64.0
	1	-41.0	-64.0
	3	-42.0	-55.0
	5	-54.0	
	7	-56.0	

**Table C3** Melting temperature, crystallization temperature, degree of crystallinity and heat of fusion data of LLDPE.

Polymer	T <sub>m</sub> (°C)	T <sub>c</sub> (°C)	%X <sub>c</sub>	ΔH <sub>f</sub> (J/g)
LLDPE	123.1±0.14	109.8±0.14	34.2±0.14	98.9±0.41

**Table C4** Melting temperature, crystallization temperature, degree of crystallinity and heat of fusion data of LLDPE/NR<sub>10</sub> blends with MA.

Blend Ratio LLDPE/NR <sub>10</sub>	MA (%wt)	T <sub>m</sub> (°C)	T <sub>c</sub> (°C)	%X <sub>c</sub>	ΔH <sub>f</sub> (J/g)
90/10	1	121.7	107.9	29.48	85.2
	3	121.5	107.8	29.34	84.8
	5	121.5	107.6	29.10	84.1
	7	121.7	106.3	29.10	84.06
80/20	1	121.6	108.0	27.19	78.6
	3	121.6	107.5	26.74	77.3
	5	121.5	106.8	25.64	74.1
	7	121.5	106.5	25.50	73.7
70/30	1	121.5	106.1	24.29	70.2
	3	121.4	105.7	23.25	67.2
	5	121.4	105.7	21.48	62.1
	7	121.4	105.6	20.86	60.3

**Table C5** Melting temperature, crystallization temperature, degree of crystallinity and heat of fusion data of LLDPE/NR<sub>10</sub> blends without MA.

Blend Ratio LLDPE/NR <sub>10</sub>	T <sub>m</sub> (°C)	T <sub>c</sub> (°C)	%X <sub>c</sub>	ΔH <sub>f</sub> (J/g)
90/10	121.7±0.07	108.4±0.07	30.2±0.09	87.3±0.28
80/20	121.7±0.00	108.4±0.00	27.4±0.41	79.2±1.18
70/30	121.5±0.03	107.9±0.07	25.2±0.16	72.8±0.46
50/50	121.4±0.03	106.2±0.07	18.2±0.14	52.6±0.41

**Table C6** Melting temperature, crystallization temperature, degree of crystallinity and heat of fusion data of LLDPE/NR<sub>25</sub> blends.

Blend Ratio LLDPE/NR <sub>25</sub>	T <sub>m</sub> (°C)	T <sub>c</sub> (°C)	%X <sub>c</sub>	ΔH <sub>f</sub> (J/g)
90/10	121.5±0.07	108.1±0.07	28.4±0.07	82.2±0.22
80/20	121.5±0.03	107.9±0.14	25.4±0.43	73.3±1.24
70/30	121.4±0.07	107.2±0.07	21.8±0.06	63.3±0.18

**Table C7** Melting temperature, crystallization temperature, degree of crystallinity and heat of fusion data of LLDPE/NR<sub>5</sub> blends.

Blend Ratio LLDPE/NR <sub>5</sub>	T <sub>m</sub> (°C)	T <sub>c</sub> (°C)	%X <sub>c</sub>	ΔH <sub>f</sub> (J/g)
90/10	122.7±0.12	109.5±0.03	33.1±0.23	95.8±0.67
80/20	122.5±0.07	109.2±0.03	31.0±0.45	89.6±1.32
70/30	122.4±0.03	109.1±0.05	27.5±0.82	79.4±2.38

## APPENDIX D

### Mechanical Properties Data

The tensile properties were studied using the Instron Universal Testing Machine model 4206 in the extension mode. Young's modulus, tensile strength, percent elongation at break and yield strength were determined according to ASTM D 638-91. The tensile temperature was 26 °C. The specimens were machined into dumbbell shapes following the ASTM D638-91 type IV for specimen dimensions, the width of narrow section was 6 mm and the gage length was 25 mm. The tests were performed at a crosshead speed of 500 mm/min with a 100 kN load cell. Raw data were shown in Table D1-D4.

**Table D1** Mechanical properties data of LLDPE.

Polymer	Tensile Strength (MPa)	Modulus (MPa)	Elongation at Break (%)	Yield Strength (MPa)
LLDPE	21.52±1.04	82.6±3.71	1728.7±17.6	12.6±0.48

**Table D2** Mechanical properties data of LLDPE/NR<sub>5</sub> blends.

Blend Ratio LLDPE/NR <sub>5</sub>	Tensile Strength (MPa)	Modulus (MPa)	Elongation at Break (%)	Yield Strength (MPa)
90/10	19±0.24	61±0.59	1628±17.6	9.5±0.009
80/20	18±0.62	51±1.72	1536±33.61	7.1±0.23
70/30	14±0.26	36±0.26	1404±47.08	5.6±0.33
0/100	0.26±0	2.7±0.36	738±12.9	0.206±0

**Table D3** Mechanical properties data of LLDPE/NR<sub>25</sub> blends.

Blend Ratio LLDPE/NR <sub>25</sub>	Tensile Strength (MPa)	Modulus (MPa)	Elongation at Break (%)	Yield Strength (MPa)
90/10	17.0±0.10	67.8±3.18	1563.5±9.18	11.3±0.54
80/20	13.8±1.16	61.0±1.33	1446.0±40.31	9.2±0.40
70/30	8.6±0.45	51.1±1.58	1220.2±64.22	8.0±0.19
0/100	0.2±0.11	3.1±0.00	653.5±14.96	0.2±0.00

**Table D4** Mechanical properties data of LLDPE/NR<sub>10</sub> blends.

Blends Ratio LLDPE/NR <sub>10</sub>	MA (%wt)	Tensile Strength (MPa)	Modulus (MPa)	Elongation at Break (%)	Yield Strength (MPa)
90/10	0	17.4±0.03	64.7±0.02	1457.4±17.60	10.0±0.09
	1	18.6±0.07	69.5±1.04	1664.2±19.60	10.8±0.04
	3	20.2±0.42	76.6±1.00	1872.3±0.14	12.5±0.09
	5	19.4±0.10	73.1±0.64	1854.2±0.24	12.3±0.12
	7	19.2±0.09	70.9±0.47	1777.3±1.10	12.0±0.03
80/20	0	15.7±0.22	56.7±0.29	1291.1±19.11	8.17±0.23
	1	16.9±0.05	60.2±1.48	1547.0±1.30	9.51±0.01
	3	17.6±0.28	65.9±0.59	1629.1±0.11	11.1±0.22
	5	18.5±0.03	70.0±0.09	1783.3±0.30	11.8±0.06
	7	17.4±0.07	65.6±0.05	1577.2±0.05	11.5±0.07
70/30	0	12.1±0.48	43.9±1.22	1073.3±4.21	7.5±0.30
	1	12.9±0.06	46.9±0.04	1272.8±0.08	8.7±0.21
	3	13.9±0.08	50.6±0.12	1450.0±0.01	9.8±0.04
	5	15.4±0.04	57.8±0.03	1481.1±0.06	10.5±0.09
	7	14.3±0.29	55.6±0.08	1375.4±0.18	10.3±0.19
50/50	0	5.1±0.06	21.0±0.09	835.4±0.15	3.4±0.03
	1	5.3±0.11	21.6±0.13	850.5±4.70	3.6±0.22
	3	5.7±0.00	26.3±0.02	876.3±2.37	3.9±0.11
	5	5.9±0.11	28.7±0.01	907.4±0.12	4.2±0.11
	7	6.6±0.24	31.0±0.23	906.2±1.59	4.4±0.01
0/100	0	0.2±0.00	2.8±0.36	738.5±12.91	0.2±0.00

## APPENDIX E

### Vicat Softening Temperature (VST) Data

The vicat softening temperature (VST) was determined at the temperature rate of 120 °C/hr with the load of 1 kg, using VST CEAST 6505 according to ASTM D1525. Table E1 provided the raw data.

**Table E1** Vicat softening temperature of LLDPE/NR<sub>10</sub> blends.

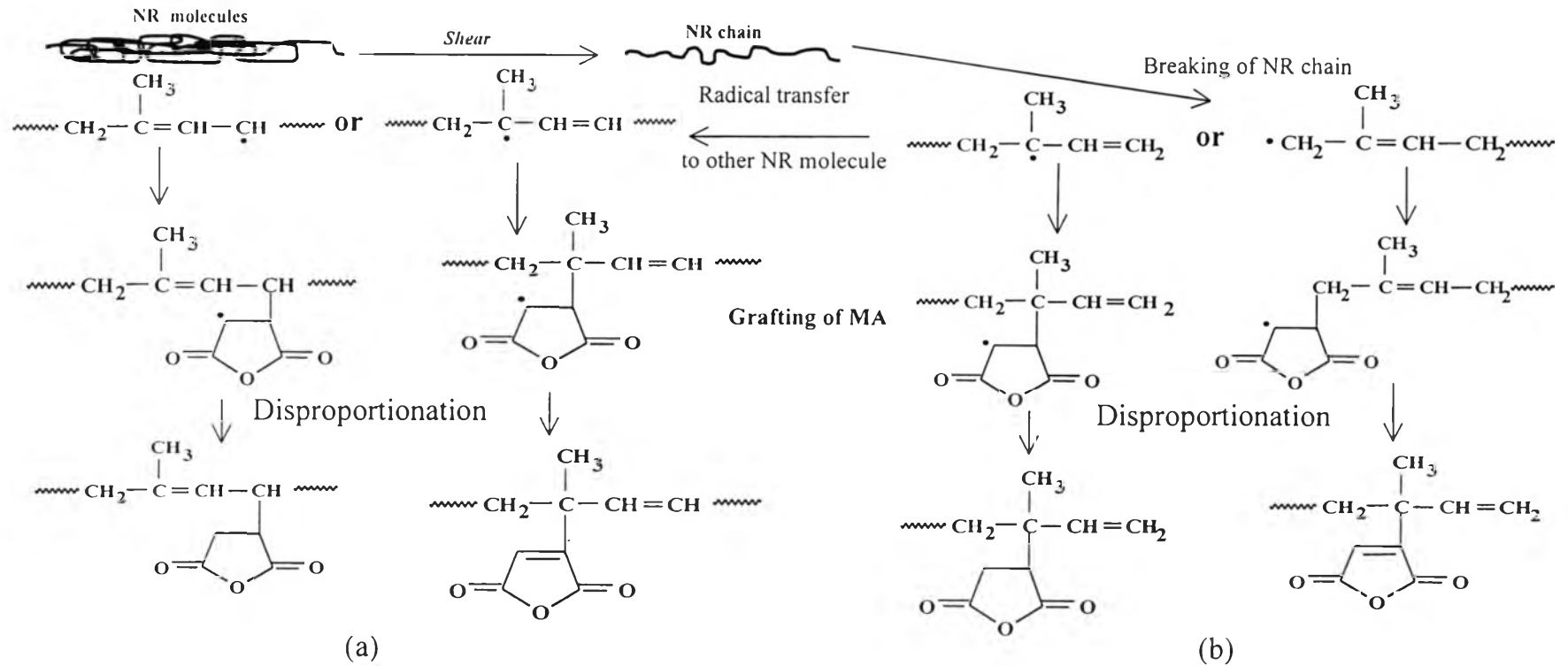
Blend Ration LLDPE/NR <sub>10</sub>	MA (%wt)	VST (°C)
100/0	0	102±0.00
90/10	0	98.8±0.14
	1	99.1±0.07
	3	99.5±0.07
	5	99.6±0.04
	7	99.7±0.07
80/20	0	91.7±0.28
	1	94.2±0.07
	3	94.3±0.07
	5	94.3±0.07
	7	94.4±0.14

Blend Ratio LLDPE/NR <sub>10</sub>	MA (%)	VST (°C)
70/30	0	83.1±0.07
	1	84.7±0.07
	3	84.7±0.14
	5	85.1±0.14
	7	84.9±0.21
50/50	0	55.1±0.11
	1	55.3±0.07
	3	55.5±0.07
	5	55.6±0.04
	7	55.6±0.01

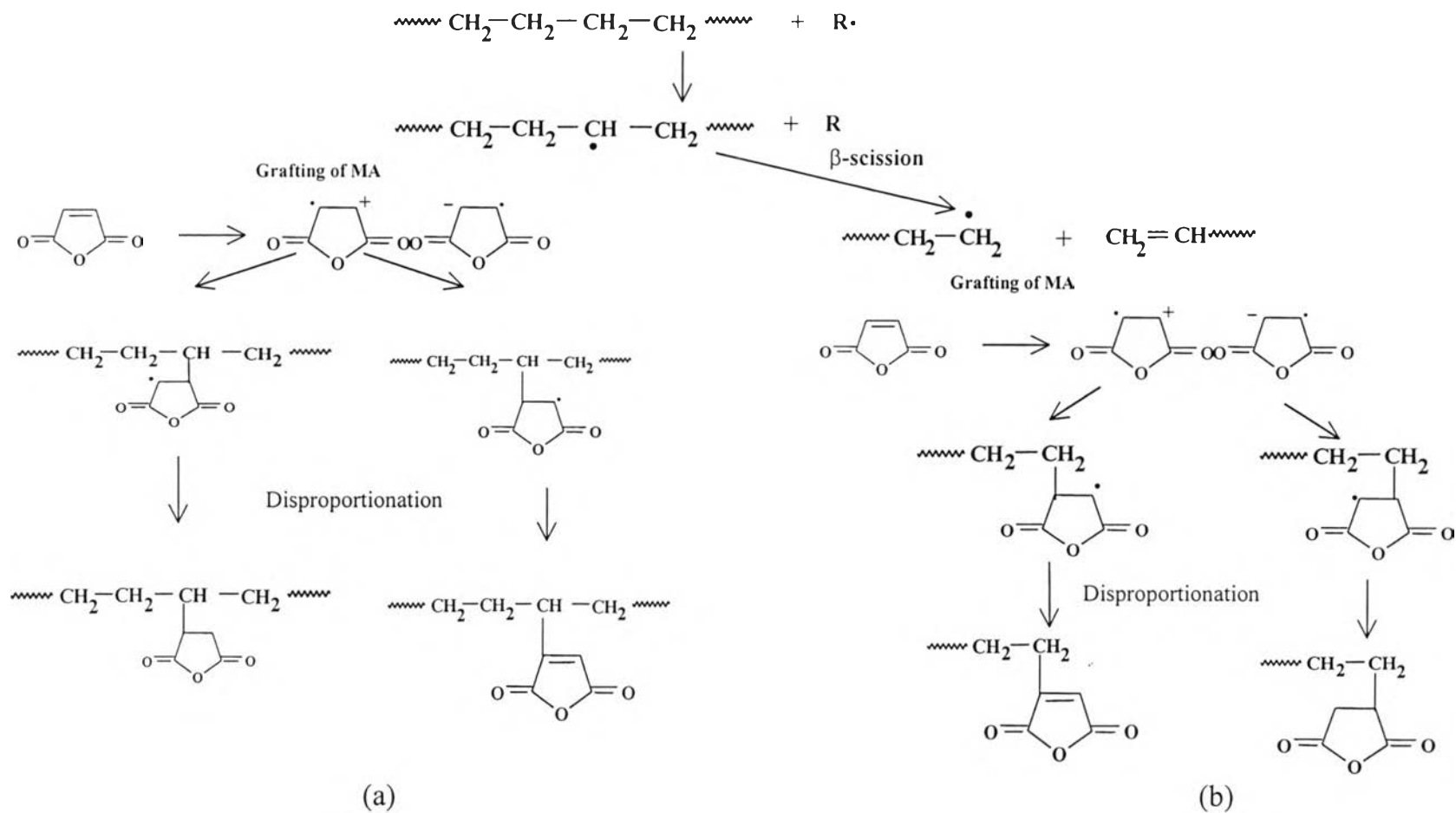


## APPENDIX F

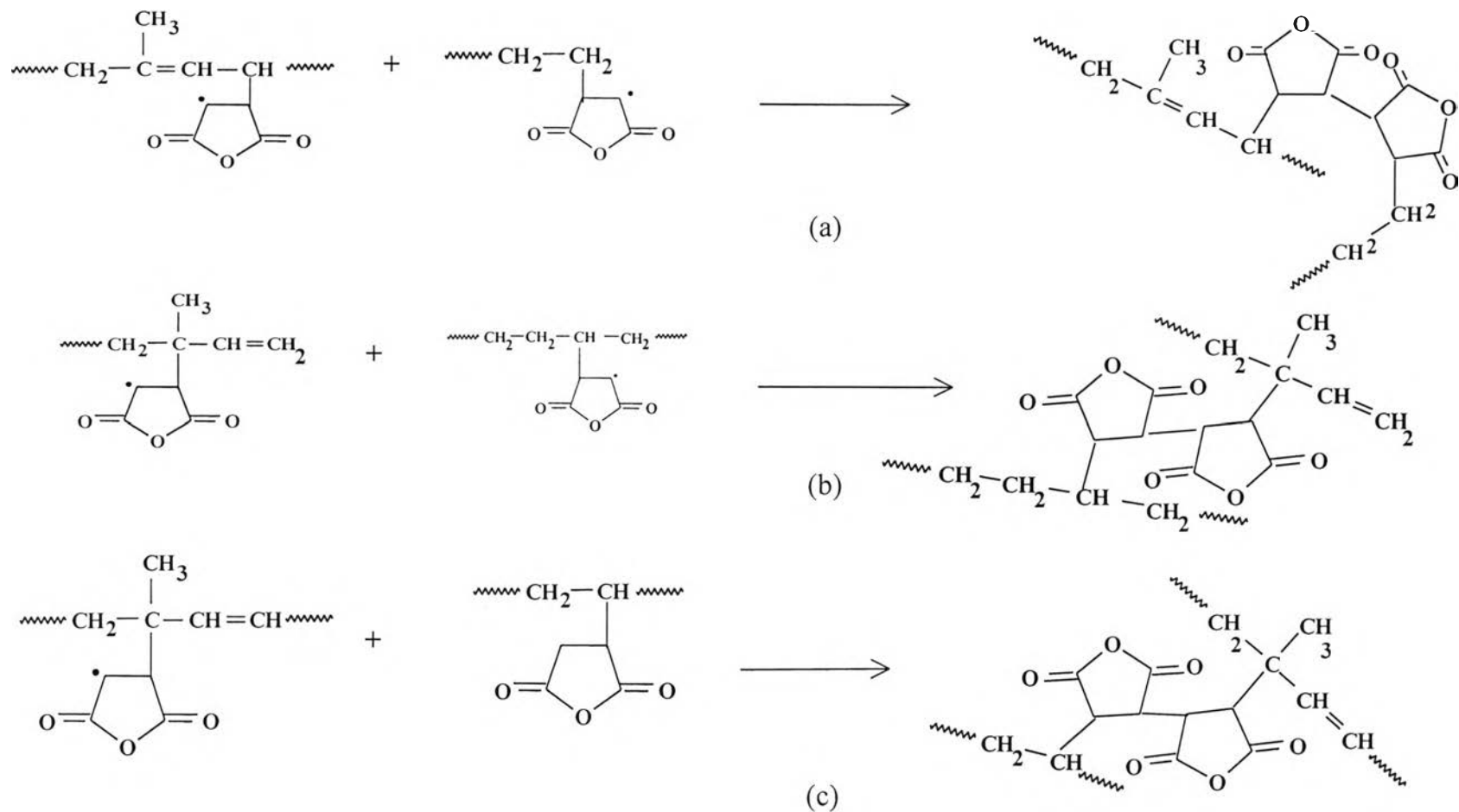
### Possible reactions of MA onto NR and LLDPE



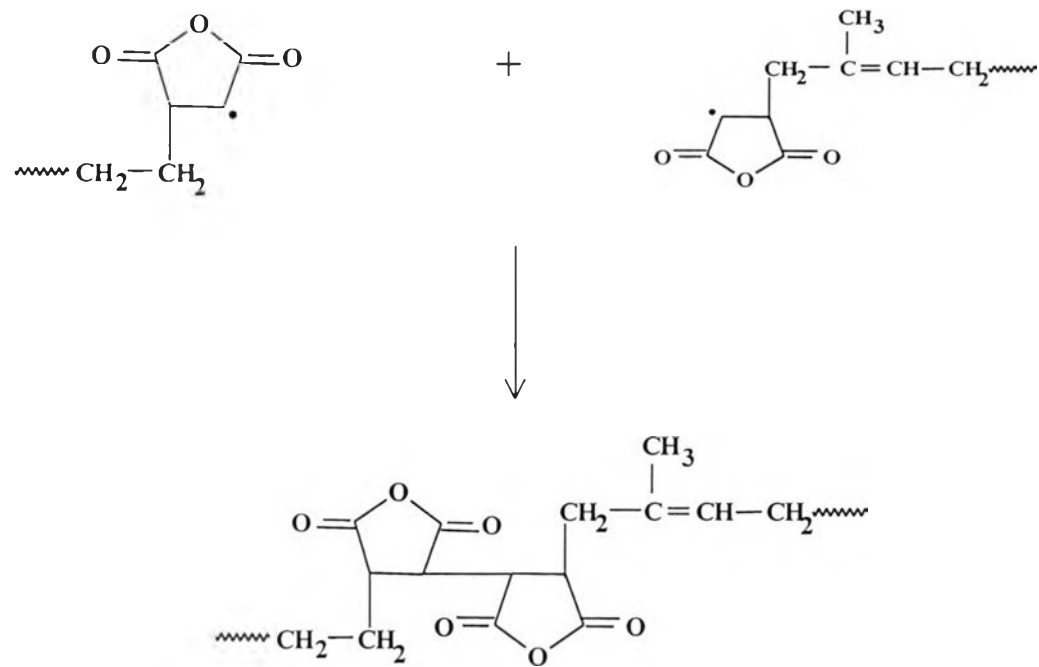
**Figure F1** Possible reaction of NR and MA to obtain NR-g-MA: (a) MA present at the middle of NR chain; (b) MA present at the end of NR chain.



**Figure F2** Possible reaction of NR and MA to obtain LLDPE-g-MA: (a) MA present at the middle of LLDPE chain; (b) MA present at the end of LLDPE chain.



**Figure F3** Grafting reaction to obtain graft copolymer of NR and LLDPE with MA linkage (LLDPE-MA-NR): (a) reaction between F1(a) and F2(b); (b) reaction between F1(b) and F2(a); (c) reaction between F1(a) and F2(a).



**Figure F4** Block copolymer of NR and LLDPE with MA linkage (LLDPE-b-MA-NR).

## APPENDIX G

## Peak Area Data from Fitting Curve Technique

Table G1 Peak area data of purified and crude of LLDPE.

MA (%wt)	Crude		Purified	
	A 1713 cm <sup>-1</sup>	A 1464 cm <sup>-1</sup>	A 1713 cm <sup>-1</sup>	A 1464 cm <sup>-1</sup>
1	8.5710	28.3265	0.2053	47.2569
3	9.4603	30.2653	0.2298	50.3625
5	11.2109	30.6952	0.2668	48.2563
7	5.1013	32.6585	0.1155	48.9635

Table G2 Peak area data of purified and crude of NR.

MA (%wt)	Crude		Purified	
	A 1713 cm <sup>-1</sup>	A 1464 cm <sup>-1</sup>	A 1713 cm <sup>-1</sup>	A 1464 cm <sup>-1</sup>
1	9.3005	60.2365	0.3076	32.6589
3	11.5633	62.3659	0.3482	30.6478
5	15.4063	59.9632	0.4678	29.6325
7	11.5348	58.6958	0.3434	28.4758

**APPENDIX H****Melt Flow Index Data**

The MFI of extrudates were measured at 190 °C, 2.16 kg loading, using a Zwick 4105 Extrusion Plastometer according to ASTM D1238. Table H1-H5 show the raw data.

**Table H1** Melt flow index data of LLDPE.

Polymer	MFI (g/10min)
LLDPE	1.04±0.07

**Table H2** Melt flow index data of LLDPE/NR<sub>5</sub> blends.

Blend Ratio LLDPE/NR <sub>5</sub>	MFI (g/10min)
90/10	0.83±0.01
80/20	0.77±0.01
70/30	0.71±0.01

**Table H3** Melt flow index data of LLDPE/NR<sub>25</sub> blends.

Blend Ratio LLDPE/NR <sub>25</sub>	MFI (g/10min)
90/10	0.96±0.003
80/20	0.92±0.005
70/30	0.88±0.004

**Table H4** Melt flow index data of LLDPE/NR<sub>10</sub> blends.

Blend Ratio LLDPE/NR <sub>10</sub>	MA (%wt)	MFI (g/10min)
90/10	0	0.91±0.003
	1	0.42±0.005
	3	0.26±0.005
	5	0.25±0.006
	7	0.24±0.003
80/20	0	0.84±0.002
	1	0.41±0.010
	3	0.17±0.002
	5	0.15±0.001
	7	0.12±0.002
70/30	0	0.77±0.010
	1	0.37±0.008
	3	0.15±0.006
	5	0.09±0.005
	7	0.04±0.003
50/50	0	0.56±0.016
	1	0.26±0.007
	3	0.12±0.004
	5	0.06±0.002
	7	0.02±0.002



## CURRICULUM VITAE



**Name;** Miss Parichart Limsila

**Birth Date:** June 29, 1975

**Nationality:** Thai

**University Education:**

1993-1996 Bachelor's Degree of Science in Department of Industrial Chemistry, Faculty of Applied Science, King Mongkut's Institute of Technology North Bangkok