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APPENDIX A

Calibration Data for Gel Permeation Chromatography (GPC)

Weight average molecular weight ($\overline{M}_w$), number average molecular weight ($\overline{M}_n$) and molecular weight distribution (MWD) of NR were determined by at room temperature gel permeation chromatography, Waters 600E. The column series of HT4 and HT5 were calibrated using narrow MWD polystyrene standard. By using flow rate 1 cm^3/min, tetrahydrofuran (THF) was used as a solvent for natural rubber and the polystyrene standard. The temperature of the column was controlled at 35°C and the injection volume was 60 µl.

Table A1 Retention time of standard polystyrene with known molecular weight at room temperature.

Retention time (min)	Specified Molecular weight	Calculated Molecular Weight
11.77	3840000	3687753
11.93	2890000	2958254
12.77	1090000	1159355
13.41	706000	688819
14.61	355000	338287
15.78	190000	191268
16.98	96400	100243
18.36	37900	38330
19.31	18100	16844
19.90	9100	9549
20.37	5970	5941

Curve Type: 4^{th} Order

Equation of Curve: $\log MW = +7.40E+01 - 1.55E+01 * R + 1.32E+00 * R^2$
$- 4.99E-02 * R^3 + 6.89E-04 * R^4$

where R = retention time (min).

Correlation Coefficient: $r^2 = 0.99964536$

Standard Error of

Estimate: 0.02387588

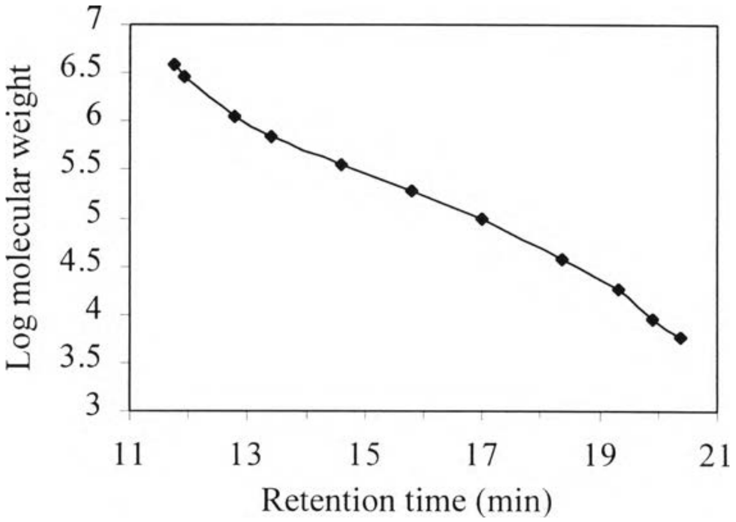

Figure A1 Calibration curve of standard polystyrene in THF at room temperature and flow rate of 1.0 ml/min.

Table A2 Effect of mastication time on molecular weight of NR.

Mastication time (min)	M_n	M_w	MWD
0	228517	1122573	4.9124
5	192404	582996	3.0301
10	183553	482715	2.6298
25	159783	365595	2.2881

Table A3 Retention time of standard polystyrene with known molecular weight at room temperature for the new lot of NR.

Retention time (min)	Molecular weight
11.532	1290000
12.306	520000
13.237	172100
13.978	66000
15.427	10850
15.965	5460

log MW = +1.23E+001 –5.37E-001 T^1

R^2 = 0.999772

R = 0.999886

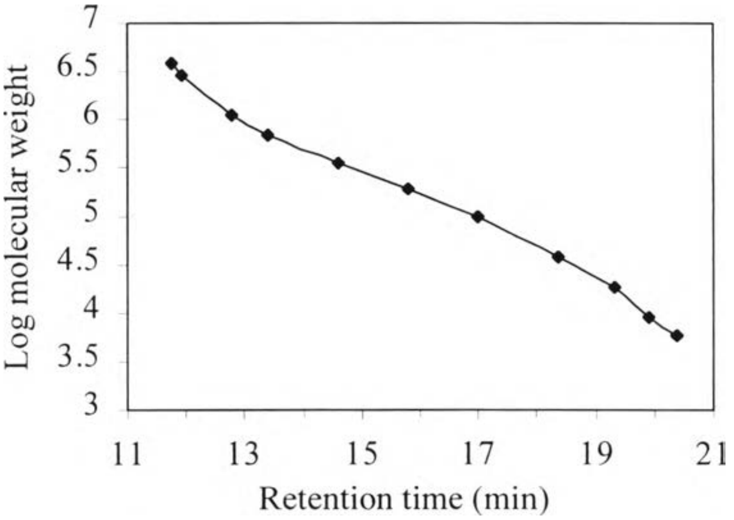

Figure A2 Calibration curve of standard polystyrene in THF at room temperature and flow rate of 1.0 ml/min for the new lot of NR

Table A4 Effect of mastication time on molecular weight for the new lot of NR.

Mastication time (min)	M_n	M_w	MWD
0	308210	2196993	7.128241
5	210461	1020572	4.849218
8	190221	786119	4.132652
10	205057	709579	3.460407
13	178759	656318	3.671518
15	176102	619219	3.515262
20	179575	519975	2.895584
25	165806	472335	2.848722

APPENDIX B

Torque-Time-Temperature Relationship of Filled-NR Compounds Prepared by Melt Technique using Brabender Plasticorder

Abbreviations

A : Loading peak
G : Inflection point
E : End
B : Minimum
X : Maximum

A-B : Loading peak to Minimum
B-X : Minimum to Maximum
X-E : Maximum to End = Fusion time (t)
A-X : Loading peak to End

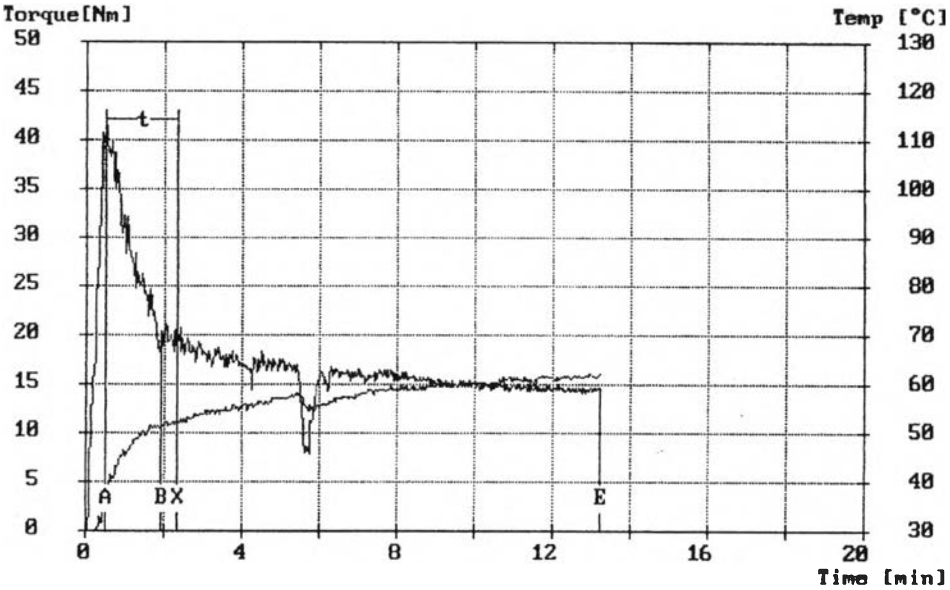

Figure B1 Torque-time-temperature relationship of NR compound

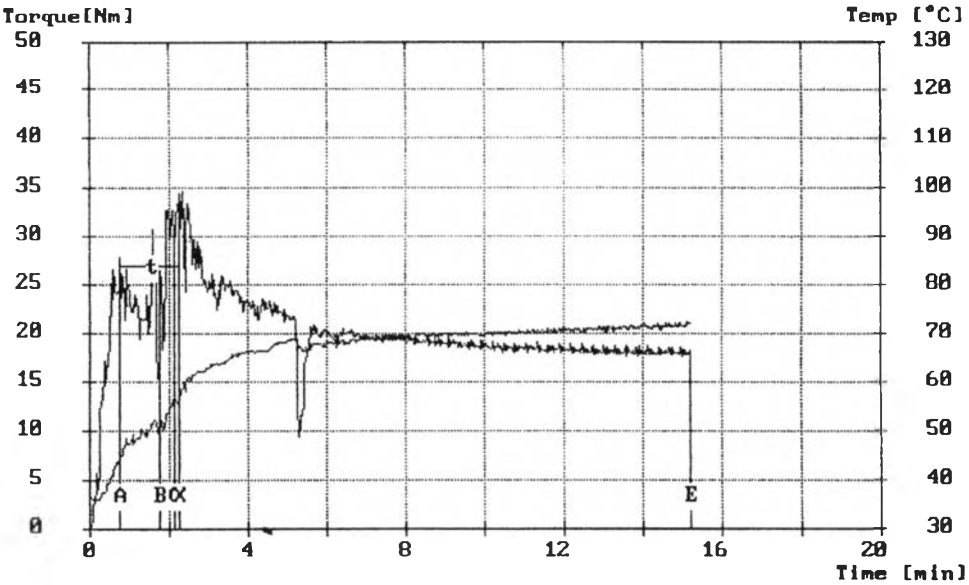

Figure B2 Torque-time-temperature relationship of NR/gypsum compound composition 80/20 in volume fraction

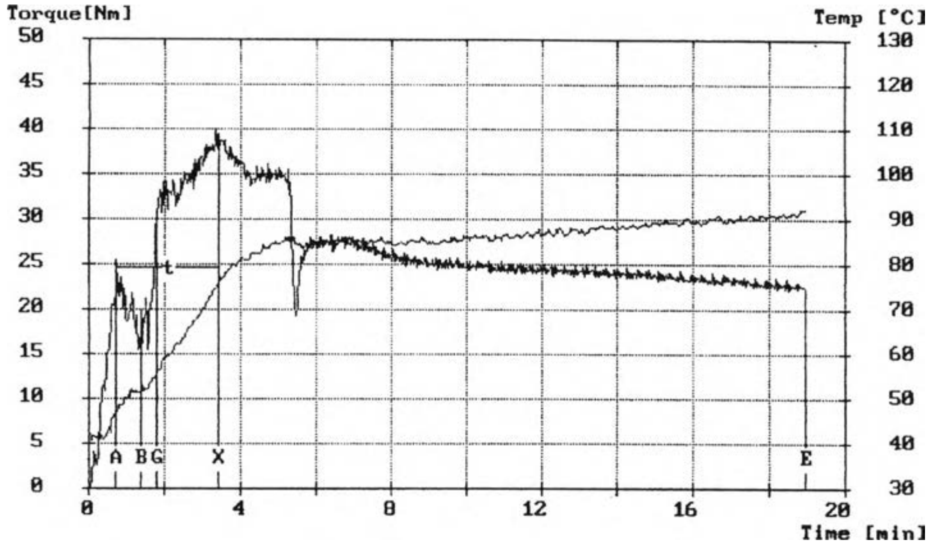

Figure B3 Torque-time-temperature relationship of NR/carbon black (N110) compound composition 80/20 in volume fraction

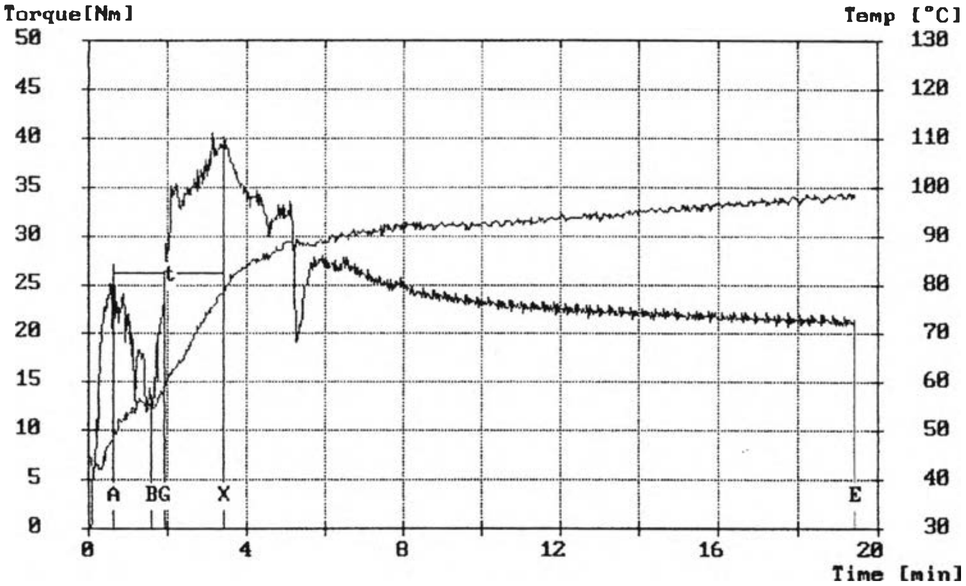

Figure B4 Torque-time-temperature relationship of NR/carbon black (N220) compound composition 80/20 in volume fraction

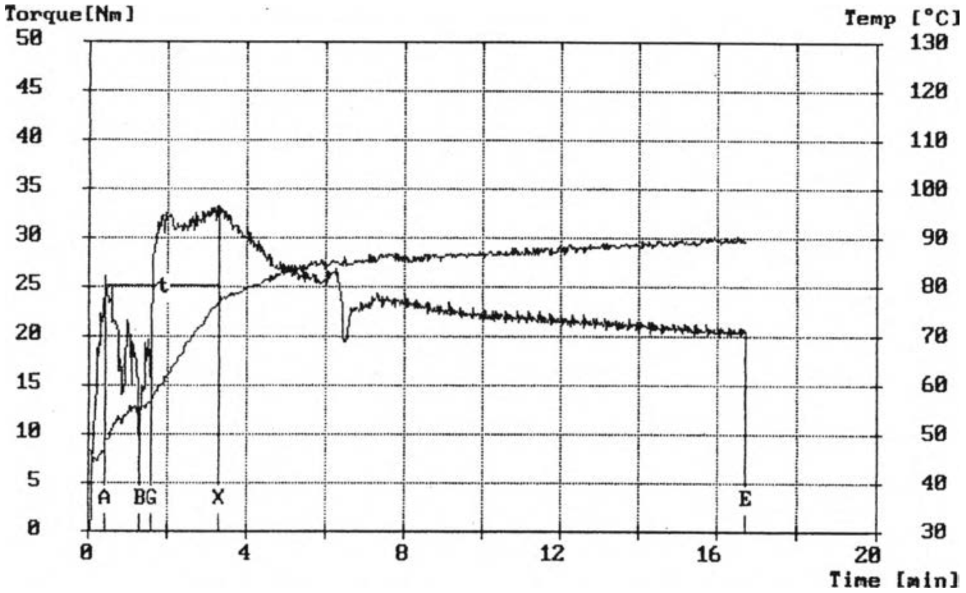

Figure B5 Torque-time-temperature relationship of NR/carbon black (N330) compound composition 80/20 in volume fraction

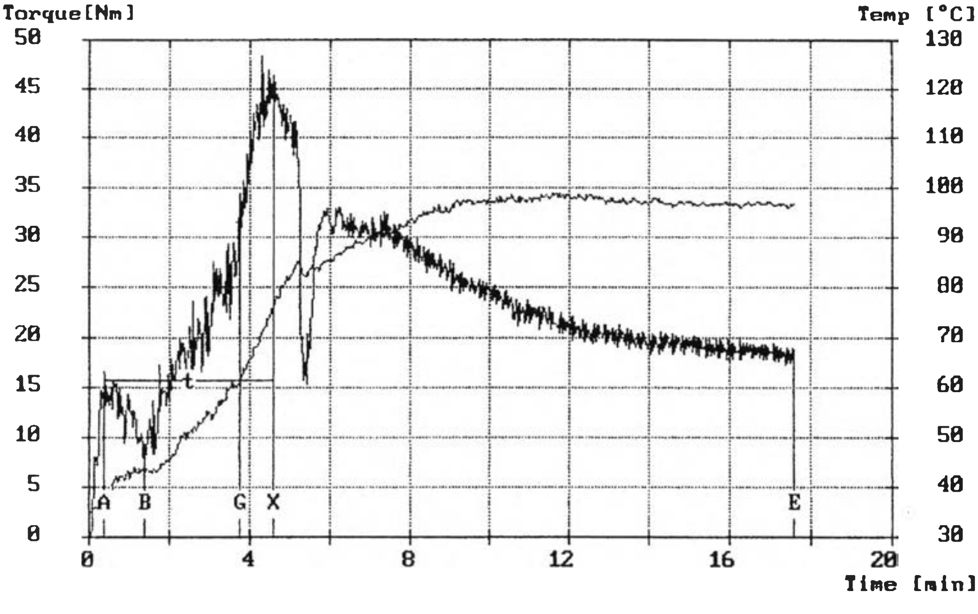

Figure B6 Torque-time-temperature relationship of NR/silica (Hi-Sil927) compound composition 80/20 in volume fraction

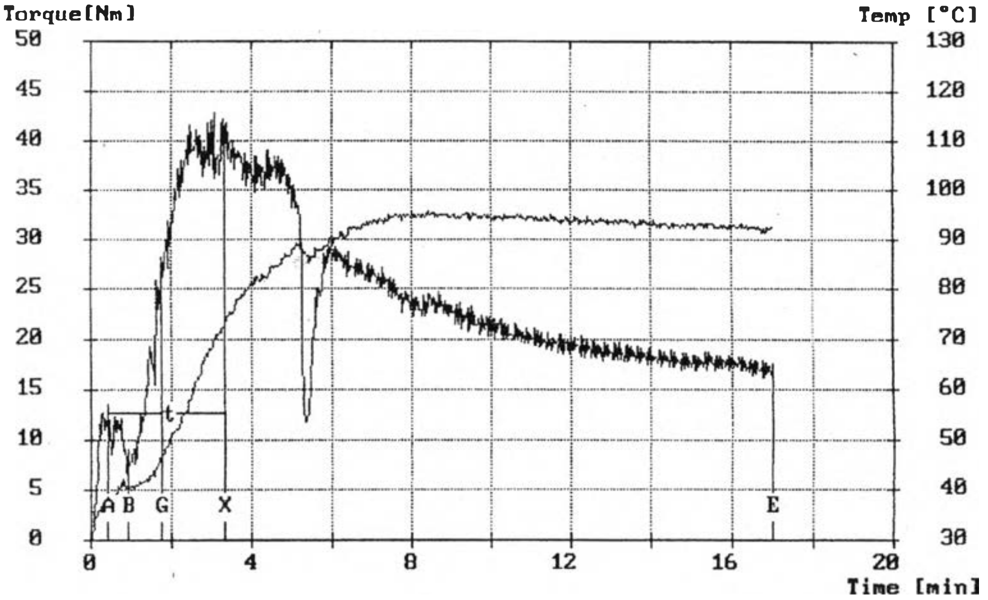

Figure B7 Torque-time-temperature relationship of NR/silica (Hi-Sil255) compound composition 80/20 in volume fraction

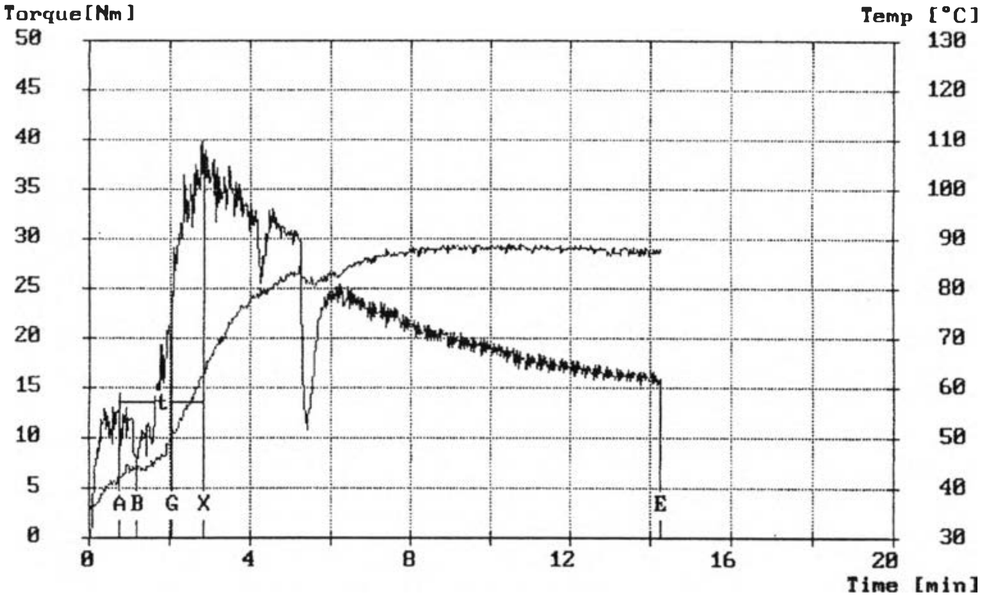

Figure B8 Torque-time-temperature relationship of NR/silica (Ultrasil-VN2) compound composition 80/20 in volume fraction

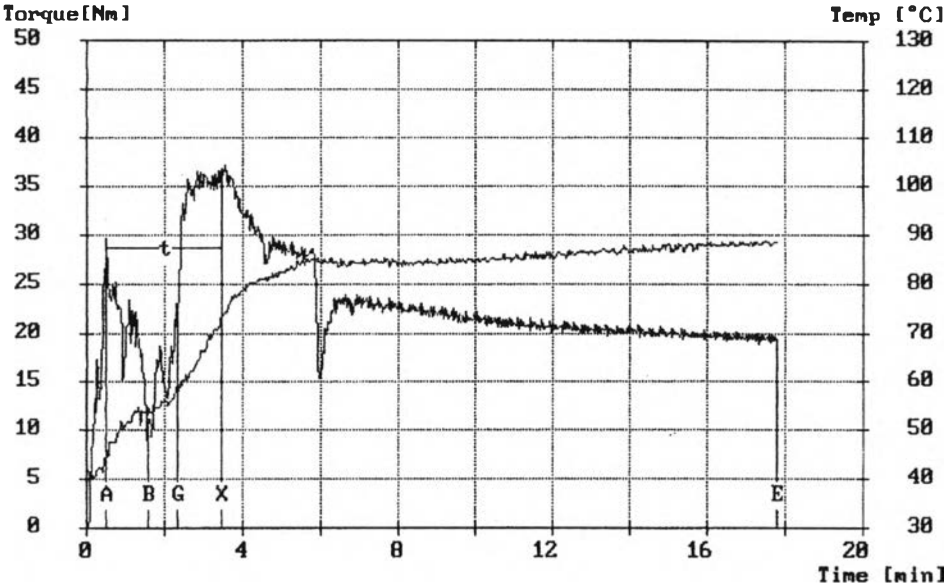

Figure B9 Torque-time-temperature relationship of NR/carbon black/gypsum compound composition 80/16/4 in volume fraction

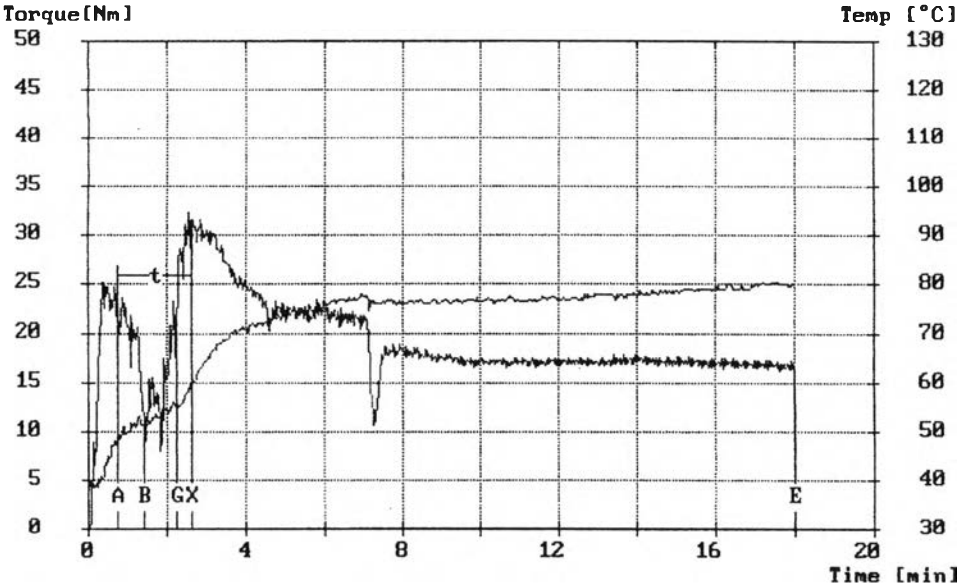

Figure B10 Torque-time-temperature relationship of NR/carbon black/gypsum compound composition 80/12/8 in volume fraction

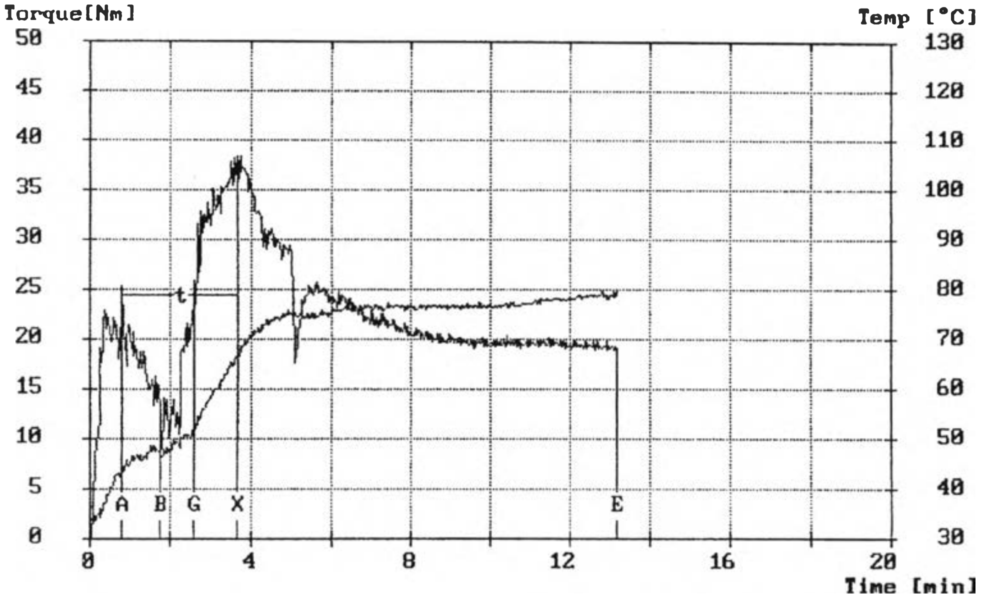

Figure B11 Torque-time-temperature relationship of NR/carbon black/gypsum/silica compound composition 80/16/2/2 in volume fraction

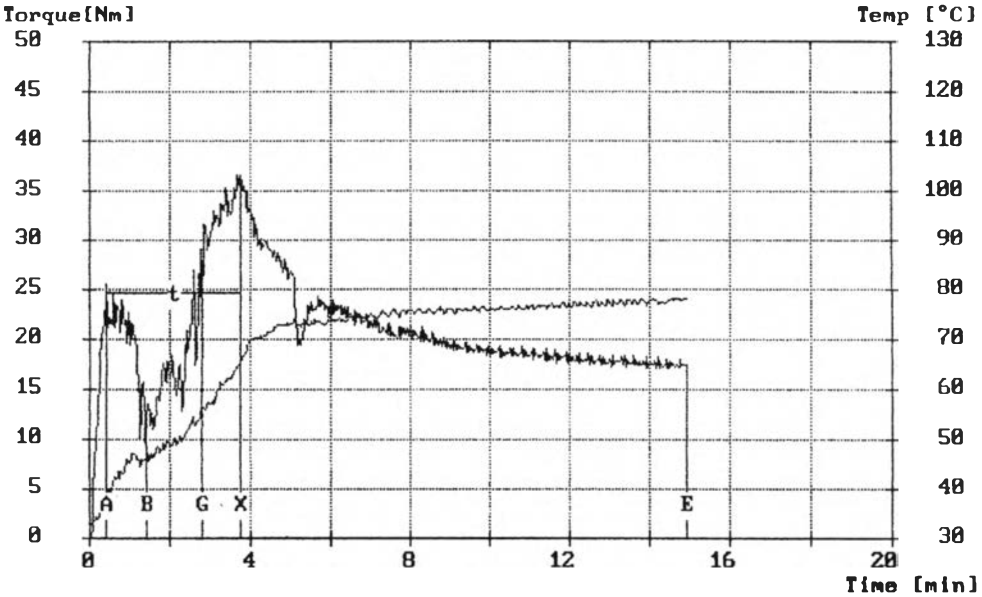

Figure B12 Torque-time-temperature relationship of NR/carbon black/gypsum/silica compound composition 80/12/4/4 in volume fraction

APPENDIX C

Calculation for Amount of Materials that used in Brabender Plasticorder

From $D = M/V$ (C.1)

Then $D_{total} = M_{total}/V_{total}$ (C.2)

And $M_{total} = (M_x+M_y+M_z+\ldots)$ (C.3)

$V_{total} = [(M_x/D_x)+(M_y/D_y)+(M_z/D_z)+\ldots]$ (C.4)

where D = density of material (g/cm^3)

M = weight of material (g)

V = volume of material (cm^3)

<u>Example of calculation</u>

For carbon black filled-NR compound consists of materials as shown below:

NR has density 0.9 g/cm^3 with loading 100 part

Carbon black has density 1.8 g/cm^3 with loading 50 part

Zinc oxide has density 5.67 g/cm^3 with loading 5 part

Stearic acid has density 0.94 g/cm^3 with loading 2 part

MBTS has density 1.54 g/cm^3 with loading 1.8 part

Sulfur has density 2.08 g/cm^3 with loading 3 part

From

$$D_{total} = M_{total}/V_{total}$$

$$D_{total} = \frac{[100+50+5+2+1.8+3]}{[(100/0.9)+(50/1.8)+(5/5.67)+(2/0.94)+(1.8/1.54)+(3/2.08)]}$$

$$= 1.1196 \text{ g/cm}^3$$

Due to a chamber of Brabender Plasticorder has volume 80 cm^3 and to achieve a good mixing the materials should be filled in 80 % of chamber volume that is

$$(80x80)/100 = 64\ cm^3$$

Then weight of materials that wanted in blending is

$$1.1196x64 = 71.6544\ g$$

Materials have total amount at 161.8 part = 71.6544 g

Then NR has 100 part = (100x71.6544)/161.8
= 44.2858 g

Carbon black has 50 part = (50x71.6544)/161.8
= 22.1429 g

ZnO has 5 part = (5x71.6544)/161.8
= 2.2143 g

Stearic acid has 2 part = (2x71.6544)/161.8
= 0.8857 g

MBTS has 1.8 part = (1.8x71.6544)/161.8
= 0.7971 g

Sulfur has 3 part = (3x71.6544)/161.8
= 1.3286 g

Volume Fraction

In this thesis, the volume fraction is calculated only from the volume of NR and the volume of each filler or is not including the volume of the other additives such as zinc oxide, stearic acid, MBTS and sulfur.

From $V_x = v_x / v_{total}$

where V_x = the volume fraction of material x

v_x = (m_x/ρ_x) = the volume of material x (cm^3)

v_{total} = $v_x + v_y + v_z + \ldots$

Example of calculation

For carbon black-filled NR compound consists of materials as shown below:

NR has density 0.9 g/cm^3 with loading 100 part

Carbon black has density 1.8 g/cm^3 with loading 50 part

$$V_{\text{carbon black}} = v_{\text{carbon black}} / v_{\text{total}}$$

$$= [(50/1.8) / ((50/1.8)+(100/0.9))]$$

$$= 0.2$$

$$V_{\text{NR}} = v_{\text{carbon black}} / v_{\text{total}}$$

$$= [(100/0.9) / ((50/1.8)+(100/0.9))]$$

$$= 0.8$$

APPENDIX D

Mechanical Properties Data of Pure and Filled NR Compounds

Each testing was done repeatedly. The average values of all tests are obtained from 3 runs.

Table D1 Tensile strength data of rubber compounds.

Rubber compound	Tensile strength (MPa)
Mix 1	19.33 ± 1.43
Mix 2	20.59 ± 0.18
Mix 3	26.56 ± 0.38
Mix 4	27.27 ± 0.52
Mix 5	28.96 ± 0.44
Mix 6	13.36 ± 0.60
Mix 7	12.75 ± 0.34
Mix 8	11.79 ± 0.35
Mix 9	27.43 ± 0.37
Mix 10	26.02 ± 0.66
Mix 11	23.22 ± 0.28
Mix 12	20.09 ± 0.53

Table D2 Elongation at break data of rubber compounds.

Rubber compound	Elongation at break (%)
Mix 1	763.20 ± 16.07
Mix 2	616.66 ± 28.47
Mix 3	560.05 ± 36.78
Mix 4	544.47 ± 14.84
Mix 5	524.08 ± 29.13
Mix 6	550.70 ± 27.45
Mix 7	556.48 ± 23.49
Mix 8	576.66 ± 18.45
Mix 9	572.68 ± 27.07
Mix 10	584.65 ± 45.00
Mix 11	602.34 ± 14.03
Mix 12	623.84 ± 22.92

Table D3 300% Modulus data of rubber compounds.

Rubber compound	300% Modulus (MPa)
Mix 1	3.45 ± 0.02
Mix 2	4.17 ± 0.28
Mix 3	11.39 ± 1.40
Mix 4	15.27 ± 0.92
Mix 5	16.64 ± 0.96
Mix 6	5.00 ± 0.28
Mix 7	5.02 ± 0.23
Mix 8	4.23 ± 0.20
Mix 9	10.17 ± 0.97
Mix 10	8.20 ± 1.31
Mix 11	7.97 ± 0.13
Mix 12	5.54 ± 0.36

Table D4 Hardness data of rubber compounds.

Rubber compound	Hardness (Shore A)
Mix 1	39.42 ± 0.19
Mix 2	49.04 ± 0.59
Mix 3	69.20 ± 0.91
Mix 4	68.98 ± 1.75
Mix 5	70.72 ± 1.78
Mix 6	68.30 ± 3.30
Mix 7	69.52 ± 8.47
Mix 8	70.04 ± 1.90
Mix 9	63.04 ± 3.37
Mix 10	62.04 ± 1.95
Mix 11	57.44 ± 1.13
Mix 12	52.02 ± 0.69

Table D5 Resilience data of rubber compounds.

Rubber compound	Resilience (%)
Mix 1	80.5 ± 6.69
Mix 2	77 ± 2.37
Mix 3	47 ± 0.53
Mix 4	48.31 ± 1.79
Mix 5	47.89 ± 3.46
Mix 6	49.9 ± 1.46
Mix 7	48.1 ± 1.13
Mix 8	46.3 ± 0.32
Mix 9	47.9 ± 0.55
Mix 10	48.6± 0.04
Mix 11	49.04 ± 0.4
Mix 12	49.46 ± 0.54

Table D6 Abrasion loss data of rubber compounds.

Rubber compound	Abrasion loss (mm^3)
Mix 1	81.01 ± 6.69
Mix 2	73.13 ± 4.68
Mix 3	48.31 ± 5.58
Mix 4	43.6 ± 5.58
Mix 5	43.0 ± 5.53
Mix 6	65.91 ± 10.88
Mix 7	75.97 ± 5.48
Mix 8	84.03 ± 5.39
Mix 9	48.31 ± 5.58
Mix 10	57.97 ± 5.58
Mix 11	49.6 ± 5.73
Mix 12	70.07 ± 5.78

Table D7 Tan delta data of rubber compounds.

Rubber compound	G' (N/m^2)	G"(N/m^2)	Tan delta
Mix 5	$4.15\text{x}10^9 \pm 1.20\text{x}10^6$	$3.85\text{x}10^8 \pm 1.13\text{x}10^4$	0.0927 ± 0.0216
Mix 9	$5.39\text{x}10^9 \pm 4.38\text{x}10^6$	$3.70\text{x}10^8 \pm 3.04\text{x}10^4$	0.0687 ± 0.0095
Mix 10	$2.09\text{x}10^9 \pm 7.07\text{x}10^3$	$6.06\text{x}10^7 \pm 58.05$	0.0290 ± 0.0185
Mix 11	$2.21\text{x}10^9 \pm 1.14\text{x}10^4$	$1.99\text{x}10^8 \pm 1.41\text{x}10^3$	0.0899 ± 0.0182
Mix 12	$2.47\text{x}10^9 \pm 7.07\text{x}10^3$	$1.44\text{x}10^8 \pm 7.07\text{x}10^2$	0.0582 ± 0.0047

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