CHAPTER 2 :   EXPERIMENTAL

2.1   Materials

The materials used are :-

- Plastic wrapper of SDR 20 CV

- Plastic wrapper of SDR 10

- Plastic wrappper of SDR 10 CV

- Plastic wrapper of KOSYN 1502

- Plastic wrapper of KOSYN 1712

- Plastic wrapper of UBEPOL 150

- Plastic bag of reclaimed rubber

- Plastic bag of rubber crumbs

- Plastic bags of curative / vulcanizing agent

- Plastic bags of small tyre ingredients such as antidegradant, activator, softener, fillers

The materials are sampling from the mixing area of DMIB Sdn Bhd. on 2001-09-18.

SDR 20CV, SDR 10, SDR 10CV are natural rubber or natural polyisoprene. KOSYN 1502
and KOSYN 1712 are styrene butadiene rubber. Ubepol 150 is butadiene rubber. The curative
used is sulphur.

2.2   Instruments

All the intruments used are located in Sime Darby Technology Centre, PJ. They are, namely,

- Perkin Elmer Model 1600 FTIR Spectrophotometer

- Mettler Toledo Model DSC822e Differential Scanning Calorimeter

- Herzog ring and ball softening point test model MC753

2.3   Procedure

2.3.1   Identification of materials by FTIR-Transmission[12]

a.   Switch on the computer and the printer.

b.   When the windows is loaded, double click on the Spectrum 2.00 icon to load the Spectrum software.

c.   Remove sample, if there is any, from the sample compartment.

d.   Display the **Instrument** menu and choose Scan **Background**. The **Scan Background** dialogue will be displayed.

e.   Enter the filename and the sample description in the **Filename** and **Description** test boxes.

f.   Choose the background spectrum to be saved as a **Single-Beam spectrum**.

g.   Set the scan parameters and the scan parameters to be used are :

Range	:	Start 4000 cm^{-1} End 450 cm^{-1}
Number of scans	:	16
Resolution	:	4.0 cm^{-1}
Interval	:	2.0 cm^{-1}
Apodization	:	Strong

h.   Choose **OK**.

i.   A window showing the background spectrum will appear when the background scan is completed.

j.   Place the sample on the KBr disc holder.

k.   Display the **Instrument** menu, choose **Scan Sample** and the dialogue will be displayed**.**

l.   Enter the filename and the sample description in the **Filename** and **Description** test boxes.

m.   Choose the sample spectrum to be collected as **Ratio spectrum**.

n.   Set the scan parameters and the scan parameters to be used as follows :

Range	:	Start 4000 cm^{-1}	End 450 cm^{-1}
Number of scans	:	16	
Resolution	:	4.0 cm^{-1}	
Interval	:	2.0 cm^{-1}	
Apodization	:	Strong	

o.   Choose OK. The spectrum of the sample will appear on the computer screen when the scan is completed. The spectrum will be automatically saved into the computer once the scan is completed.

p.   The spectrum obtained can be customised by selecting the commands in the **View**, **Process** and **Setup** menus.

q.   The spectrum can be printed by choosing **Print** from the File menu.

2.3.2   Identification Of Materials By FTIR-ATR[12, 13]

a.   Switch on the computer and the printer.

b.   When the windows is loaded, double click on the Spectrum 2.00 icon to load the Spectrum software.

c.   Remove sample, if there is any, from the sample compartment.

d.   Choose a suitable crystal to be used. For the project, Germanium crystal was used.

e   Place the ATR accessory in the sample slide holder of the FTIR instrument.

Place the sample holder, containing the crystal but without the sample, in the **45°** position of the ATR accessory.

g. On the keys pad of the FTIR instrument, select **Shift** key followed by **Scan** key. The screen of the instrument is changed and select **Energy** from the screen. Upon doing this, an energy bar and the percentage of energy throughput will be displayed on the screen of the instrument.

h. Use thumb screw T1 to adjust the rotation of the flat mirror M1, thumb screw T2 to rotate the toroidal mirror M2 and thumb screw T3 to rotate the mirror M3 until maximum energy throughput to the instrument is obtained.

i. When the maximum energy throughput is obtained, select **Stop** from the instrument screen.

j. Display the **Instrument** menu and choose **Scan Background**. The **Scan Background** dialogue will be displayed.

k. Enter the filename and the sample description in the **Filename** and **Description** test boxes.

l. Choose the background spectrum to be saved as a Single beam spectrum

m. Set the scan parameters.

Range	:	Start 4000 cm⁻¹	End 450 cm⁻¹
Number of scans	:	16	
Resolution	:	4.0 cm⁻¹	
Interval	:	2.0 cm⁻¹	
Apodization	:	Strong	

n. Choose OK.

o. Remove the sample holder from the ATR accessory.

p. Loosen the nut hand screw on the sample holder so that you can pull the two pressing blocks away from the crystal.

q. Mount the sample piece on both side of the crystal.

r. Place the pressure pads wrapped in non coated aluminium foil, between the sample and the pressing block.

s. Rotate the nut hand screw of the sample holder so that the pressing blocks and the pressure pads are just in contact with the crystal.

t. Place the sample holder in the ATR accessory and optimize the optical alignment by referring to g, h, i of section 2.3.2.

u. Proceed with k to q in section 2.3.1.

2.3.3 Thermal analysis of a material by using Differential Scanning Calorimeter[14]

a. Switch on Printer, PC, Screen, Liquid nitrogen valve and DSC 822e module.

b. Press Ctl-Alt-Delete to log on.

c. Log in to Window NT with username and password as follows :

> Username : dscau
>
> Passwored : CTLeq53

d. Double click the STAR Software icon on the desktop to start the Software.

e. Click **Function** in the main menu bar.

f. Click **Install** for testing option

g. Click on the button at the lowest row of the window labelled as DSC 822e/700/090/41491

h. In method window, click **select** and **new**.

i.   Perform Indium check by selecting "In check exo"method. Take an Indium tablet and record down the mass of Indium.

j.   Click **send experiment** button.

k.   Click **show buffer** to see experiment buffer.

l.   Wait for the beeping sound or " wait for sample insert " statement to appear on the monitor.

m.   Remove furnace lid. Place the selected indium tablet in the left position (S) of the blue DSC sensor and ensure that the reference pan is on the right position (R).

n.   Press **OK** to start the experiment.

o.   If the heat flow and the temperature are within the specification, proceed with testing sample.

p.   Select a method to test sample. For the project, the sample was analysed from 50°C to 150°C at 10K/min at air atmosphere.

q   Make sample tablet for testing by using aluminium pans. Weigh the sample. For the project, the weight of sample is 10 to 13 mg.

r.   Enter sample name and sample size in the appropriate field.

s.   Repeat j to n in section 2.3.3.

t.   To interpret the data obtained, open evaluation window by clicking **Function** and **Evaluate** from the Star Software main menu bar.

u.   Click **File** and **Open Curve** in the menu bar. Select the file name and click **Open** to open the curve.

v.   Make the desired setting and define the thermal effect (melting point) by making a frame on the curve.

2.3.4 Softening point determination by using the automatic ring and ball tester MC 754[15, 16, 17]

a. Clamp the sample with two rings.

b. Place the rings that contain the samples into the ring holder of the test support.

c. Place the magnetic stirrer and the test insert support into 600ml beaker. Pour glycerol into the beaker.

d. Place the steel balls in glycerol.

e. Condition the beaker with glycerol at $30 \pm 1°C$ for 15 min.

f. With a pair of forceps, place the steel balls in the centre of the upper surface of the material in the ring.

g. Switch on the test apparatus

h. Place the beaker with glycerol, the magnetic stirrer and the test insert with the sample to be tested, on the heater of the test unit.

i. Place the resistance thermometer in the test insert.

j. Select the **ANALYSER** key, then press key 1.

k. Press the **W/G** key until "**G**" is appeared on the display of apparatus.

l. Press the **START** key to activate the analyser unit.

m. The test is immediately terminated after the two balls have fallen through the rings and come into contact with the bottom plate. At that moment, a "pi" sound is heard. Press the orange button in the test unit to stop the siren. Record the softening points indicated in the display.