EXPERIMENTAL TECHNIQUES

The materials used and the experimental procedures adopted in the present investigations are given in this chapter.

2.1 Materials Used

2.1.1 Elastomers

a) Natural rubber (NR)

ISNR-5 was supplied by the Rubber Research Institute of India, Kottayam and had a Mooney viscosity [ML (1+4), at 100°C] of 82.

b) Acrylonitrile-butadiene rubber (NBR)

Acrylonitrile-butadiene rubber was obtained from Apar Polymers Ltd., India. Grade N553 with 33% acrylonitrile content and Mooney viscosity [ML (1+4), at 100°C] of 45.
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c) Styrene-butadiene rubber (SBR)

Styrene-butadiene rubber (SBR, 1502) was obtained from Korea Kumho Co. Ltd. and had a Mooney viscosity [ML (1+4), at 100°C] of 45.

d) Isobutylene-isoprene rubber (IIR).

Isobutene - Isoprene rubber (IIR, Exon: 301) was supplied by polysar, Canada and had a Mooney viscosity [ML (1+4), at 100°C] of 50.

e) Chloroprene rubber (CR)

Chloroprene rubber (CR) was supplied by Distugil, Paris and had a Mooney viscosity [ML (1+4), at 100°C] of 45.

f) Polybutadiene (BR)

Polybutadiene rubber was obtained from Indian Petrochemicals Corporation Ltd, Baroda. The grade used was cisamer 1220 having a Mooney Viscosity [ML (1+4) at 100°C] of 45.

g) Natural rubber latex

Natural rubber latex (centrifuged) was obtained from Rubber Research Institute of India, Kottayam conforming to the specifications of the Bureau of Indian Standards (BIS 5430 -1981).

2.1.2 Antioxidants

a) Pilflex-13

Commercial antioxidant pilflex-13 [N-(1,3-dimethyl butyl) N'- phenyl-p- phenylenediamine], was obtained from Polyolefin Industries Ltd., India.

b) Accinox TQ

Polymerised 1,2 dihydro 2,2,4-trimethyl quinoline was supplied by ICI India Ltd. West Bengal, Melting point 78-90°C.
c) **Vulkanox 4020**

Vulkanox 4020 [N-(1,3-dimethyl butyl)-N\(^1\)-phenyl- p-phenylenediamine], was obtained from Bayer India Ltd.

d) **Accinox DN**

Phenyl- \(\beta\)-napthylamine was supplied by ICI India Ltd. West Bengal. It had a melting point of 105°C.

e) **Vulkanox SP**

Commercial phenolic type antioxidant vulkanox SP (Mixture of styrenated phenols) was obtained from Bayer India Ltd.

f) **Phenol**

S.D. Fine Chemicals, India, supplied phenol. It had a melting point of 41°C.

g) **2,6 di-t-butyl-4-methyl phenol**

2,6 di-t-butyl-4-methyl phenol was supplied by BDH laboratories India Ltd.

h) **Diphenylamine**

Diphenylamine was supplied by BDH Laboratories India Ltd. The melting point of the sample was 54°C.

i) **Paraphenylenediamine**

Paraphenylenediamine used in the study was supplied by CDH Laboratories Ltd., India and a melting point of 141°C.

**2.1.3 Catalysts**

a) **Anhydrous zinc chloride (An.ZnCl\(_2\))**

Anhydrous zinc chloride was obtained from Ranbaxy Laboratories. It had a melting point of 290°C.
be) *Anhydrous aluminium chloride (An.AlCl₃)*

Anhydrous aluminium chloride used was white powder obtained from Qualigens Fine Chemicals, India.

2.1.4 Fillers

a) *High abrasion furnace black (HAF, N330)*

High abrasion furnace black (HAF, N 330) used in these experiments was supplied by M/s. Philips Carbon, Kochi (India) Ltd. It had the following specifications:

- DBP absorption: 102 ± 5 cc/100g
- Iodine number: 82

b) *Precipitated Silica*

Precipitated silica used in this study was GSL-150 Grade obtained from Gujarat.

2.1.5 Compounding Additives

a) *Zinc oxide*

Zinc oxide (ZnO) was supplied by M/s. Meta Zinc Ltd. Mumbai. It had the following specifications: zinc oxide content - 98%, acidity-0.4% max., heat loss (2h at 100°C) - 0.5% max.

b) *Stearic Acid*

Stearic acid used in the study was supplied by Godrej Soaps (Pvt.) Ltd., Mumbai and had the following specifications:

- Melting point-50-69°C, acid number-185-210, iodine number-9.5 max., specific gravity 0.85 ± 0.01, ash - 0.1 % mass.

c) *Mercaptobenzthiazole (MBT)*

Mercaptobenzthiazole having the following specifications was supplied by Bayer Chemicals, Mumbai with melting point - 160-180°C.
d) Dibenzthiazyl Disulphide (MBTS)

Dibenzthiazyl disulphide was supplied by Bayer Chemicals, Mumbai. It had a melting point of 165°C.

e) Tetramethyl Thiuram Disulphide (TMTD)

Tetramethyl thiuram disulphide used was supplied by polyolefins industries Ltd., Mumbai. It had a melting point of 136°C.

f) Benzthiazyl 2- Sulphenmorpholide (MOZ)

Benzthiazyl 2- sulphenmorpholide (MOZ) was supplied by ICI Ltd., India.

g) Aromatic Oil

Aromatic oil was supplied by Hindustan Petroleum Corporation. It had the following specifications: Specific gravity 0.95-0.98, viscosity gravity constant (VGC) -0.907, aniline point 38°C.

h) Napthenic Oil

It was supplied by M/s. Hindustan Petroleum Ltd. India. It had the following specifications:

Viscosity gravity constant (VGC) - 0.85 - 0.9
Aniline point - 75°C

i) Paraffinic Oil

M/s. Hindustan Petroleum Ltd., India, supplied paraffinic oil. It had the following specifications:

Viscosity gravity constant (VGC) - 0.85
Aniline point - 96°C.

j) Dioctyl Phthalate (DOP)

Dioctyl Phthalate used was commercial grade supplied by Rubo - Synth Impex Pvt. Ltd., Mumbai. The viscosity of the sample was 60 mPa.s.
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**k) Sulphur**

Sulphur was supplied by Standard Chemical Company (Pvt.) Ltd., Madras, and had the following specifications: acidity 0.01 % max., ash 0.01 % max., solubility in CS₂ - 98 % max.

**l) PF resin**

Phenol formaldehyde resin supplied by Bakelite Hylam, Hyderabad. The used grade was 6417 having the following specifications:

- Softening point 88-102°C
- Methylol group 14-16 %

### 2.1.6 Other Additives

**a) Bitumen**

Bitumen with penetration 80/100 was supplied by Cochin Refineries Ltd., Ambalamugal.

**b) Chlorinated paraffin wax (CPW)**

Chlorinated paraffin wax was supplied by KLJ Organic Ltd., Gujarat, India.

- Sp. gravity 1.280
- Chlorine content 51.5%
- Viscosity 159 mPa.s

**c) Polyethylene glycol - 600**

Polyethylene glycol - 600 with molecular weight range 570-630 was supplied BDH Laboratories India Ltd.

- Specific gravity 1.128
- Viscosity 105 mPa.s

**d) Polyisobutene (PIB-24)**

Polyisobutylene(PIB) with molecular weight 934 was supplied by Cochin Refineries, Balmer Lawrie Ltd., Ambalamugal, Kerala.
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Viscosity 248 mPa.s
Molecular weight 930

Sodium carbonate, sodium hydroxide, sodium thiocyanate, sodium thiosulphate, sodium peroxide, potassium carbonate, potassium chromate, potassium iodide, potassium dichromate, starch, silver nitrate, dil. nitric acid, ferric alum, fused calcium chloride, thionyl chloride used were analytical grade.

2.1.7 Solvents

Dioxan, toluene, carbon tetrachloride, methanol, tri ethyl amine, carbon disulphide acetone, iodine monochloride, aniline, 2-butanolone were of analytical grade.

2.2 Experimental Methods

2.2.1 Characterisation methods

a) Infrared spectroscopy (IR)

IR spectra were taken on Perkin - Elmer Model 377 IR spectrometer.

b) Proton magnetic resonance spectroscopy (\(^1\)H NMR)

The \(^1\)H-NMR measurements were carried out using a JEOL-JNM spectrometer and the spectra were measured at 20°C using 10% solution in CDCl\(_3\) with tetramethylsilane as the internal standard.

c) Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was carried out using Shimadzu and Du pont (1080) TG-DSC standard model at a heating rate of 10°C per minute.

d) Thin layer chromatography (TLC)

Thin layer chromatography was done using silica gel as the adsorbent (supplied by E.Merck). Eluent - detector system was selected according to the characteristics of the particular product.
Chapter 2

e) Optical microscopic analysis

Carl Zeiss optical microscopic model Stemi 2000C with 2.5 magnification was used to take the photographs of the ozone cracked samples.

f) Vapour phase osmometry (VPO)

Molecular weights of the samples were determined by VPO method using Knauer Vapour Phase Osmometer. The principle is the Raoult's law i.e. the lowering of the vapour pressure of a solvent by a solute is proportional to the mole fraction of the solute.

g) Determination of Iodine Value

The iodine value is determined as follows 0.2g of sample was dissolved in 25 ml of CCl₄ in an iodine flask. 20 ml of 0.1N solution of iodiinemomonochloride in glacial acetic acid was added. It was stoppered and allowed to stand in darkness for 30 minutes. After that 100 ml 10% potassium iodide solution was added. The resultant 2 phase solution was titrated against thiosulphate using starch as indicator. A blank was run separately. Iodine value was calculated as,

\[
\text{Iodine value} = \frac{(V_1 - V_2) \times 12.69 \times N}{m}
\]

Where \( V_1 \) and \( V_2 \) - the volumes (ml) of 0.1N sodium thiosulphate used for titration of the sample and the blank respectively, \( N \)- normality of sodium thiosulphate and \( m \)- weight of the polymer sample in g.

h) Determination of chlorine content

A mixture of Na₂CO₃, K₂CO₃ and Na₂O₂ in the ratio 1: 1: 0.2 was prepared. About 5g of above mixture was used to cover the sample taken in a platinum crucible. Weight of the sample taken was in the range 0.1-0.3 g. The platinum crucible was kept in a muffle furnace at 900°C for 4h.

After 4h the crucible was taken out, cooled and placed in a beaker and 150ml water added. The contents were heated until the fusion mixture dissolves in water. Added conc. HNO₃ drop wise to neutralise the solution. Then added excess and boiled off. 35ml of AgNO₃ (0.1N)solution was added and the precipitate was filtered off. The filtrate was titrated with 0.1 N NaSCN. AgNO₃ was standardised.
with NaCl using K$_2$CrO$_4$ indicator. Similarly a blank was also done. Chlorine content was calculated using the following equation.

\[
\text{Chlorine content} = \frac{(\text{Blank} - \text{sample volume}) \times 35.5 \times 100}{\text{W} \times 1000}
\]

\(N\) - Normality of NaSCN

\(W\) - Weight of the sample.

i) **Determination of chemical crosslink density**

The chemical crosslinks was estimated from the equilibrium swelling data. Samples of approximately 1cm diameter, 0.20 cm thickness were punched out from the central portion of the vulcanizate, accurately weighed and allowed to swell in excess solvent. In the vulcanizates 'without antioxidant' swelling is done in the solvent containing 0.1% accinox DN. The swollen samples were taken out of the solvent after 24h and weighed. Samples were dried in vacuum oven and samples weighed again.

The volume fraction of rubber \((V_r)\) in the swollen network was then calculated by the method reported by Ellis and Welding from the following equation:

\[
V_r = \frac{(D - FT) \rho_r^{-1}}{(D - FT) \rho_r^{-1} + A_s \rho_s^{-1}}
\]

where,

\(T\) - weight of the specimen

\(D\) - Deswollen weight of the specimen

\(F\) - weight fraction of insoluble components

\(A_s\) - weight of the absorbed solvent corrected for the swelling increment

\(\rho_r\) - density of rubber

\(\rho_s\) - density of solvent.

The value for \(\rho_r\) and \(\rho_s\) taken were,

\(\rho_r, NR\) - 0.921 g/cm$^3$

\(\rho_r, SBR\) - 0.94 g/cm$^3$

\(\rho_r, NBR\) - 1.0118 g/cm$^3$

\(\rho_r, IIR\) - 0.917 g/cm$^3$
\[ \rho_{BR} = 0.93 \text{ g/cm}^3 \]
\[ \rho_{Toluene} = 0.886 \text{ g/cm}^3 \]
\[ \rho_{Methyl ethyl ketone} = 0.804 \text{ g/cm}^3 \]

In the case of vulcanizates containing HAF black the value of \( V_r \) obtained as above, was converted into \( V_o' \), by means of the following equation, which was derived by Porter.\(^3\)

\[
\frac{V_o'}{V_r} = 0.56 \times e^z + 0.44
\]

where \( z \) - weight fraction of filler.

The crosslink density \( \frac{1}{2M_c} \) was determined from \( V_m \) using the Flory - Rehner Equation\(^8\)

\[
\frac{1}{2M_c} = -\ln \left(1 - \frac{V_m}{V_o}\right) + \frac{\chi V_m^2}{\rho_s (V_m)^{\nu_3}}
\]

where,

\[
V_s \quad - \text{molar volume of solvent}
\]

\[
V_s (\text{toluene}) = 106.2 \text{ cc/mol}
\]

\[
V_s (\text{methyl ethyl ketone}) = 89.5 \text{ cc/mol}
\]

and \( \chi \) - parameter characteristic of interaction between rubber and solvent. Values of parameter \( \chi \) taken for calculations were the following:

For NR - toluene - 0.39
For SBR - toluene - 0.32
For IIR - toluene - 0.56
For BR - toluene - 0.34
For NBR - Methyl ethyl ketone - 0.21

For NR/SBR and NR/SBR blends, \( \chi \) was found to vary linearly with composition.\(^6\)

Hence average value of \( \chi \) was taken for 70/30 blends of these polymers.
2.2.2 Mixing and homogenization using mixing mill

Mixing and homogenization of elastomers and compounding ingredients were done on a laboratory size (15 x 33 cm) two roll mill at a friction ratio of 1:1.25. The elastomer was given one pass through the nip (0.002 x 100)". Then it was given two passes through the nip (0.002 x 10)" and allowed to band at the nip of (0.002 x 55)". The temperature of the rolls was maintained at 70±5°C during mastication. After the nerve had disappeared, the compounding ingredients were added as per ASTM D 3184 (1980) and ASTM (1982) in the following order: activators, fillers, accelerators and curing agents. The batch was thoroughly cooled before the addition of accelerator and sulphur.

After complete mixing, the compound was homogenised by passing six times endwise through tight nip of 0.8mm and finally sheeted out at a nip gap of 3mm by passing the stock through the rolls four times folding it back on itself each time.

2.2.3 Determination of cure characteristics

The cure characteristics of the elastomers were determined using a Göttfert elastograph model 67.85. It is a microprocessor controlled rotorless cure meter with a quick temperature control mechanism and well defined homogeneous temperature distribution in the die or test chamber. In this instrument, a specimen of definite size is kept in the lower half of the cavity which is oscillated through a small deformation angle (±0.2). The frequency is 50 oscillations per minute. The torque is measured on the lower oscillating die half. A typical elastograph cure curve is shown in Fig.2.1 and the following data can be taken from the torque time curve.

![Torque-time curve](image)
(i) Minimum torque: Torque shown by the mix at the test temperature before the onset of cure.

(ii) Maximum torque: This is the maximum torque recorded.

(iii) Scorch time ($t_{10}$): This is the time taken for attaining 10% of the maximum torque.

(iv) Optimum cure time ($t_{90}$): This is the time taken for attaining 90% of the maximum torque.

The elastograph microprocessor evaluates the vulcanization curve and prints out these data after each measurement.

### 2.2.4 Moulding of test specimens

The test specimens were prepared in standard moulds by compression moulding on a single day light, electrically heated press having 30×30 cm platens at a pressure of 200 kg/cm$^2$ on the mould. The rubber compounds were vulcanised up to their respective optimum cure times at 150°C unless otherwise specified. Mouldings were cooled quickly in water at the end of the curing cycle and stored in a cold and dark place for 24h and were used for subsequent physical tests. For samples having thickness more than 6 mm (compression set, abrasion resistance etc.) additional curing time based on the sample thickness was given to obtain satisfactory mouldings.

### 2.2.5 Physical test methods

**a) Tensile stress-strain behaviour**

Tensile properties of elastomers were determined according to ASTM D 412-98 using dumbbell specimens on a Zwick universal testing machine model 1445. All the tests were carried out at 28°C. Samples were punched out from compression moulded sheets using a dumbbell die (C-type). The thickness of the narrow portion was measured by bench thickness gauge. The sample was held tight by the two grips, the upper of which was fixed. The rate of separation of the power actuated lower grip was fixed at 500 mm/min. for elastomeric specimens. The tensile strength, elongation at break and modulus were evaluated and printed out after each measurement by the microprocessor.
b) Tear resistance

This test was carried out as per ASTM D 624-98 using unnicked 90° angled test pieces. The samples were cut from the compression moulded sheets parallel to the mill grain direction. The test was carried out on Zwick universal testing machine. The speed of extension was 500 mm/min and the test temperature 28 ± 2°C.

c) Hardness

The hardness (shore A) of the moulded samples was tested using Zwick 3115 hardness tester in accordance with ASTM D 2240-97. The tests were performed on a mechanically unstressed sample of 300mm diameter and minimum 6mm thickness. A load of 12.5N was applied and the readings were taken after 10 seconds of indentation after firm contact has been established with the specimens.

d) Compression set

The samples (6.25mm thick and 18 mm diameter) in duplicate, compressed to constant deflection (25%) were kept for 22h in air oven at 70°C. After the heating period, the samples were taken out, cooled at room temperature for half an hour and the final thickness was measured. The compression set was calculated as follows:

\[
\text{Compression set} \% = \left( \frac{t_0 - t_1}{t_0 - t_s} \right) \times 100
\]

where \(t_0\) and \(t_1\) are the initial and final thickness of the specimen respectively and \(t_s\) the thickness of the spacer bar used. The procedure used was ASTM D 395-98 (method B).

e) Abrasion resistance

The abrasion resistance of the samples was tested using a DIN Abrader. Sample having a diameter of 12 ± 0.2 mm and a thickness of 16 to 20 mm, was kept on a rotating sample holder and 10N load was applied. Initially a pre run was given for the sample and its weight was taken. The weight after final run was also noted. The difference in weight is the weight loss of the test piece after its travel through 42 m in 132 seconds on a standard abrasive surface. It is expressed as volume loss(cc/h). The abrasion loss was calculated as follows:

\[
V = \frac{\Delta m}{\rho}
\]
where $\Delta m$ - mass loss, $\rho$ - density of the sample and $v$ - abrasion loss.

f) **Heat build-up**

The Goodrich flexometer conforming to ASTM D 623-99 (Method A) was used for measuring heat build-up. The test was carried out with the cylindrical sample of 2.5 cm in height and 1.9 cm in diameter. The oven temperature was kept at 50°C. The stroke was adjusted to 4.45mm and load 10.9kg. The sample was pre-conditioned to the oven temperature for 20 minutes. The heat developed was sensed by thermocouple and relayed to a digital temperature indicator. The temperature rise ($\Delta T°C$) at the end of 20 minutes was taken as heat build-up.

g) **Resilience**

The resilience was determined by vertical rebound conforming to ASTM D 2632-96 method. It is determined as the ratio of rebound height of a metal plunger of prescribed weight and shape which is allowed to fall on the rubber specimen. The test specimens of 12.5 ± 0.5 mm. thickness were molded. Level the instrument and raise the plunger to the top of the guide rod. Position the resilience scale so that its full weight rests upon the specimen. Lock it in this position and release the plunger making sure that it slides freely on its guide. Record the height after first three rebounds.

h) **Flex resistance**

The test is intended to compare the resistance of rubbers to the formation and growth of cracks and is recommended when flexing is encountered in service as in the case of a side wall. The apparatus used was a Wallace Demattia flexing machine as per ASTM D 430 - 57 T. Moulded test specimens with a circular groove, conditioned for 24 hours were tested in duplicate. The number of flexing cycles for crack initiation and for complete failure were noted on the counter. The test was conducted at ambient temperature.

i) **Ozone resistance**

Ozone ageing studies were conducted according to ASTM D 518 Method D in a Mast Model 700 - 1 ozone test chamber at 41°C. Ozone concentration in the chamber was adjusted to 50 parts per hundred million (pphm). Samples were
placed in the chamber and the time for crack initiation and total failure were noted by checking the samples using a magnifying lens at regular intervals of 30 minutes.

**j) Density**

The densities of the samples were estimated by the method of displacement by the method of displacement of liquid (ASTM D 792). In this method the weight of the specimen in air was first noted and then the specimen was immersed in a liquid and its loss of weight in liquid was determined. The density is given by,

\[
\text{Density} = \frac{\text{Weight of specimen in air}}{\text{Weight loss of specimen in liquid}} \times \text{Density of the liquid}
\]

**k) Solvent extraction studies**

Dumbbell samples for evaluation of physical properties were prepared and kept in different solvents at pre determined temperatures for specified periods. Retention in physical properties was measured after ageing at predetermined time and temperature.

**l) Ageing studies**

Dumbbell samples for evaluation of physical properties were prepared and kept in an air oven at pre determined temperatures for specified periods. Retention in physical properties like tensile strength, elongation at break, modulus etc. was evaluated after conditioning the samples at room temperature for 24h according to the procedure given in ASTM D 573.

**2.2.6 Determination of softening point (IP 58/82)**

The softening point is the temperature at which the substance acquires a particular degree of softening under specified conditions of the test.

The material is heated to a temperature 75 to 100°C above its expected softening point. It is stirred until it is completely changed into fluid and is free from air bubbles and water and filtered through IS sieve 30. The rings, previously heated to a temperature approximately to that of the molten material, were kept on a metal plate coated with the mixture of equal parts of glycerine and dextrose. The ring was then filled with sufficient melt to give an excess above the level of the ring when cooled. After cooling for 30 minutes in air, the material is levelled in the ring by removing the excess with a warmed sharp knife.
The apparatus is assembled with the rings, thermometer and ball guides in position. The bath was then filled with distilled water (for materials having softening point below 80°C) to a height of 50mm above the upper surface of the rings. The bath is then brought to a temperature of 5°C and maintained at that temperature for 15 minutes. Then the ball previously cooled to a temperature of 5°C is kept by means of forceps, in each ball guide. Now heat is applied to the liquid with stirring so that the temperature rises at a uniform rate of 5.0 ± 0.5°C per minute till the material softens and allows the ball to pass through the ring. For materials of softening point above 80°C glycerine is used in place of water in the bath and the starting temperature of the test is 35°C. For each ring and ball the temperature shown by the thermometer is recorded at the instant the sample surrounding the ball touches the bottom plate of the support. This temperature is reported as softening point.

2.2.7 Determination of penetration (IP 49/79)

The penetrometer of Precision Scientific Co. (USA) was used for the purpose. Penetration of a bituminous material is the distance in tenths of a millimetre that a standard needle will penetrate vertically into a sample of the material under standard conditions of temperature (25°C), load (100g) and time (5sec).

The penetration needle is a highly polished, cylindrical hard steel rod, coaxial, having the following shape, dimensions and tolerances as shown in the figure below:
The needle is provided with a shank about 3mm in diameter into which it is fixed immovably. The taper shall be symmetrical and the point shall be 'blunted' by grinding to a truncated cone.

**Preparation of sample**

The material was softened to a pouring consistency between 75°C and 100°C above the approximate softening point and stirred thoroughly till it was homogeneous and was free from air bubbles and water. The melt was then poured into the container to a depth 10mm in excess of the expected penetration. The sample was protected from dust and allowed to cool at a temperature between 15 to 30°C for 1 to 1.5 hours. It was then placed along with the transfer dish in the water bath at 25 ± 0.1°C and allowed to remain for another 1.5 hour. The sample was then placed on the base of the penetrometer and the needle point was kept in contact with the surface of the sample. The needle was then loaded with the weight required to make a total moving weight (i.e. the sum of the weights of the needle, carrier and superimposed weights) of 100 ± 0.25 g.
The pointer in the dial was brought to zero. Little water at 25°C was poured on the surface of the sample and released the needle for exactly five seconds. The distance in tenths of a millimetre vertically penetrated the sample is reported as the penetration. Two more determinations are made at points on the surface of the sample not less than 10mm apart and not less than 10mm from the side of the dish. The mean value of these three determinations is reported as penetration.

2.2.8 Determination of ductility (IP 32/55)

The ductility meter of Humboldt Manufacturing Company, (USA) was used for the purpose. The ductility of a bituminous material is the distance in centimetres to which it will elongate before breaking when ends of a briquette specimen of the material of the specified form is pulled apart at a specified speed and at a specified temperature. The test was conducted at a temp of 27 ± 0.5°C and at a rate of pull of 50 ± 2.5 mm per minute. The bituminous material was heated to a temperature about 75 to 100°C above the approximate softening point, till it became thoroughly fluid. It was then filtered through IS : sieve 30 and then poured to the mould of the type (total length = 75mm,distance between the clips = 30 mm, width at mouth of clip = 20mm and width at minimum cross section = 10mm) as shown above.
The mould was assembled on a brass plate and in order to prevent the material under test from sticking the surface of the plate as well as inferior surface of the sides of the mould were coated with the a mixture of equal parts of glycerine and dextrin. The material was poured as a thin stream back and forth from end to end of the mould until it was then allowed to cool at room temperature for 30 to 40 minutes and then placed in a water bath maintained at the specified temperature for 30 minutes. The excess bitumen was then cut off by means of a hot, straight - edged knife, so that the mould was just level full.

The brass plate and mould with briquette specimen was placed in the water bath at 27 ± 0.5°C for about 85 to 95 minutes. The briquette was then removed from the plate, the side pieces were detached and the rings at each end of the clips were attached to the pins in the testing machine. The two rings are then pulled apart horizontally at a uniform speed of 50 ±2.5 mm per minute until the specimen ruptures. The density of water in the bath was adjusted by the addition of sodium chloride so that the bituminous thread formed during the test does not touch the bottom of the bath at any time during the test. The distance is centimetres through which the standard briquette elongated before the bituminous thread formed breaks is reported as ductility.
2.2.9 Determination of flash point of bitumen (IP 34/85)

The flash point of a material is the lowest temperature at which the vapour of a substance momentarily takes fire in the form of a flash under the specified conditions of the test.

Pensky Martens Closed Method was used for the determination of the flash point of bitumen in this study. The flash cup was filled with the material to be tested up to the level indicated by the filling mark. The flash cup was then kept on the heater with the lid carrying the thermometer in position. The test flame was adjusted in such a way so that it is of the size of a bead of 4mm in diameter. The heating rate was adjusted in such a way so that it is of the size of a bead of 4mm in diameter. The heating rate was adjusted in such a way that the temperature recorded by the thermometer increases not less than 5°C and not more than 6°C per minute. The stirrer was turned at a rate of approximately 60 revolutions per minute. The test flame is applied at each temperature reading which is a multiple of 1°C up to 104°C. For the temperature range above 104°C the test flame is applied at each temperature reading which is a multiple of 3°C, the first application of the test flame being made at a temperature approximately 17°C below the actual flash point. The test flame is applied by operating the device controlling the shutter and test flame burner so that the test flame is lowered in 0.5°C seconds, left in its lowered position for one second and quickly raised to its high position. The stirring has to be discontinued during the application of the test flame. The duplicate results should not differ by more than 3°C of the mean.
Experimental Techniques

References


