CHAPTER 3
MATERIALS AND METHODS

This chapter deals with the details of various material and methods used through the research. With that this chapter also discussed the test methods used to determine the physical, rheological and performance properties of bitumen and crumb rubber modified bitumen. The physical test include penetration, softening point, elastic recovery, viscosity etc. Rheological properties deal with Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR) and Multiple Stress Creep Recovery (MSCR) tests. Performance properties is related to bitumen aggregate mixture, include Marshall Strength and Wheel Tracking tests.

3.1 MATERIALS

3.1.1 BITUMEN

Bitumen is a black viscous liquid, containing polycyclic hydrocarbons soluble in trichloroethylene, toluene etc. It is produced from crude petroleum oil by a refinery process, or found as a natural deposit.

3.1.1.1 BITUMEN COMPOSITION AND STRUCTURE

Whiteoak (1990)[63] and Airey (1997)[64] explained the four main chemical compositions of bitumen are: Saturates, Aromatics, Resins and Asphaltenes.

Resins are normally black or brown solid with high polarity having molecular weight range between 500 to 50,000. Due to the high polarity of the resins, it is very adhesive in nature. The resins portion of the bitumen play like a peptizing agent for the asphaltenes and work as stabilizers, which can keep everything unitedly in the bitumen.[65]

Asphaltenes are generally black or brown amorphous solids with highly polar having high molecular weight (between 1,000 and 100,000). There is a relation between molecular weight and size of the asphaltenes molecule i.e. higher the molecular weight, larger the molecules. The rheological properties of bitumen are strongly affected by asphaltenes content. As bitumen heated the gel structure (Figure 3.1, A-sol structure, B-gel structure) of the micelles are decomposed on heating and regenerated on cooling. The asphaltenes micelles may break down when it goes through a long-term heating and decrease the molecular weight. Moreover, asphaltenes content also determine the rigidity and stiffness of the bitumen.

Aromatics are mainly dark brown viscous liquid with low polarity and molecular weight range between 300 to 20,000. It plays like a dispersion medium for the asphaltenes and gives gum characteristics to the bitumen.

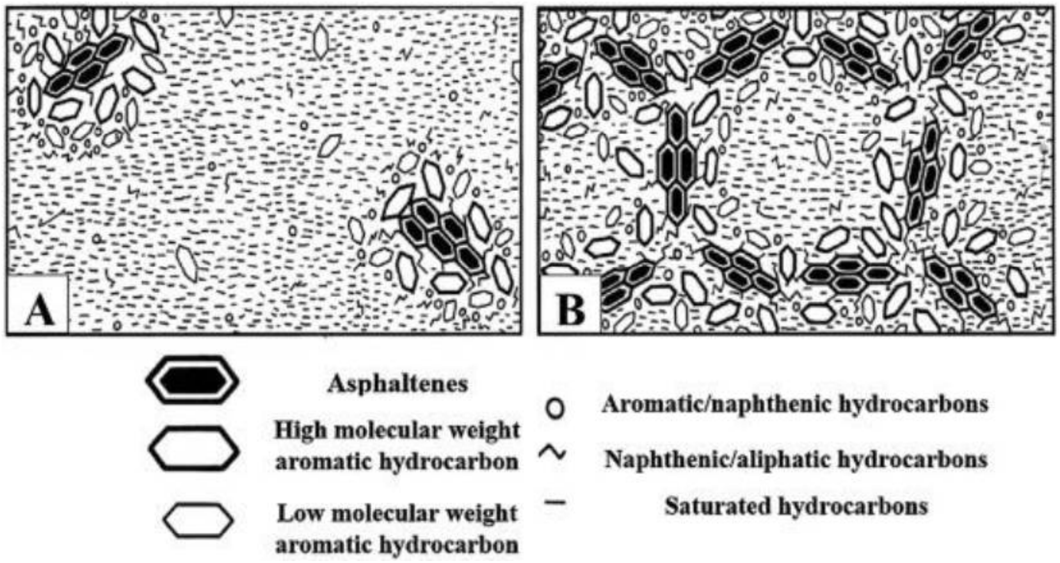


Figure 3.1: Sol-Gel structure of bitumen (Whiteoak, 1990)

Saturates are like oil which increases the liquid characteristics of the bitumen. They are generally straw or white in colour with lowest molecular weight (like aromatics). A representative structure of bitumen composition is given in Figure 3.2.

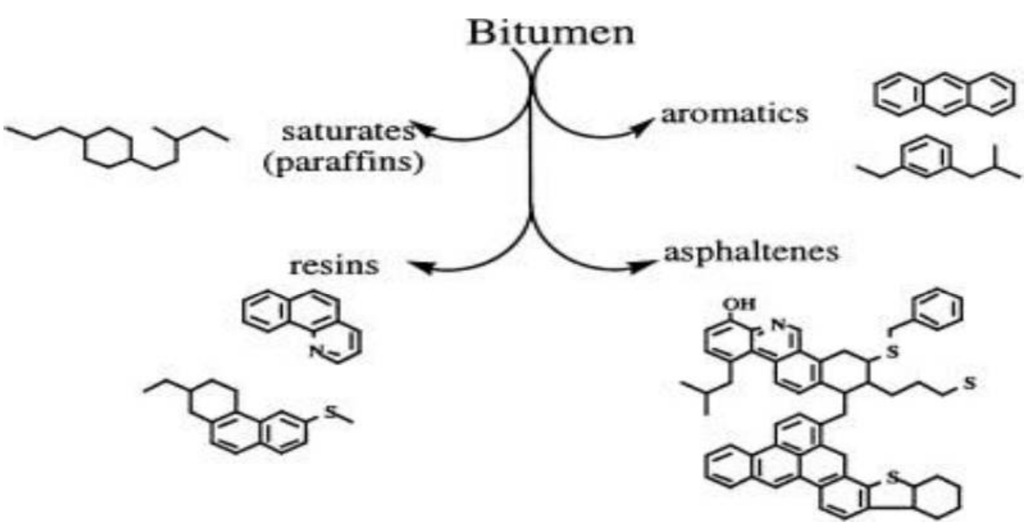


Figure 3.2: Representative structure of bitumen fractions (J F Masson et al., 2001)

3.1.1.2 PRODUCTION OF BITUMEN

The refining process separates the lighter fractions of bitumen from the residues. The production methods of bitumen depend on the crude source and processing methods available. The bitumen production scheme is given in (Figure 3.3). Bitumen is manufactured by using Distillation, Solvent de-asphalting, Oxidation and blending processes.

Distillation is the most common refining process for bitumen production in which straight reduction of crude oil occurred by using atmospheric and vacuum distillation (Figure 3.3). At starting the atmospheric distillation is generally used to separate lighter fractions of fuel and heavier component at the bottom. The heavier fraction left out in the kettle after distillation is known as an atmospheric residue. The atmospheric residue is entered to a vacuum distillation unit, where the lighter fraction is recovered without any significant thermal cracking of the molecules due to pressure reduction. Depending upon the specific grade required, the remaining residue i.e. short residue or vacuum residue can be used as such or further processed or blown further to the desired penetration or viscosity grade of bitumen.

Solvent de-asphalting process is used to separate the lubricant and bitumen components of crude without hampering their chemical structure by some specific solvents. Generally, propane or butane solvents are used to produce different categories of bitumen.

Oxidation process is followed by blowing air through bitumen at an elevated temperature (averagely 280 °C) to produce bitumen with desired physical properties for commercial purpose.

Blending process is the final method for bitumen production, where bitumen product can be produced as per required technical specification either directly using refining process or by blending bitumen with appropriate additives

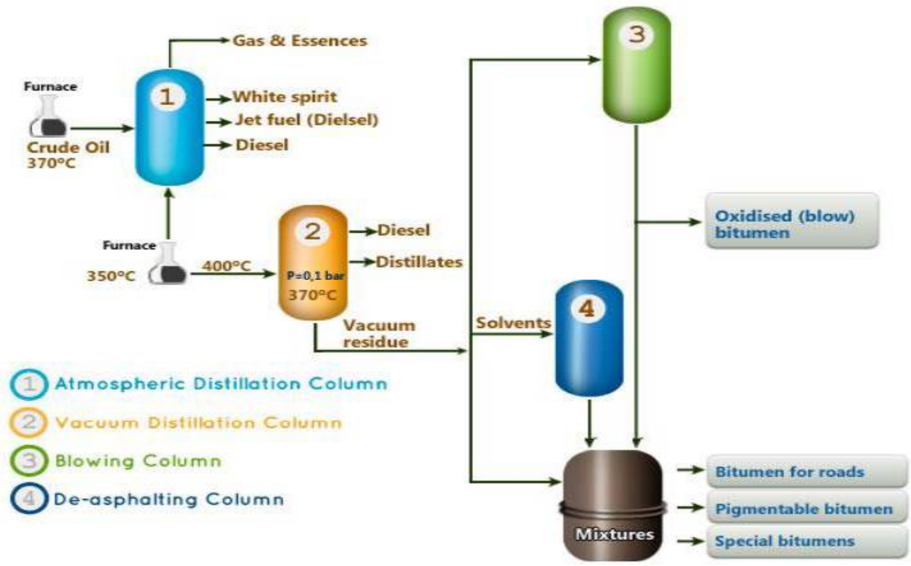


Figure 3.3: Typical Bitumen Production Scheme

3.1.2 CRUMB RUBBER

Crumb rubber is the term generally used for any material which is derived from scrap tires to uniform rubber granules by removing other contaminants. (Figure 3.4). The scrap rubbers can be reduced to a preferable size, by using a mechanical grinder. Crumb rubber can be blended with bitumen to improve the performance properties of the bitumen used in road pavement.

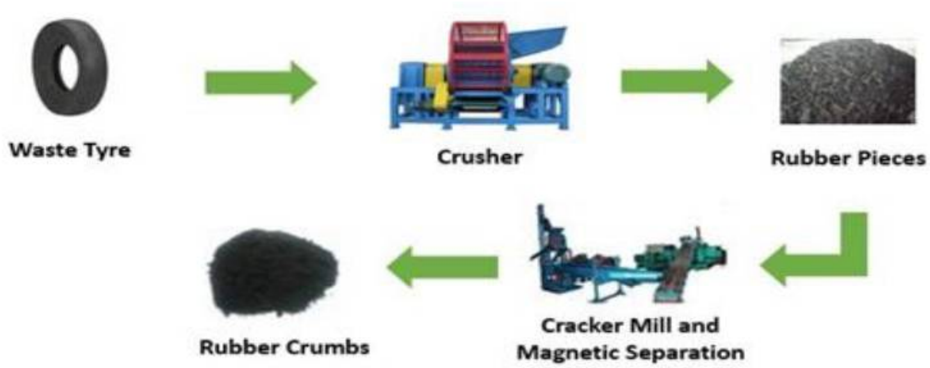


Figure 3.4: Crumb rubber production from waste tire

3.1.2.1 PROCESS FOR CRUMB RUBBER PRODUCTION

There are two main methods are used for production of crumb rubber like ambient and cryogenic process.

Ambient method is generally required to prepare preferably size, lacerate particles with large surface areas to upgrade the interaction with the paving bitumen (Figure 3.5). This mechanical grinding is executed by the help of rotating blades and knives, where the separation of the fibres is a critical step. It is the cost-effective and widely used method of processing end of life tyres.

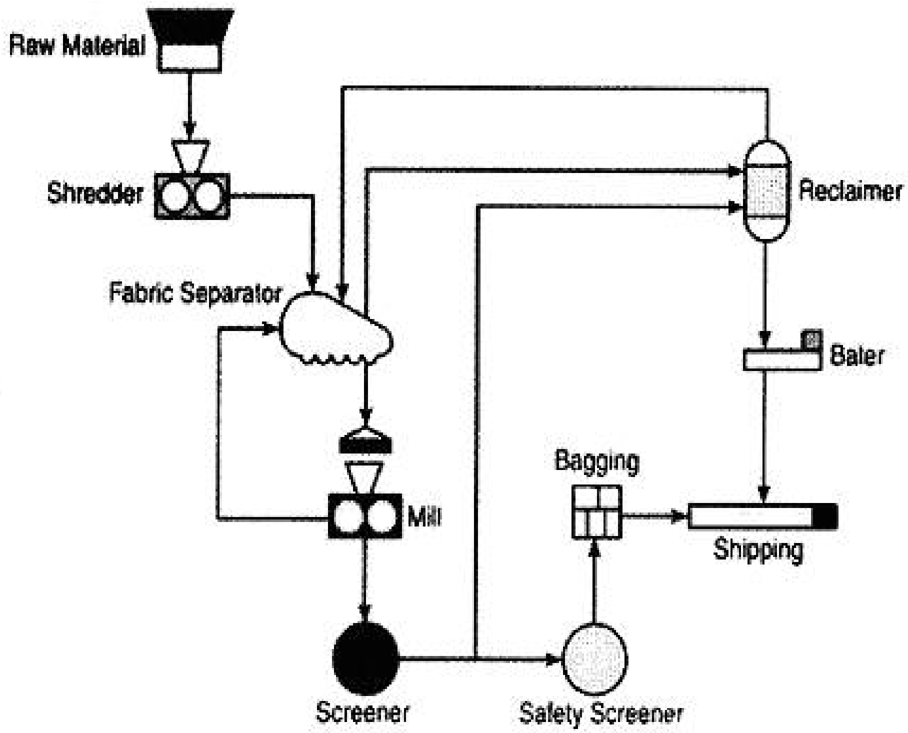


Figure 3.5: Schematic representation of ambient grinding[66]

Cryogenic method is the grinding process of the scrap tire at temperature between -87 to -198 ^{0}C using liquid nitrogen, where the rubber is grinded into smaller elements with relatively lower surface area than those obtained by ambient grinding (Figure 3.6).

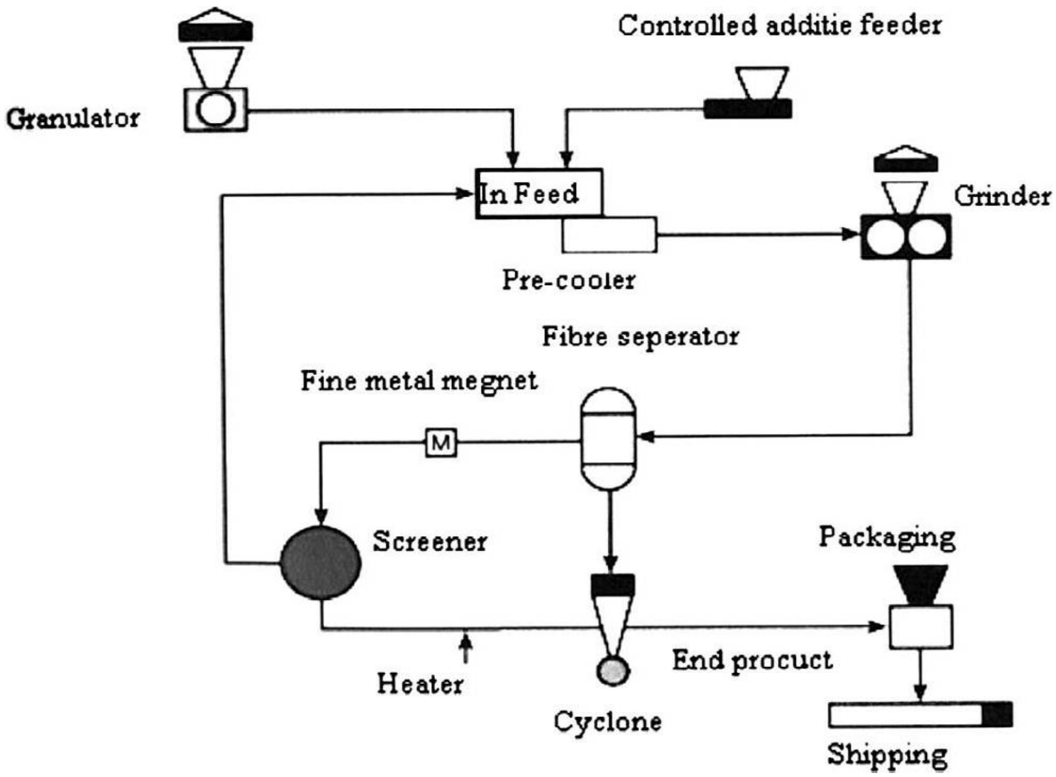


Figure 3.6: Schematic representation of cryogenic grinding[66]

3.2 TEST METHODS

Several test methods explored to evaluate the physical characteristics of bitumen and crumb rubber modified bitumen using many standard specifications such as European Standards, ASTM, AASHTO and Indian Standards. Crumb rubber modified bitumen is generally evaluated by various physical, rheological and performance related test methods as per the required paving bitumen specification.

Generally, bitumen is a mixture of hydrocarbons i.e. saturates, aromatics, resins and asphaltenes with about 300 - 2000 chemical components. Due to this complex structure of bitumen, it is very difficult to analyse the modified bitumen product using spectroscopic techniques. Therefore, in the present work we have used physical and rheological test method for the evaluation of modified bitumen blends. Following test method has been used to carry out physical, rheological and performance related evaluation of prepared CRMB blends

3.2.1 PHYSICAL TEST METHODS

3.2.1.1 PENETRATION

The consistency of the bitumen is measured using penetration test by the help of a penetrometer. In this research, we are using the test method which is described in ASTM D5.[67] Penetration may be defined, as the distance travelled by a standard needle with 100g weight in to a bitumen sample for 5 seconds. Generally, penetration is carried out at average service temperature (25 ^{0}C) for hot mix asphalt (HMA) pavements. Schematic diagram of penetration method is given in Figure 3.7.

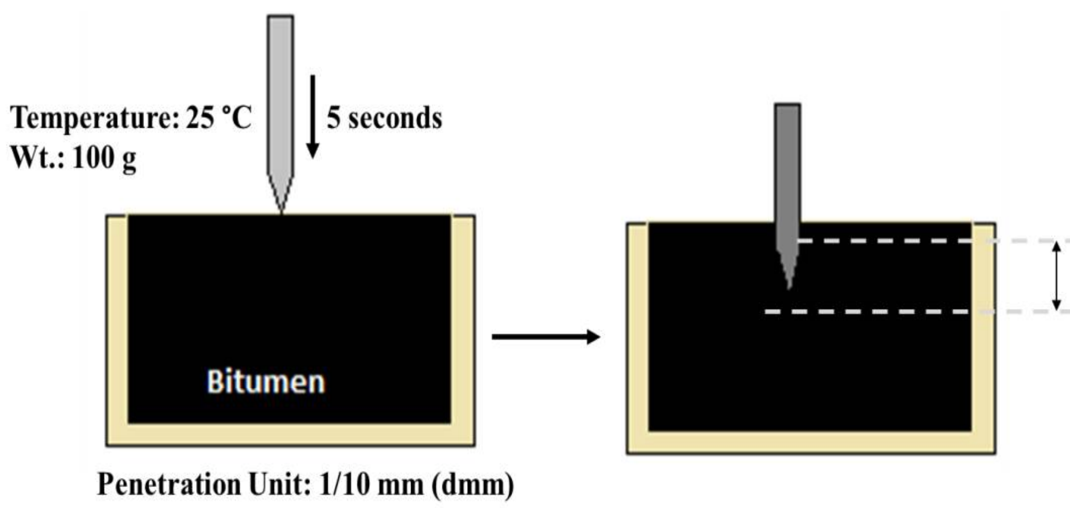


Figure 3.7: Schematic diagram of penetration method

3.2.1.2 SOFTENING POINT

The softening point (SP) test is also a physical test method used to predict the consistency of bitumen as per ASTM D36 method. Schematic diagram of softening point test is given in Figure 3.8.

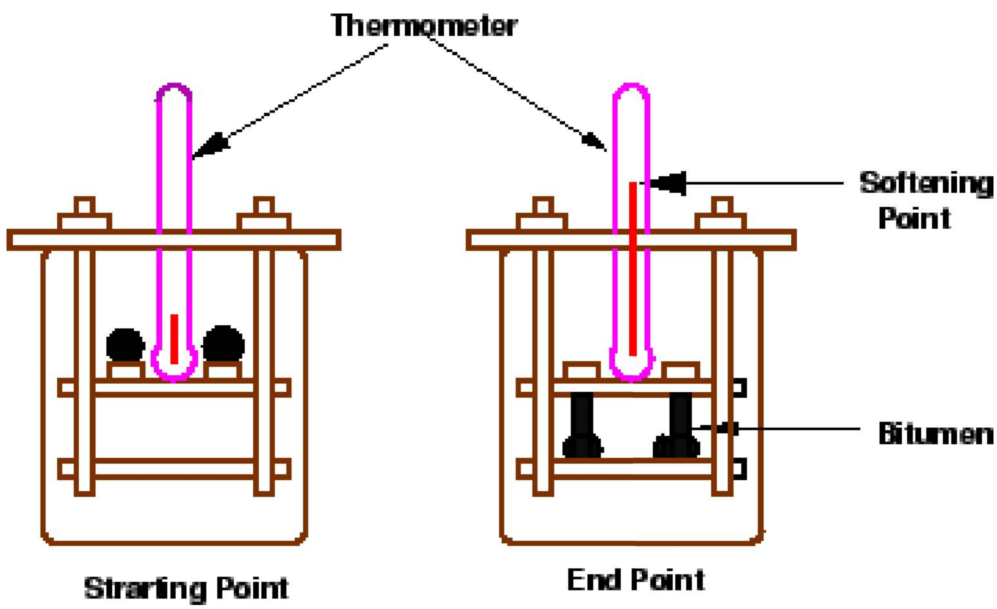


Figure 3.8: Schematic diagram of softening point test

In this test two steel balls are kept on two discs of bitumen in a water or glycerol bath at the starting point of this test. Then system is heated with a rate of heating 5 ^{0}C/min to increase the temperature of liquid. Generally, water is needed for bitumen with SP below or equal to 80 ^{0}C) and glycerol is required for bitumen with SP more than 80 ^{0}C. The temperature at which the bitumen softens much more to allow the balls kept in bitumen to fall 25 mm into the bottom plate is known as softening point of bitumen. Moreover, this test method evaluates a temperature where a phase transformation of bitumen occurred from semi-solid to liquid.

3.2.1.3 ELASTIC RECOVERY

The elastomeric characteristic of a material is measured by using this test method as per ASTM D6084-06. The elastic recovery of a bituminous material measured the recoverable strain produced after severing elongation of bitumen specimen. In this test, specimens are pulled to a 10-cm distance with a speed of 5 cm/min at a temperature of 15 °C in a Ductilometer apparatus.

3.2.1.4 VISCOSITY

The viscosity of bitumen binders is measured by Rotational Viscometer (RV) at high temperature as per Super pave performance grade (PG) bitumen binder specification. About 15-gram sample is taken in a cylindrical vessel and then a torque is applied to the spindle with constant speed at a temperature. This torque is converted in to viscosity by RV. In this research, the viscosity test of bitumen binder has been carried out at 150 °C as per ASTM D4402. Schematic diagram of rotational viscometer is given in Figure 3.9.

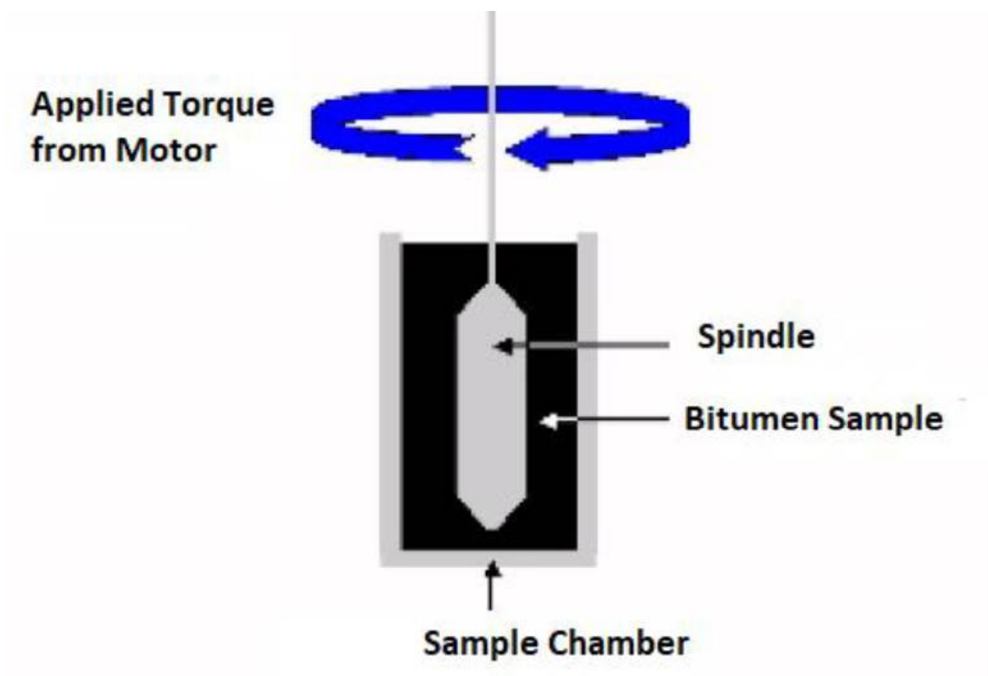


Figure 3.9: Schematic diagram of rotational viscometer

3.2)

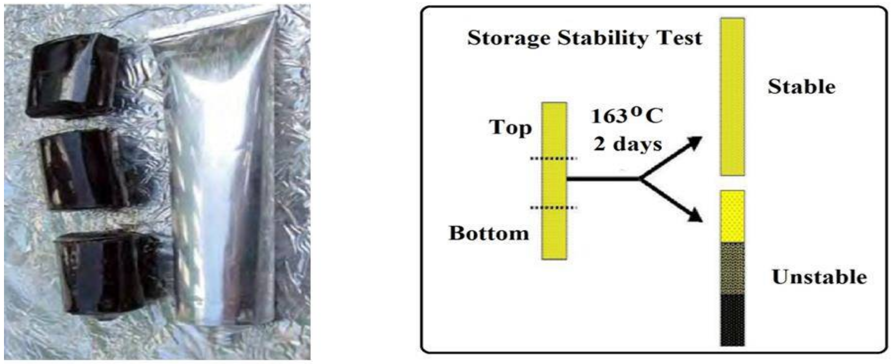
The test indicates approximate changes occur in bitumen during hot-mixing process of road construction. Test is carried out according to ASTM D2872. The test is conducted at temperature163 °C with air flow rate-4 L/min for 85 minutes. Around 35 g of sample taken in glass container heated in RTFO under this test conditions. Effect of heat and air is determined from the change occurred in the value of absolute viscosity of the residue. After RTFO test modified bitumen sample is subjected to DSR evaluation which determines high temperature rutting resistance. The tests were conducted in rolling thin film oven (Matest Company, Italy).

3.2.1.6 PRESSURE AGING VESSEL (PAV)

This test is used to predict the long term aged characteristics of bitumen binder as per AASHTO R 28. From this test, it can be known the aging properties of bitumen as it is exposed to heat and pressure to simulate in-service aging over a 7 to 10 years period. In this test, the RTFO aged bitumen binder samples are taken in stainless steel pans and then kept it in a heated vessel with pressure 2.10 MPa for 20 hours. After PAV test, modified bitumen sample is subjected to BBR evaluation studies which determines low temperature thermal cracking properties. Tests were carried out by using PAV-9300 system, Prentex Company, USA.

3.2.1.7 STORAGE STABILITY TEST

The Storage stability test is a method to check the separation of CRMB's under storage. It is characterized according to ASTM D7173 (Figure 3.10). In this test samples are put in aluminium tube and heated at 163 °C for 48 hours without disturbance. After that, sample was placed in the freezer for 4 hours to solidify CRMB. Then the tube was removed from freezer and cut into three equal parts by spatula and hammer. The softening points of top and bottom part of the sample were tested separately. Then the difference in softening points between top and bottom part of the tube were measured as separation value in °C.



(b)

(a)

Figure 3.10: Schematic diagram of storage stability test

According to IS 15462:2004, if the softening point differences between the top and the bottom part are less than 4 °C, then sample could be considered with good storage stability. Which represent lower the separation value between top and bottom portion, higher the storage stability of CRMB.

3.2.2RHEOLOGICAL TESTS

The study of deformation and flow behaviour of matter is known as rheology and is a vital characteristics to predict the behaviour of bitumen.The DSR and BBR are essential parts of the hot mix asphalt (bitumen) specification and are recognized as the most potentially valuable tools. For hot mix, DSR testing is done at the high pavement temperature, and BBR testing is done at a temperature to characterize the rheology at the low pavement temperatures.

3.2.2.1 DYNAMIC SHEAR RHEOMETER (DSR) TEST

Rheological properties were characterized with the help of dynamic shear rheometer (DSR, MCR102, Anton Paar Co. Ltd. of Austria). This test includes parallel plate configuration with a gap width of 1 mm and is used to determine the viscosity and elastic behaviour of a material at different temperatures for Performance Grade (PG Grade) specification of bitumen binder. In this test, a thin layer of bitumen sample is placed between two circular plates of DSR. The lower plate of DSR is fixed whereas the upper plate oscillates back and forth across the sample for the creation of a shearing action (Figure 3.11). DSR tests are carried out on unaged, RTFO aged and PAV aged bitumen binder samples. All the DSR testing was performed by using the method AASHTO T315-10. This method is used to evaluate complex shear modulus (G*) and phage angle (δ) at 10 rad/s which correlate the rut resistance of the sample. According to this method, the minimum requirement of rutting factor (G*/Sinδ) value is 1 kPa for unaged sample and 2.2 kPa for RTFO aged sample to pass at a particular temperature.

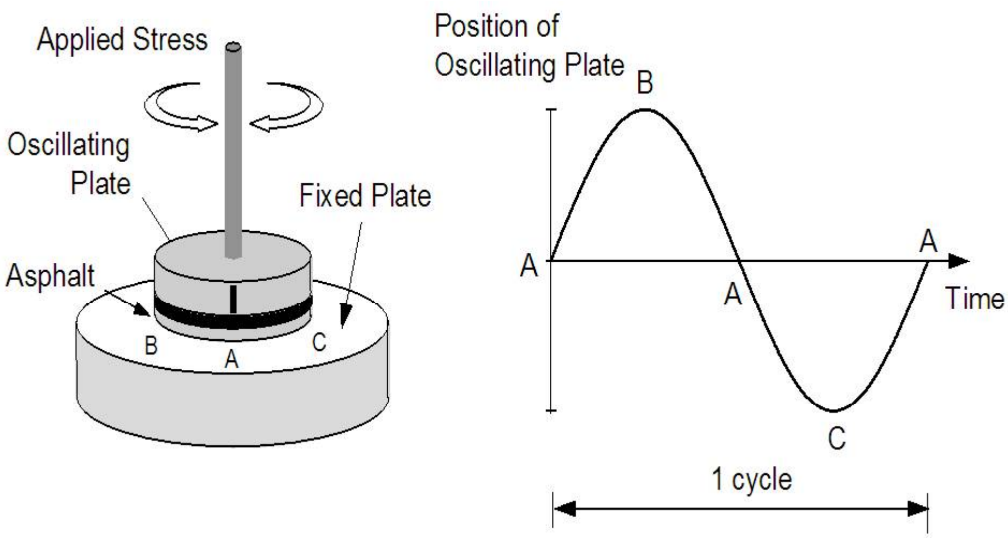


Figure 3.11: Schematic diagram of DSR

3.2.2.2 BENDING BEAM RHEOMETER (BBR) TEST

This test **is** used to determine low temperature creep stiffness of bitumen binder using BBR apparatus (Canon Instrument Company). In this test, 100 gm load is applied on PAV aged bitumen beam for 240 seconds and the creep stiffness (S) with rate of change of creep stiffness (m- value) are measured at a particular temperature (Figure 3.12).

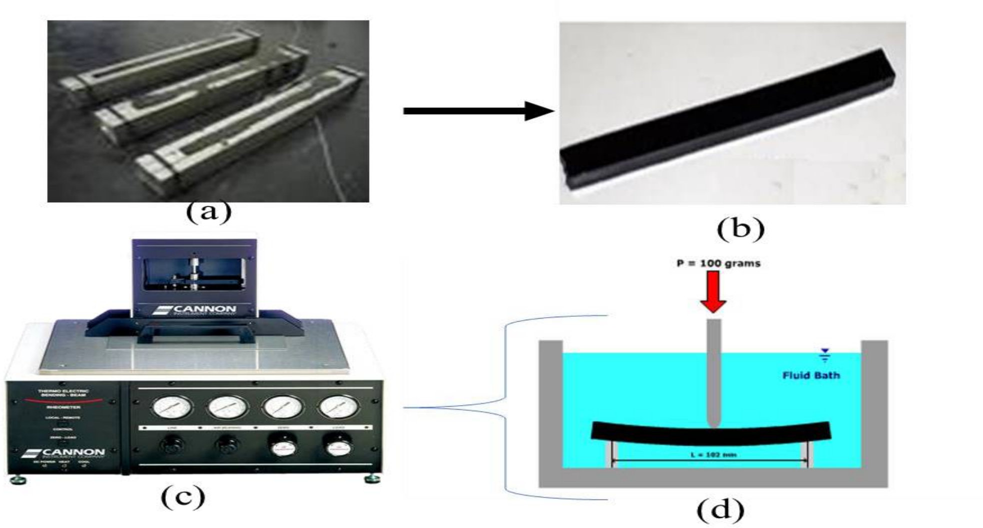


Figure 3.12: Beam formation and analysis

These parameters showed the ability of bitumen binder to resist low temperature cracking. The test was performed by using AASHTO T313-10 test method. According to this method sample of bitumen binder is moulded into a beam having (6.25 x 12.5 x 127 mm) size. Then sample is immersed in a low temperature liquid bath supported at two points (102 mm) apart. After that load is given at the midpoint of bitumen beam and measured the stiffness and m-value at that particular temperature.

3.2.2.3 MULTIPLE STRESS CREEP RECOVERY (MSCR) TEST

The multiple stress creep recovery (MSCR) test is the latest and more accurate test for Performance Graded (PG) specification of bitumen binder. This test is conducted by using AASHTO TP70 and AASHTO MP19 methods which indicates the rutting performance of the bitumen binder more accurately at high temperature. The MSCR test deals with creep and recovery test concept to determine the binder's potential for permanent deformation. This test is characterized by using the dynamic shear rheometer (DSR), the same equipment which is used for PG specification. In this test, a creep load (0.1 kPa) is applied to the bitumen binder sample for one second, then load is removed and the sample allowed to recover for 9 seconds. It is repeated up to 10 cycles. Similarly, test is conducted at 3.2 kPa stress level and repeated for an additional 10 cycles.

3.3.3 PERFORMANCE TEST

3.3.3.1 MARSHALL STABILITY TEST

In this test, Marshall Stability with flow values of the pavement are measured by using Marshall Apparatus (Controls Company). Preparation of Marshall specimen and test apparatus are given in Figure 3.13

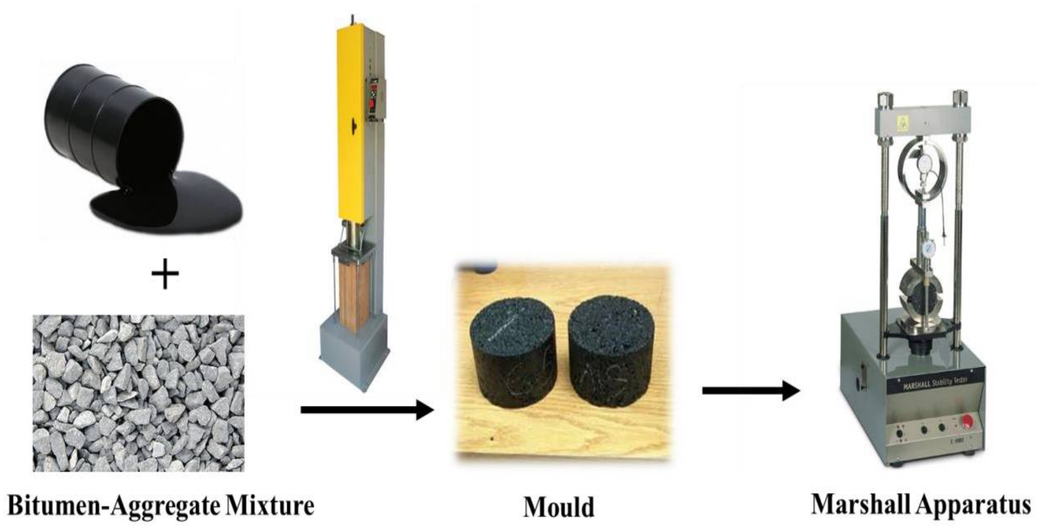


Figure 3.13: Mould Preparation and Marshal apparatus

The compacted bituminous mix specimens of diameter (100 mm) and thickness (63.5 ± 0.5mm) were prepared as per ASTM D6926 and ASTM D6927 methods. The specimen is immersed in a water bath having temperature of 60 ± 1 °C for a period of 30 minutes called (before conditioning, S1) and when the same is for 24 hours then it is called (after conditioning, S2). Finally, Marshall Strength and flow values and retained Marshall Stability was calculated.

3.3.3.2 RUTTING RESISTANCE TEST

The rutting resistance test was carried out by using wheel tracking apparatus (Cooper Company) as per EN12697–22:2003+A1. A mix slab with dimension (300 × 300 × 40) mm was prepared for testing. The mixes were compacted by a roller compactor at 150–160 °C by maintaining percentage of air void 4-6%. Then the prepared slab could cure at room temperature for 48 hours. After that slab was put into an oven at 60 °C for 5 hours for conditioning before testing. Then a wheel is moved with a frequency of 53 passes per minute on the slab up to 10000 cycles. During test period, rut depth was measured at different cycles. Samples with rutting and without rutting are given in Figure 3.14.

Figure 3.14: Bitumen Mixed Samples; a) Before Rutting; b) After Rutting

Table 3.1: Specification for Paving Bitumen (IS 73: 2013)

Characteristics	Paving Grade				Reference to IS No.
	VG 10	**VG 20**	**VG 30**	**VG 40**	
Penetration at 25ºC, 100g, 5 s, 0.1 mm, *Min*	80	60	45	35	IS 1203
Absolute viscosity at 60ºC, Poises,	800–1200	1600-2400	2400-3600	3200 4800	IS 1206 (Part 2)
Kinematic viscosity at 135ºC, cSt, *Min*	250	300	350	400	IS 1206 (Part 3)
Flash point, (Cleveland open cup), ºC, *Min*	220	220	220	220	IS 1448 P:69
Solubility in trichloroethylene, percent, *Min*	99.0	99.0	99.0	99.0	IS 1216
Softening point (R&B), ºC, *Min*	40	45	47	50	IS 1205
Tests on residue from rolling thin film oven test (RTFOT)					
Viscosity ratio at 60ºC, *Max*	4.0	4.0	4.0	4.0	IS 1206 (Part 2) IS 1208
Ductility at 25ºC, cm, *Min*,	75	50	40	25	IS 1206 (Part 2) IS 1208

Table 3.2: Specification for Crumb Rubber Modified Bitumen (IS 15462: 2004)

Characteristics	**Grade and Requirements**			**Method of Test**	**Ref to**
	CRMB 50	**CRMB 55**	**CRMB 60**	**IS No.**	**Annex**
Penetration at 25 °C, 0.1 mm, 100g, 5s	<70	<60	<50	1203	-
Softening Point, (R&B), °C Min.	50	55	60	1205	-
Flash Point, COC, °C. Min.	220	220	220	1209	-
Elastic recovery of half thread in ductilometer at 15 °C, percent, Min.	50	50	50	-	A
Separation, difference in softening point (R&B) °C Max.	4	4	4	-	B
Thin film oven tests and test on residue (TFOT):					
Viscosity at 150 °C, Poise	1-3	2-6	3-9	1206 (Part 1)	-
Loss in mass, percent Max.	1.0	1.0	1.0	9382	-
Increase in softening point, °C. Max.	7	6	5	1205	-
Reduction in penetration of residue, at 25 °C, percent, Max.	40	40	40	1203	-
Elastic recovery of half thread in ductilometer at 25 °C, percent Min.	35	35	35	-	A