CHAPTER 3. MATERIALS AND RESEARCH METHODOLOGY

3.1 Preamble

This chapter provides the description of materials used in this study and research methodology to complete the objectives of the research. The materials chosen for the study, including the base asphalt binders, aggregates from different sources, and WMA additives are discussed in length in this chapter. Along with this, the preparation of WMA modified asphalt binders have been described. Additionally, a detailed research methodology has been provided.

In this chapter, the traditional tests, including viscosity, penetration value, and softening point, elastic recovery, zero shear viscosity have been briefly described. The different methods used of MT and CT has been discussed in this chapter. Along with this, the tribological assessment of WMA modified binders has been introduced for determination of MT and CT of WMA additives. Following determination of MT and CT using tribological approach, the validation of MT and CT has been introduced in this chapter. The different aspects related to the aging of WMA binders has been explored. The methodology of the tests used for determining chemical composition (Fourier transform infrared spectroscopy) has been described. The various performance tests on WMA modified binders, such as multiple stress creep and recovery (MSCR) and linear amplitude sweep test (LAS), bitumen bond strength (BBS) test have also been stated. Further, the strategies for selection of WMA additives have been discussed. Finally, this chapter also shows the results of physical properties of WMA modified binders and compared it with the base binders. Figure 3-1 shows the methodology adopted for the present research study.

This chapter has been divided in following phases:

1) Phase 1: Selection of materials for the study
2) Phase 2: Preparation of WMA modified asphalt binder
3) Phase 3: Development of experimental program
4) Phase 4: Strategy for selection of WMA additives

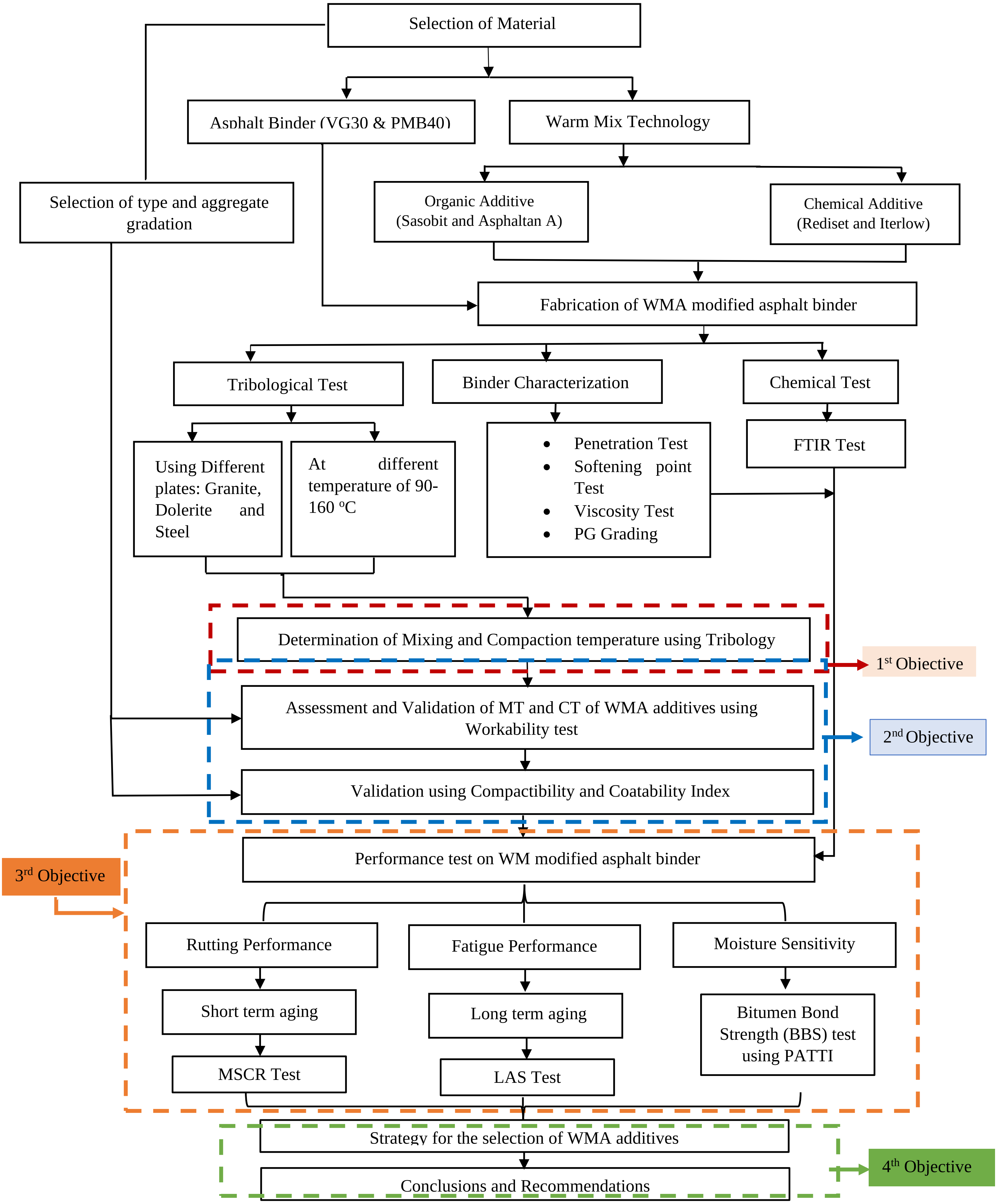

Figure 3-1. Methodology adopted in the study

3.2 Phase 1: Material Section

The following materials have been chosen for carrying out the research work in different phases.

3.2.1 Selection of Asphalt Binder

One viscosity graded (VG) asphalt binder VG 30 and polymer modified asphalt binder PMB 40 have been used for the study [307]. These are the most widely used binders in India for road construction. Also, selection of two binders will facilitate in understanding the effect of base binder on the performance of WMA modified binders. Physical properties were performed on these base asphalt binders in accordance with IS 73:2013 [308] and IS15462 [309]. The results of physical properties are shown in Table 3-1. In addition, fourteen viscosity grade binders were selected to establish reference coefficient of friction (CoF) values for estimating production temperatures using the tribological approach. Furthermore, four polymer modified binders were used to assess the suitability and applicability of this approach for predicting production temperatures of polymer modified binders containing WMA additives. The details of these binders have been discussed in Chapter 5.

Table 3-1. Physical properties of the asphalt binders

Property	**Values Obtained**		**Test Standard**
	VG 30	**PMB40**	
Penetration at 25°C (dmm)	64	49	IS 1203 [310]
Softening point (°C)	49	70	IS 1205 [311]
Absolute Viscosity at 60°C (Pa.s)	280	-	IS 1206 (Part 2) [312]
Viscosity at 135°C (Pa.s)	0.51	0.92	ASTM D2493 [133]
Elastic Recovery of half thread in ductilometer at 15°C (%)	-	81.4	IS15462:2019 [309]

Specific gravity	1	1.02	IS 1202 [313]
High temperature performance grade	PG70-XX	PG82-XX	ASTM D7175 [314]

3.2.2 Selection of WMA Additives

Based on two different groups of WMA i.e. wax/organic additives and chemical additives, four different WMA additives have been selected. Organic additives selected for the study are: Sasobit and Asphaltan A. Chemical additives selected for the study are: Iterlow and Rediset. These warm mix additives were used to prepare different warm mix modified asphalt binders. The additives were mixed at different doses with VG 30 and PMB 40 binders as per the manufacturer's recommendations. Figure 3-2 (a-d) shows the visual appearance of WMA additive. The two additives selected under organic and chemical category were different from each other. This can be confirmed by their chemical spectra determined from the Fourier transform infrared spectroscopy. The details regarding the FTIR test have been discussed in detailed in subsequent section of this chapter. Figure 3-3 to Figure 3-6 shows the FTIR spectra of WMA additives used in the study.

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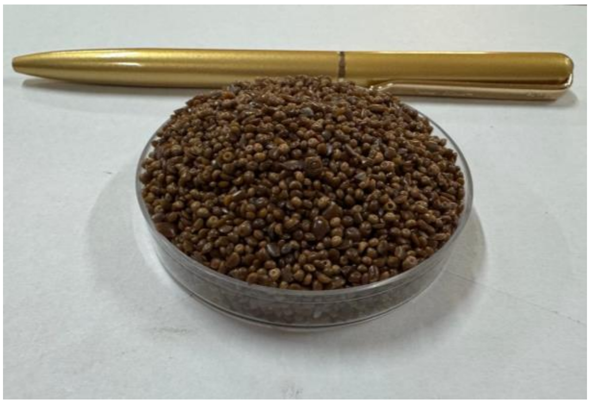

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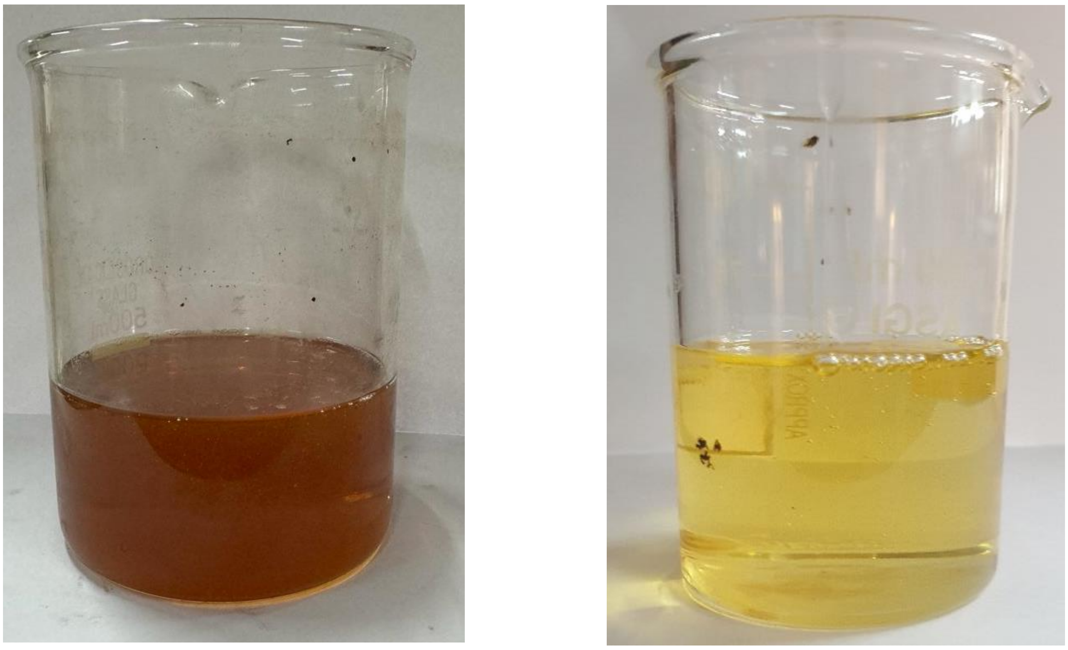


(c) (d)

Figure 3-2. Warm mix additives a) Sasobit b) Asphaltan A c) Iterlow d) Rediset LQ1102CE

3.2.2.1 Sasobit

According to past literatures [2,52,136], Sasobit is a long-chain (40-115 carbon atoms) aliphatic hydrocarbon with a fine crystalline structure that is generated from natural gas by the Fischer Tropsch process. It is also called as synthetic microcrystalline wax having carbon atoms greater than paraffin wax. In this study, Sasobit has been studied and classified based on FTIR spectra. The FTIR spectra of Sasobit has been shown in Figure 3-3. The two peaks appeared between 3000 and 2800 cm^{-1} wavenumber corresponds to the asymmetric and symmetric stretches of C-H in CH_2 and CH_3 [196,315]. The C-H showed asymmetric stretching vibration at 2921 cm^{-1} whereas symmetric stretching vibration was found at 2852 cm^{-1} [315]. The peaks of the scissoring vibrations of the C-H in CH_2 and CH_3 were also located in the 1500-1350 cm^{-1} region [315]. From Figure 3-3, the scissoring vibrations of the C-H in CH_2 and CH_3 was found at 1473 cm^{-1} [316]. The presence of scissoring vibrations at 1473 cm^{-1} indicates the packing of all *trans*-hydrocarbon chain in a triclinic sub-cell lattice and also the hexagonal packing of all *trans*-methylene chains [317,318]. Another peak was also observed at 720 cm^{-1}

due to the rocking of the same vibrational group (C-H). The occurrence of C-H at these wavelengths signifies the presence of wax-based structure in additive [319]. Based on the overall FTIR spectra details, the Sasobit can be classified as wax type linear aliphatic hydrocarbons having lattice structure.

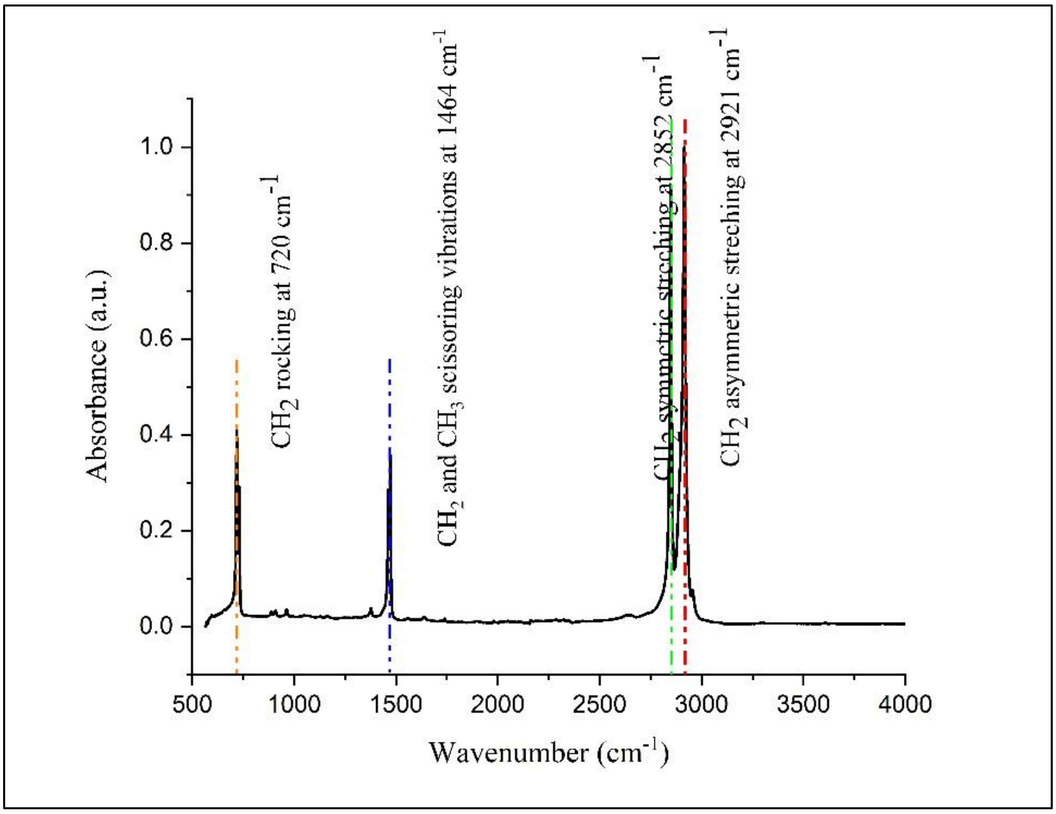


Figure 3-3. FTIR spectra for Sasobit

3.2.2.2 Asphaltan A

Asphaltan A is obtained from the blend of Montan wax (obtained from solvent extraction of lignite or brown coal) and fatty acid amides [221]. The FTIR spectra has been shown in Figure 3-4. Most of the peaks in Asphaltan A were similar to the Sasobit but only difference in the peaks of amine group and fatty acids. The peak at 3257 cm^{-1} indicates the presence of stretching of N-H in amine group [320,321]. The presence of this peak generally follows by the presence of the aliphatic amid group at 1045 cm^{-1} [320]. The two peaks obtained at 1714 and 1738 cm^{-1} corresponds to the symmetric stretching of C=O whereas peak at 1245 cm^{-1} corresponds to the stretching of C-C-O (ester group) [320]. The presence of the C=O and C-O in Asphaltan A indicates the presence of esters and free fatty acids. The presence of these peaks verified that the Asphaltan A is a combination of wax and fatty acid amides. Therefore, the Asphaltan A can be classified as Amine based wax type linear aliphatic hydrocarbons.

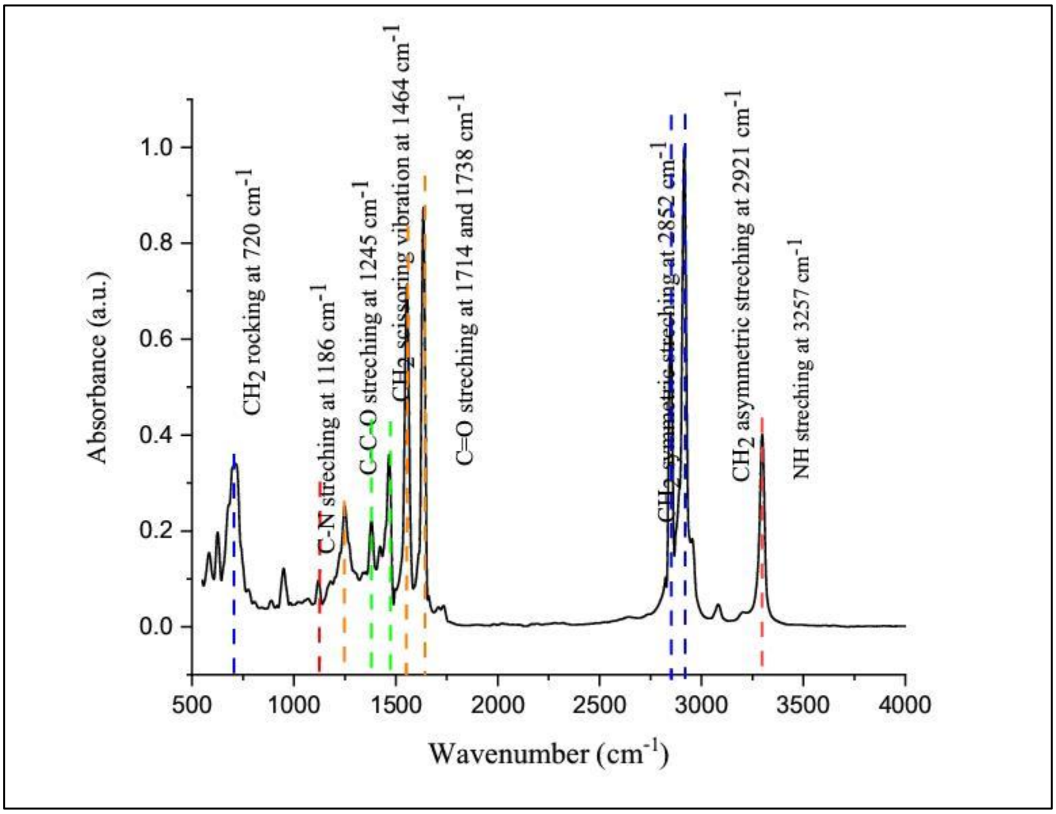


Figure 3-4. FTIR spectra for Asphaltan A

3.2.2.3 Iterlow

Iterlow is surfactant type chemical additive used for lowering the production temperature of asphalt mixture around 120ºC [206]. FTIR spectra of Iterlow has been shown in Figure 3-5. The peaks at 2852 and 2921 cm^{-1} corresponds to the asymmetric and symmetric vibration of C-H in CH_2, respectively [319]. The peaks at 1370 and 1450 cm^{-1} indicates the bending vibrations of C-H in methyl group i.e.CH_3 and methylene i.e.CH_2 group, respectively [321]. The presence of ester in Iterlow can be identified by C-C-O vibration at 1245 cm^{-1} [320]. The presence of peaks at 1652 and 3410 cm^{-1} indicates the presence of N-H in Iterlow [321]. The peak especially observed at 3410 cm^{-1} indicates the vibration of silane in amine group (i.e. Si-NH) [322]. This group (Si-NH) along with the peaks at 2852, 2921, 1370, and 1450 cm^{-1} acts as basic building block for the surfactant kind material. These entire structure can be referred to as *Tris*-(trimethylsilyl) amine [322]. The peak between 1000 and 1100 cm^{-1} corresponds to the Si-O-Si vibration which indicates the presence of organo-silica in the structure of Iterlow [323,324]. The results of FTIR showed that the Iterlow contains fatty amine and organo-silica in its structure. Based on this, Iterlow can be classified as amine-silane based chemical additive.

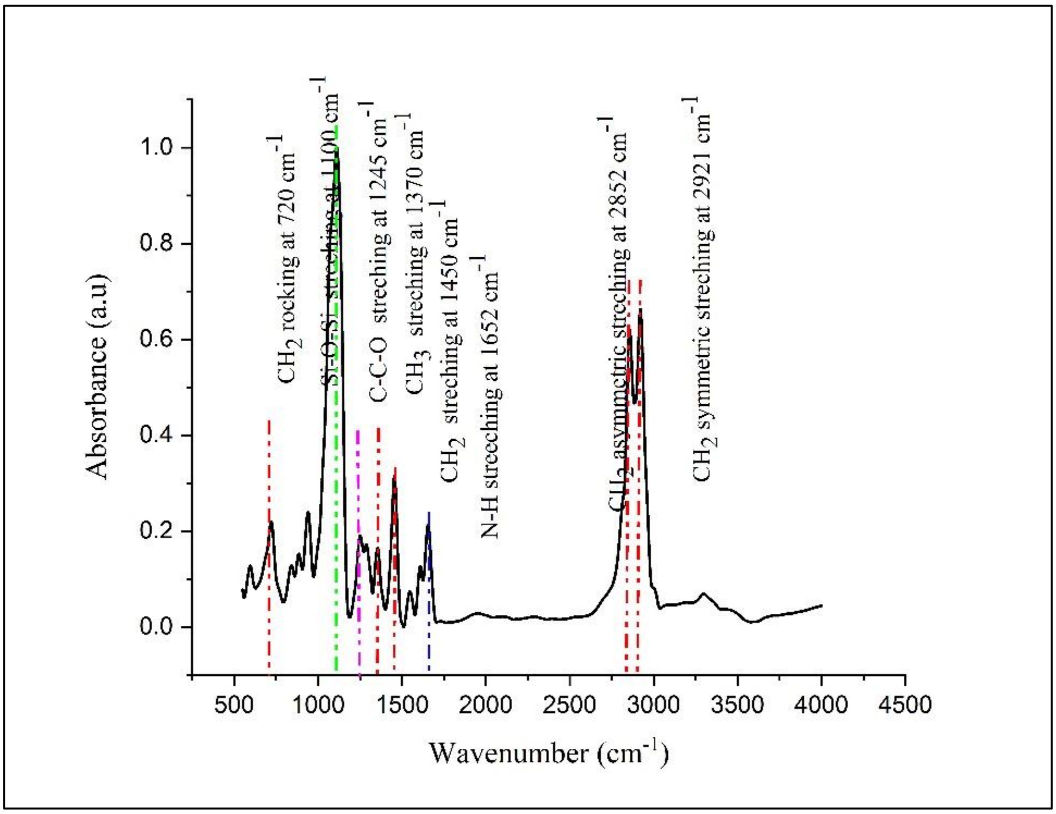


Figure 3-5. FTIR spectra for Iterlow

3.2.2.4 Rediset LQ1102CE

Rediset is a chemical additive which contains fatty amine and a long chain aliphatic hydrocarbon structure that imparts the surfactant type behaviour to the additive [58]. The FTIR spectra of Rediset has been shown in Figure 3-6. The presence of amine in the Rediset was identified based on the simultaneous occurrence of N-H peak at 3410 and 1652 cm^{-1} [320,321].

The peaks obtained at 2852, 2921, 1370, and 1450 cm^{-1} indicates the different vibrations of C-H in CH_2, which indicates the presence of long chain aliphatic hydrocarbon. The vibrations of C-N were observed at wavelength of 1045 and 1186 cm^{-1} indicates the presence of aliphatic amide bond in the structure of Rediset [320]. The combination of N-H and C-H presented in Rediset imparts surfactant type behaviour to Rediset [325]. Due to the presence of these functional groups, the Rediset can be referred to as Polyethylene amines. Based on spectra, it can be classified as amine based chemical additive as it contains only amine group in its structure.

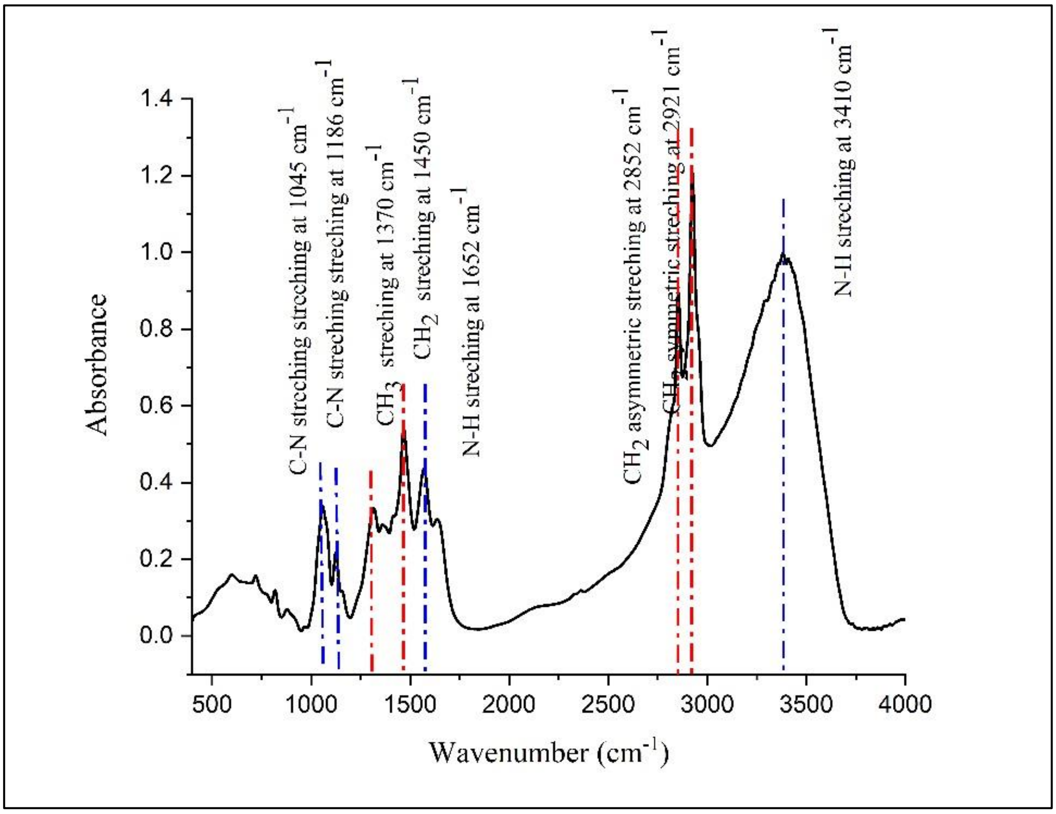


Figure 3-6. FTIR spectra for Rediset

The FTIR spectra allow the differentiation of WMA additives used in the study. Organic additives can be classified into two groups: wax type organic additives and amine-based organic additives. On the other hand, chemical additives are divided into two groups: amine-silane based chemical additives and amine-based chemical additives.

3.2.3 Physical Properties of WMA Additives

The physical and chemical properties of all WMA additives used in the study are shown in Table 3-2. All organic additives were solid in nature whereas chemical additives were liquid in nature. The colour and physical appearance of all the additives are mentioned in Table 3-2 and shown in Figure 3-2. It can be found that the Asphaltan A showed slightly higher melting point than Sasobit. Rediset exhibits melting point near to 0^oC except for Iterlow (-5^oC). The density of all chemical additives was found to be near 1 g/cm³ except for Iterlow RT, which had a density of 0.85 g/cm³. Organic additives showed a density range between 0.9–1 g/cm³. The Asphaltan A showed slightly lower density than Sasobit. These higher melting point and densities of organic additives can be attributed to the higher number of carbon atoms in their structure [52]. For chemical additives, the higher densities might be due to the presence of silica (in the form of Silane group) in their structure. Iterlow was found insoluble in the water except for the Rediset (partial soluble). The pH value showed that the Sasobit was neutral in nature whereas Asphaltan A showed basic nature. The basic nature of Asphaltan A can be attributed to the presence of amine group in its structure [326]. Chemical additives showed the basic nature. This property of WMA additives can be confirmed by the FTIR spectra discussed in the previous section.

Table 3-2. Physical and chemical properties of WMA additives

Property/ WMA Additives	**Organic**		**Chemical**	
	Sasobit	**Asphaltan A**	**Iterlow RT**	**Rediset LQ1102CE**
Visual color	White	Brown	From yellow to brown	Pale yellow
Physical State	Pellets	Flakes	Liquid	Liquid
pH value	Neutral	8	8-10	10

Flash Point (°C)	285	>240	>105	165
Melting Point (°C)	>90	90-95	-5	0
Solubility in water	Insoluble	Insoluble	Insoluble	Partially soluble
Density at room temperature (g/cm^3)	0.9	0.85-0.9	0.85	0.99-1.00

3.2.4 Aggregates

Aggregates are important ingredient of flexible pavement which forms the strong skeleton to carry required traffic loading. The properties of aggregates are greatly influenced by their mineral composition [327]. Therefore, the selection of aggregate is important prior to construction of the pavement. One of the objectives of the study is to study the moisture sensitivity of WMA modified asphalt binders using different types of aggregates. Therefore, three basic aggregates i.e. Dolomite, Dolerite, and Basalt and two acidic aggregates i.e. Granite and Sandstone were used in this study, as shown in Table 3-3. Among these aggregates, Granite and Dolerite were used for workability study to assess their effect on workability characteristics of WMA modified asphalt binders. The details about these are discussed in Chapter 5. The physical properties of the aggregates are shown in Table 3-4.

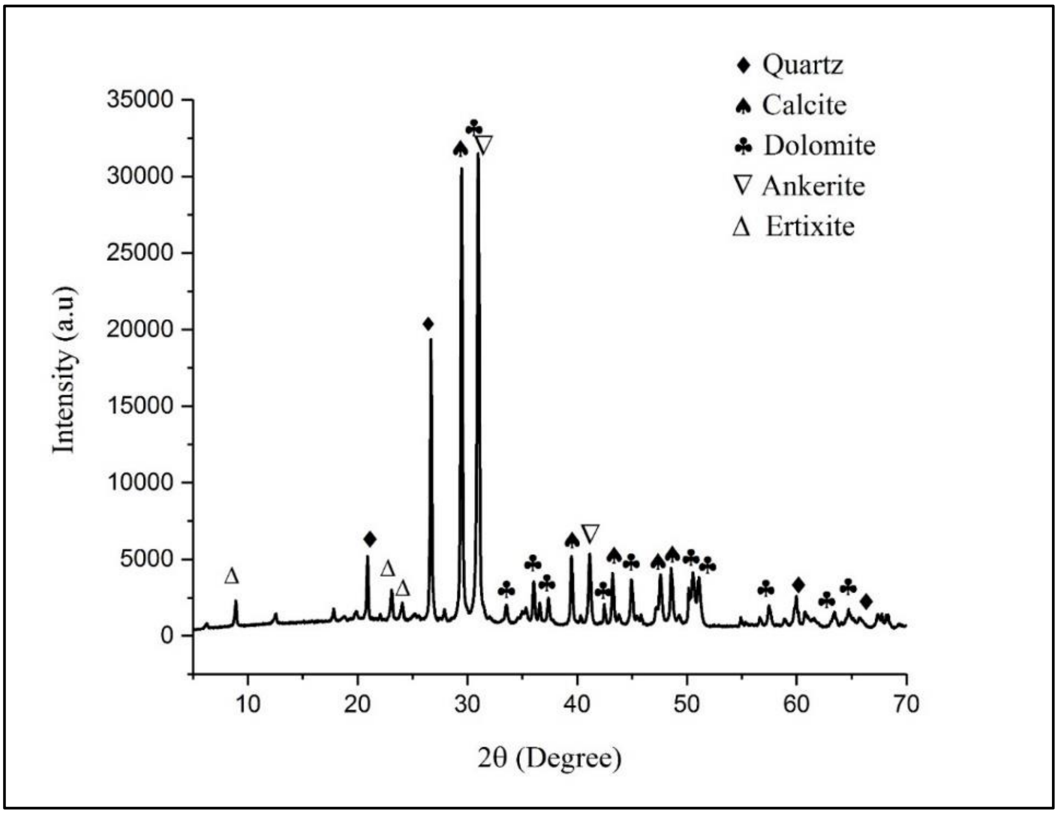
Table 3-3. Nature of different aggregate type

Aggregate Name	**Rock Type**	**Nature**
Dolomite	Sedimentary	Basic
Dolerite	Igneous	Basic
Basalt	Igneous	Basic
Granite	Igneous	Acidic
Sandstone	Sedimentary	Acidic

Table 3-4. Physical properties of aggregates

Tests	Value Obtained					Specified Limit for Wearing Course	Test Standard
	Dolomite	Dolerite	Basalt	Granite	Sandstone		
Aggregate Impact Value (AIV), %	14	20	13	18.5	23.5	Max 24%	IS:2386 (Part-4) [328]
Los-Angeles Abrasion Value (LAV), %	19	22	16	21.0	28.4	Max 30%	IS:2386 (Part-4) [328]
Combined Flakiness and Elongation Index, %	18	21	18	20.0	17	Max 35%	IS:2386 (Part-1) [329]
Water Absorption, %	0.6	0.8	0.55	0.6	1.6	Max 2%	IS:2386 (Part-3) [330]
Soundness (using Sodium Sulphate),%	7.5	9.2	7.2	8.1	11.5	Max 12%	IS:2386 (Part-5) [331]

The mineral present in the aggregates was evaluated in this study using x-ray diffraction (XRD) technique. Rigaku SmartLab 9kW Powder type device (Rigaku Company, Japan) was used to measure the XRD data of five different types of aggregates (rocks), specifically Dolomite, Dolerite, Basalt, Granite and Sandstone. This machine adopts Cu as target, while the X-Ray wave length λ is 1.5406 Å with 40 kV operating voltage and 40 mA operating current, and the test range of diffraction angle 2θ is 10–100° [332]. In this study, qualitative examination of X-Ray was analyzed using XPERT high score software in which different mineral present in aggregates were identified. This identification was performed by comparing the standard data of the well-known mineral in the powder diffraction file (PDF) with the measured phase diffraction spectrum. Once the well-known minerals match with the peak of measured diffraction spectrum, then the match minerals were considered to be present in the given aggregates. The X-ray diffractograms of the different aggregates are shown in Figure 3-7. In this study, efforts were made to calculate approximate percentage of the different minerals present in the aggregates using *Match!* (version 2) software. Figure 3-8 (a) shows interface of the software and Figure 3-8 (b) shows percentage of minerals present in Dolomite aggregate. Similar kind of analysis was also performed for other aggregates. Figure 3-7 showed that all aggregates consist of different mineral composition and form the structure of the aggregates. These mineral composition plays important role in adhesion mechanism between aggregate and asphalt binder [327]. Most of the aggregates contain silica in the form of quartz in various concentrations. The presence of higher amount of silica in aggregate have potential of moisture damage. The aggregate with higher silica content is referred to as siliceous aggregate. The aggregate used in the study such as Dolomite and Basalt have calcite in their mineral composition. The presence of calcite compound has ensured better bond with asphalt binder in presence of moisture. Table 3-5 shows major mineral present in the X-ray diffractograms and quantified using "*Match!* (version 2)" software. From Table 3-5, it can be seen that the Dolomite aggregate has predominant phases of Dolomite (46.1%) and Calcite (36.8%) as compared to other minerals. The presence of these mineral in Dolomite aggregate makes it calcareous in nature and has potential to resist the moisture damage. The Dolerite is made up of a different mineral phase. The Albite and Anorthite are the dominant minerals in Dolerite with a presence of 35.7% and 34.2 %, but Plagioclase and Quartz also have significant quantities. Along with mineral phases, some traces of Calcite have been observed in Dolerite. For Basalt aggregate, the Plagioclase (44.7%) is the predominant phase when compared to the other minerals present. The Calcite (20.1%) and Quartz (14%) showed significant presence in Basalt. The Granite and Sandstone have higher percentage of Quartz and Albite. Granite contains 36.4% and 28.3% of Quartz and Albite, respectively whereas Sandstone has 44.1% Quartz and 25.5% Albite, 14.4% Glauconite, and 13.7% Illite in the structure. The Sandstone has higher silica content among all the aggregates which showed higher susceptibility of this aggregate towards moisture damage [333]. The XRD analysis showed that most of the aggregates has difference in their mineral composition especially in Quartz, Albite and Calcite content. It is thought that the high proportion of Albite and Quartz phases could result in moisture damage due to the poor adhesion between Quartz and asphalt binder [334]. Albite and asphalt binder can form strong bond when it is dry, however in presence of moisture, this bonding dissolves very quickly [327]. There is also evidence that the aggregate with higher Calcite content is responsible for better adhesion between aggregate and asphalt binder due to formation of hydrophobic salt which are insoluble in water [333,335]. Presence of these various minerals in aggregate affects the bonding mechanism of aggregate and asphalt binder. The effect of these minerals on bond strength for various aggregate-binder combinations are discussed in Chapter 6.



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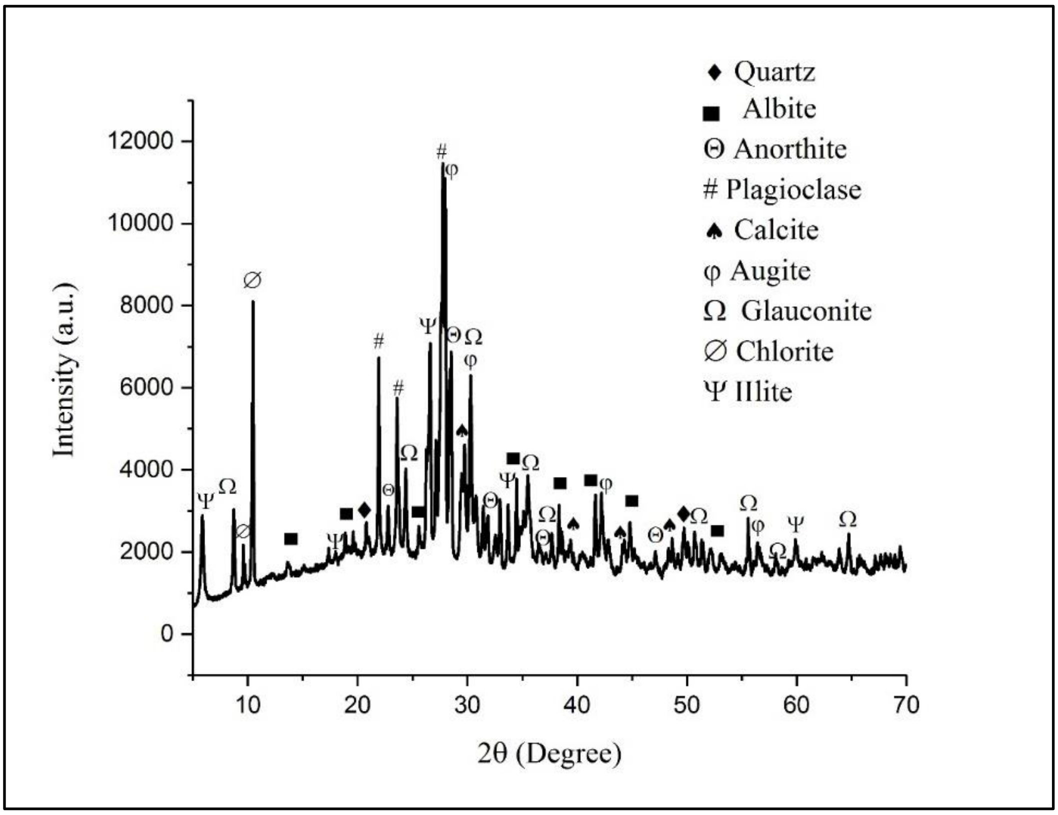
♦ Quartz
■ Albite
Θ Anorthite
Plagioclase
♠ Calcite
φ Augite
Ω Glauconite
∅ Chlorite
Ψ Illite
Intensity (a.u.)
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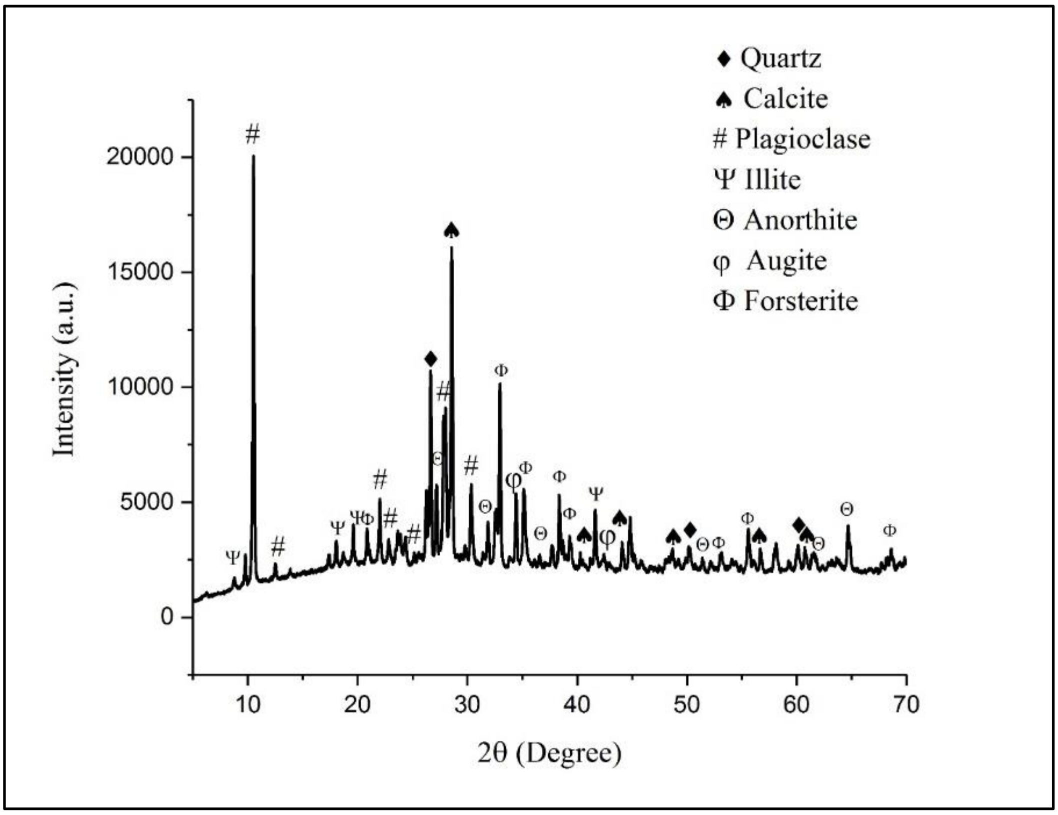
♦ Quartz
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Ψ Illite
Θ Anorthite
φ Augite
Φ Forsterite
Intensity (a.u.)
2θ (Degree)

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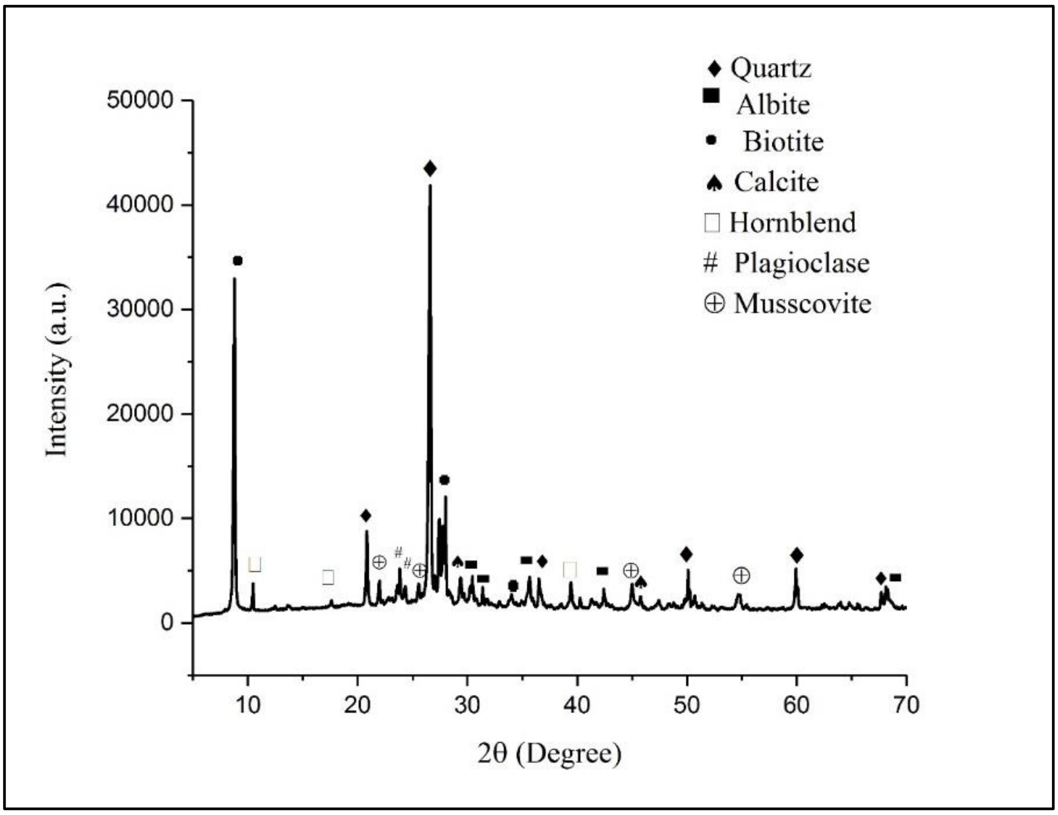

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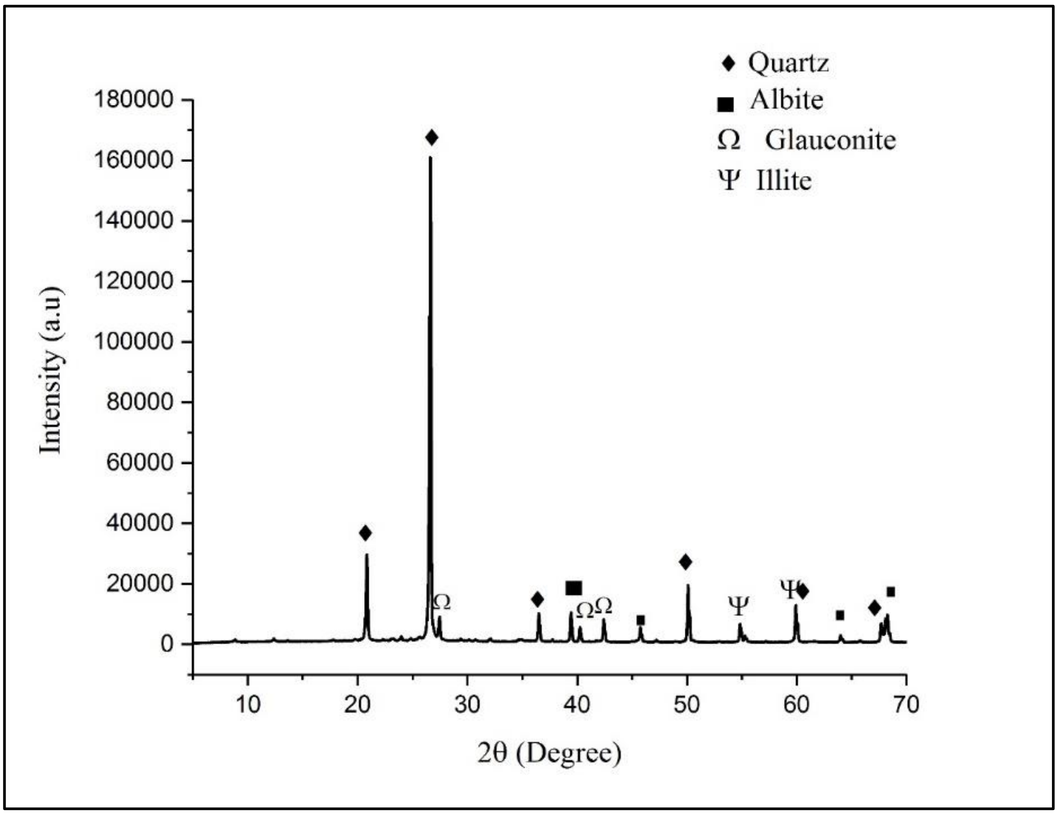
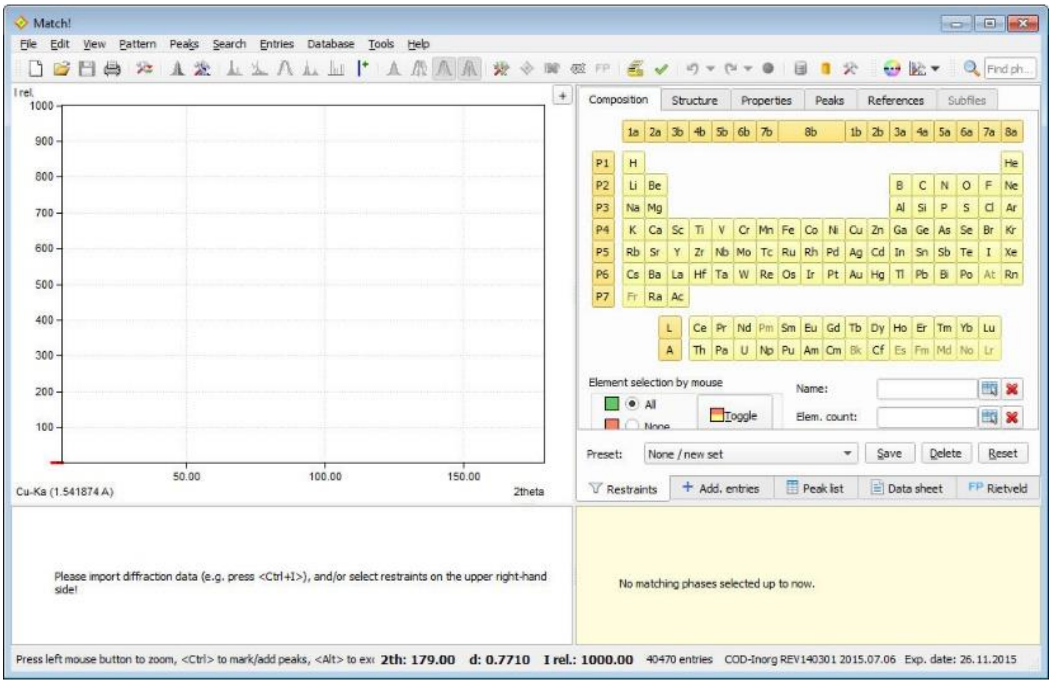


(e)

Figure 3-7. XRD pattern of aggregates a) Dolomite b) Dolerite c) Basalt d) Granite e) Sandstone



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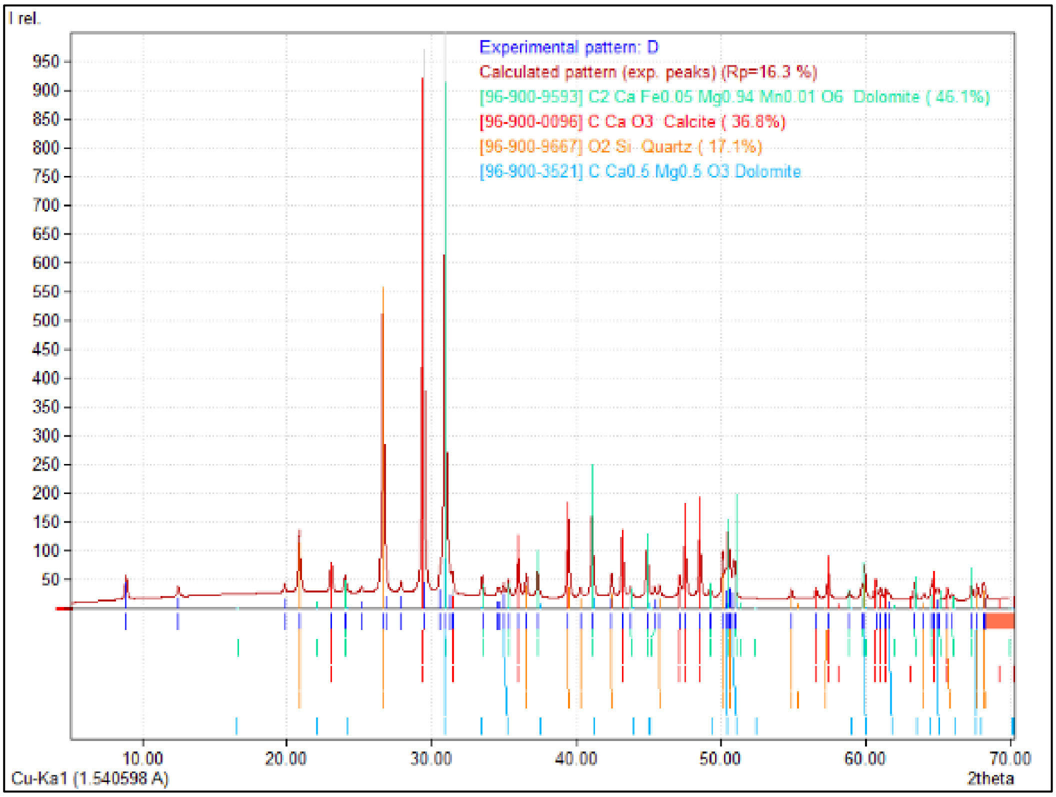


(b)

Figure 3-8. Match software a) Interface of Match software b) XRD pattern of Dolomite obtained from Match Software

Table 3-5. Mineral composition of various aggregate type

Aggregates	**Minerals Composition (%)**								
	Quartz	**Albite**	**Anorthite**	**Plagioclase**	**Glauconite**	**Calcite**	**Biotite**	**Dolomite**	**Illite**
Dolomite	17.1	-	-	-	-	36.8	16.1	46.1	
Dolerite	8.7	35.7	34.2	14	-	4.6	2.8	-	
Basalt	14	-	9.2	44.7	-	20.1	-	-	12
Granite	36.4	28.3		15.2		4	16.1		
Sandstone	44.1	25.5			14.4	2.3			13.7

3.3 Phase 2: Preparation of WMA Modified Asphalt Binders

To prepare WMA binders (organic and chemical-based), base asphalt binders were heated using a temperature-controlled heating mantle until they became fluid enough to be poured. 500 gm of base asphalt binders were taken and appropriately stirred to remove the entrapped air. After that, the selected dosages of the WMA additives were added to the preheated base asphalt binders. The dosages of the WMA additives were selected based on the manufacturer's recommendations and prior literature. For organic additives (Asphaltan A and Sasobit), dosages of 1%, 2%, and 3% by weight of binder were used, with a 1% increment as suggested by the manufacturer and previous studies. For chemical additives, Rediset was tested at 0.4%, 0.5%, and 0.6%, and Iterlow RT at 0.3%, 0.4%, and 0.5% by weight of binder. These dosages were selected using a 0.1% increment also in line with manufacturer guidance and earlier research findings. Mixing was commenced using a high-shear propeller mixer (as shown in Figure 3-9) operated at a speed of 500 rpm. In the case of VG30, mixing was continued for 30 minutes under a controlled temperature range of 130-140°C, as per previous literatures

[201,336,337]. For PMB40, mixing was done in two stages. First stage involved the stirring of base PMB40 for 15 minutes to ensure proper dispersion of polymers within the asphalt binder, followed by the second stage, wherein the blending with WMA additives was done for 30 minutes at a temperature of 140-150°C [34]. To ensure equivalent aging conditions during the mixing process, VG30 and PMB40 were also subjected to the same mixing protocol (as adopted for respective WMA binders) prior to testing. Figure 3-9 shows the stepwise preparation of WMA modified binders using high shear mixer. Table 3-6 shows the abbreviations used (in this study) for different combinations of WMA additives and asphalt binders.

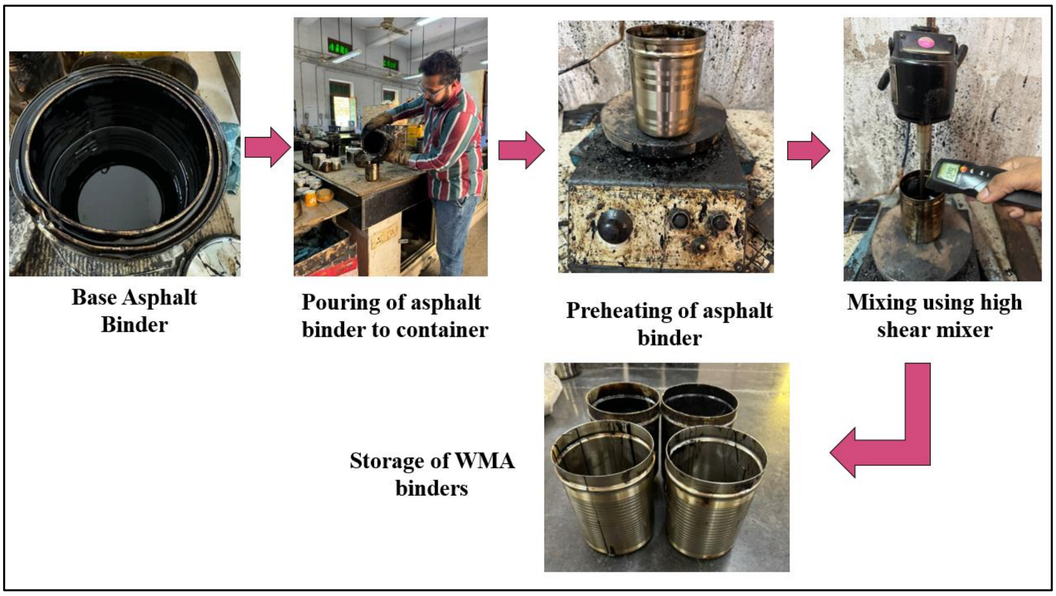


Figure 3-9. Stepwise preparation of WMA modified binders

Table 3-6. Terminology used in the present study

Binder Type	Additives (Type)	Dosages	Description
VG30	Sasobit (S) (Organic)	1%,2%, and 3% w/b	VG30+1%S (S1), VG30+2%S (S2), VG30+3%S (S3)

	Asphaltan A (A)	1%,2%, and 3% w/b	VG30+1%A (A1), VG30+2%A (A2), VG30+3%A (A3)
	Iterlow (I) (Chemical)	0.3%, 0.4%, and 0.5% w/b	VG30+0.3%I (I0.3), VG30+0.4%I (I0.4), VG30+0.5%I (I0.5)
	Rediset (R) (Chemical)	0.4%, 0.5%, and 0.6% w/b	VG30+0.4%R (R0.4), VG30+0.5%R (R0.5), VG30+0.6%R (R0.6)
PMB 40	Sasobit (S) (Organic)	1%,2%, and 3% w/b	PMB40+1%S (PS1), PMB40+2%S (PS2), PMB40+3%S (PS3)
	Asphaltan A (A)	1%,2%, and 3% w/b	PMB40+1%A (PA1), PMB40+2%A (PA2), PMB40+3%A (PA3)
	Iterlow (I) (Chemical)	0.3%, 0.4%, and 0.5% w/b	PMB40+0.3%I (PI0.3), PMB40+0.4%I (PI0.4), PMB40+0.5%I (PI0.5)
	Rediset (R) (Chemical)	0.4%, 0.5%, and 0.6% w/b	PMB40+0.4%R (PR0.4), PMB40+0.5%R (PR0.5), PMB40+0.6%R (PR0.6)

Note: w/b indicate by weight of the asphalt binder

3.4 Phase 3: Development of Experimental Program

This section discussed the various experimental tests carried out on the WMA modified asphalt binders to study the effect of WMA additives on the various performance parameters of pavement. For effective utilization of WMA additives for the construction of flexible pavement, the formation of proper experimental program is necessary. After preparing various WMA modified asphalt binders, the preliminary tests such as penetration, softening point, viscosity, elastic recovery and high-temperature performance grading (PG) were performed to characterize the WMA modified asphalt binders. Following the basic material characterization, various viscosity based approaches, phase angle method, and tribological approach were used to evaluate the mixing and compaction temperatures of WMA modified asphalt binders. Appropriate approach for determination of mixing and compaction temperatures of WMA modified asphalt binders were identified and validated using workability test, coating ability test, and compactibility test. Following the determination of mixing and compaction temperature, the performance of WMA modified asphalt binders were evaluated at reduced aging temperature using various performance tests on asphalt binders. Advanced tests such as MSCR, Phase angle test, LAS are recommended to clearly comprehend the rutting and fatigue behaviour of WMA modified asphalt binders. The fatigue behaviour of the WMA modified asphalt binders was assessed using the phase angle test, LAS test over the temperature range of 10–30^{o}C, while the rutting potential was evaluated through MSCR test at temperatures ranging from 40–70^{o}C. This study also focused on the moisture sensitivity of WMA modified asphalt binder. Recently, the bitumen bond strength (BBS) test is being used to assess the influence of moisture conditioning at the interface of asphalt binder and aggregate matrix. Further, the results of these tests were analysed to determine the effectiveness of WM technology for asphalt pavements. Along with performance tests, Fourier transform infrared spectroscopy test was also performed on the WMA binders to evaluate the effect of WMA additives on the aging potential of the binders. All the tests were performed thrice, and the average of obtained results are presented in this study. It was found that the coefficient of variation for all the measurements was under 10%.

3.4.1 Basic characterization of WMA Modified Binders

After obtaining the various combinations of WMA modified asphalt binders, following basic tests have been carried out to assess the properties of each WMA modified binders.

a) **Penetration test:** Penetration is a consistency test which measures the penetration of a standard needle (100 g) inside an asphalt binder sample at 25 ℃ in 5 seconds. The test was carried out on all the prepared binders as per IS 1203-1978 [310]. Figure 3-10 (a) shows the experimental setup for penetration test.

b) **Softening point test:** Softening point is also a consistency test which measures the temperature at which the substance attains a particular degree of softening under specified condition of the test. This was carried out using a ring and ball apparatus as per IS 1205-1978 [311]. Figure 3-10 (b) shows the experimental setup for softening point test.

c) **Viscosity test:** Viscosity is one of fundamental flow property of material. The viscosity of the binders was studied using a Cannon Manning viscometer at a temperature of 60^{o}C (maximum temperature of the in-service pavement) in accordance with IS 1206-1978. The viscosity of the asphalt binder is determined by measuring how long it takes the binder to cross certain timing markers within a viscometer tube at standard temperature and pressure. In this study, the test was only carried out on viscosity grade binders. Figure 3-10 (c) shows the experimental setup for viscosity test.

(a) (b) (c)

Figure 3-10. Test setup (a) Penetration, (b) Softening point, and (c) Viscosity

d) Elastic recovery (ER) test: Elastic recovery test is used to determine the elasticity of polymer modified binders imparted by polymers used in it. This property tells about the amount of recovery of polymer modified binders when load is removed. Higher elastic recovery indicates the better performance of polymer modified binder against the distresses. In this study elastic recovery test was carried out on polymer modified binders containing WMA additives. Elastic recovery of modified binder can be evaluated by comparing thread recovery (after cutting in two equal half) when the specimen is elongated in ductility machine after conditioning of 1 hour at specified temperature (15^{o}C) for a deformation of up to 10 cm. This test was conducted on polymer modified binders using the guidelines mentioned in IS: 15462 [309]. The elastic recovery of WMA modified binder can be calculated using equation 3-1. The Figure 3-11 shows the experimental set up for the elastic recovery test

$$Elastic\ Recovery\ (\%) = \frac{10-X}{10} * 100 \qquad 3\text{-}1$$

Where,

X= length of recombined specimen after recovery (cm)

a) Initial stage b) Final stage

Figure 3-11. Experimental setup for elastic recovery test using ductilometer a) elongation phase b) recovery phase

e) **Performance grading (PG) using dynamic shear rheometer (DSR):** PG grading is the outcome of Strategic Highway Research Program (SHRP) carried out in USA. Superpave performance grading (PG) is based on the idea that an HMA asphalt binder's properties should be related to the conditions under which it is used. For asphalt binders, this involves expected climatic conditions as well as aging considerations. Therefore, the PG system specifies that a particular asphalt binder must pass these tests at specific temperatures that are dependent upon the specific climatic conditions in the area of use. The empirical nature of tests like penetration and softening point makes them unsuitable for accurately describing the viscoelastic behaviour of asphalt binders. Despite being essential, viscosity tests typically don't reveal information on the asphalt binders' time dependency. This issue with conventional tests lead to the development of the PG grading system. In this study, the high temperature PG was determine using dynamic shear rheometer, shown in Figure 3-12. The purpose of the test is to evaluate the temperature up to which all WMA modified binders suitable. Generally, the performance grade is indicated as PGXX-

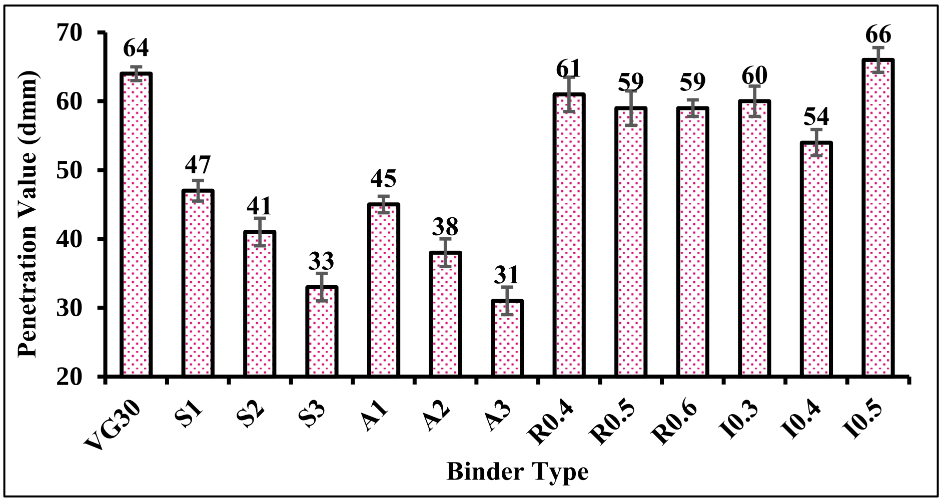
YY. In which XX represent the average seven-day maximum pavement temperature, whereas the YY is the expected minimum pavement temperature. For example, PG 70-10 indicates that the asphalt binder with this grade can be used where the average seven-day maximum pavement temperature is 70°C, and the minimum pavement temperature likely to be experienced is -10°C. In the present research work, only the high-temperature PG was evaluated. The performance grade was performed on the unaged asphalt binders considering the strain level of 12% and frequency of 1.59 Hz as per ASTM D7175. The test was performed using a parallel plate geometry with a 25 mm diameter and 1 mm gap width. The temperature at which G*/sinδ is greater than 1 kPa is considered as true failure temperature. The lowest PG corresponding to the true failure temperature is reported for the asphalt binder. G^* indicates the complex modulus and is defined as the ratio of maximum applied shear stress to the maximum value of the resulting strain. On the other hand, δ represents the phase lag between the shear stress and shear strain response of the asphalt binder when subjected to an oscillatory test in DSR. In this study, the true failure temperature was evaluated for all WMA modified binders.

Figure 3-12. Dynamic shear rheometer (DSR)

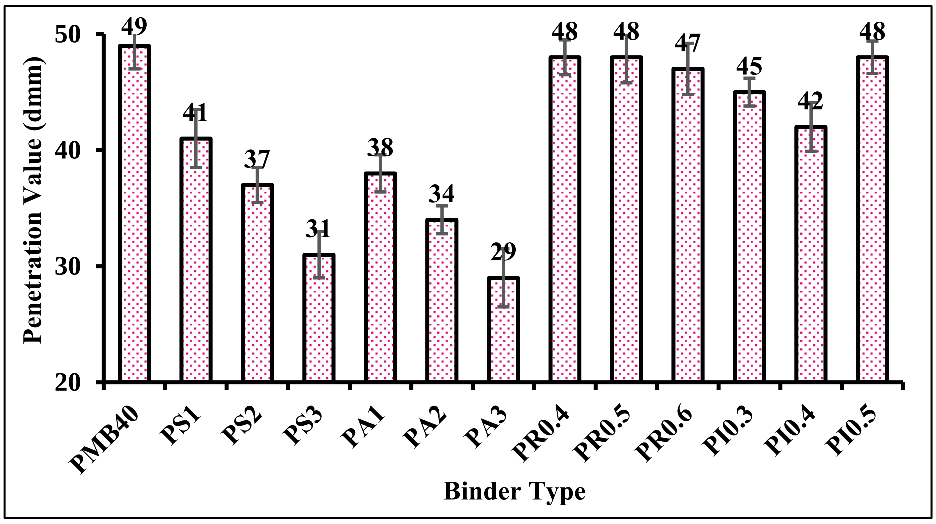
3.4.2 Results of Basic Properties of WMA Modified Binders

3.4.2.1 Effect of WMA Additives on Penetration Value

Figure 3-13 (a) and Figure 3-13 (b) presents the penetration test results for different WMA binders prepared with VG30 and PMB40, respectively. It can be seen that the addition of organic additives (Sasobit and Asphaltan A) reduced the penetration value whereas chemical additives (Rediset and Iterlow) did not show significant change in penetration values. These results were true for both the base asphalt binder. The addition of Sasobit and Asphaltan reduced the penetration value approximately by 25-50% for VG30 binder whereas it was reduced by 15-40% for PMB40 binder. The decrease in the penetration value was found to be increase with the increase in dosages of organic additives. This can be attributed to increase in the stiffness of base binders due to the formation of crystalline lattice structure of waxy particles in presence in Sasobit and Asphaltan A [338]. Remarkably, the effect of WMA additives was more prominent for VG30 than PMB40. This could be associated with the dominance of crosslinking polymeric network in PMB40 [201]. On the other hand, Rediset and Iterlow showed insignificant changes in penetration value for the both base binders. It can be seen that the increase in Rediset dosages reduces the penetration values whereas Iterlow reduces values up to dose of 0.4% and increases with higher dose of 0.5 %. This can be due to softening of the base binder due to addition of higher dosage of Iterlow. However, the results showed that the addition of chemical additives did not affects the stiffness of the base binders.



(a)

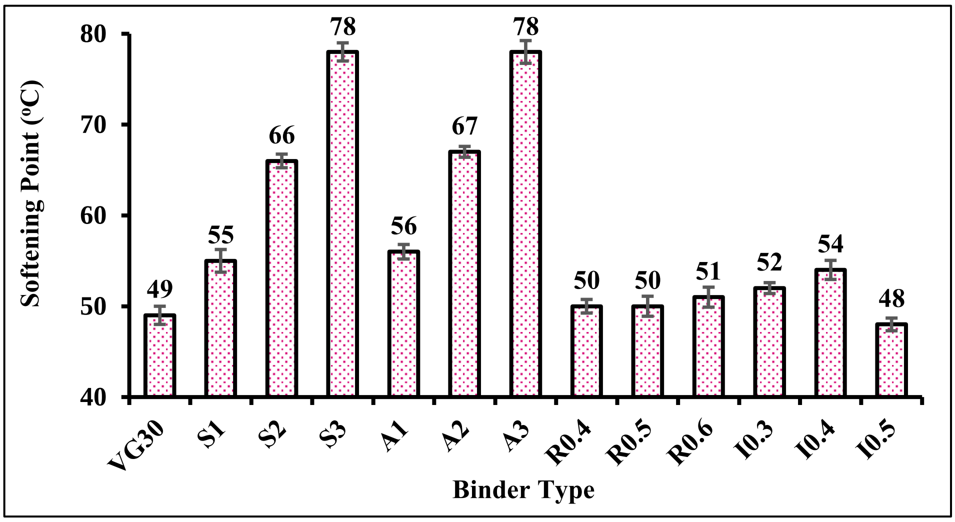


(b)

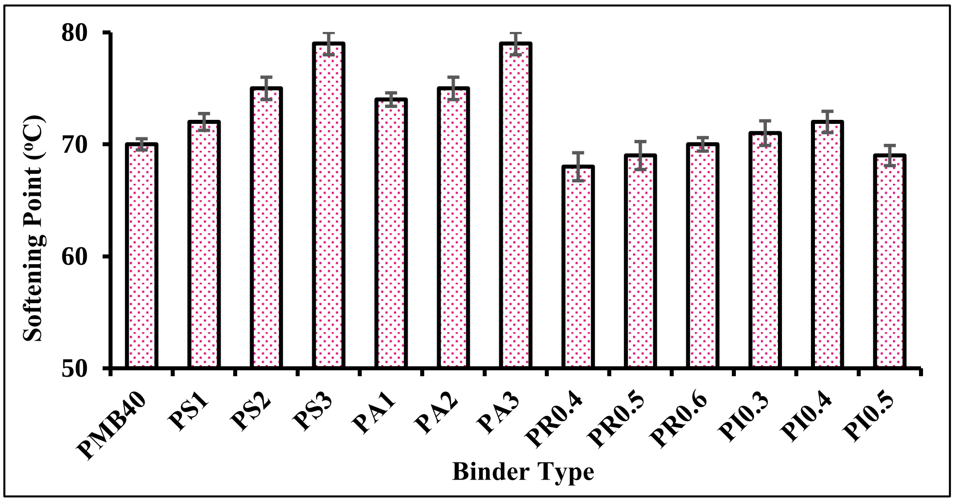
Figure 3-13. Penetration value of different WMA binders (a) VG30 and (b) PMB40

3.4.2.2 Effect of WMA Additives on Softening Point

Figure 3-14 (a) and Figure 3-14 (b) present the softening point results for different WMA binders prepared with VG30 and PMB40, respectively. It can be seen that the addition of organic additives (Sasobit and Asphaltan A) increased the softening value whereas chemical additives (Rediset and Iterlow) did not show significant change in softening point. These results were true for both the base asphalt binder. The addition of Sasobit and Asphaltan increased the softening point approximately by 10-50% for VG30 binder whereas it was increased by 5-15% for PMB40 binder. The softening point was found to be increased with the increase in dosages of organic additives. The higher value of softening point indicates the higher stiffness of binder indicating better performance of binder against rutting. The addition of chemical additives showed marginal improvement in the softening point of base asphalt binders. However, the improvement in softening point was found to be insignificant with addition of chemical additives.



(a)



(b)

Figure 3-14. Softening point of different WMA binders (a) VG30 and (b) PMB40

3.4.2.3 Effect of WMA Additives on Absolute Viscosity

Figure 3-15 presents the absolute viscosity values for different WMA binders prepared with VG30. The addition of Sasobit and Asphaltan increased the viscosity value of base binders. The values were increased with the increase in dosages of organic additives. The addition of organic additives increases the viscosity of base binder more than 100% at all the dosages. This can be attributed to increase in the stiffness of based binder due to fiber like reinforcement effect (crystallization effect) of wax present in the organic additives. On the other hand, both the chemical additives showed slight increase in the viscosity values of base binder. This can be due to the poly-functional function of both chemical additives, in which the stiffness is imparted by the crystallization component and the flow is influenced by the surfactant part [58]. The test was not performed on polymer modified binders due to clogging of Cannon-Manning tube due to the presence of polymers in their structure.

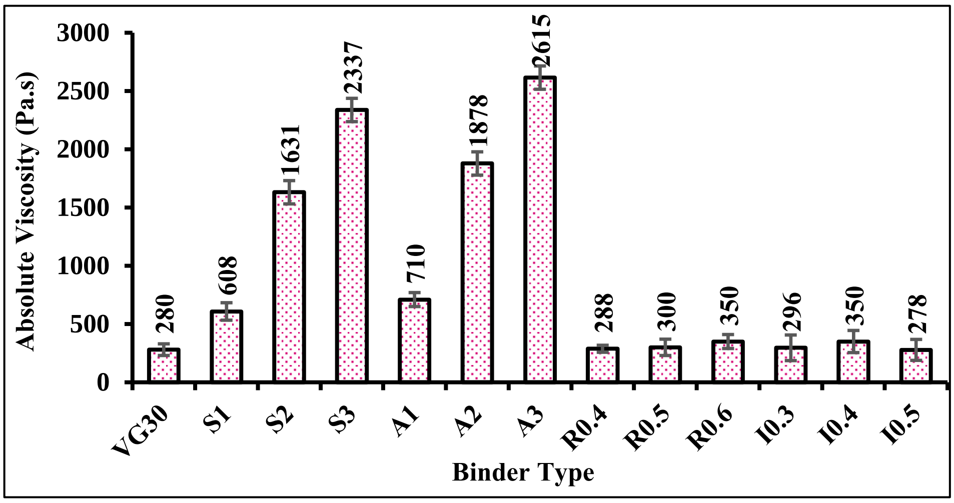


Figure 3-15. Effect of WMA additives on absolute viscosity of VG30

3.4.2.4 Effect of WMA Additives on Elastic Recovery

The effect of different WMA additives on the elastic recovery of PMB40 are shown in Figure 3-16. The minimum recovery value of 85% is desirable for better performance of polymers present in PG82 (PMB40 in this study) as mentioned in IS15462:2019. From the Figure 3-16, it can be seen that the elastic recovery of PMB40 increased with addition of Sasobit and Asphaltan A indicating better elastic properties against fatigue cracking. The improvement in elastic recovery was increased with increase in dosages of organic additives. However, the improvement was found to be insignificant with addition of organic additives. Only 5% improvement was observed with addition of higher dosages of organic additives. On the other hand, the chemical additives did not indicate any remarkable change in the elastic recovery values of base asphalt binder. The results of elastic recovery indicate that all WMA additives satisfy the criteria of minimum elastic recovery indicating that the addition of WMA additives did not influence the polymer network present in PMB40.

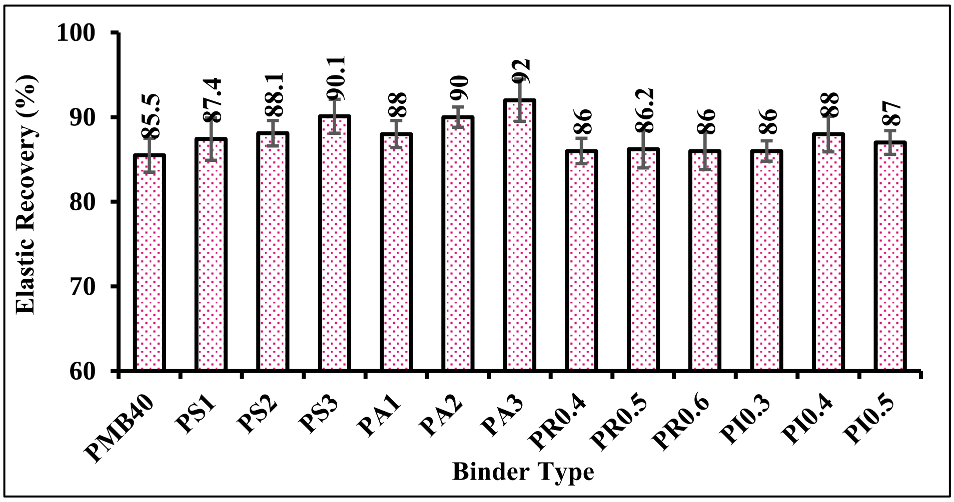
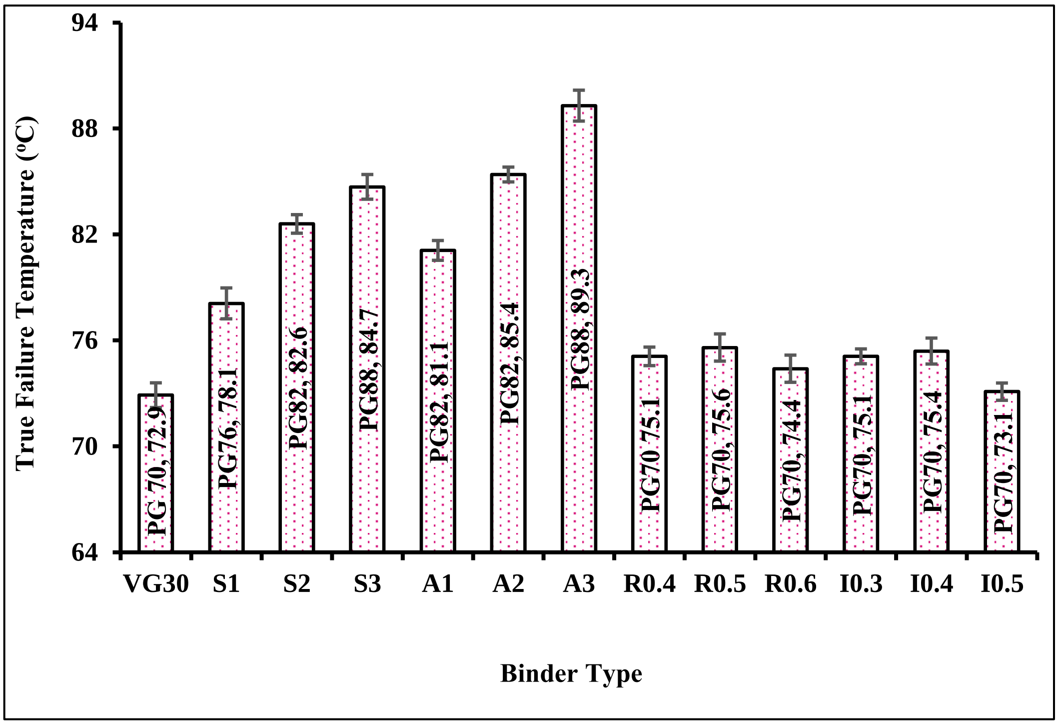


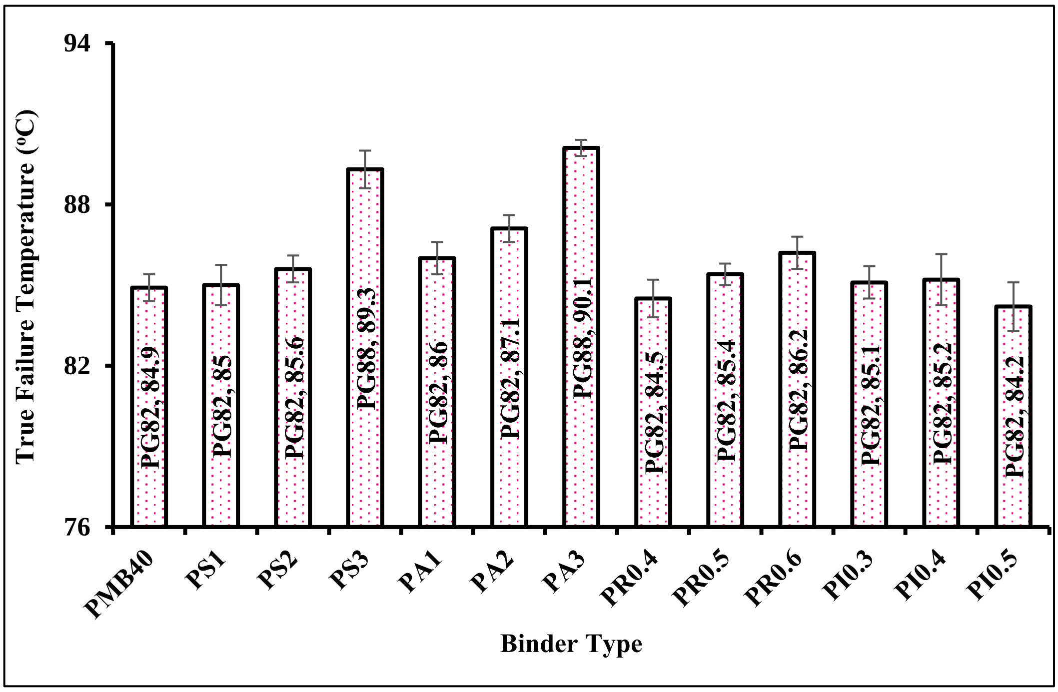
Figure 3-16. Effect of WMA additives on elastic recovery of PMB40.

3.4.2.5 Effect of WMA Additives on Performance Grade

In this study, the stiffness of WMA modified asphalt binders were assessed using the true high temperature performance grade based on rheological parameter, $G^*/\sin\delta$. This property of asphalt binder was related with the rutting performance of the binder. Figure 3-17 (a) and (b) shows the true high temperature performance grade based on $G^*/\sin\delta$ parameter at unaged condition for VG30 and PMB40, respectively. The improvement in the grade indicates the better performance of the asphalt binder against the rutting [339]. Two important observations were made from the results, first, the addition of the organic additives (Sasobit and Asphaltan A) improved the high temperature grade by one full grade with addition of 1% organic additives) and two full grades with addition of 2% and 3% organic additives to the VG30 binder. The grade was improved by one full grade in case of PMB40 with the addition of 2% and 3% organic additives. Although the true failure temperature was higher for 1% organic additives in PMB40, but lower dose (1%) of organic additives did not affect the grade of PMB40. Second, the addition of the chemical additive did not show any significant change in the high temperature grade for both base binders as they do not alter the mechanical properties of the binders. The improvement in the grade (from PG70 to PG 82 in case of VG30 and PG82 to PG88 in PMB40) due to the organic additive can be attributed to the crystallization effect of wax present in the additive structure which impart stiffness to the binder [52]. From the Figure 3-17, it can also be observed that the improvement in high temperature grade was function of base binder, the improvement in the grade was more prominent in case of VG30 than PMB40. The higher grade of PMB40 can be due to its higher stiffness as result of polymeric network of elastomers present in the PMB40 [340]. The results of PG grading showed that the addition of organic additives improved the grade of binder indicating improved rutting performance whereas chemical additives did not affect the rutting performance of the base binders.



(a)



(b)

Figure 3-17. True failure temperature and PG of different WMA binders (a) VG30 and (b) PMB40

3.4.3 Determination of Mixing and Compaction Temperatures

Compared to HMA, the application of WMA significantly reduces the production temperature by 20-40°C, thus reduces fuel consumption, and greenhouse gas emissions [2]. Despite lower production temperature, WMA extends hauling distance, improves workability and coatability of asphalt mixtures. Notably, the extension in hauling distance of asphalt mixture near to cold plateau zones reflects good social and economic benefits. The application of WMA also provides various technical and environmental benefits that leads to sustainable development. The successful implementation of WMA technology depends on the accurate determination of production temperatures (mixing and compaction temperature). Inappropriate determination of mixing and compaction temperatures lead to premature failure of the pavement [2,127,341]. Lower mixing temperature may lead to insufficient coating of the asphalt binder over the mineral aggregates, whereas lower compaction temperature can cause difficulty during field compaction, and may lower the in-field density [2,341]. On the other hand, higher production temperatures may result in oxidative hardening of the asphalt binder, leading to premature failure of the pavement [125,342]. Past studies have shown that there are no standards available for the determination of production temperatures (mixing and compaction temperature) of WMA [132,343]. Therefore, the various methods have been employed in this study to determine the mixing and compaction temperature of WMA modified asphalt binders. The viscosity of asphalt binder has been adopted to measure the mixing and compaction temperature of HMA. ASTM D2493 specify the guidelines for the measurement of mixing and compaction temperature and is purely based on the viscosity values obtained using Rotational Viscometer (RV) [133]. Figure 3-18 shows the Brookfield viscometer used for the test. The temperature corresponding to 0.17±0.002 Pa.s and 0.28 ±0.003 Pa.s is taken as mixing and compaction temperature, respectively, for the production of asphalt mixtures. Previous literatures stated that ASTM D2493 concept popularly known as equiviscous method is appropriate for determining the production temperatures for Newtonian fluids whose behaviour remain same with respect to shear rate [132,343] . Traditionally for the determination of mixing and compaction temperature of warm mix modified binders, the equi-viscous criteria are used. However, the researchers shown that the equiviscous criteria is fails to determine the production temperature of modified asphalt binder [135–137]. This is attributed to the shear thinning behaviour of modified asphalt binder where viscosity is function of shear rate. To address these issues with modified asphalt binders, researchers come up with new methods for appropriate determination of production temperature of modified asphalt binder. The various methods have been used for determining mixing and compaction temperatures of modified asphalt binder along with Equiviscous method (ASTM D2493) are discussed in Chapter 4.

Figure 3-18. Brookfield viscometer used for determination of MT and CT

3.4.4 Tribological Assessment of WMA Modified Binders

Few literatures adjudged that viscosity is not sufficient to estimate the production temperature, lubricity and overall workability of WMA modified asphalt binders. Past studies also showed that the mechanism of WMA technology can be related to reduction in friction at contact zone of aggregate and binder [18,186]. The reduction in friction can be associated with lubricating behaviour of WMA additives. The friction between mineral aggregate and asphalt binder can be studied using the science of tribology [18]. Tribology can be defined as the study of lubrication, friction, and contact zone between particles in relative motion. Mixing and densification of WMA asphalt mixture led to the reduction in friction, wherein mineral aggregate particles are in relative motion with a thin film of asphalt binder acting as the lubricating medium. In recent studies, the fundamentals of tribology has been successfully used to describe the such mechanism [15,193]. In this study the tribology test was performed to determine the coefficient of friction for understanding the lubricating behavior of WMA additives. The test was performed through a tribological fixture fixed on the MCR 102 DSR (from Anton Paar). The fixture consisted of a ball-on-three-plates geometry also known as ball-on-pyramid, shown in Figure 3-19. This test used the ball of radius 6.35 mm and the plates with $3 \times 6 \times 16$ mm^3 dimensions. Both balls and plates were exchangeable after each test. The main purpose of the test was to measure coefficient of friction for studying the lubricating behavior of the asphalt binder. The coefficient of friction μ was determined by imposing the axial load N and the sliding speed of different magnitudes. The different values of normal load and sliding speed result in different torque values and these values were used for the calculation of μ. The primary goal of the tribology test is to ascertain the coefficient of friction in order to comprehend the lubricating properties of WMA additives. The coefficient of friction (CoF) is not a material intrinsic property, but it is a property of a tribo-system that comprises at least two bodies in contact, along with the surrounding environment and the interface referred to as lubricant. In the pavement industry, asphalt binder acts as the interface (lubricant) between mineral aggregates. The frictional behavior of asphalt binders during the test is influenced by various parameters such as normal load, sliding speed, surface roughness, temperature, etc. To study the effect of parameters on the coefficient of friction, the normal load of (1N, 3N, 5N, and 10 N), and sliding speed of (0.05 m/s, 0.1 m/sec, and 0.3 m/sec) were selected for tribological assessment of WMA modified asphalt binders. The testing temperature was varied from 90°C to 160°C with an increment of 10°C. The selection of these temperatures covered the entire domain of the production temperatures for VG30, PMB40, WMA modified asphalt binders. The outcomes from tribology test were used to determine the mixing and compaction temperature of WMA modified binders. The detailed description of tribology test has been discussed in chapter 5.

(a) (b)

Figure 3-19. a) Ball-on-three plates arrangement mounted on DSR b) Schematic of the ball-on-three-plates test

3.4.5 *Validation of Tribology Based Approach*

To check the suitability of production temperature obtained from tribology-based approach, the validation of MT and CT is necessary. In this study, the tribology-based approach was validated with a workability test, coating ability test and compactibility test.

3.4.5.1 Workability Test

The workability test was used in this study to validate the MT and CT determined from tribology approach. Workability of asphalt mixture is defined as easiness in mixing and compaction of asphalt mixture. Workability is directly related to the production of asphalt mixtures. Past studies showed that the mechanism of WMA technology can be related to reduction in friction at contact zone of aggregate and binder [15,193]. The reduction in friction can be associated with improved workability of asphalt mixture. As workability is also related to the friction between aggregate and asphalt binders, so it can be used for the validation of the tribology approach. Mayank et al. developed workability test to overcome the drawbacks of the equi-viscous method for the determination of MT and CT in the case of modified asphalt binders [117]. The workability in the asphalt mixture was quantified in terms of the torque required to rotate the mixture. Using a similar concept, the workability of WMA modified binders was determined in this study. The torque measurement was done using a workability device fabricated in the lab. The single-sized aggregate gradation was used (9.5 mm-6.3 mm) for the quantification of workability. This was used to avoid the formation of the shear plane, where the fine aggregates get deposited at the edge of the container. The two types of aggregates: Granite and Dolerite were used for the preparation of asphalt mixtures. The technical details and detailed procedure adopted for the workability study can be found in Chapter 5. The power required to rotate the asphalt mixture was measured over the temperature ranges from 130°C to 160°C (at an interval of 5°C). The torque, which represents the workability of the mixture at different temperatures, is calculated using the following relationship given in Equation 3-2. For each sample, readings were taken 5 times at each temperature to assess the variability in the result. The average values were finally taken for further analysis. The torque-temperature profile for various WMA modified binders obtained from workability test was used for determination of MT and CT. These production temperatures obtained from workability-based approach were used for the validation of MT and CT obtained from the tribology-based approach

$$\tau = \frac{60\times P}{2\times\pi\times N} \qquad 3\text{-}2$$

Where,

τ = Torque generated in N-m

P = Power in watt

N= Speed of the shaft in rotation per minute (rpm)

3.4.5.2 Coating Ability Test

The coating test was performed to validate the mixing temperature determined from the tribology approach. At the mixing temperature, the asphalt binder is expected to sufficiently coat the aggregate particles. It is expected that all WMA modified binder show similar or better coating as compared to the base binders. The concept of coating index (CI) was used in this study to ensure coating at mixing temperature. The measured CI was normalized to the CI of the base binder to understand the impact of WMA, independent of base asphalt binder and aggregate. To measure the coating index, the image analysis was used. The image acquisition of asphalt mixture was carried out using experimental setup that was developed in laboratory. The experimental setup used for capturing image under constant illumination is shown in Figure 3-20. The setup was consisting of camera installed at top to capture the image of asphalt mixture. The camera was aligned perpendicular to horizontal plane so that it will capture image at same angle and height. The asphalt surface exhibits high reflectance behaviour which is in combination with dark colour causes difficulties in photography. It has been found that the lack of light causes an underexposed image in which texture is not capture in detailed whereas even more minimal illumination causes overexposed image due to reflectance of incident light. To overcome these difficulties, diffuse light source was used to captured detailed texture of coated and uncoated aggregates. In experimental setup, light source was mounted along the boundary at top of the setup. To reduce the subjectivity in this study, all images were captured under constant lighting condition. To ensure the same position of each asphalt mixture, the setup consists of arrangement that kept each asphalt mixture with same orientation and alignment. The coating test was carried out on the mixture prepared using WMA modified binders at their mixing temperatures using coating apparatus. The detailed determination of coating index has been discussed in Chapter 5.

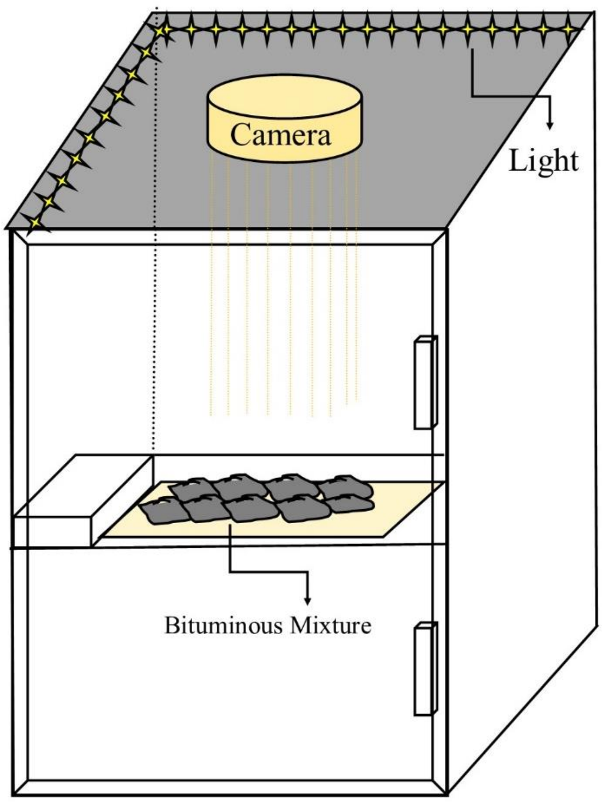


Figure 3-20. Schematic of coating apparatus

3.4.5.3 Compactibility Test

The compactibility test was performed to validate the compaction temperature determined from the tribology approach. At the compaction temperature, it is expected to have sufficient air voids and required density in asphalt mixture. It is expected that all WMA modified binder show similar or better compactibility as compared to the base binders. Air voids in a compacted mixture were taken as an indicator of compactibility. In this test, all WMA modified mixture were prepared at optimum binder content using the Marshall mix design according to Asphalt Institute specification (MS-2), utilizing bituminous concrete (BC) gradation with a nominal maximum size of aggregate of 19 mm, mentioned in Ministry of Road Transport and Highways (MoRTH) [344]. All WMA mixture were compacted at their compaction temperature determined using tribology test. After compaction, the air voids for all asphalt mixtures were computed and compared to check the effect of WMA on compactibility characteristics. The detailed information regarding the compactibility test has been provided in Chapter 5.

3.4.6 Aging of WMA Modified Binders

Exposure of pavement to high temperature, atmospheric oxygen and moisture, affects the durability of pavement. This is due to the changes in the behaviour of asphalt binder over the period of time [220]. There are evidences that supports the hardening or oxidation of asphalt binder occurring during construction and in service life of pavement [217]. This indicates an adverse influence on the performance of the compacted asphalt mixtures. Oxidation or hardening of bitumen, generally referred as aging, is defined as the change in behaviour of asphalt binder and mixture due to the shift in micro-structure and chemical composition during the construction and in service life at different environmental conditions. Aging occurs at both binder and mixture level, indicates a negative impact on the long-term performance of asphalt mixture [345]. Aging of asphalt binder affects the viscoelastic properties and leads to the development of micro cracks that is converted into macro cracks with the application of traffic. This premature failure of flexible pavement may be the function of aging process of asphalt binder [346]. In short, the durability of flexible pavement is recognized as the resistance against aging process.

The aging kinetics of bitumen is mainly explained by three mechanisms: Volatilization, exudation and oxidation [256,347]. The volatilization generally includes the loss of volatiles during construction of pavement. The second mechanism is physical hardening of bitumen referred as exudation. This is a reversible process caused by molecular rearrangement due to reduction in temperature. The exudation causes change in the physical properties of bitumen without affecting the chemical composition of bitumen. The third aging mechanism is oxidation that occurs partially during construction and in service life of pavement. This is irreversible phenomenon causes change in the chemical structure of bitumen. This is caused by an auto-oxidation of bitumen due to atmospheric oxygen, moisture and UV radiation. The oxidation leads to a conversion of non-polar to the highly polar group, thereby causing increases in molecular weight that subsequently cause hardening of bitumen [347]. Aging can be broadly characterised into two stages. The primary stage initializes at a very rapid rate during the production of asphalt mixture. This stage is referred as short-term aging (STA). STA is caused due to the loss of volatile components present in the asphalt binder and oxidation process at elevated temperatures. On the other hand, the second stage of aging is observed after the exposure of pavement to environmental oxygen and atmospheric moisture throughout its service life. The second stage mostly occurs after 8-10 years of construction and designated as long-term aging (LTA). Figure 3-21 shows the initialization of aging at different stages. It can be observed that the rate of oxidation in second phase is relatively lower than the first phase. The second phase indicates a linear increase with time.

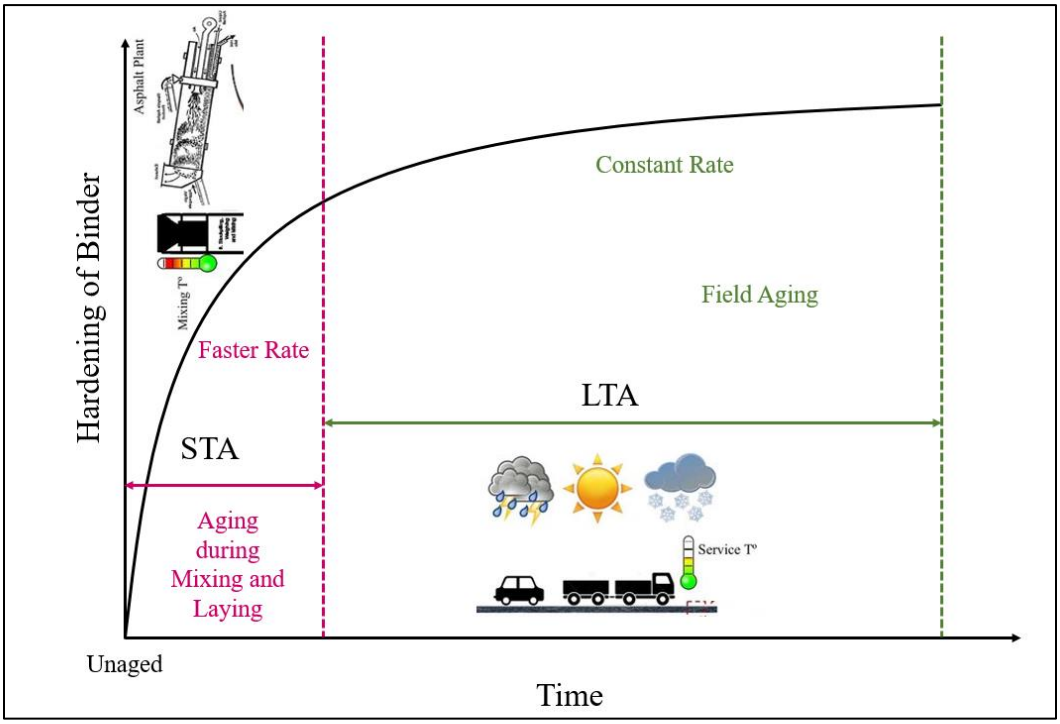


Figure 3-21. Short term aging and long term aging of asphalt binders

WMA has gained interest all over the world owing to their environmental and economic benefits. Various studies have been carried out to ascertain the behaviour of WMA at binder as well as mixture level to facilitate its implementation. WMA inclusive asphalt binder and mixtures exhibits reduced aging due to the lower production temperature as compared to HMA [21]. Various studies revealed that binders modified with WMA additives resulted in similar performance despite of reduced aging and concomitant oxidation. The reduced aging in case of WMA modified binder during the initial period may also affect the structural performance of the binders. Attributable to lower production temperature, Bennert et al. concluded that WMA becomes highly susceptible to rutting because of reduced aging and can possibly lead to distortion under heavy traffic loading [12,21]. On the other hand, previous studies also divulged that the reduced production temperature significantly improves the resistance to fatigue cracking of asphalt binders and mixtures due to lower aging of asphalt binders [348,349]. In addition, the comparison results associated with aging mechanism between HMA and WMA are sometimes contradictory in the previous literatures [21,34,228]. Notably the change in the performance at binder and mixtures level is dependent on the type of WMA additive and source of asphalt binder. It is noteworthy that change in the performance of WMA pavements over the time may be affected by numerous other factors such as increased traffic, asphalt film thickness, change in air voids, and moisture-induced damage. To this end, complete fundamental understanding of aging kinetics of WMA modified bitumen helps in increasing the durability of pavement. To understand the aging consequence, the aging of WMA modified binders has been carried out at the temperatures determined from the tribology approach.

The standard techniques for simulating STA and LTA of asphalt binders are a Rolling Thin Film Oven (RTFO) and a Pressurised Aging Vessel (PAV), respectively. Nonetheless, a number of other test techniques have been used in the literature to replicate the aging of asphalt binders. Western Research Institute (WRI) devised the Universal Simple Aging Test (USAT) technique, which was used in this study to simulate aging [350]. USAT uses a draft oven because it's easy to use and convenient for aging the asphalt binder. Farrer et al showed that the characteristics employed in USAT such as aging duration, aging temperature, and aging pressure are similar to the RTFO and PAV methods [350]. Details of the USAT protocol can be found elsewhere [350,351]. In this study, all the WMA modified asphalt binders were short-term aged (STA) using a thin film of binders (300 μm) kept in draft oven as per the universal simple aging test (USAT) protocol. According to protocol, the thin film of binder was kept in draft oven at fixed temperature of 150°C for hot mix asphalt and 130°C for warm mix asphalt for a period of 50 minutes. Farrar's study chose these two values considering that the average mixing temperatures of all the HMA and WMA binders were around 150°C and 130°C, respectively [350]. Since the present study involves the application of WMA technology, wherein the asphalt mixtures are produced at lower temperatures relative to conventional HMA mixtures, it is obvious that STA of WMA binders would occur at relatively lower temperatures.

Therefore, the short-term aging of WMA modified asphalt binders was carried out at the mixing temperature determined using the tribology approach. After STA, the long-term aging (LTA) of binders was also carried out in accordance with the USAT protocol. As per the protocol, the LTA of binders was carried in the draft oven at 100°C for a duration of 40 hrs. Asphalt binders with different aging conditions (i.e., unaged, STA, and LTA) were stored separately for further investigations. Figure 3-22 shows the schematic Figures of STA and LTA using USAT protocol.

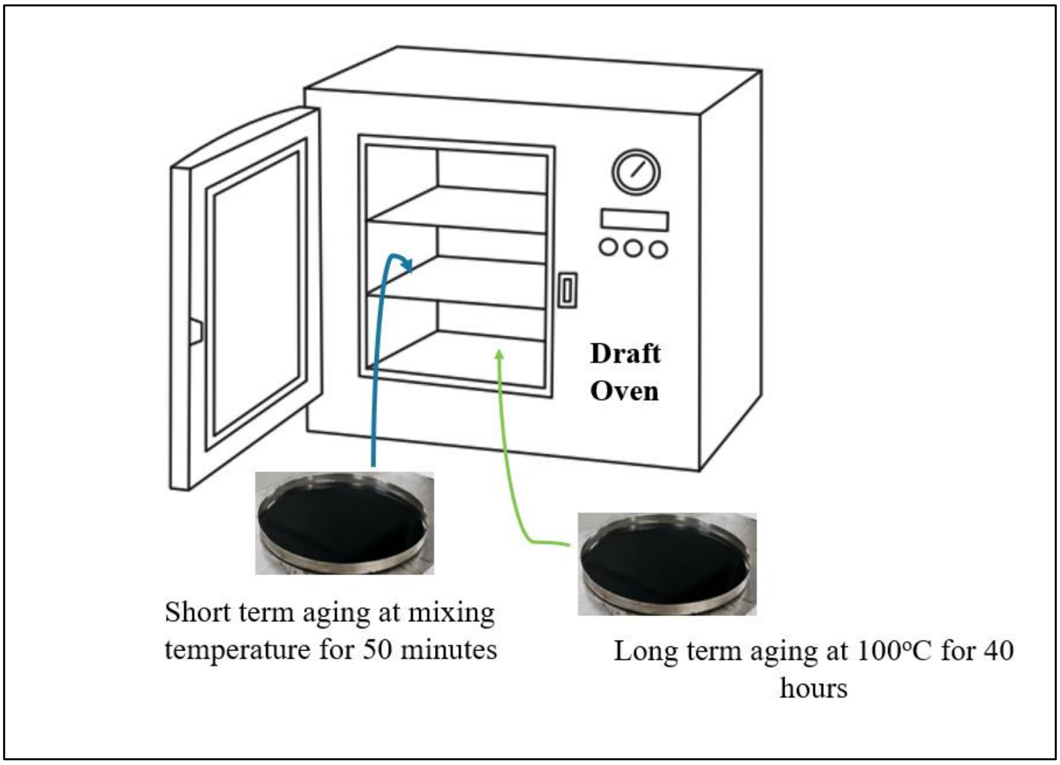


Figure 3-22. STA and LTA of asphalt binders using USAT protocol

3.4.7 Fourier Transform Infrared Spectroscopy (FTIR) Test

FTIR test was performed to determine the chemical composition of asphalt binders [352]. This was useful in understanding the effect of chemical composition of base binders on the performance of WMA modification. Also, it was used to assess the variations in chemical composition of the WMA modified binders after short term and long term aging [352]. In this study, FTIR test was done using Potassium Bromide (KBr) pellet method. Figure 3-23 shows the experimental setup for the FTIR test. To prepare the specimens for spectroscopy, a drop of 10 wt% (0.5 g of binder dissolved in 5 ml of carbon disulphide) was placed on the pellet, which was then allowed to dry for 10 minutes under an air blower. FTIR spectroscopy measures the amount of light absorbed at various infrared light wavelengths (400–4000 cm^{-1}) by placing the sample in a chamber to interrupt the beam coming from infrared source. The process includes measurement of absorbance in a range of wavenumbers between 400 cm^{-1} to 4000 cm^{-1}. This helps in understanding the presence of specific functional groups which belongs to a specified and well-defined wavenumber. The detailed procedure of KBr pellet method can be found elsewhere [353–355]. The purpose of the FTIR test was to understand the changes in chemical composition of all binders due to lower aging offered by WMA additives. The most significant functional groups affected by aging are the carbonyl group (C=O), and the sulfoxide group (S=O) [41,216]. It is found that these groups increase with aging. This can be due to the conversion of generic fractions (i.e., maltenes) into high molecular weight fractions (i.e., asphaltenes). Saturates (aliphatic), aromatics (naphthalene and polar), resins and asphaltenes are the components of asphalt binders [347]. The molecular weights of these constituents increase from saturates to asphaltenes. Asphalt binder age as a result of reactions between ambient oxygen and lower molecular weight molecules (Saturates and aromatics) that create subsequent higher molecular weight compounds (asphaltenes). The amount of aging experienced by asphalt binder is thus indicated by changes in the concentration of saturates and aromatics due to their oxidation. This may have resulted in increase in the binder's oxide groups such as sulfur dioxide and carbonyl. Therefore, in this study the amount of aging due to WMA additives has been analyzed by determining the changes in carbonyl group (C=O), and sulfoxide group (S=O). These functional groups were analysed with respect to the reference functional group to determine the aging resistance of all WMA binders. In this study, area between 1350 cm^{-1} and 1525 cm^{-1} wavelength range, was considered as reference area which remains stable during aging [356]. The carbonyl and sulfoxide index has been determined based on the area below the absorbance spectrum for the peaks of functional groups. Following equations (Equation 3-3 and 3-4) are used for calculation of these indices. The higher values of carbonyl and sulfoxide indicates the higher aging of asphalt binders. The results and discussions on the aging indices of different WMA asphalt binders are presented in chapter 6.

$$Carbonyl\ Index\ (I_{\mathrm{C=O}}) = \frac{A_{\mathrm{C=O}}}{A_{\mathrm{Ref}}} \quad 3\text{-}3$$

$$Sulfoxide\ Index\ (I_{\mathrm{S=O}}) = \frac{A_{\mathrm{S=O}}}{A_{\mathrm{Ref}}} \quad 3\text{-}4$$

Where,

$A_{C=O}$= area around carbonyl group (Area around 1700 cm^{-1})

$A_{S=O}$= area around sulfoxide group (Area around 1030 cm^{-1})

A_{Ref}= area around aliphatic group (Area between 1350 cm^{-1} and 1525 cm^{-1})

Figure 3-23. FTIR test setup

3.4.8 Performance of WMA Modified Binders

It is expected that WMA technology performs same or even better than HMA. If this objective is not achieved, then all of its above-mentioned benefits will be worthless. Till date, the previous literatures have shown that WMA perform same as that of HMA. But there are certain zones where WMA performance still needs to be enhanced [37,223,261,264]. The performance of WMA after aging is needs to be explored in greater depth since this may be the area where there are the greatest differences between HMA and WMA. Although the technology offer reduction in production temperatures with numerous benefits, but the implementation of the technology may create the potential issues. The most repeated concerns raised in the literature are related to reduced aging of WMA modified asphalt binders [261,264]. While offering reduced production temperatures, WMA technology minimizes the oxidative aging of the asphalt binder, which may possibly affect the structural performance of the binders Therefore, it is necessary to check the performance of asphalt binder against rutting, fatigue cracking, and moisture due to reduced aging offered by WMA additives. In this study, the various performance tests have been carried out on the warm mix modified binders to check their suitability. The selection of particular type of warm mix additive for different type of distresses has been decided based on the performance parameters determined from the tests. This section described various performance tests carried out on WMA binders.

3.4.8.1 Multiple Creep and Recovery (MSCR) Test

Rutting performance of WMA modified asphalt binders was evaluated using PG plus test called MSCR test. The test was conducted on short term aged WMA modified binders using DSR in accordance with AASHTO T350 [357]. The test was performed at the high working temperature of 40^{o}C, 50^{o}C, 60^{o}C and 70^{o}C using a 25-mm diameter spindle and a 1 mm gap. These temperatures correspond to the high pavement temperatures at which rutting could be the predominant failure. The test protocol consists 10 alternative cycles, each at stress level of

0.1 kPa and 3.2 kPa. Single cycle consists of 1 sec creep period and 9 sec rest period. The test was initiated with the lower stress level of 0.1 kPa and increased to 3.2 kPa stress level after 10 cycles without any rest period. The strain values were recorded during the MSCR test to determine non-recoverable creep compliance J_{nr} (kPa^{-1}) and elastic recovery R (%). These parameters at each stress level were averaged over ten cycles of creep and recovery. Figure 3-24 illustrates the typical result of MSCR test for one cycle. Equations 3-5 and 3-6 show the expression of J_{nr} and R (%) respectively. Three replicates of each combination were tested to guarantee the repeatability of the test. To ensure positive performance of the binder against rutting, it is desirable to have a lower value of non-recoverable creep compliance (Jnr) which indicates reduced permanent deformation under repeated loading and a higher percentage of elastic recovery (%R), reflecting the binder's ability to recover after deformation.

$$J_{nr} = \frac{\epsilon^{nr}}{\sigma} \qquad 3\text{-}5$$

$$\text{R (\%)} = \frac{\epsilon^{1} - \epsilon^{10}}{\epsilon^{1}} \qquad 3\text{-}6$$

Where, J_{nr} = non-recoverable creep compliance (kPa−1)

σ = applied stress level (kPa) during the creep loading

ϵ^{nr} = non-recoverable strain recorded at the end of the rest period

R (%) = percent recovery

ϵ^{1} = strain recorded at the end of 1 s

ϵ^{10} = strain recorded at the end of the 10-s creep phase.

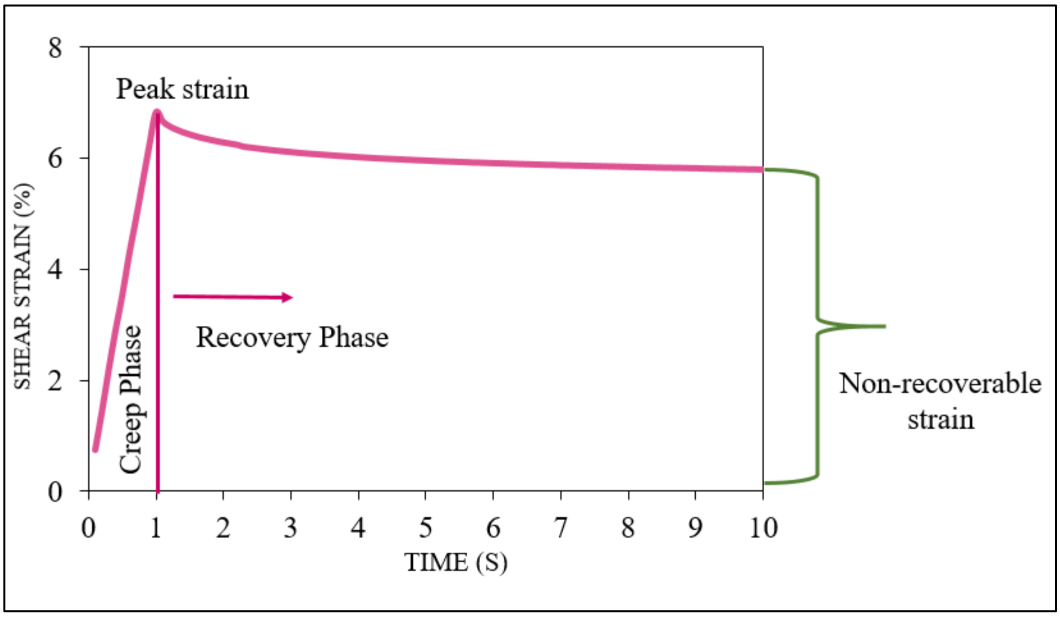


Figure 3-24. MSCR data for one cycle

In this study, the Jnr values at 3.2 kPa stress level were used to grade the WMA modified asphalt binder as per [358]. Table 3-7 shows the guidelines of binder gradation based on Jnr at 3.2 kPa for different levels of traffic and speed. The results and discussions regarding the effect of WMA additives on the J_{nr} and R (%) of base binders are presented in chapter 6.

Table 3-7. Asphalt binder grading based on J_{nr} at 3.2 kPa stress level

Traffic level (ESALs) and Speed (load rate)	**Designation**	**Interpretation**	**Recommended Jnr, kPa^{-1}**
>30 million and <20 kmph	E	Extremely high traffic	0-0.5
>30 million or < 20 kmph	V	Very High traffic	0.5-1
10-30 million or 20-70 kmph	H	High traffic	1-2
<10 million and > 70 kmph	S	Standard Traffic	2-4

Note: kmph denotes kilometre per hour.

3.4.8.2 Linear Amplitude Sweep (LAS) Test

LAS test was used for fatigue evaluation of WMA modified asphalt binders. This is an accelerated fatigue test performed on long term aged asphalt binders in accordance with

AASHTO TP101 [359]. The test was conducted using dynamic shear rheometer with an 8-mm diameter spindle and a 2-mm gap. The test was performed at three intermediate temperatures: 10°C, 20°C and 30°C. These temperatures were selected with a view that fatigue cracking is most expected to occur between these ranges, which corresponds to the intermediate pavement temperature. The analysis of LAS test is based on viscoelastic continuum damage (VECD) theory and details of the theory can be found elsewhere [270,360,361]. The fatigue assessment of asphalt binder is based on parameters derived from following fatigue performance model presented in Equation 3-7 and Equation 3-8. Three replicates of every combination were examined in order to guarantee repeatability in this assessment.

$$N_f = A(\gamma_p)^B \qquad 3\text{-}7$$

$$N_f = \frac{f\left(D_f\right)^k}{k(\pi C_1 C_2)^\alpha}(\gamma_p)^{-2\alpha} \qquad 3\text{-}8$$

Where, N_f = number of cycles to fatigue failure;

γ_p = Maximum strain induced in asphalt binder for given pavement structure (%);

A = $\frac{f\left(D_f\right)^k}{k(\pi C_1 C_2)^\alpha}$

B = -2α are the fatigue law parameters indicate asphalt binder properties;

$D_f = \frac{f(C_0 - C_{at\ peak\ stress})^k}{C_1}$ = damage at failure corresponding to peak stress;

k= 1 + (1-C_2)α and C_1 and C_2 are curve fitting coefficients.

The parameter “A” was determined from amplitude sweep test while parameter B was determined from frequency sweep test [362]. Therefore, the test was performed using a two-step procedure in accordance with AASHTO TP101. First, a frequency sweep test is done by applying oscillatory shear loading at a low stain amplitude of about 0.1% over a range of frequencies from 0.2 to 30 Hz. During this test, asphalt binder remains undamaged as strain of 0.1% is within linear viscoelastic limit. The output of this test is used to determine undamaged parameter α, which is used to calculate the fatigue law parameter "B" (B= -2α). The second step consists of amplitude sweep test which is performed by applying shear loading with increased strain amplitude from 0.1% to 30% in 300 secs at a frequency level of 10 Hz. This step uses VECD theory for calculation of fatigue law parameter "A" with the help of analysis from the frequency sweep test. In this study, the dissipated energy approach was used to calculate the damage and the details about it can be found in AASHTO TP101 [359,363]. The results and discussions regarding the effect of WMA additives on the fatigue life of base binders are presented in chapter 6.

3.4.8.3 Bitumen Bond Strength (BBS) Test

Bitumen bond strength (BBS) test is used to determine the bond strength between aggregate and asphalt binder [364]. In this study, this test was performed on all WMA modified binders by using Pneumatic Adhesion Tester (PAT) in accordance with AASHTO T361 [365]. A F-8 piston and zero mm cavity pull-out stub was used for asphalt bond strength evaluation. The test was conducted at room temperature by employing a normal force to pull-out stub which is adhered to an aggregate substrate. Figure 3-25 shows experimental setup for BBS test. The normal force was created by applying pneumatic pressure at rate of 100 psi/sec. The maximum pull-out pressure exerted by the instrument was recorded and defined as the pull-out tensile strength (POTS). The bond strength between asphalt binder and aggregate is indicated by POTS and calculated as follows:

$$POTS = \frac{(BP \times A_g) - C}{A_{PS}} \times 100 \qquad 3\text{-}9$$

In equation 3-9, A_g is the contact area of reaction plate with the gasket (mm^2); BP is the burst pressure (kPa); A_{ps} is the area of the pull-out stub (mm^2) and C is the piston constant.

Recently, this test gained more popularity for evaluation of moisture damage between binder and aggregate and found that the POTS value is good indicator of moisture damage [366–368]. Hence, test was conducted on both conditioned and unconditioned aggregate-binder samples. Dry conditioning was carried out by placing the sample in air for 24 hr prior to test while wet conditioning of samples was carried out in water bath at 40^oC for 24hr in accordance with AASHTO T361. After that, wet conditioned samples were kept at room temperature for 1 hr prior to test. To assess the effect of moisture conditioning, bond strength ratio (BSR) was determined. The Equation 3-10 shows expression of BSR. To ensure repeatability in this test, five replicates of each combination were tested. In this study, the failure pattern was also examined using micro camera to determine the mode of failure i.e. adhesive or cohesive. In a cohesive failure, the aggregate surface was not exposed which typically occurs within the asphalt binder indicating its internal strength. On other hand, an adhesive failure occurs at the interface of binder-aggregate system and hence the aggregate surface was exposed. Most of the times, the actual failure was a combination of both. The images of failure patterns has been discussed in Chapter 6.

$$BSR = \frac{POTS_{WET}}{POTS_{DRY}} \times 100 \qquad 3\text{-}10$$

Where, BSR= Bond strength ratio

$POTS_{DRY}$ = Pull-out tensile strength for unconditioned sample

$POTS_{WET}$ = Pull-out tensile strength for conditioned sample

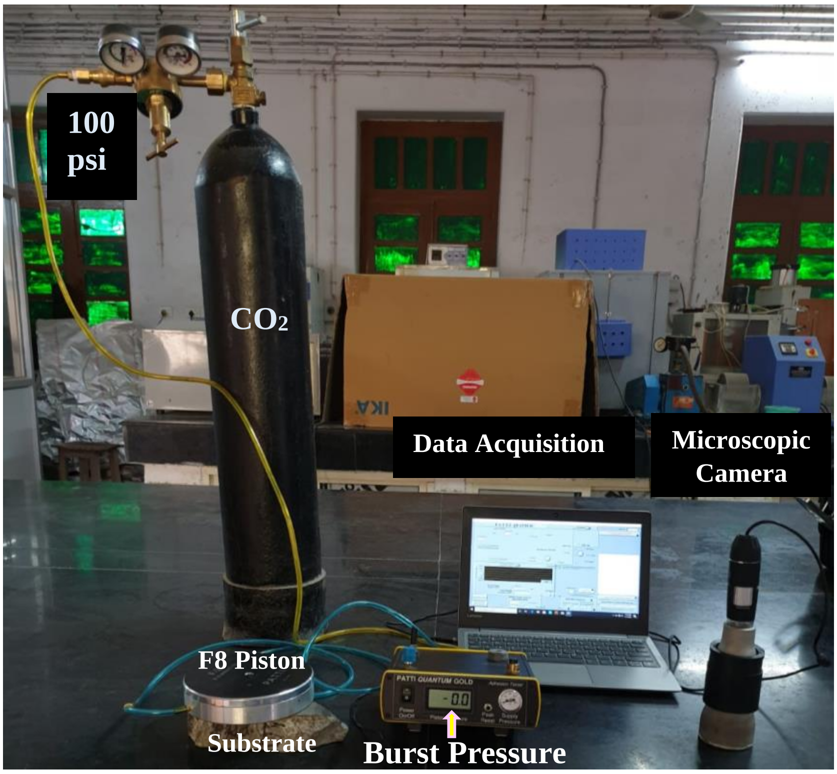


Figure 3-25. Bitumen bond strength test setup used for assessement of moisture damage of WMA modified binders

3.4.8.4 Sample Preparation for BBS Test

The aggregates used for this study were collected from local quarry. The five types of aggregates were selected for substrate preparation: Dolomite, Dolerite, Basalt, Granite, and Sandstone. The collected aggregates were cut using stone cutter in such way that it should accommodate enough number of pull-out stub on it. After cutting, the aggregates substrates were polished using 280 silicon carbide (SiC) grit mentioned in AASHTO T361. The polishing was done to remove irregularities on surface due to cutting. The polished substrate then cleans using ultrasound bath and placed in oven for drying. Thereafter, the prepared substrates along with pull-out stub were placed in oven at mixing temperature (suggested by AASHTO T361) for preparation of BBS test samples. The asphalt binder samples were prepared using silicon mould so as get samples of specific volume. After this, aggregate substrate and pull-out stub were removed from oven and prepared sample of binder was placed on stub for 10 sec. Then, binder sample was pressed perpendicular against aggregate substrate. Figure 3-26 shows steps for samples preparation for BBS test. This prepared aggregate-binder system with pull-out was subjected to wet and dry conditioning for further experimentation. The test was conducted on prepared samples using PAT in accordance with AASHTO T361. Figure 3-27 shows the samples prepared by considering various type of aggregates for BBS test.

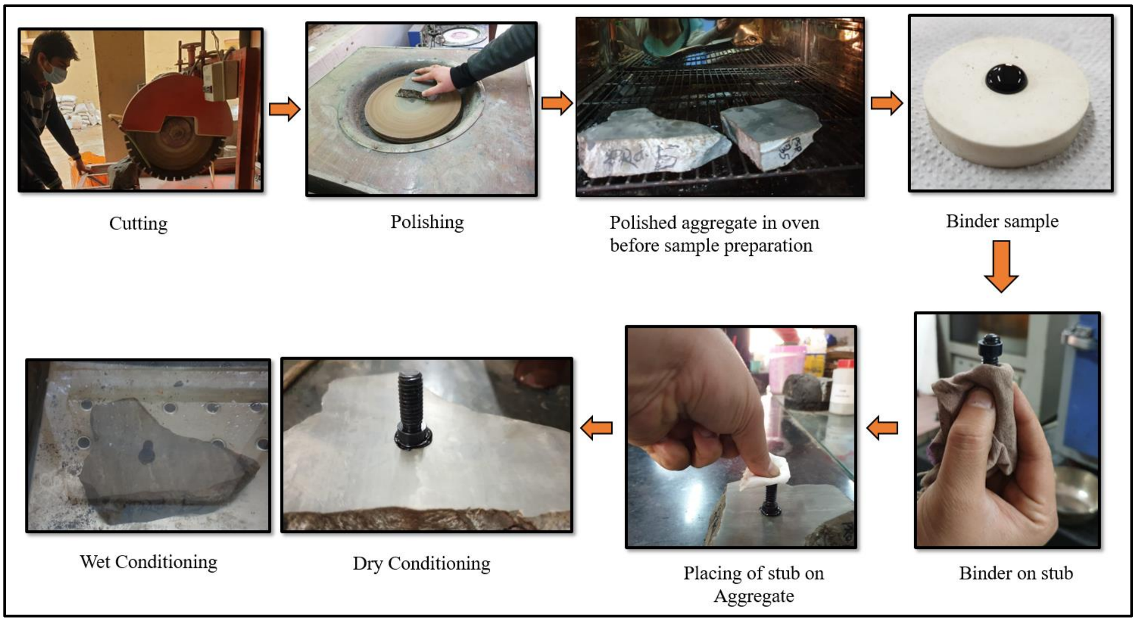


Figure 3-26. Sample preparation for BBS test

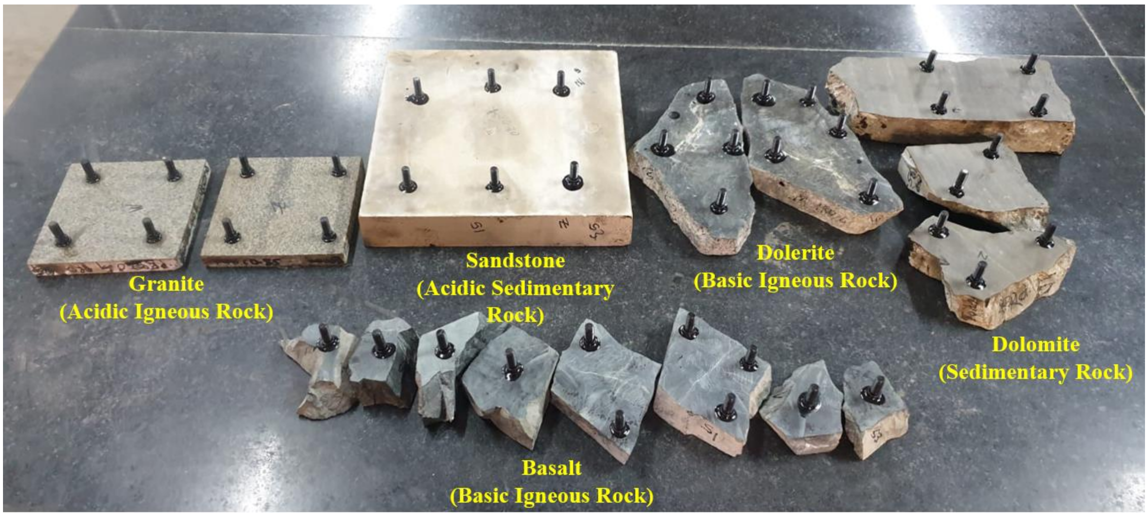


Figure 3-27. Sample for BBS test by considering different aggregate types

3.5 Phase 4: Strategy for Selection of WMA Additives

The selection of particular WMA additive is majorly based on its performance characteristics. It is required that the WMA additive should improve the performance of the binder against rutting, fatigue cracking, and moisture damage. The selection of WMA additive for particular location or project is a function of service conditions such as traffic loading and ambient temperatures [369]. The location is prone to rutting when it has higher traffic loading and ambient temperature exceeds 40^{o}C. On the other hand, fatigue cracking is more likely to occur at intermediate temperatures where the binder exhibits a higher stiffness. The moisture damage can be a serious concern at the location where rainfall is high, improper surface drainage, and aggregates used for the construction are highly siliceous in nature. In this study, the warm mix additives were selected based on the performance requirement of the location. From the earlier discussion, it has been found that the performance of the WMA additive is a function of base binder. It can be seen that the effect of WMA modification found to be more prominent with VG30 than PMB40. Therefore, the selection of WMA additive is also greatly influenced by the type of base binder. The different locations according to the predominant distress has been considered for selection of WMA additive based on their performance. The WMA additive selection framework adopted in this study is based on the assumption that the construction practices followed for the different locations are identical. Ranking framework has been adopted in the study to rank the WMA additives for their selection corresponding to the types of distress. Ranking was carried out separately for both the base binders. This can be due to their different interaction with WMA additives. While ranking the additives, performance of WMA and the primary function of WMA additive i.e. reduction in production temperature was considered. It is desirable that the additives should ensure superior performance against distresses with maximum temperature reduction. The additive with this behaviour can be considered as the best additive. The detailed discussion regarding the selection of WMA additives according to their temperature reduction and performance has been presented in Chapter 7.

3.6 Summary

The materials and experimental methodology adopted in this research work were described in depth in this chapter. A viscosity graded (VG) binder, VG-30, and a polymer modified binder, PMB-40, were used as base binder in this study to prepare WMA modified asphalt binders. Four different warm mix additives (WMA): two organic additives and two chemical additives were selected for the research work. Organic additives selected for the study are: Sasobit, and Asphaltan A. Chemical additives selected for the study are: Rediset, and Iterlow. These warm mix additives were used to prepare different warm mix modified asphalt binders. The additives were mixed at different doses with VG 30 and PMB 40 binders as per the manufacturer's instructions. Three basic aggregates i.e. Dolomite, Dolerite, and Basalt and two acidic aggregates i.e. Granite and Sandstone were used to study the moisture sensitivity of WMA additives. Following the preparation of WMA modified binders, the various tests has been carried out on binders to determine the production temperatures. This study also covers the use of tribology-based approach for determination of MT and CT asphalt binder blended with WMA additives. The influence of WMA additives on the aging behavior of asphalt binders was evaluated regardless of the type of base binder. Unaged, STA, and LTA asphalt binders were subjected to FTIR in order to evaluate the influence of WMA additions on the aging properties of asphalt binders. The performance of WMA modified asphalt binder has been studied using various performance test such as MSCR test, LAS test, and BBS test for selection of WMA additives. Some primary material characterization results were also described in this chapter. It was found that the addition of organic additives increased the softening point, viscosity, and true fail temperature and reduced the penetration value, irrespective of the base asphalt binder. The effect of dosages of organic additives have been clearly seen through the primary material characterization. On the other hand, the chemical additives did not show significant changes in basic properties of the base binders.