CHAPTER 2

REVIEW OF THE LITERATURE

2.1 Preamble

In this chapter, literatures pertaining to the utilization of fillers in bituminous mastics and mixes are discussed in detail. Various requirements for a material to be good filler as per Indian (MoRTH, 2013) and other pavement specification is discussed in initial section. The focus is on analyzing influence of various physical and chemical properties of fillers as well as their quantity on bituminous mastics and mixes. Primary pavement distresses (rutting, cracking, moisture sensitivity, and ageing), their responsible mechanisms, major affecting factors, various test methods for their evaluation, and influence of fillers on them is then reviewed. Finally, the performance of various mixes made with 30 different waste materials as alternative fillers was reviewed. This section helps to identify the gaps in current literature and to devise suitable research methodology.

2.2 Role of Filler

2.2.1 Specifications of Filler

Filler is an integral part of the bituminous concrete mix which can be defined as the finest part of aggregates. As per the ASTM D242 (2009), the filler consists of "finely divided mineral matter," which should be dry enough to flow and have no agglomerations. According to the clause 505.2.4 of the current Indian mix design guidelines (MoRTH, 2013), filler should consists of finely divided mineral matter (such as rock dust, hydrated lime, or cement), which can be measured by 75μm sieve and have gradation within limits indicated in Table 2.1. It should be free from organic impurities and shouldn’t have plasticity index greater than 4 (except for cement and hydrated lime). For research purposes, Indian researchers consider filler as the mineral matter which is finer than 0.075 mm sieve (Chandra and Choudhary, 2013; Kuity et al., 2014; Mistry and Roy, 2018; Sharma et al., 2010).

Table 2.1 Gradation for mineral filler (MoRTH, 2013)

IS Sieve (mm)	Cumulative percent passing by weight of total aggregates
0.6	100
0.3	95-100
0.075	85-100

The optimum quantity of filler in the mixture is decided by filler bitumen ratio, which according to MoRTH (2013), should be in the range of 0.6-1.2 by weight of the aggregates. The other global specifications (ASTM D242, Asphalt Institute, 2014) also prescribed the similar allowable range. However, some European and Asian guidelines also suggest different permissible range of filler bitumen ratios for optimum mix design. Material specification of Portugal, JAE (Junta Autónoma de Estradas), has prescribed the allowable range filler bitumen ratio to be 1.1-1.5, while Chinese specification JTG F-40 (2004) has suggested that the filler bitumen ratio of mixes should be in the range 0.6-1.5 (Matos et al., 2014). German specification TL Asphalt-StB 07 has recommended the range of filler bitumen ratio as 1.2-1.8 in mass (Rochlani et al., 2019; TL Asphalt-StB 07, 2007). Hence this area of research is open for wide speculation.

Various specifications (AASHTO M17, ASTM D242, EN 13043, MoRTH, 2013) has established the selection criteria for fillers based on the primary characteristics such as particle size distribution, plasticity index, water content and organic content (Melotti et al., 2013). These properties might be good enough for the general quality control for filler, but they are not sufficient to predict the expected performance of bituminous mastic and mixes. Various studies have observed the influence of several other physical (particle gradation, shape, size, surface area, texture, specific gravity, and porosity) and chemical (hydrophobic nature, clay content, chemical composition, mineralogical composition, and loss on ignition) properties of fillers on the behavior of bituminous mastics and mixes. This problem is more pronounced for alternative fillers, which often exhibit unique traits. Hence there is a need to improve the selection criteria for filler in Indian specification by adding simple, reliable, and inexpensive test methods.

2.2.2 Influence of Fillers on Bituminous Mastic and Mixes

Intentional use of filler in bituminous mixes was traced back to DeSmedt in the 1870s. Until 1893, filler was added in the bituminous mixes to fill the interstices between aggregates to improve their density and impermeability (Richardson, 1905). Richardson (1905) has implied that role of filler in bituminous mixes was not just void-filling, but some physicochemical phenomenon exists in the filler-bitumen system, which increases the stiffness of bitumen. Einstein (1911) has hypothesized that the inclusion of filler in bitumen filler suspension linearly increases its stiffness with a rate specified as the Einstein coefficient. Later it was understood that filler plays dual roles in mixes. They not only primarily act as inert material which fill interstices between larger aggregates in mixes but also finer particles of filler along with bitumen form bituminous mastic. Bituminous mastic or simply mastic is the particulate composite material in which bitumen, the continuous phase or bitumen is mixed with filler particles. Mastic is thermorheologically simple viscoelastic material which coats the aggregates in the mix to hold the mix together (Anderson and Goetz, 1973; Huang et al., 2006). The behavior of the mastic affects nearly every aspect of bituminous mixture design, construction, and performance.

Several studies have observed a good influence of physical and chemical properties of fillers and performance of bituminous mastics (Buttlar et al., 1999; Das and Singh, 2018; Kim et al., 2003; Rigden 1947; Smith and Hesp, 2000). The fine filler particles stiffen mastic and improve the strength and density of the bituminous mixes. During the design of bituminous mixes, the mastic affects lubrication of the aggregate particles and thus influences its voids in the mineral aggregate, compaction properties, and optimum bitumen content (Huang et al., 2007; Zulkati et al., 2012). Finer particles of filler having a size smaller than the thickness of bitumen film also act as bitumen extender in mastic and make mix behave such as if there is additional bitumen present (Chandra and Choudhary, 2013). This can reduce the optimum bitumen content of the mix, which ultimately can reduce the cost of the pavement. However, if not properly monitored, this behavior can also result in problems like stability loss, bitumen bleeding, and fat spots. During the construction of bituminous pavements, the mastic should have adequate stiffness to prevent drain-down (downward migration of the mastic due to gravitational forces during storage and handling). This aspect is particularly important in the case of open and gap graded mixes like stone mastic asphalt (SMA) mixtures. However, mastic should also not be excessively stiff to hinder proper compaction. The stiffness of the mastic in the field also influence the ability of the mixes to resist permanent deformation at higher temperatures, stress development and fatigue resistance at intermediate temperatures, and stress development and fracture resistance at low temperatures (Airey et al., 2006; Buttlar et al., 1999; Wang et al., 2011). Furthermore, depending upon their mineralogical composition, fillers act as anti-stripping agents preventing moisture damage, or they also can make their bituminous mixes more moisture susceptible due to their interaction with bitumen and aggregates (Chandra and Choudhary, 2013; Lesueur et al., 2013). Finally based on its physical and chemical nature, the filler also catalyzes or inhibit the diffusion of oxygen in the mastic and thus affect the ageing of the bituminous mastic and the mixes (Gubler et al., 1999; Lesueur et al., 2016; Moraes and Bahia, 2015). The primary properties of the fillers and their primary testing specifications are stated in Table 2.2.

Table 2.2 Influence of filler characteristics and quantity on the bituminous mastic and mixes

Name of the test	Test specification	Parameters obtained
Specific gravity	ASTM D854-14 (2014)	Specific gravity
Particle size distribution	ASTM D422-63 (2007)	Particle size distribution curve
		D_{10}, D_{60} and D_{30} values
Blaine's air permeability test	IS 4031-2 (1999)	Specific surface area of filler
BET Surface Area Test	ASTM C1069-09 (2014)	
Methylene blue value (MBV) test	ISSA (1989); EN 933-9 (2009)	Active clay content in filler
Plasticity Index	IS 2720 (Part 5) (1985)	
Fractional voids (Rigden voids)	EN 1097-4 (2008)	Porosity of filler
German filler test	NAPA (1999)	
Scanning Electron Microscopy	ASTM E986-04 (2017)	Size, Shape and Texture
X-Ray Diffraction (XRD)	ASTM C1365-18 (2018)	Mineralogical composition of the filler
X-Ray Fluoroscopy (XRF)	ASTM E1621-13 (2013)	Chemical composition of the filler
Energy Dispersive X-Ray spectroscopy (EDS)	ASTM E1508-12a (1209)	Elemental composition of the filler
Hydrophilic Coefficient	JTG E42 (2005)	The affinity of filler towards water and bitumen
pH Value	ASTM D4972-19 (2019)	Acidic/basic nature of the filler

It is necessary to provide an adequate amount of filler in the mixes to ensure satisfactory performance of mixes. Several studies have observed that the increase in filler content in the mix show increase in Marshall stability, rutting resistance, indirect tensile strength as well as lowering of optimum bitumen content (OBC) (Akbulut et al., 2012; Chandra and Choudhary, 2013; Huang et al., 2007; Sharma et al., 2010). However, the excessively high filler content can also lead to lower moisture resistance, lower fatigue life, and poor workability of the mixes (Chandra and Choudhary, 2013). Table 2.3 discussed the influence of various characterization properties of filler and their quantity on the performance of the bituminous mastic and mixes.

Table 2.3 Influence of filler properties and quantity on the bituminous mastic and mixes

Filler parameter	Inferences	Reference
Rigden Voids (RV)/ porosity	Fillers having high Rigden voids (RV) can convert free bitumen into structural bitumen (bitumen that fills the voids among filler particles), thus creating a stiffer structure of bitumen filler mastic which may result in higher rutting resistance of bituminous mix.	Rigden (1947); Faheem et al., (2012); Kandhal et al. (1998);Wang et al. (2011)
	Fillers having excessively high RV have a detrimental effect over workability and compaction properties of bituminous mixes.	Brown and Cooley (1999); Brown et al., (2009); Melloti et al., (2013)
	Rigden voids of fillers failed to correlate with rutting potential of bituminous mixes	Mogawar and Stuart (1996)
Particle shape	The particle shape of filler significantly affects permanent deformation of bituminous mix.	Tayebali et al. (1998)
	Fillers having large particle size and regular shape acts as friction lubricant agent by acting as tiny rollers during the compaction process, thus resulting in lowering of compaction resistance of bituminous mix.	Zulkati et al. (2012)
	Sphericity/roundness of filler particles has significant influence over the viscosity of bituminous mastic.	Grabowski & Wilanowicz (2007)
	Non-spherical filler particles increase shear strength	Al-Hdabi

Filler parameter	Inferences	Reference
	and stiffness of bitumen which in turn increases resistance to plastic flow of bituminous mix and improves its Marshall stability.	(2016)
	Fillers having irregular shape may negatively affect workability and fluidity of bituminous mix	Melloti et al., (2013)
	Geometrical irregularity of filler could lead to a decrease in effective bitumen which may cause detrimental effect over stripping resistance of bituminous mix.	Pasandin et al., (2016)
	Morphological parameters of fillers like angularity index, average diameter, and fractal dimension influence rutting resistance of mastics at high temperature and fatigue life of mastics at intermediate temperatures	Xing et al., (2019)
Particle size	Finer filler particles can act as bitumen extender which may lead to the production of over-rich mixes which can cause problems such as loss of stability, bleeding, and high rutting and flushing.	Kandhal et al., (1998)
	Filler having low D_{60} values stiffens the bitumen and thus increases rutting resistance of bituminous mix.	Kandhal et al., (1998)
	Bituminous mixes prepared with finer filler have higher stiffening which increases stripping resistance.	Kandhal et al., (1998)
	At similar filler bitumen ratio, fillers having medium and coarse particles improved rutting and fatigue resistance of bituminous mix.	Muniandy et al., (2013)
Particle size distribution (gradation)	The particle size distribution of filler influences the stiffening level of bituminous mastic.	Brown et al. (1996)
	Fillers having homogenous particle increases mastic viscosity & rutting resistance of bituminous mixes.	Clopotel and Bahia (2013)
Particle texture	Filler having rough surface texture has higher bitumen adsorption, which increases the stiffness of the mastic and forms high strength mixes.	Craus et al. (1978)
	Mixes prepared with filler having a rough texture and high surface area have high OBC.	Zulkati et al., (2012)
	Fillers with rough texture improves the rutting resistance of the bituminous mixes	Chen et al., (2011)
	Aggregates and filler with rough texture provide act as an anchor for the bitumen for adherence which increases the moisture resistance of the mix.	Zhu et al., (2000)
Surface area	The larger surface area of filler absorbs a higher quantity of bitumen which influences the performance of bituminous mix.	Antunes et al.,(2016);Clopotel and Bahia (2013); Taylor (2007)
	Interaction between bitumen and filler due to its higher specific area influence compaction properties and void content of the bituminous mix.	Zulkati et al. (2012)

Filler parameter	Inferences	Reference
	The higher surface area of filler may also improve bitumen-aggregate interaction that subsequently enhance resilient modulus of the mixes	Modarres and Rahmanzadeh (2014)
Specific gravity	Fillers with lower specific gravity occupy higher volume in the compacted mix and leave lesser space for bitumen to be accommodated, thus reducing OBC of the mix.	West and James (2006); Korayem et al., (2018)
	Fillers with lower specific gravity form mastics with higher viscosity, higher complex modulus, and lower phase angles at intermediate temperatures which may increase their fatigue resistance. However, this effect diminished at the higher temperature due to the increase in the effect of test temperatures.	Xu et al., (2019)
	Filler with lower specific had higher adsorption that resulted in the formation of moisture-resistant mixes.	Chen et al., (2011a)
Chemical and mineralogical composition	Hydrated lime and some other basic fillers improved the moisture resistance of bituminous mix by interacting with carboxylic acid present in bitumen and forming water-insoluble calcium salts.	Little and Jones (2003); Read & Whiteoak (2003)
	Hydrated lime slows down bitumen ageing due to the acid-base reaction between polar molecules of bitumen and lime surface.	Petersen et al. (1987)
	Fillers with strong bases like $Ca(OH)_2$, CaO and $Mg(OH)_2.Ca(OH)_2$ reduces ageing of bitumen. Fillers with weak base like $Mg(OH)_2$ doesn't reduce ageing.	Lesueur et al. (2016)
	Siliceous fillers form weak mechanical (Vanderwall force) bond with bitumen, while basic fillers like lime form strong chemical bond with bitumen.	Antunes et al., (2015)
	Basic fillers (hydrated lime and limestone) improved tensile bond strength of mastic than acidic filler.	Jakarni (2012)
	CaO content in filler displayed good correlation with the rutting potential of bituminous mixes.	Wang et al. (2011)
	In the case of bitumen emulsion-filler system, the basic filler displayed higher stiffness due to their ability to rise the pH of the system which lead to faster breaking or development of cohesion in the emulsion	Robati et al., (2015)
	Pozzolanic compounds like SiO_2 & Al_2O_3 with lime in the presence of water can form cementitious compounds & can improve moisture resistance of mix.	Modarres and Rahmanzadeh (2014)
	Chemical composition of filler doesn't have any direct influence over stiffening of bituminous mastic.	Antunes et al., (2015)
	Excessive plastic fines may increase the fatigue susceptibility of the bituminous mix.	Kandhal et al., (1998)
	Excessive plastic fines may increase the moisture susceptibility of the bituminous mix.	Kandhal et al., (1998); Chandra and

Filler parameter	Inferences	Reference
		Choudhary (2013)
	Good physiochemical interaction between bitumen and filler can improve fatigue life of mix by providing better resistance to micro cracking by lowering the rate of damage evolution and damage accumulation.	Kim et al., (2003)
	Mineralogy of mineral fillers has significant influence over active and passive adhesion between aggregate and bitumen.	Pasandin and Perez (2015)
	Presence of minerals such as mica, chlorite and hydrated iron oxides in filler may significantly lower the performance of bituminous mix against moisture sensitivity and freeze-thaw action.	Loorents and Said (2009)
	Chemical composition of fly ash and other coal combustion products as the filler has a significant influence on stiffening potential of bitumen filler mastic. Higher quantities of CaO, SO_3, and LOI (loss in ignition) increased the stiffening rate and formed stiffer mastics. Increase in Al_2O_3 and SiO_2 decreases the stiffening of mastic.	Bautista et al. (2015)
	Filler stiffening effect variation depends on the filler mineralogy and their concentration in the mastic	Faheem and Bahia (2010); Winniford (1961)
	Optimum filler bitumen ratio of mix is dependent upon surface mineralogy of aggregate and surrounding temperature.	Zejiao et al., (2017)
Filler quantity	A higher concentration of filler may result in stronger bituminous mixes due to improvement in mix's cohesively and internal stability caused by good packing.	Tunnicliff (1967); Tayebali et al., (1998)
	An excessive amount of filler may require a higher amount of bitumen to coat aggregates completely, which may weaken the bituminous mix.	Kandhal et al., (1998)
	Mixes having an excessive amount of fillers may find difficulty in mixing and compaction.	Dukatz and Anderson (1980); Matos et al., (2014)
	OBC of bituminous mixes decreases with increase in filler content. Higher filler content in mix requires less amount of bitumen to form the same amount of mastic to lubricate the aggregates.	Chandra and Choudhary (2013); Huang et al. (2007); Kandhal et al., (1998); Tayebali et

Filler parameter	Inferences	Reference
		al., (1998)
	Excessive filler content in bituminous mix may negatively affect fracture behavior of pavement by causing brittle failure and by promoting oxidation and hardening of bitumen.	Kandhal et al., (1998)
	Quantity of filler influences viscoelastic properties of bitumen and affect the stiffness of bituminous mixes.	Zulkati et al. (2012)
	Increase in viscosity due to the inclusion of filler decreases the severity of short-term aging of bitumen	Lesueur et al. (2016)
	Increase in filler bitumen ratio increases the stiffness of the mastic as well as the rutting resistance and resilient modulus of the bituminous mixes	Diab and Enieb (2018)
	Filler type and filler content have a significant influence on the moisture sensitivity of the bituminous mixes.	Aljassar et al. (2004)
	Cracking resistance of mixes increases with the increase in the filler content due to improvement in the strength of mastic by increasing concentration of filler.	Huang et al. (2007); Diab and Enieb (2018)
	Fatigue resistance of bituminous mixes decreases with the increase in the filler content in mix. This is due to the increase in the brittle nature of mastic by the inclusion of a larger amount of filler.	Huang et al. (2007)
	Fatigue resistance of bituminous mastic may increase with the increase in the filler content. Despite the stiffening of the bitumen inclusion of filler may provide better resistance against microcracking due to lower rate of damage evolution and higher capability of total damage accumulation	Kim et al., (2003)
	Moisture susceptibility of bituminous mixes increases with the increase in filler content due to subsequent lowering of their OBC.	Chandra and Choudhary (2013); Huang et al. (2007)
	Increase in filler bitumen ratio increases the viscosity of mastic, which requires more compaction energy to produce uniformly compacted bituminous mixes.	Anani et al., (1989)
	Rheological behaviour of bituminous mastic at low and high temperatures is significantly influenced by the volumetric composition of filler.	Lackner and Spiegl (2005)
	If the volumetric concentration of filler is kept lower than the critical concentration, the addition of filler lead to the moderate decrease in failure strain as well as a significant increase in the stiffness modulus, which ultimately improve the fatigue response of the mixture.	Miro et al., (2017)

2.3 Primary Distresses of Bituminous Pavements

2.3.1 Rutting

2.3.1.1 *Mechanism of Rutting*

Permanent deformation/Rutting can be identified as a longitudinal surface depression with or without transverse displacement along the wheel path. The bituminous layer in the pavement undergoes deformation due to application of wheel load. This deformation consists of two components the recoverable deformation and non-recoverable (plastic deformation). This plastic deformation gets accumulated after each load repetition and manifests in the form of rutting. The rutting mechanisms (Figure 2.1) can be explained as (a) one-dimensional inelastic displacement (densification), (b) two-dimensional inelastic displacement (vertical or lateral flow).

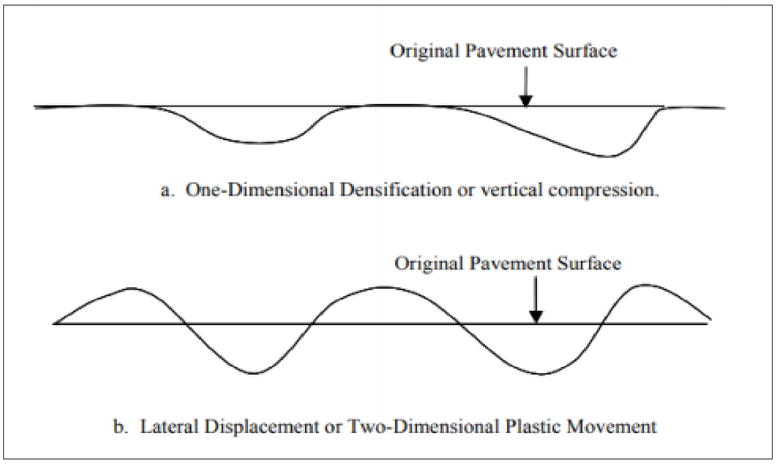

Figure 2.1 Mechanisms of rutting failure (NCHRP 1-37 A, 2004)

The densification of pavement layers and/or the consolidation of unbound granular layer or soil lead to the vertical inelastic deformation in the pavements. At high pavement temperature and due to excessive compressive stresses from wheel load, bituminous mixes become more susceptible to additional densification. On the other hand, lateral deformation is caused due to the local shear failure by virtue of the application of excessive tyre pressures. This usually attributed to the inadequate shear strength or low void contents in the bituminous mix. The mixes compacted to the lower air void contents (less than 3%) and have excess bitumen content resulted in the loss of the inter-granular friction in between the aggregates that lead to higher chances of lateral flow (Hodges 2002; Huber and Heiman 1987). The Sousa et al., (1994) has compiled a list of various influential factors and their effect on the rutting behavior of the bituminous mixes which is stated in Table 2.4

Table 2.4 Various factors affecting rutting resistance of the mixes (Sousa et al., 1994; Radhakrishnan, 2017)

Material/test condition	Factor	Change in variable	Effect of change on the rutting resistance
Aggregate	Surface Texture	Smooth to Rough	Increase
	Shape	Rounded to Angular	Increase
	Gradation	Gap to Continuous	Increase
	Size	Increase in Maximum Size	Increase
Bitumen	Stiffness	Increase	Increase
Mixture	Air void content	Increase	Decrease
	Bitumen content	Increase	Decrease
	VMA	Increase	Decrease
Test/Field Condition	Temperature	Increase	Decrease
	Tyre contact pressure	Increase	Decrease
	Load Repetition	Increase	Decrease
	Water	Dry to wet	Decrease if mix is moisture sensitive

2.3.1.2 *Evaluation of Rutting Resistance of Bituminous Mastics*

The rutting resistance of bitumen and the bituminous mastic majorly influence the performance of their bituminous concrete mix. There are several test methods/parameters to assess the rutting susceptibility of the bitumen. However, as per the author's knowledge, there is no well-defined method exist which can specifically be used to determine rutting resistance of mastics. So, the testing protocols defined for the bitumen are usually followed for the assessment of the bituminous mastics by the researchers (Kim et al., 2003; Das and Singh, 2017). The rutting resistance of bitumen is usually determined using the Superpave rutting parameter (G*/sinδ) (G* is Complex shear modulus and δ is the phase angle) determined as per the strategic highway research program guidelines (Anderson and Kennedy, 1993; Kennedy et al., 1994). This parameter is determined by applying a cyclic oscillatory shear stress/strain (within the linear viscoelastic limit of bitumen) on a bitumen sample and observing its responses at different temperatures and frequencies using a Dynamic Shear Rheometer (DSR). Bitumen/mastic sample having higher rutting parameter found to show higher rutting resistance and thus sample with higher G* and lower δ is desirable. The minimum value of G*/sinδ for unaged and short-term aged bitumen was specified as 1 and 2.2 kPa, respectively (Zaniewski and Pumphrey, 2004). The specification is laid out in AASHTO M 320 and found to be well correlated with the rutting resistance of the bituminous mixes. (Dongre et al., 2007; Stuart et al., 1999). However, Superpave rutting parameter was found to be insufficient to determine rutting performance of modified bitumen, since they exhibit delayed elasticity and exhibit lower amount of plastic strain than unmodified bitumen at similar loading and temperature conditions (Bahia et al., 2001; Bouldin et al., 2001; Carswell and Green, 2000; Dongre and D'Angelo, 2003).

The need for the evaluation of bitumen responses in non-linear viscoelastic domain is primarily highlighted by Bahia et al. (1999) by providing several reasons as follow. (a) Different types of bitumen which display similar behavior in linear domain can behave differently in non-linear domain.

(b) The fixing of bitumen parameter entails the consideration of pavement structure. In general, the weak flexible pavement structure allows more deformation and larger strain, as compared to the flexible overlay over rigid concrete bases. Hence, the definition of strain level is necessary to define their behavior in non-linear domain.

c) In non-linear domain, the strain is accumulated much faster (in both rutting and cracking), with each traffic load application.

d) The modified bitumen is not homogenous but exists as a multi-phase system, hence its behavior depends upon the magnitude of the applied stress.

e) The bitumen parameters evaluated in linear domain did not correlated well with mixture properties.

Considering all aforesaid reasons, Bahia et al. (2001) proposed Repeated Creep and Recovery (RCR) test for the analysis of bitumen. The parameters obtained from this test were found to be well correlated with the large number of mixes (Hrdlicka and Tandon 2007).

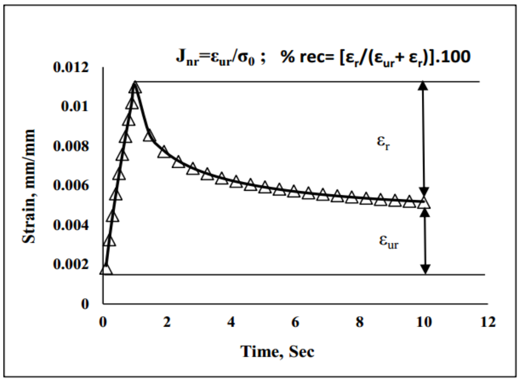

Figure 2.2 Schematic representation of a creep and recovery cycle (Saboo and Kumar, 2016)

D'Angelo et al. (2007, 2010) proposed a new test method known as "multiple stress creep and recovery test (MSCR)" to introduce the non-linearity associated with modified bitumen. This method was introduced as a part of new Superpave grading system (AASHTO MP 19-10). Various laboratory and field investigations have proved this method to be applicable for both unmodified and modified bitumen as well as in mastics (Dongre and D'Angelo, 2003; Marasteanu, 2005; Saboo and Kumar, 2016; Wang and Zhang, 2014; Xing et al., 2019). MSCR test is conducted as per the procedure stated in AASHTO T 350. This test is performed using a DSR with 25 mm diameter spindle having 1 mm gap. The test is typically conducted at 64°C on rolling thin film oven (RTFO) samples. The sample is subjected to ten cycles of creep loading and unloading of 1 second and 9 second duration respectively, at stress levels of 0.1 kPa and 3.2 kPa. The test utilizes two parameters, non-recoverable creep compliance (J_{nr}) and percent recovery (%R) to evaluate the rutting resistance of bitumen and mastics. The lower J_{nr} and higher %R value of samples signifies their better rutting resistance. Figure 2.2 shows a classical creep and recovery curve for a single cycle and the corresponding calculations. However, the average of all the ten cycles at each stress level is used in practice. Over years, several researchers (Oliver and Tredrea, 1998; Dongre and D'Angelo, 2003; Marasteanu, 2005; Wang and Zhang, 2014; Saboo and Kumar, 2016) have predicted the rutting susceptibility of bitumen using MSCR test.

2.3.1.3 *Evaluation of Rutting Resistance of Bituminous Mixes*

Indian mix design guideline (MoRTH, 2013) doesn't specify any performance based method to assess rutting resistance of the bituminous mixes. MoRTH (2013) prescribed an empirical parameter known as Marshall quotient to form mixes with adequate stiffness and prevent excessive brittleness. It is the ratio of Marshall stability (kN) to flow (mm) value of compacted Marshall specimen at failure. Marshall quotient value displays superior stiffness as well as better load distribution capability, which ultimately results in its improved resistance against creep or rutting. Marshall quotient has been used by various researchers to assess the rutting resistance of different types of bituminous mixes (Arabani et al., 2017; Bostancioğlu and Oruç, 2016; Moghaddam et al., 2014; Whiteoak, 1991). MoRTH (2013) suggested an allowable range of Marshall quotient (2-5 kN/mm) for dense graded bituminous mixes.

Extensive research works have been carried out in the past to determine appropriate method for deciding rutting resistance of bituminous mixes in the lab, which can also correlate well with field conditions. The static creep test is one of the simplest and inexpensive tests used for evaluation of rutting resistance of bituminous mixes. In creep test, the vertical strain in the specimen subjected to creep load at a constant temperature is measured during and after the application of the load and the residual strain is then calculated. The static creep tests can be done in uni-axial mode or in triaxial state of stress, which applies confining stress. However, in the majority of cases, it is performed in uni-axial mode. Static/repeated creep has been used by several researchers to evaluate the rutting resistance of bituminous mixes (Dongre et al., 2009; Faheem et al., 2005; Kaloush et al., 2002; Kandhal and Cooley, 2003; Sousa et al., 1991; Witczak et al., 2002). Since pavement undergoes repeated traffic loading, the repeated load permanent deformation test is more simulative of field loading condition. The test consists of applying a repeated compressive haversine loading (0.1 s loading time and 0.9 s rest period) and measures accumulated permanent strain as a function of load cycles. This test protocol is usually performed as per protocol given in NCHRP 9-19 project report.

Apart from the above mentioned destructive tests (static and dynamic creep), evaluation of the rutting resistance can also done using parameters obtained from non destructive tests like Dynamic modulus test. Dynamic modulus test measures the stress-strain relationship of compacted specimen under a continuous sinusoidal loading. It is an indicator of the viscous property of the material which can be related to the permanent deformation characteristics of the mix. Various non-destructive test parameters determined by this test at high temperatures like dynamic modulus (E*), phase angle (φ) and E*/sinφ has been used by researchers to evaluate rutting resistance of the mixes (Pellinen and Witczak, 2002; Witczak et al., 2002; Shenoy and Romero, 2002; Walubita et al., 2012; Zhang et al., 2013; Hou et al., 2016). Several researchers have also determined the rutting resistance of bituminous mixes by measuring the permanent strain accumulated in repeated simple shear tests at constant height (Tayebali et al., 1999; Brown et al., 2001; Sousa et al., 2002).

Apart from aforesaid mentioned test parameters, Wheel tracking can also be effectively used for quantifying the rutting resistance of bituminous mixes, the test results of wheel rut testing are often reported as pass or fail specification. The wheel rut testing can be performed either in a dry state or in submerged state. The results can also be reported in term of dynamic stability, rut depth, number of passes at maximum impression, creep slope, strip slope, and maximum inflection point (Chandra and Choudhary, 2013; Katamine, 2000). There are several protocols of wheel rut testing designed by several different agencies used to determine rutting resistance of bituminous mixes. Few of which are, Georgia loaded wheel tester, Asphalt Pavement Analyzer (APA), Hamburg wheel rut tester, French rutting tester, Purdue university laboratory wheel tracking device (PURWheel), Wessex dry wheel tracker, HEART wheel load simulator, IIT-KGP wheel tracker, Dry wheel tracker, KNTU wheel rut tester, etc.

2.3.1.4 *Effect of Fillers on Rutting Susceptibility of Bituminous Mastic and Mixes*

The rutting resistance of bituminous mixes is dependent on the stiffness of bitumen, volumetric properties of mixes, and the bonding interaction between mastic and the aggregates. Several early studies (Einstein 1911; Rigden 1947; Winniford 1961;

Anderson et al., 1983; Buttlar et al., 1999) have suggested that the incorporation of filler in the mix tends to stiffen the bitumen and affect the stiffness of the mastic. There are three mechanisms responsible for the stiffening of the mastic, which are stated as volumetric reinforcement, physicochemical interaction, particle interaction reinforcement (Buttlar et al., 1999). The stiffening of the mastic by all these mechanisms depends upon various physical (particle size, shape, texture, gradation, and porosity) and chemical characteristics (mineralogy and chemical nature) of the filler as well as on their relative proportion in bituminous mixes.

Rigden (1947) had studied different filler characteristics which could influence the stiffening of mastic at low and high filler concentrations. He has calculated the bulk volumes of the dry compacted specimen and termed the bitumen required to fill the voids in the dry compacted state as "fixed" bitumen while the bitumen in excess to this volume as free bitumen. He named this concept as "fractional voids concept" and suggested that the volume of voids in the filler in the dry compacted state alone affect the flow behavior of mastic and optimum bitumen content of the mixes. He also suggested that the stiffening of the mastic is independent of the chemical differences between the fillers. The influence of Rigden Voids (RV) on the stiffening of bituminous mastics and mixes were later verified by other studies as well (Antunes et al., 2015; Faheem et al., 2012; Wang et al., 2011). Stiffening effect of mastic prepared with filler and modified bitumen were found to be significantly affected by RV in fillers and somewhat affected by fineness modulus and CaO content of filler (Wang et al., 2011). Mogawer and Stuart (1996) in the contrary did not find any correlation between rutting potential and Rigden voids.

The portion of mineral filler finer than the thickness of bitumen film thickness blends with bitumen and strengthens mastic, which contributes to improved rutting resistance of mix. Kavussi and Hicks (1997) have stated that viscosity of filler-bitumen mastic is attributed to the particle size of filler since finest blends were found to have the highest viscosity value at a given temperature. Tayebali et al., (1998) stated that rutting resistance of bituminous mix is also affected by particle shape and size of the filler. Kandhal et al., (1998) had found correlations between rutting resistance of bituminous mastic and D_{60} (particle size corresponding to 60% passing of filler), and active clay content in the filler. Fillers with lower D_{60} and higher clay content formed higher rut resistant mixes. Arabani et al., (2017) have found that the fine size and angular shape of filler particles might be responsible for the higher rutting resistance of their mixes. Chandra and Choudhary (2013) have also suggested that the fillers having angular particle form mixes with higher rutting resistance. Xing et al., (2019) have investigated the effect of ten different morphological parameters of fillers on the rheological parameters of bituminous mastics at high temperatures using MSCR test. The percentage recovery of bituminous mastics was found to be highly sensitive to porosity, angularity index, average diameter, aspect ratio, and fractal diameter of the filler particles. Some studies have observed that the larger specific surface area of filler led to higher absorption of polar groups of bitumen (asphaltenes and resins) which is responsible for change in mechanical (stiffness and viscosity) as well as thermo-volumetric (coefficient of thermal contraction) properties (Antunes et al., 2015; Clopotel and Bahia, 2013).

Tunnicliff (1960) postulated that the mineral fillers have the same shape, size, surface texture, and size distribution, but different mineralogy or surface chemistry may result in different stiffening effects. Bautista et al., (2015) has studied the mastics prepared with coal combustion products and concluded that the stiffening of the bituminous mastic is dependent on both physical and chemical properties of the fillers. They have concluded that the fillers having higher Rigden voids and lower D_{10} display higher stiffening. While the other physical properties such as specific gravity, fineness modulus, and the surface area don't have significant influence on the stiffness. They have also observed that the increase in the quantities of CaO, SO_3, and loss on ignition tends to increase the stiffening rate of the mastic, while the increase in the quantities of Al_2O_3, and SiO_2 soften the mastic. Das and Singh (2017) have analyzed the rutting resistance of mastics prepared with the combination of basalt filler with the different amount of hydrated lime. It was observed that the increase in the proportion of lime tends to increase the rutting resistance of the mastic due to the agglomeration tendency of lime particles in the mastic.

2.3.2 Cracking

2.3.2.1 *Mechanism of Cracking*

The fatigue in bituminous layers manifests itself in the form of cracking under repeated traffic loading. Fatigue failure is a three-stage process which includes: crack initiation (development of microcracks), crack propagation (development of macrocracks from the microcracks), disintegration (catastrophic failure of the material due to unstable crack growth) (Smith and Hesp, 2000). It initiates with adhesive and cohesive micro-cracking at the bottom of the layer, which propagates upwards as micro-cracks grow and coalesce (Figure 2.3). These appear in the form of interconnected alligator cracks which are usually referred to as a bottom-up cracking.

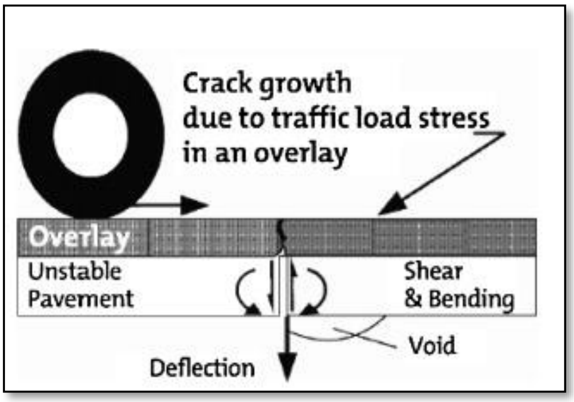

Figure 2.3 Mechanism of fatigue cracking (Arabani and Mirabdolazimi, 2011)

On the other hand, the cracking which initiates from the surface of the bituminous layer and propagates towards the bottom is determined as top-down cracking. It is primarily caused due to age hardening and the consequent brittleness of bitumen in the upper few mm of the top layer. Another reason for top down cracking is the application of high tensile stresses near the edges of tyres and the application of horizontal surface stresses due to acceleration and braking of vehicles. The predominance of top-down and bottom-up cracking depends on whether tensile stresses/strains is at the top or bottom of the bituminous layer that will be exceeding the allowable flexural tensile stress or strain. Usually, there is a higher probability for the initiation of top down cracking in the thin pavement, whereas, cracks are more likely to start from the bottom in the thicker pavements. The third type of cracking, which is usually considered as a less matter of concern in the majority of parts of India is low-temperature cracking. It is caused due to the restraint provided to the contraction of the bituminous layer, which caused the resulting thermal stress and strain to exceed the tensile strength and failure strain of the material. It can be initiated by the combination of factors such as traffic loading, number of cycles of change in temperature and even by a single significant drop in temperature. The considerable differences in the day-night temperatures can also generate cracks which started from the top and moved to the bottom (Ferne, 2006).

2.3.2.2 *Evaluation of Fatigue Characteristics of Bituminous Mastics*

Similar to the rutting resistance, there are no well-defined protocols for analyzing fatigue behavior of bituminous mastics, and analysis is usually conducted as per the protocols designed for bitumen (Das and Singh, 2018; Liao et al., 2012). The current Superpave specification employs the fatigue parameter ($G^*\sin\delta$) to determine the fatigue resistance of bitumen, with lower fatigue parameter corresponds to better fatigue resistance. The method is based on the dissipated energy concept, which suggested that the less dissipated energy per loading cycle ($\pi.\gamma_o^2.G^*\sin\delta$) will lead to a lower accumulation of distress. According to Superpave Performance Grade (PG) guideline, fatigue parameter determined for aged bitumen at 10 rad/s should not exceed 5000 kPa. However, several studies have considered $G^*\sin\delta$ was not a useful measure for fatigue cracking resistance, and reported a weak correlation between the parameter with laboratory performance of mixes (Hajj and Bhasin, 2018). This test is unable to provide any insight to actual complicated fatigue phenomena where bitumen is exposed to higher strain levels and varied frequency levels at different temperatures (Saboo, 2015). To avoid the limitations of the PG grading system, several researchers have attempted more effective methods to evaluate fatigue resistance of bitumen. Hajj and Bhasin (2018) have classified these methods under four broad categories:

(a) Time sweep test: In time sweep test, the sample is loaded with multiple cycles repeated at same amplitude, frequency and temperature. The failure of the sample is defined by the reduction in modulus or physical breakdown of the sample. Although, this approach is superior over conventional PG specification, but it takes a substantial amount of time for completion and its results get affected by factors such as the sample's geometry, thickness, and edge effects.

(b) Ductility based test methods: The ductility based test methods used rheological indicators measure fatigue resistance of bitumen. Two popular examples of such methods are Double Edge Notched Tensile (DENT) test and Direct Tension Test (DTT). Studies have shown that ductility parameters determined from the above tests can correlate well with the fatigue cracking issues in the field for unmodified bitumen (Rowe et al, 2014; Zhou et al., 2012). However, this correlation was not found universal in the case of polymer modified bitumen (Glover et al., 2005; Hajj and Bhasin, 2018).

***(c) Methods based on amplitude sweep*:** To overcome the drawbacks of the time sweep test, Johnson (2009) has proposed a new method known as Linear Amplitude Sweep (LAS) test. This method involves cyclic loading of bitumen in DSR having parallel plate geometry, but with increasing amplitude after a predetermined number of load cycles. Due to this arrangement, this test creates the same type of damage accumulation as time sweep test but in a quicker fashion. This test is conducted as per the AASHTO TP 101-14 guidelines and use viscoelastic continuum damage (VECD) theory to interpret the results and determine the fatigue characteristics of bitumen at any strain amplitude as per the structure of the pavement. Researchers have observed a good correlation between the results from the LAS test for a set of bitumen from Long Term Pavement Preservation database and fatigue performance of these pavement sections (Hintz et al., 2011).

(d) Measuring the strength or fatigue cracking resistance of bitumen in a realistic stress state (e.g., using thin films):The testing methods stated above used test bitumen in a bulk state (e.g., 1 or 2 mm thick samples in between parallel plates of

DSR). Under these conditions, the bitumen doesn't experience the same state of stress as it is when it is confined in small volumes (i.e., between rigid aggregates in bituminous mixes). These test methods allow direct measurement of bitumen strength in its realistic level of confinement because cracks occur upon the bitumen, reaching stress which exceeds its tensile strength. The examples of such tests are the Double Cantilever Beam (DCB) Test (Harvey and Cebon, 2003) and Poker Chip Test (Motamed et al., 2014; Sultana and Bhasin, 2014).

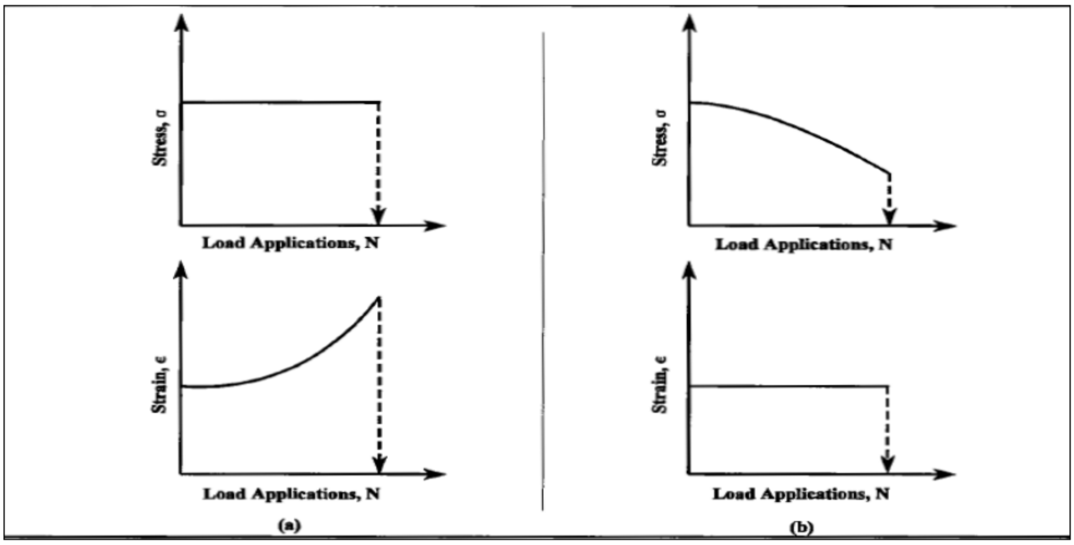

Figure 2.4 Principle of applying (a) control stress (b) control strain during testing (Epps and Monismith, 1972)

2.3.2.3 *Evaluation of Fatigue Characteristics of Bituminous Mixes*

The resistance of bituminous mixes to the fatigue cracking can be determined in two modes of loading, either by controlled-stress (force/load) or controlled-strain (displacement). In case of the controlled-stress mode of testing, the applied stress to the sample is kept constant, while the strain keeps on increasing with the load repetitions. While, in the controlled-strain mode test, the strain is kept constant, and the stress need to produce this constant strain producing decreases with load repetitions due to the lowering in the stiffness of the material owing to the gradually accumulating damage (Figure 2.4).

The constant stress test and constant strain test methods are found to be more suitable for the thick and thin pavement sections respectively (Monismith and Deacon 1969; AUSTROADS, 2010). Monismith et al., (1990) has compiled the differences between the controlled stress and controlled strain mode of testing (Table 2.5). However, the effect of type of bitumen (hard or soft) on fatigue performance of bituminous mix in the field may depend upon the type of the pavement structure. The use of a stiffer mix may reduce the strain at the bottom of the bituminous layer, which will increase the fatigue life of the bituminous mix (Harvey and Tsai 1997; Castel and Pintado 1999).

Table 2.5 Difference between controlled stress and controlled strain mode of testing

Variable	Controlled stress test	Controlled strain test
Definition of failure	Well defined as specimen fracture	Arbitrary; usually defined as 50% reduction of stiffness modulus
Scatter in fatigue test data	Less scatter	More scatter
Required number of tests	Fewer	More
Influence of stiffness	Increased stiffness results in increased fatigue life	Increased stiffness results in decreased fatigue life
Magnitude of fatigue life	Shorter life	Longer life
Effect of mixture variables	More sensitive	Less sensitive
Rate of crack propagation	Faster	Slower
Degree of healing	Larger beneficial effect	Smaller beneficial effect

The anti-cracking behavior of bituminous mixes has also been widely analyzed using a simple indirect tensile strength test, and mix with higher indirect values correspond to its better cracking resistance (Bennert et al., 2018; Christensen and Bonaquist, 2004; Si et al., 2016). There are several standard protocols specified to evaluate fatigue resistance of bituminous mixes, two of them are prescribed by standard guidelines and used by researchers flexural beam fatigue testing and indirect tensile fatigue test. Flexural fatigue testing is usually conducted as per the standard protocols like AASHTO T321-07 and ASTM D7460-10 in controlled strain mode (AASHTO, 2007). Indirect tensile fatigue test is usually conducted on standard Marshall specimen, which has been subjected to uniaxial loading as per EN 12697-24 specification in controlled stress mode. Currently, the testing of semicircular beam bending by the application of static and dynamic loading is also gaining popularity amongst several researchers (Biligiri et al., 2012; Saha and Biligiri, 2015) and could be used as standard protocol in future for fatigue testing. Tangella et al. (1990) have reviewed different fatigue testing methods based on their advantages and disadvantages. The test methods examined were: simple flexure test, supported flexure test, direct axial test, indirect tensile test, and wheel tracking test. They have ranked the test methods based on their simulation of field conditions and their simplicity. Out of these methods, the flexural beam test methods were ranked as the best, and indirect tensile test methods were ranked as second best because of its simplicity.

2.3.2.4 *Effect of Fillers on Cracking of Bituminous Mastic and Mixes*

The fatigue performance of bituminous mastic and mixes is strongly related to properties of bitumen, filler, interaction between bitumen and filler, and the phenomenon that influence the development of micro-crack and its growth in mastic (such as crack pinning) (Bahia et al., 1999; Kim et al., 2003; Smith and Hesp, 2000). The physicochemical interaction between bitumen and filler is linked to the fineness and the surface characteristics of filler which influence fracture characteristics of the mix. Crauss et al., (1978) has stated that the physicochemical interaction is related to adsorption intensity at bitumen-filler interface, and fillers having higher surface activity form stronger bonds with bitumen. Few studies have observed that the mixes containing fine filler particles dispersed well in the mixes, which lead to the improvement of their fracture properties (Smith and Hesp, 2000; Modarres and Bengar, 2017). Smith and Hesp (2000) have suggested that the fine filler display crack-pinning behavior in the mastic due to which it stops the propagation of the micro-crack in the mastic. They also have suggested that the fatigue life of mastic increases with the decrease in the size of the filler particles, however, they don't observe any such trend in the case of a gap and dense-graded bituminous mixes. Sobolev et al., (2014) have conducted the micro-structural investigation of mastics containing fine coal fly ash, which revealed crack arresting behavior of fine ash particles by pinning and deflecting brittle cracks. Das and Singh (2018) have compared the fatigue lives of mastics prepared basalt fillers in combination with regular-sized hydrated lime and nano-sized hydrated lime at different proportions. It was observed that the mastic containing a higher proportion of nano-sized hydrated lime displayed superior fatigue life. It was attributed to its higher interaction with the bitumen due to its higher surface area. Xing et al., (2019) have investigated the effect of ten different morphological parameters of fillers on the rheological parameters of bituminous mastics at high temperatures using LAS test. The fatigue parameters of bituminous mastics were found to be highly sensitive to porosity, angularity index, average diameter, aspect ratio, and fractal diameter of the filler particles. However, Kandhal et al., (1998) haven’t found any correlation between fatigue cracking and various characterization properties such as Rigden voids, German filler test values, particle size distribution, methylene blue value and plasticity indices of filler types (Kandhal et al., 1998).

Kim et al., (2003) has observed that the inclusion of fillers in the mastic improved their fatigue life in strain controlled testing, even though the stiffening in the bitumen is observed. It was observed that the inclusion of filler reduces the rate of damage to evolution. Hydrated lime was also found to be more effective filler than limestone, and hence it was observed that the other than the surface activity and volume reinforcement, the physicochemical interaction is also dependent on the type of filler and bitumen (Kim et al., 2003; Lesueur and Little, 1999). The potential of hydrated lime to retard fatigue cracking was also observed by Lee et al., (2010). However, Johansson and Isacsson (1998) and Lackner et al., (2005) have stated in their studies that the hydrated lime is very porous in nature due to which it can also cause excessively stiffening if used in high proportion.

The volume of filler content in the mastic and mixes also influence their fracture properties. Some studies have observed that the indirect tensile strength of the bituminous mixes increases with the increase in filler content (Huang et al., 2007; Choudhary, 2008). However other studies have reported that the excessively high filler bitumen ratio in the mastic can increase its brittleness which negatively affects the fracture properties and fatigue lives of mastic and mixes (Vale et al., 2016; Mazzoni et al., 2016). Liao et al., (2012) has observed that the testing method of fatigue resistance also significantly affects their fatigue life. They have prepared six mastics with three fillers (limestone, cement, and gritstone) with a mass percentage of fillers to be 35% and 65% respectively and compared with standard 40/60 penetration bitumen. The testing was done both at controlled stress and controlled strain mode. It was observed that in case of controlled stress mode fatigue life were found to increases with the filler volume and mastics containing 65% displayed the highest life, followed by 35% and plain bitumen. On the other hand, reverse trend was observed in the case of testing at controlled strain mode in which plain bitumen displayed the best, and 65% mastic displayed the worst fatigue life.

2.3.3 Moisture Damage

In bituminous mixes, the adhesive and cohesive forces between the aggregates and bitumen are responsible for holding the mix together. Water can infiltrate into the layer of the bituminous mix due to the rainwater, rising groundwater table, and due to the absorption of water vapors from the surrounding environment or a combination thereof. This ingression of water impair the adhesion between the bitumen-aggregate interface which reduce the strength of mix and increase the possibility of failures such as rutting, cracking, ravelling and potholing (Bhasin and Little, 2009). The removal of bitumen film thickness due to the ingression of water is termed as stripping (Bagampadde 2004, Kakar et al., 2015). Bitumen molecules containing functional group orient and get adsorbed to the aggregate adsorption site. When water moves to the interface, it dislodges the bond between the aggregate and bitumen which strips off bitumen from the aggregate surface (Figure 2.5).

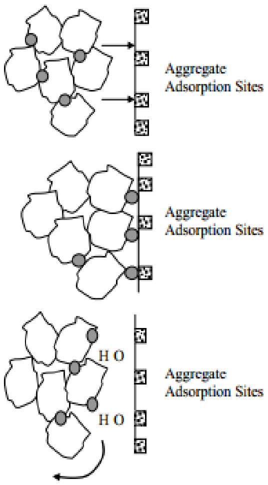

Figure 2.5 (a) movement of bitumen molecule towards aggregate site (top image) (b) adsorption of bitumen to the aggregate site (middle image) (c) stripping of bitumen (bottom image)

2.3.3.1 *Material Properties affecting the Moisture Damage*

Kiggundu and Roberts (1988) defined stripping as the progressive deterioration of bituminous mix due to loss of adhesive bond between the bitumen and aggregate surface and/or loss of the cohesive resistance within the bitumen due to the action of water. The chemistry occurring at the interface strongly affects the stripping which is highly influenced by the properties of all materials at the interface

(a) Effect of Bitumen: Bitumen mainly consists of hydrocarbons with heteroatoms containing polar functional groups and organometallic complexes of iron, vanadium and nickel. Some of the primary functionalities are phenolics, polynuclear aromatics, pyrrolics, sulphides, sulfoxides, pyridinics, 2-quinolones, ketones, carboxylics, and anhydrides (Bagampadde, 2004; Petersen et al., 1974). These functional groups interact with aggregate adsorption sites through, Vander Waals forces, π-π bonding, and hydrogen bonding. Bagampadde (2004) reviewed several older studies and observed the affinity of various polar functional groups towards dry and wet aggregates. Polar groups such as carboxylics, anhydrides and sulfoxides were found to be most water sensitive. On the other hand, nitrogen bases, phenolics, and ketones were associated with superior water resistance. Other than the chemical properties, proper adhesion at the interface also demands appropriate rheological properties during mixing, compaction operations, and during overall service life. The higher viscosity of bitumen is associated with its poor wettability of the aggregates. However, during service life, high viscosity also offers more resistance to moisture damage (Majidzadeh, 1968)

***(b) Effect of Aggregates*:** Aggregates are composed of definite mineralogical composition based on which they can be classified in five broad categories. These minerals and their relations to stripping are summarized in Table 2.6. Apart from mineralogy, chemical nature of aggregates also influences the stripping. Various studies have shown that bitumen groups that adsorbs on aggregates are mainly naphthenic acids. Hence, basic aggregates form stronger bonds with bitumen while acidic aggregates form weak bond. In addition to this, Fe, Ca, Mg and Al are generally beneficial regarding the stripping resistance, while alkali metals were found to be detrimental (Stuart, 1990).

Table 2.6 Type of minerals and their effect on stripping (Bagampadde, 2004)

Category	Type of mineral	Type of rock	Inference	Reference
Silica	Quartz-(SiO_4)	Granite, Sandstone, Quartzite, Rhyolite	Poor bitumen adherents as water attaches them to H-bonding	Majidadeh (1968); Rice, (1958); Stuart, (1990)
Limestone	Calcite-($CaCO_3$) Dolomite-($CaMg(CO_3)_2$)	Chalk, Dolomite, Limestone	Good adherends and undergo strong acid-base and electrostatic interactions with bitumen.	Curtis (1990); Stuart, (1990)
Ferro-magnesian	Olivine-$(MgFe)_2SiO_4$; Augite-$(Ca,Mg,Fe)(Si, Al)_2O_6$; Biotite-$K(Mg,Fe^{2+})_3(Al,Fe^{3+})$-$Si_3O_{10}(OH)_2$	Andesite, Basalt, Diabase, Diorite, Gabbro, Mica	Augite and Olivine form insoluble Mg and Ca salts while biotite makes soluble K salts.	Majidadeh, (1968); Rice, (1958); Stuart, (1990)
Clays	Kaolinite, Illite, Montmorillonite	Dust, Baghouse Fines	Can readily take up the water. Form stable bonds with lime.	Balghunaim (1991); Kandhal et al., (1998)
Feldspar	Albite- $NaAlSi_3O_8$ Orthoclase- $KAlSi_3O_8$ Anorthite- $CaAl_2Si_2O_8$	Diabase, Gabbro, Granite, Gneiss, Rhyolite, Quartzite, Sandstone	Anorthite forms insoluble Ca salts that are resistant to stripping.	Scott (1978); Stuart, (1990)

(c) Effect of Water: Water displays hydrogen bonding which affects its adhesive and cohesive properties. Most of the surfaces of the aggregates have electrostatic charges and comparison to the polar groups of bitumen; water molecules display stronger force to satisfy unbalance charges. The effect of water on the bitumen aggregate interface is aggregate specific. Siliceous aggregates form weak bonds with bitumen and thus are hydrolytically unstable. In such cases, water adsorbs onto aggregates through hydrogen bonding. On the other hand, calcareous aggregates provide free calcium ions forming water-resistant bonds with bitumen (Bagampadde, 2004; Kiggundu et al., 1986).

2.3.3.2 *Various Mechanisms of the Stripping*

The primary mechanisms of stripping are briefly explained below.

(a) Detachment: It is defined as the microscopic separation of bitumen film from the aggregate surface without causing any rupture in the film itself (Taylor and Khosla, 1983). It may be caused due to the incomplete drying of the aggregates, which leads to the movement of water within their interstitial pore and cause detachment.

(b) Displacement: Displacement is the separation of bitumen film around the aggregate with the action of water. It happens when absorption of water takes place to the aggregates through the crack in the bitumen film (Kiggundu and Roberts, 1988). The rupture in bitumen film at field might occur due to the traffic loading, aggregate fracture, or by the environmental actions like freeze-thaw.

(c) Spontaneous Emulsification: An inverted phase emulsion of water droplets formed within the bitumen mastic causes the spontaneous emulsification which leads to the cohesive failure. The emulsification rate depends upon the nature and the viscosity of the bitumen. The softer bitumen emulsify much faster than, the harder bitumen (Kakade, 2015; Kiggundu and Roberts, 1988).

(d) Pore Pressure: The water entrapped in the air void system can exhibit pore water pressure due to the repeated traffic loading. The high pore pressure (higher than the tensile strength of mix) may result in separation of bitumen film from the aggregate surface. This also might result in the development of micro-cracks in the mastic, which may lead to the adhesive and cohesive failure of mix (Little and Jones, 2003).

(e) Hydraulic Scour: The repeated movement of traffic on a saturated pavement surface causes hydraulic scouring. Due to the traffic movement water entrapped in the pavement create compressive stress within interconnected voids of the mixes. Once the wheel passes, a vacuum create in that place which pulls water back out of the voids. This repeated compression and tension produced due to continuous loading and unloading of vehicles can lead to moisture damage (Kakade, 2015).

(f) Chemical Disbonding: The removal of bitumen from the aggregates due to the chemical and electrostatic interaction between aggregates and water is referred to as the chemical debonding (Bagampadde 2004; Kakar et al., 2015).

(g) Microbial Activities: The metabolic process of some microbial may form by-products that break adhesion at the bitumen aggregate interface (Brown et al., 1990).

(h) Osmosis: The concentration gradient across the bitumen film may also transport the water to the interface (Thunqvist, 2001).

2.3.3.3 *Evaluation of Moisture Damage of Bituminous Mixes*

Analysis of moisture susceptibility of bituminous mixes in the laboratory is usually done by conducting tests on loose aggregates and/or on compacted bituminous mixes. Kakar et al. (2015) has further categorized several laboratory testing methods under these categories and are presented in Table 2.7 and Table 2.8.

Table 2.7 Test methods for loose mix and mixture components

Qualitative measures of stripping	
Test method	Test description
Static immersion	Percent of aggregates surface that has maintained their bitumen coatings after static immersion in water
Dynamic immersion	Percent of aggregates surface that has maintained their bitumen coatings after being agitated in water
Methylene Blue	The amount of harmful clays of the smectite (montmorillinite) group, organic matter and iron hydroxides present in fine aggregates
Boiling water test	Percent of stripped aggregates after immersion in boiling water
Indirect qualitative methods	
Test method	Test description
Chemical immersion	A quantitative index based on the concentration of chemical material for the initiation of moisture damage
Net adsorption	A quantitative index based on the difference of the adsorbed bitumen to aggregate surface in presence and in the absence of moisture
Tack Test System (TTS)	Measuring the required force to cause cohesion failure in the bitumen
Surface reaction	A quantitative index based on the pressure of produced gas due to the reaction of a chemical with the stripped surface of aggregates
Energy based methods	
Test method	Test description
Pneumatic Adhesion Tensile Testing Instrument (PATTI)	Measuring the required force to cause adhesion failure between bitumen and aggregate in presence and absence of moisture
Peel test	Measuring the adhesive fracture energy of bitumen and aggregate
Dynamic mechanical analyzer	Controlled-strain; cyclic torsional experiment on bituminous mastic
Wilhelmy plate	Measuring the surface energy of bitumen
Universal Sorption Device (USD)	Measuring the surface energy of aggregate
Contact angle sessile drop test	Measuring the surface energy of bitumen and aggregate
Fourier Transform Infra-Red (FTIR)	Measuring the adsorption/desorption of water in thin bitumen films
Nuclear Magnetic Resonance (NMR)	Measuring the changes in chemical composition/molecular mobility of bitumen due to moisture
Micro calorimeter	Measuring the energy adhesion components of aggregate and bitumen

Table 2.8 Test methods for moisture sensitivity analysis of compacted bituminous mixes

Destructive mechanical tests on compacted mixes	
Test method	Test description
Haveem stability	Measuring the ratio of Heveem stability after moisture conditioning to that before conditioning
Immersion compression	Measuring the ratio of compressive strength after moisture conditioning to that before moisture conditioning
Marshall immersion	Measuring the ratio of Marshall strength after moisture conditioning to that before moisture conditioning
Modified Lottman/ Tensile Strength Ratio	Measuring the ratio of diametric strength after moisture conditioning to that before moisture conditioning (with freeze and thaw cycles)
Freeze thaw pedestal	The number of freeze thaw cycles to crack initiation in the sample before and after moisture conditioning
Root-Tunnicliff	Measuring the ratio of diametric strength after moisture conditioning to that before moisture conditioning (without freeze and thaw cycles)
Double punch	Measuring the ratio of punch shear strength after moisture conditioning to that before moisture conditioning
Dissipated Creep Strain Energy (DSCE)	Fracture mechanics/energy
Wheel tracking	Number of cycles corresponding to the intersection point of the slop of second and third part of creep curve (known as stripping turning point)
Direct tensile test	Measuring the ratio of direct tensile strength after moisture conditioning to unconditioned
Beam fatigue test	Fatigue life before and after moisture conditioning
Cantabro test	Measuring the ratio of abrasive strength loss after moisture conditioning to unconditioned
Non destructive mechanical tests on compacted mixes	
Test method	Test description
Environmental Conditioning System (ECS)	Measuring the permeability of compacted bituminous mixtures and changes of their resilient modulus during application of thermal cycles and cyclic loading
Resilient modulus	Retained resilient modulus, the ratio of conditioned to unconditioned resilient modulus of bituminous mixtures
Dynamic modulus	Use of Simple Performance test to measure the dynamic shear factor of bituminous mixtures
SATS	Measuring the ratio of resilient modulus after moisture conditioning to that before moisture conditioning
Non destructive mechanical tests on compacted mixes	
Test method	Test description
Permeability, CT Scan, Diffusion, Capillary rise	An indirect measure of moisture sensitivity of bituminous mixtures based on the interconnectivity of air voids

The tests conducted on loose mixes or aggregates involve the analysis of the amount of bitumen coating retained after mix/aggregates have been subjected to specific conditions of moisture damage. Whereas in the case of the compacted mix, the moisture damage is assessed by evaluating its effect on different compacted mix parameters like stability, indirect tensile strength, rut depth, stiffness, fatigue life, Cantabro loss etc. Despite the availability of several methods, no test method entirely correlates laboratory results with field performance (Bagampadde, 2004). Several agencies have qualified the Modified Lottman test (AASHTO T283) as the most reliable method available at the moment to detect moisture sensitivity of the compacted bituminous mixes. It compared the effect of moisture permeation on the mixes by comparing their tensile strengths before and after the moisture permeation. The results are expressed in the term of the ratio of the tensile strength of the mixes after moisture conditioning and before moisture conditioning (Tensile strength ratio (TSR)). Indian pavement specification (MoRTH 2013) has also prescribed the Modified Lottman test to identify moisture resistant mixes and fixed the minimum requirement of TSR for moisture resistant mix to be 0.8. Aschenbrener and McGennis (1995) have analyzed the effectiveness of different moisture damage evaluation methods, and the results were compared with the performance of twenty different pavement sections which were failed due to moisture damage. Modified Lottman test was found to be fairly successful in identifying the mixes which are good performing or extremely moisture susceptible.

2.3.3.4 *Effect of Fillers on Moisture Resistance of Bituminous Mastics and Mixes*

There are numerous physical and chemical properties of fillers which influence the performance of bituminous mastic and mixes against moisture. Bitumen has slightly acidic tendencies, so it forms the stronger bond with basic fillers such as limestone and hydrated lime, while it forms weak bond with acidic fillers consisting silica (Airey et al., 2008; Bidgoli et al., 2019; Sakanlou et al., 2018). Hydrated lime is one of the most popular anti-stripping agents which significantly improve the moisture sensitivity of the mix. Airey et al., (2008) has investigated the effect of filler type, aggregate type, and volumetrics on the moisture sensitivity of the bituminous mixes. They have used Saturation Ageing Tensile Stiffness (SATS) test which simulates the effect of moisture submersion and the field ageing simultaneously, and then they have compared the results using Modified Lottman test. Both protocols displayed similar results, and as expected, the moisture resistance of bituminous mixes containing acidic aggregates was found to be lower than basic aggregates. However, the moisture resistance of mixes was improved dramatically by replacing the 2% conventional fillers with hydrated lime. Hence it was observed that even the addition of 2% hydrated lime could significantly improve the moisture resistance of the bituminous mix.

Fillers having hydrophobic (water repelling) nature also adheres well to bitumen since they absorb a greater amount of bitumen on their surfaces as compared to hydrophilic fillers (Chen et al., 2011b; Modarres et al., 2015). Hydrophilic fillers degrade the quality of bituminous mix and reduce strength, impermeability, and heat resistance (Géber and Gömze, 2010). Mineralogical composition of filler also affects its adhesion with the bitumen and thus moisture sensitivity of the mix. Partially or completely water-soluble minerals such as halite, sylvite, thernardite, cesanite, and quartz led to poor adhesion since they increase moisture penetration in the mix when exposed to moisture (Bidgoli et al., 2019; Pasandin et al., 2016). Therefore, fillers with insoluble minerals such as calcite, dolomite, and portlandite are preferred for bituminous mixes (Little and Epps, 2001; Bagampadde, 2004). Elemental composition of aggregates and fillers also found to have a correlation with moisture damage. Bituminous mixes prepared from aggregates and fillers containing sodium, potassium, and mica were found to exhibit relatively higher moisture sensitivity (Bagampadde et al., 2005; Said et al., 2009). The converse was apparent for aggregates with calcium, iron, and magnesium. No significant correlation was found between the strength ratios and contents of Al_2O_3 and SiO_2 (Bagampadde et al., 2005). Clay content in the filler also has a negative effect on moisture sensitivity of mix. Clay present on the surface of aggregates expand in the presence of water and form a barrier to adhesion, thus weakening the mix (Chandra and Choudhary, 2013; Kuity et al., 2014).

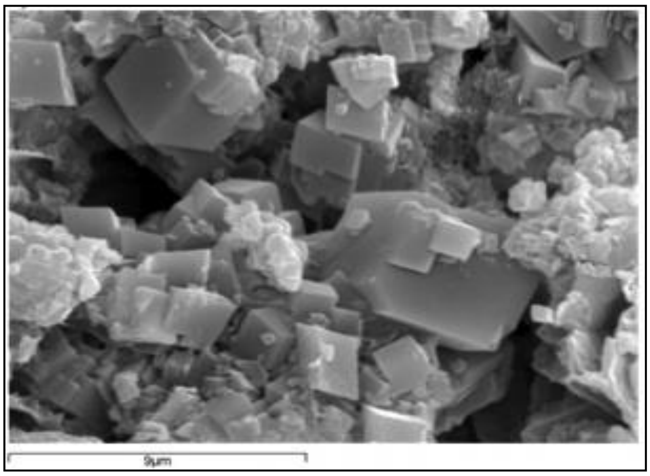

Figure 2.6 SEM image of moisture sensitive dreg waste from paper industry showing geometrical irregularities (sharp edges) (Pasandin et al., 2016)

Apart from chemical characteristics, geometrical irregularities of filler lead to decrease in effective bitumen which can also cause detrimental influence over their moisture sensitivity. For example, it was observed that when dreg waste obtained from paper industry was utilized as filler, its sharp edges (Figure 2.6) was considered as one of the primary reasons for poor moisture sensitivity of mix (Pasandin et al., 2016). Apart from these, Fillers which have high natural water content tends to agglomerate, which makes coating of bitumen difficult and has a detrimental effect over stripping potential (Pasandin et al., 2016). Kandhal et al., (1998) has stated that fineness of filler indicated by its D_{10} value stiffens the mastic, thus providing increased resistance to stripping. Filler having finer particles also evenly distribute in the mix and enhanced adhesion between aggregate and bitumen (Mistry et al., 2018). Das and Singh (2018) have investigated the effect of fillers on the interfacial bond strength of aggregates and mastics with the help of bitumen bond strength test performed in wet and dry state. They have prepared mastics containing basalt fillers in combination with regular sized hydrated lime and nano-sized hydrated lime at different proportions. It was observed that the mastic containing a higher proportion of nano-sized hydrated lime displayed superior bond strength in both dry and wet state. It was attributed to its higher interaction with the bitumen due to its higher surface area. The filler content in the bituminous mixes also affects its moisture resistance. Several studies have suggested that an increase in filler proportion in the mix reduces the moisture optimum bitumen content of the mix. This, in turn, also reduces the bitumen film thickness on the aggregates, which is responsible for the durability of the mixes against moisture. This ultimately reduces the moisture resistance of the bituminous mixes (Chandra and Choudhary, 2013; Huang et al., 2007)

2.3.4 Ageing

During the mixing, construction, and their service life, the bitumen and bituminous mixes observed hardening, which influences the physical characteristics of the mixes and their performance against various distresses. This hardening of the bitumen and the mixes can be termed as ageing. The initial aging in the first stage happens at a rapid rate when a bituminous mix is exposed to high temperatures during the mixing and is referred to as short-term ageing. It is also known as chemical ageing, and it happened due to exposure of thin bitumen film to the air at the elevated temperatures which modifies its rheological properties. The aging of the bitumen occur during the service period of the pavements when the mix is exposed to the atmospheric conditions for a long period of time is termed as long-term ageing. It occurs at a slower rate for a longer period of time. Due to the hardening of the bitumen, the bituminous mix becomes brittle, and it lost its ability to support traffic-induced stresses and strains, which leads the formation of cracks that lead to the deterioration of the pavement. Excessive hardening can also reduce the adhesion between the bitumen and aggregate, which increase the possibility of distresses like moisture damage and rutting. Hence it can be said that ageing itself is not distress, rather it can act as a catalyst for the other pavement distresses. Sirin et al., (2018a) in his study compiled the influence of several intrinsic (characteristics of mixes) and extrinsic (variable outside the mixes) parameters on short and long-term ageing of bituminous mixes which are given in Table 2.9.

Table 2.9 Effect of various factors on the ageing of bituminous mixes

Short-term aging		
Factors	Inference	Reference
Bitumen chemistry	Major effect	Traxler (1961)
Bitumen type and Source	Significant effect on lab and field ageing	Lund and Wilson (1984); Topal and Sengoz (2008)
Bitumen film thickness	Significant effect	Kandhal and Chakraborty (1996)
Aggregate gradation	No effect	Chipperfield and Welch (1967)
	Important effect	Morian et al.,(2011)
	Major effect	Traxler (1961)
Aggregate absorption	Important effect	Morian et al., (2011)
Inclusion of recycled materials and reheating	Significant effect	Mogawar et al., (2012)
Production temperature and silo storage	Significant effect	Mogawar et al., (2012)

Long-term aging		
Factors	Inference	Reference
Aggregate source	No effect on lab ageing	Morian et al., (2011)
Aggregate porosity	Significant effect	Kemp and Predoehl (1981)
Bitumen source	Significant effect	Morian et al., (2011)
Bitumen content	Significant effect	Kari (1982)
	No effect	Rolt (2000)
Filler type and content	Major effect	Al-Hdabi (2016); Movilla-Quesada et al.,(2017); Recans et al., (2005)
Air voids	Significant effect	Harrigan (2007); Houstan et al., (2005)
	No effect	Rolt (2000)
Pavement permeability	Significant effect	Kari (1982)
In-service temperature	Significant effect	Rolt (2000); Sirin et al., (2018a)
Exposure time	Significant effect	Rolt (2000)
Ultraviolet rays	Significant effect	Lee (1973)

2.3.4.1 *Various Mechanisms of Ageing*

Ageing of bitumen during its service life can be classified in two categories of reversibility: (a) reversible physical ageing (hardening) caused by the change in temperature (b) irreversible oxidative ageing caused due to oxygen exposure. There are several mechanisms associated with the ageing, which are primarily classified in the following categories (Moraes and Bahia 2015):

(a) Oxidation: Oxidation is the reaction of the bitumen with the oxygen present in the surrounding environment. Oxidative aging form oxygen-containing polar functionalities on bitumen molecules which can agglomerate among molecules because of higher physico-chemical associations like coulomb force, Vander Waal force, and hydrogen bonding such as hydrogen bonding (Petersen and Harnsberger, 1998). Its rate depends upon the composition of bitumen and the ambient temperature. It is a major responsible factor for the irreversible ageing of the bitumen.

(b) ***Volatilization:*** Volatilization is evaporation of the light bitumen components, when mixed at temperature greater than 150°C. During volatilization, asphaltene function in bitumen increases while aromatic fractions rapidly evaporate (Farcas, 1996).

(c) ***Steric hardening:*** Steric hardening is the progressive hardening of the bitumen due to structural reorganization of molecules within the bitumen at low temperatures over a period of time. Steric hardening is more pronounced at lower temperatures and it can be destroyed by reheating and working the bitumen (Peterson, 2000).

(d) ***Polymerization:*** Polymerization is the progressive hardening of bitumen due to the increase in molecular weight caused by the combination of smaller molecules to form a larger molecule. At lower temperatures, the rate of this association is slow as a result of higher viscosity of the bitumen (Moraes and Bahia 2015).

(e) Syneresis: Syneresis is the separation of less viscous fraction from the more viscous bitumen molecular network. Due to the loss of liquid, bitumen tends to harden due to shrinkage or rearrangement of bitumen structure by physical and chemical changes (Roberts et al. 1996).

(f) Separation: Separation is the removal of asphaltenes, resins, or oily components from the bitumen due to its absorption in porous aggregates (Roberts et al. 1996).

2.3.4.2 *Ageing of Bitumen Mastics*

There is no well-defined protocol for simulation of short-term and long-term ageing of the bituminous mastic and usually, the methods used to simulate the ageing in bitumen also applied for the mastics. Some major test protocols prescribed by different reviewers to simulate short-term and long-term ageing are described in the Table 2.10

Table 2.10 Various bitumen ageing methods (Airey, 2003)

Test method	Temperature (°C)	Duration	Film thickness	Extra features
Thin film oven test (ASTM D1754)	163	5h	3.2mm	-
Modified thin film oven test (Edler et al., 1985)	163	24h	100µm	-
Rolling thin film oven test (ASTM D2872; EN 12607-1)	163	75m	1.25mm	Air flow-4000ml/m
Rotating flask test (EN 12607-3)	165	150m	-	Flask rotation-20 rpm
Shell microfilm test (Griffin et al., 1955)	107	2h	5µm	-
Modified shell microfilm test (Traxler, 1963)	107	24h	15µm	-
Modified rolling microfilm oven test (Schmidt, 1973)	99	48h	20µm	1.04 mm dia opening
Tilt oven durability test (Kemp and Prodochl, 1981)	113	168h	1.25mm	-
Thin film accelerated aging test (Petersen, 1989)	130 or 113	24 or 72h	160µm	3 mm dia opening
Modified rolling thin film oven test (Bahia et al., 1998)	163	75m	1.25mm	Steel rods
Pressure Aging Vessel (PAV) (Christensen and Anderson, 1992)	90-110	20h	3.2 mm	2.07 MPa-air
High pressure aging test (Hayton et al., 1999)	85	65h	3.2 mm	2.07 MPa-air
Short-term ageing in normal oven (Behera et al., 2013)	163	3-4.5 h (bitumen dependent)	650µm	-
Long-term ageing in normal oven (Behera et al., 2013)	85	3-5 days	650µm	-

Majority of the researchers have used the thin film oven to age bitumen by applying extended heat and blowing air. Two types of ageing protocols are prescribed by many agencies to simulate short-term ageing which are: Thin Film Oven Test (TFOT) (ASTM D1754) and Rolling Thin Film Oven Test (RTFOT). Pressure aging vessel (PAV) is used to simulate long-term aging of bitumen at the field. In the current Superpave bitumen specifications, bitumen that need to be evaluated has to be subjected to RTFOT at 163°C for 85 minutes for short-term aging and then by a PAV to simulate several years of field aging (Sirin et al., 2018a).

2.3.4.3 *Ageing of Bituminous Mixes*

The current AASHTO R30 guidelines covers different three types of conditioning to simulate short-term ageing as well as long-term ageing synonymous to the field conditions (AASHTO, 2002). The guidelines are as follows:

(a) Conditioning of mixes for volumetric mixture design is done by placing bituminous mix at its compaction temperature in an oven for two hours.

(b) To simulate short-term ageing that occurs during the mixing and placement, mixes are kept in an oven maintained at 135°C for four hours.

(c) To simulate long-term ageing which occurs during the service life of the pavement, samples are kept in an oven maintained at 85°C for five days.

(a) Short-Term Ageing Protocol: Several researchers have investigated the effectiveness of short-term ageing protocol specified in AASHTO R30 specification, and most of them have concluded that it simulates short-term ageing very well except for some rare exceptions (Bell et al., 1994; Epps Martin et al., 2014).

(b) Long-Term Ageing Protocol: Researchers have simulated long-term ageing of the bituminous mixes by subjecting either loose or the compacted mix specimens to the ageing conditions in the oven. The oven ageing on the compacted samples is generally preferred to simulate ageing conditions. However, in the compacted samples, due to the geometry of cylindrical geometry of the specimen, it usually experiences the oxidation gradient in radial direction and along with the height of sample (Housten et al., 2005). Hence, some researchers have preferred to condition loose mix at high temperatures to maintain homogeneity and efficiency of the ageing. However, aged loose mixes often face difficulties in compaction due to their excessive stiffness caused by loss of volatile functional groups of the bitumen (Reed, 2010). Few researchers have also suggested several different protocols (Table 2.11) by taking different climatic conditions in consideration.

Table 2.11 Various long-term ageing protocols for bituminous mixes (Sirin et al., 2018a)

Ageing condition	Inferences	References
0, 2, 4, and 8 days at 85°C;	2 days at 85°C or 1 day at 100°C = 1-3 years of field ageing.	Kliewer et al., (1995)
1, 2, and 4 days at 100°C	8 days at 85°C or 4 days at 100°C = 9 years of field aging	
4 and 5 days at 85°C	4 days at 85°C simulates 15 years old pavement in the US.	Brown and Scholz (2000)
	5 days at 85°C simulates long-term aging of UK pavements.	
5 days at 80, 85, and 90°C	5 days at 85°C = aging 7–10 years of field	Harrigan (2007), Houston et al. (2005)
1 to 16 weeks at 60°C	4–8 weeks at 60°C = first summer of field aging	Epps Martin et al. (2014)
2 weeks at 60°C, 3 days at 85°C and 5 days at 85°C	2 weeks at 60°C = 7-12 months field aging 5 days at 60°C = 12-23 months field aging	Yin et al. (2017)
0, 3, 7, 15, 30, 45, 60, 90, and 120 days at 85°C on compacted specimen	45 and 75 days at 85°C = 5 years field aging in Middle East condition for wearing and base course, respectively	Sirin et al. (2018b)
0, 1, 2, and 3 days at 135°C on loose mixtures	2-3 and 1-2 days at 135°C = 5 years field aging in Middle East condition for wearing and base course, respectively	

2.3.4.4 *Effect of Fillers on the Ageing of Bituminous Mastics and Mixes*

Short-term and long-term aging of bituminous mastic and mixes alter their physical and chemical properties, which are reflected in their rheological and performance parameters. The ageing of bituminous mastics and mixes are usually computed by comparing any specific mastic/mix parameter before and after ageing. The ratio of the value of the studied parameter of mastic/mix after being aged to that of the same in un-aged condition is termed as the aging index. It can be expressed as in equation given below:

$$Ageing\ Index = \frac{Value\ of\ Parameter\ after\ Short\ Term\ or\ Long\ Term\ Aged}{Value\ of\ Parameter\ in\ Unaged\ or\ Short\ Term\ Aged\ Condition}$$

In case of asphalt mixes, properties such as Marshall Stability, Marshall quotient, indirect tensile strength, resilient modulus, and Cantabro loss is used for ageing susceptibility analysis of bituminous mixes (Al-Hdabi 2016; Masoudi et al., 2017; Zhang et al., 2018).

The ageing of bitumen in the mastic is highly influenced by the physical and chemical properties of bitumen and filler and also by the molecular interaction between the two components. The filler particles act as an obstacle to the diffusion of oxygen in the bitumen, which affect the ageing of bitumen (Glover et al., 2009; Gubler et al., 1999; Miro et al., 2005), which in turn influences the performance of mastic and the mix. Glover et al., (2009) have claimed that since fillers are impervious to the oxygen, it needs a more tortuous path through the bitumen, which lengthens the diffusion path thus effectively reduce the oxidation of bitumen. Arambula et al., (2010) has compared the diffusion of gases in between bitumen and fillers. Interestingly it was observed that diffusion of gases in filler has higher values than that in bitumen. Similarly, Bautista (2015) has analyzed the effect of filler concentration on their ageing index of the mastics having different coal combustion products as filler. He discovered the increase in the value of ageing indexes with the filler concentration in the mastic, which suggested that the inclusion of fillers in mastic increase their ageing susceptibility. Anderson and Tarris (1982) have stated that fillers have a very large surface area which gets activated by the application of heat during the mixing process.

Hence fillers might influence the ageing of bitumen by promoting oxidation or side reactions like dehydration or polymerization. Moraes and Bahia (2015) have investigated the effect of filler on the long oxidative ageing of mastics. Three different types of fillers (limestone, granite, and dolomite) were mixes at 10 and 40% by volume of two bitumen (PG 64-16 and PG 64-22). The ageing indices of mastics were determined using complex modulus as parameter. It was observed that at a similar filler volume and film thickness, the filler which has higher surface area, absorb higher asphaltene, which resulted in lowering of lower of the ageing index of mastic.

Chemical composition of filler also significantly affected the ageing of the bitumen in mastics and mixes. Hydrated lime is widely recognized as active filler which inhibit ageing due to acid-base reaction between the polar molecules of the bitumen and the lime surface (Lesueur et al., 2013; Lesueur et al., 2016; Petersen 2009; Petersen et al., 1987). It was observed that fillers consisting of strong bases like $Ca(OH)_2$ (hydrated Lime), CaO (quick lime), and $Mg(OH)_2.Ca(OH)_2$ (dolomitic hydrated lime) seems to act as active fillers and reduce the bitumen ageing (Petersen et al., 1987; Lesueur et al., 2016). While the fillers containing weak bases like $Mg(OH)_2$ doesn’t act as an active filler (Johansson et al., 1996). Alfaqawi et al. (2017) has investigated the effect of mineralogy of fillers on the ageing of mastic. Two different mastics were prepared by mixing granite and limestone at 0.5 filler bitumen ratio by mass. Then four more mastics were prepared by replacing 10 and 20% of each fraction with hydrated lime. All six mastics were subjected to short and long-term ageing and their subsequent ageing indices were compared by taking complex modulus as the parameter. Replacement of granite and limestone with the hydrated lime was found to reduce the change in carbonyls and reduced the long-term ageing by up to 50%. Petersen et al.,

(1974) has observed that presence of minerals like limestone and quartzite can catalyze the oxidation of lighter components of bitumen (saturates and aromatics), however, the ageing effect is negligible when heavier fractions (asphaltenes and resins) or whole bitumen is aged.

Although there are several studies which investigate the effect of fillers on the ageing behavior of bituminous mastics, there is surprisingly limited number of studies which investigated the influence of the type of filler and their quantity on the performance of the bituminous mixes. Al-Hdabi (2016) has investigated the effect of long-term ageing on the Marshall stability of the bituminous concrete mixes containing ordinary Portland cement and rice husk ash as fillers. The Marshall stability of both mixes was found to be increased after the ageing, which was possibly due to the stiffening of bitumen ageing and mixes containing rice husk ash displayed higher stability. Zhang et al. (2018) have investigated the effect of long-term ageing on the ravelling resistance (Cantabro test) of the porous bituminous mixes containing limestone powder and red mud as fillers. It was observed that the ravelling resistance of both mixes improved after being subjected to long-term ageing and higher improvement (lower Cantabro loss) was shown by the mix containing red mud filler. Interestingly in both studies, instead of analyzing the ageing index, the mixes which displayed higher stability and higher retained Cantabro weight is considered as more age resistant, this might be debatable amongst the researchers.

2.4 Type of Fillers

There is no well-defined classification of fillers, but in this study, fillers are classified in two heads based on their utilization named as standard or conventional fillers and waste or unconventional fillers.

2.4.1 Standard or Conventional Fillers

Standard fillers are fillers which are traditionally utilized in bituminous mixes over a period of time. These fillers are intentionally produced by grinding and crushing of parent material to suitable sizes. Since they are produced to serve a suitable purpose, their performance came out to be fairly good, and their cost is also higher. Some of the standard fillers utilized globally for pavement construction are stated below.

2.4.1.1 *Stone Dust*

Stone dust is one of the most widely used filler in the bituminous mixes. It is obtained by crushing stones at stone quarries. It is usually non plastic in nature and its quality varies with its parent stone, and the type and performance of crusher employed. Its plastic nature is also get affected by the presence of foreign impurities. Limestone and dolomite are two most commonly used stones for preparation of stone dust. They are widely preferred in preparation of bituminous mix due to their alkaline nature and high good proportion of calcium-based water-insoluble materials like calcite and dolomite, which form strong bond with bitumen.

2.4.1.2 *Ordinary Portland Cement*

Ordinary Portland cement is arguably the second most widely used anti-stripping agent in the bituminous mixes. It is manufactured by mixing calcareous and siliceous rock at very high temperatures (1000-1200°C). It is alkaline in nature and consists of high percentages of CaO due to which it exhibits anti-stripping nature. It has higher specific gravity, well-graded nature, and relatively lower porosity due to which it absorbs a relatively lower amount of bitumen. However, due to it's the relatively higher cost, it has been utilized in a limited amount at the field. MoRTH (2013) recommends to substitute up to 2% of stone dust with the cement if dense-graded mix fails to fulfill the requirements of adequate coating and moisture resistance.

2.4.1.3 *Hydrated Lime*

Hydrated lime is the most widely used anti-stripping agent in the bituminous mixes. It has fine size, uniform grain size distribution, high porosity, alkaline nature, and high affinity towards bitumen. It is produced by slaking or hydrating quick lime (CaO) with water and primarily composed of calcium dihydroxide ($Ca(OH)_2$) (Kakade 2015; Lee, 2007). It is usually added in bituminous mixes as an anti-stripping agent to improve their moisture resistance. MoRTH (2013) recommends that 2% of stone dust filler used in dense-graded mix can be substituted with the hydrated lime in case they fail to fulfill the requirements of adequate coating and moisture resistance. In the USA, the hydrated lime has been used in the range of 1 to 2.5% (with 1 to 1.5% being the most common range) of the weight of the aggregates in the mix (Hicks and Scholz, 2003).

Advantageous Mechanisms of Hydrated Lime

The benefits of using hydrated lime in the mix may be associated with its following mechanisms:

(a) Filling the voids between the aggregates: Hydrated lime occupies the voids between the aggregates and reduces their size as well as their interconnectivity. It will also provide additional contact points between the aggregates, thus improving the strength and impermeability. This, in general, increases the density and stability of the mixes as well as reduces its optimum bitumen content (Anderson 1987; Brown et al., 1983; Kandhal 1981). Hydrated lime is comprised of fine particles, and filler particle having size lower than bitumen film thickness act as a bitumen extender thus can reduce the optimum bitumen content of the mixes.

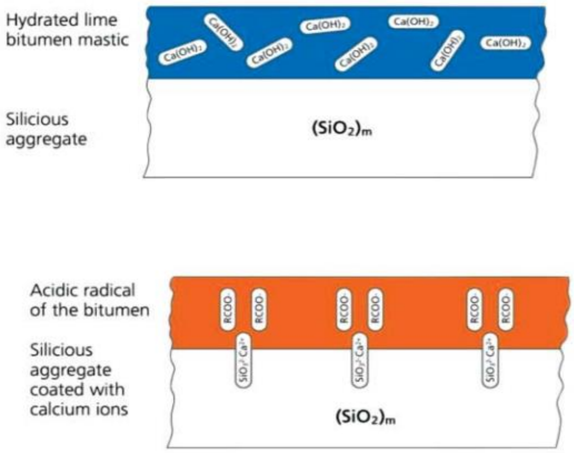

Figure 2.7 Effect of hydrated lime on aggregate surfaces (Ishai and Crauss, 1977; Lesueur et al., 2016)

(b) Modification of the surfaces of the aggregates: Hydrated lime can also improve the moisture resistance of the siliceous aggregates by modifying their surfaces. Siliceous aggregates are known to form weak bonding with the bitumen in comparison to calcareous aggregates. Bitumen consists of both cationic and anionic surfactants, and both of these surfactants strongly bond with the calcium ions in the calcareous aggregates. In the case of siliceous aggregates, only cationic surfactants form a strong bond with the silica atoms, while anionic surfactants get easily displayed by the water on the siliceous aggregates making aggregates prone to stripping (Curtis et al., 1990). Hydrated lime precipitated the calcium ions on the aggregate surfaces and improved its bonding with the bitumen (Ishai and Crauss, 1977) (Figure 2.7). When hydrated lime comes in the contact of water, it forms calcium carbonate which precipitates on the surface of the aggregates and enhances its roughness which improves its bonding with bitumen (Little, 1995). Other than that, hydrated lime can also treat any clay particles adhering to the aggregate surface, thus limiting their damaging on the moisture resistance of the mix (Little, 1995).

(c) Physical interaction with the bitumen: Hydrated lime usually has higher porosity than the ordinary mineral fillers due to the presence of the internal voids in it (Lesueur et al., 2016). Hence it results in the higher bitumen absorption, which in turn increases its stiffening effect than other fillers. This higher bitumen absorption and the consequential increase in the effective volume of solid particles affect various mechanical properties of bituminous mastic (Das et al., 2018; Lesueur and Little 1999).

(d) Chemical interaction with the bitumen: Several studies have investigated the chemical interaction of the bitumen with the hydrated lime and concluded that a part of the bitumen got strongly absorbed on the hydrated lime particles. This selective adsorption significantly affects the ageing characteristics of the bitumen. Petersen et al., (1987) has observed that hydrated lime reduced the quantity of anhydrides, ketones, and most of carboxylic acid that formed after ageing. It was also found to inhibit the oxidation catalyzers like the Vanadium compounds in the bitumen (Petersen et al., 1987). The polar molecules present in the bitumen and that are prone to ageing get adsorbed on to lime particles which inhibits their reaction to further chemical ageing, and reduce the rate of ageing of the bitumen.

2.4.2 Waste or Unconventional Fillers

Waste or unconventional fillers are obtained as a by-product of another process and treated as wastes or a low-cost material. Their performance in bituminous mixes is not pre-decided and depends upon the source of the parent material and process of their production, so extensive testing is a sheer necessity before their utilization. They are very cheap and often available in free of cost, so they got undivided attention from various researchers around the world. This section discussed 30 widely produced waste materials from the different origin which can be utilized as filler in bituminous mixes. They can mainly be classified under following heads (a) Agricultural wastes (b) Industrial wastes (c) municipal/household/domestic wastes (d) construction and demolition wastes (e) Drilling and mining wastes. The details of regarding the process of generation of various wastes are stated in Table 2.12. Comparison between various physical and chemical properties of different wastes materials utilized as filler in previous studies is stated in Table 2.13. Finally the comparison between the performance of conventional mixes and waste filler incorporated mixes are shown in Table 2.14. It provided the idea regarding the influence of waste filler on the performance of primary aspects of different types of bituminous mixes. Comparison between different mixes is made in terms of their Marshall stability, rutting resistance, cracking resistance, moisture sensitivity and optimum bitumen content (OBC). For each aspect, the improvement or the decline in parameter is mentioned with respect to conventional mixes when replacement of conventional filler is done with the stated waste. The symbol “I” and “D” represented the increase and decline of the property while the magnitude of change is stated in parenthesis. For example, “I (60%)” in rutting resistance suggested the improvement in the rutting resistance of the mix when conventional filler is replaced with waste filler.

Table 2.12 Various types of wastes and their process of generation

Category of waste	Type of waste	Process of generation	References
Agricultural	Rice Husk Ash (RHA)	Produced after incineration of rice husks as a fuel source for a boiler at 600°-800°C to produce energy. It can also be produced by uncontrolled open burning of rice husk.	Al-Hdabi (2016); Mistry and Roy (2018)
	Sugarcane Bagasse Ash (SCBA)	Produced after incineration of crushed sugarcane bagasse as boiler fuel.	Kumar et al., (2012)
	Palm Oil Fuel Ash (POFA)	Produced after combustion of palm husk, fibre and oil palm shell generated during the production of crude palm oil.	Ranjbar et al., (2014)
Industrial	Coal Fly Ash (CFA)	Mineral by-product obtained after combustion of pulverized coal in thermal power plant for electricity generation.	CPCB (2007); Chandra and Choudhary (2013)
	Ceramic Waste (CW)	Produced during the cutting, grinding, dressing and polishing operation of ceramic materials in the ceramic industry.	Muniandy et al., (2009)
	Cement Kiln Dust (CKD)	Collected from electrostatic precipitators in large volumes during production of cement clinkers in cement industry.	Kontasta-Gdoutos and Shah (2003)
	Lime Kiln Dust (LKD)	By-product generated in high temperature rotary kiln and capture by baghouse and electrostatic precipitator in lime industry.	West and James (2006)
	Copper Slag (CS)	Produced during the process of smelting for sulphide ore and collected as the material that floats on the top of the molten copper in a furnace.	Modarres and Bengar (2017); Nazer et al., (2016)
	Copper Tailing (CT)	Copper tailing is the waste rock remaining after ore has been processed to remove the copper. It is usually pulverized to the size of fine sand.	
	Baghouse Fine (BF)	Fine dust generated from heating of mineral aggregate during the production of the bituminous mix in hot mix plant. It is collected from Baghouse installed for dust collection in hot mix plant.	Lin et al., (2006)
	Marble Dust (MD)	Produced during the cutting and polishing operations of larger stone slabs.	Akbulut et al., (2012); Chandra and Choudhary (2013)
	Granite Dust (GD)		
	Phosphogypsum (PG)	Produced as byproduct during production of phosphoric acid from rock phosphate in the fertilizer industry.	CPCB (2007)
	Glass Powder (GP)	Can be produced after crushing glass waste to suitable fineness. It can also be generated during cutting and polishing operations of large glass slabs in glass industry.	Arabani et al., (2017); Saltan et al., (2015)
	Blast Furnace Slag	By-product obtained when iron ore, coke, and limestone are superheated in blast furnace to	Bocci (2018)

		produce pig iron.	
	Ladle Furnace Slag (LFS)	Produced in the secondary metallurgy process, during the final stages of steelmaking.	
	Red Mud (RM)	Red mud or bauxite residue is solid waste produced by aluminium industries after digestion of bauxite ore with caustic soda in Bayer’s process.	Paramguru et al., (2005)
	Brake Pad Waste	After crushing old brake pad of a heavy duty truck to suitable fineness	Hu et al., (2017)
	Recycled Tyre Rubber	After shredding waste tyres to suitable fineness	Chen et al., (2015)
	Carbon Black	After incineration of waste tyre in the absence of oxygen.	Lesueur et al., (1995)
Construction and Demolition Waste	Brick Dust (BD)	After efficient crushing of waste bricks from demolished structures.	Chen et al., 2011(a)
	Concrete Dust (CD)	After efficient crushing of waste concrete from demolished structures.	Chen et al., 2011(b)
	Plaster Board Waste	Indirect burning of waste plaster board obtained from construction industry.	Kuttah et al., (2015)
Domestic and Municipal Waste	Municipal Solid Waste Incineration Ash (MSWIA)	Solid by-product produced during efficient combustion of municipal solid waste in a well-maintained incineration facility.	Tasneem (2014); Xue et al., (2009)
	Sewage Sludge Ash (SSA)	Produced by efficient combustion of dewatered sewage sludge from the wastewater treatment plant in a well-maintained incineration facility.	Tenza-Abril et al., (2014)
Drilling and Mining Waste	Coal Mine Waste (CMW)	Generated during coal preparation processes which include operations such as screening, cleaning, crushing, and separation in coal industry. These wastes can be reduced to suitable fineness.	Modarres et al., (2015a);
	Boron Mine Waste	Produced during the separation of boron mineral from the boron ores.	Gurer and Selman (2016)
	Oil shale Waste	Waste sedimentary rock which contains various organic matters, especially kerogen, while after heating at 500ºC yields oil, gases, and other carbon residues.	Katamine (2000)
	Oil Shale Waste Ash	Direct combustion of oil shale waste can produce fly ash that could be utilized as filler in bituminous mixes.	Hamdan and Azzam (1999)

Table 2.13 Characterization properties of major waste fillers

Category of Waste	Material Type	Characterization Property								
		Specific Gravity (g/cm^3)	Water Absorption (%)	MBV (g/kg)	Particle Shape	Specific surface area (m^2/kg)	Primary mineralogical composition	SiO_2 content (% by weight)	CaO content (% by weight)	References
Agricultural Waste	Rice Husk Ash	2.05	1.0	NA	H	231	Quartz; Crystobalit; Anorthite	86.67	1.88	Melotti et al., (2013); Farooque et al., (2009).
	Sugarcane Bagasse Ash	2.52	NA	NA	Fs and G	514	Quartz; Calcite; Corundum; Halite	62.43	11.80	Zainudin et al., (2016)
	Palm Oil Fuel Ash	2.22	NA	NA	I	492	NA	43.6	8.4	Borhan et al., (2010); Hamada et al., (2018)
	Bio Mass Ash	2.18-3.13	0.2-32	0.7-5.3	I	NA	NA	5-95	3-65	Melotti et al., (2013)
Industrial Waste	Coal Fly Ash	1.97-2.23	0.13	1.24	R	300-600	Quartz; Mullite; Dolomite; Rutile	37.05-59.70	1.71-2.3	Chandra and Choudhary (2013); CPCB (2006).
	Marble Dust	2.69	0.97	4.5	SA	1140	Dolomite; Quartz; Calcite; Chlorite; Tremolite	15-30	20-40	Chandra and Choudhary (2013); Kumar et al., (2003).
	Granite Dust	2.79	0.92	2.25	SA-SR	131	Quartz; Kyanite; Sphene; Albite	37.49	7.84	Barra et al.,(2014); Chandra and Choudhary (2013)
	Phosphogypsum	2.3-2.6	NA	NA	Fs	NA	Calcite; Quartz; Gypsum	3.70	35.7	CPCB (2006); Katamine (2000)
	Cement Kiln	2.75	NA	NA	A	460	Portlandite;	15-18	45-52	Modarres et al.

	Dust						Lamite; Hartrurite,			(2015(b)); Ramadan and Ashteyat, (2009).
	Ceramic Waste	2.07	0.97	2.0	R	588	Quartz; Mullite; Zircon; Hematite; Orthoclase.	76.41	7.1	Medina et al., (2012); Munaindy et al., (2009)
	Steel Slag	3.4	0.2	0.3	A	238	NA	41.41	4.25	Muniandy et al., (2009)
	Glass Powder	2.62	NA	1.25	A	NA	Quartz	71.42	7.462	Arabani et al., (2017)
	Red Mud	3.12	NA	2.875	G	NA	Hematite; Quartz; Rutile; Sillimanite; Calcite.	9.60	15.44	CPCB (2006); Choudhary et al., (2019)
Construction & Demolition Waste	Brick Dust	2.68	1.0	1.67	A.	256	Quartz; Chromite.	68.1	2.05	Chen et al., (2011a); Kuity et al., (2014).
	Concrete Dust	2.637	0.9	1.10	A	372	Quartz; Calcite; Gobbinsite.	35.59	29.20	Chen et al., (2011b); Kuity et al., (2014).
Domestic and Municipal Waste	Sewage Sludge Ash	2.78	NA	NA	I	630	Quartz; Hematite; Thernardite.	17.21	29.88	Aburkaba and Munaindy (2010); Tenza-Abril et al., (2014)
	Municipal Solid Waste Ash	2.18	NA	NA	I	NA	Quartz; Calcite; Portlandite.	18.81	38.92	Tasneem (2014); Xue et al., (2009).
Mining Waste	Coal Mine Waste	2.324	NA	NA	NA	NA	Quartz; Kaolinite; Pyrite; Calcite;	34.8	0.51	Modarres and Rahmanzadeh (2014)

Abbreviations **A-** Angular; **SA-**Subangular; **R-** Rounded; **SR-**Sub rounded; **G-**Granulous; **Fs**: Fibrous; **Fy-**Flaky; **H-** Honeycombed; **I-**Irregular, **C-** Cubicle; **NA:** Not available.

Table 2.14. Effect of waste fillers in the performance of bituminous mixes

Type of waste	Type of mix	Type of standard filler used	Substitution rate of standard filler (%)	Comparison of performance of waste filler incorporated mixes with standard mixes					Other remarks	References
				Marshall stability	Rutting resistance	Cracking resistance	Moisture resistance	OBC		
Rice Straw Ash (RHA)	BC	Limestone	25-100	D (30%) – I (18%)	-	-	-	Same	RHA mixes with 50% replacement rate displayed best properties.	Sargin et al., (2013)
	BC	Cement	100	I (65%)	-	-	I (30%)	Same	RHA also improved the performance of mixes against long-term ageing.	Al-Hdabi (2016)
	BC	Stone Dust	100	D (11%)	D (26%)	D (2%)	-	Same	Porous nature of RHA negatively affects performance of mixes.	Arabani et al., (2017)
	DBM	Hydrated Lime	100	-	I (19-41%)	I (3-47%)	D (5%) – I (21%)	D (0-5%)	Bitumen reinforcement by RHA improves the properties of mixes.	Mistry and Roy (2018)
Sugarcane Bagasse Ash (SCBA)	BC	Stone Dust	100	I (1%)	-	-	-	Same	SCBA mixes displayed 17% higher resilient modulus than control mixes. SCBA has lighter weight and lower silica than RHA	Zainudin et al., (2016)
Palm Oil Fuel Ash (POFA)	BC	Stone Dust	100	D (8-26%)	I (8-36%)	I (100%)	-	Same	POFA has cementitious properties in mixes due to pozzolanic properties.	Borhan et al., (2010)
	BC	Cement	100	I (30%)	-	-	-	I (15%)	Mixes with POFA has 54% higher ravelling resistance than control mix.	Maleka et al., (2015)
Coal Fly Ash (CFA)	BC	Stone Dust	100	-	I (20-24%)	I (21-51%)	I (4-7%)	D (1-3%)	Lower active clay and high bulk volume of fines in CFA improved the moisture & rutting resistance of mixes	Chandra and Choudhary (2013)
	BC	-	-	-	-	I (182-448%)	I (4-7%)	D (10%)	Fly ash act as bitumen extender to reduce OBC of the bituminous mixes without adversely affecting the overall workability of the mix.	Faheem et al., (2017)
	DBM	Hydrated Lime	100	-	I (2-26%)	D (3%) – I (81%)	D (7%) – I (19%)	D (3-10%)		Mistry et al., (2018)
	BC	Limestone Dust	20-80	D (4-20%)	D (16-60%)	I (7-18%)	I (4-7%)	Same	CFA has Calcite, Portlandite & lime that improve its bonding with bitumen	Xue et al., (2019)

Type of waste	Type of mix	Type of standard filler used	Substitution rate of standard filler (%)	Comparison of performance of waste filler incorporated mixes with standard mixes					Other remarks	References
				Marshall stability	Rutting resistance	Cracking resistance	Moisture resistance	OBC		
Ceramic Waste (CW)	BC	Hydrated Lime	25-75 (by volume)	-	D (15-18%)	I (0-83%)	-	Same	CW is mostly made from Kaolin clay which has lower moisture susceptibility. CW due to high strength and porous nature absorbs higher amount of bitumen and form mixes with higher OBC and resilient modulus.	Huang et al (2009)
	SMA	Limestone Dust	100	Same	I (30%)	-	-	I (1%)		Aburkaba & Muniandy (2010)
	SMA	Limestone Dust	100	I (19%)	I (18%)	-	-	I (1%)		Muniandy et al., (2013)
Cement Kiln Dust (CKD)	BC	Limestone Dust	100	D (17%)	-	-	-	D (2%)	Leaching analysis suggested that bitumen has a stabilizing effect over CKD particles which limits the leaching of heavy metal from the mix	Taha et al., (2002)
	BC	Limestone Dust	25-100	I (14-29%)	-	I (79-158%)	-	Same		Ahmed et al., (2006)
	BC	Limestone Dust	25-100	-	-	I (2-20%)	I (0-3%)	I (1-7%)		Modarres et al., (2015)
Lime Kiln Dust	SMA	Rock Dust	100	-	-	-	I (2%)	D (3%)	LKD mixes has lower OBC due to their lower specific gravity.	West and James (2006)
Copper Industry Waste (CW)	BC	Limestone Dust	33-100	-	I (5-8%)	I (1-10%)	-	D (2%)	Finer size and finer gradation of copper slag improved the stiffness & cracking resistance of bituminous mixes. Bitumen in mixes prevented leaching of heavy metals from the copper waste.	Modarres and Bengar (2017)
	BC	Stone Dust	100	I (4%)	I (6%)	I (11%)	D (5%)	D (2%)		Choudhary et al., (2019)
Baghouse Fine	BC	Lime	0-75	D (2-14%)	D (63%) – I (67%)	-	D (0-9%)	Same	Baghouse fines are not used in some states of USA since their mixes causes problems related to stripping, compaction, bleeding and flushing.	Anderson (1987); Wilanowicz et al., (2013)
Marble Dust MD)	BC	Stone Dust	100	-	I (8-11%)	I (54-83%)	I (3-7%)	D (2-7%)	Marble dust has calcite & dolomite that improves moisture sensitivity of its mixes. It has lower porosity which reduces OBC.	Chandra and Choudhary (2013)

Type of waste	Type of mix	Type of standard filler used	Substitution rate of standard filler (%)	Comparison of performance of waste filler incorporated mixes with standard mixes					Other remarks	References
				Marshall stability	Rutting resistance	Cracking resistance	Moisture resistance	OBC		
Granite Dust (GD)	BC	Stone Dust	100	-	I (19-23%)	I (3-22%)	D (2-3%)	D (1%) - I (19%)	Granite dust has angular particles & rough texture that improve rutting resistance & increase OBC of its mixes.	Chandra and Choudhary (2013)
Phosphogypsum (PG)	BC	Limestone Dust	100	I (9%)	I (12%)	-	-	I (11%)	Phosphogypsum mixes loose mechanical interlock due to softening of its microcrystalline structure in the presence of water.	Katamine (2000a)
Glass Powder (GP)	BC	Limestone Dust	100	D (11%)	-	-	-	I (2.4%)	Glass powder has low absorption and primarily consists of silica.	Saltan et al., (2015)
	BC	Stone Dust	100	I (21%)	I (51%)	I (13%)	-	Same		Arabani et al., (2017)
Steel Slag	SMA	Limestone Dust	100	I (25%)	I (50%)	-	-	I (1%)	Utilization of slag as filler also significantly improve resilient modulus of mixes	Muniandy et al., (2013)
	BC	Limestone Dust	100	-	-	I (13%)	-	Same	Slag is good filler due to its alkaline nature & well edges & coarse texture. It form good bonding with bitumen.	Li et al., (2016)
Ladle Furnace Slag (LFS)	BC	Hydrated Lime	100	-	-	I (19%)	I (3%)	I (2%)	Mixes containing LFS has lower compactability due to higher viscosity and stiffness of bitumen filler mastic.	Bocci (2018)
Red Mud (RM)	PA	Limestone Dust	100	-	I (23%)	-	-	Same	Mixes containing RM also displayed better adhesion & ravelling resistance.	Zhang et al., (2018)
	BC	Stone Dust	100	I (9%)	I (1%)	I (21%)	D (8%)	I (6%)	Higher porosity, finer size, & mineralogical composition of RM is responsible for good performance of mixes	Choudhary et al., (2019)

Type of waste	Type of mix	Type of standard filler used	Substitution rate of standard filler (%)	Comparison of performance of waste filler incorporated mixes with standard mixes					Other remarks	References
				Marshall stability	Rutting resistance	Cracking resistance	Moisture resistance	OBC		
Brake Pad Waste (BPW)	BC	Limestone Dust	100	-	I (41%)	I (245%)	I (15%)	Same	Use of BPW in mastic also improved high temperature stability & its elastic recovery.	Hu et al., (2017)
	BC (WMA)	Limestone Dust	100	-	I (175%)	I (65%)	D (4%)	Same	BPW improved the high temperature properties of sulphur modified bitumen & worsen low temperature properties.	Hu et al, (2019)
Recycled Tyre Rubber	BC	Limestone Dust	100	-	I (595%)	-	D (1%)	I (2%)	Use of recycled tyre rubber as filler also reduces thermal conductivity of bituminous mixes.	Chen et al., (2015)
Carbon Black	BC	Limestone Dust	100	I (5%)	I (27%)	I (24%)	I (1%)	D (1%)	Use of carbon black as filler also improves mix's electrical conductivity.	Ahmedzade et al., (2007)
Brick Dust (BD)	BC	Limestone Dust	100	-	I (33%)	I (20%)	I (3%)	D (1%)	High contact area & low hydrophilic coefficient is responsible for better moisture & rutting resistance of its mix.	Chen et al., (2011)(a)
	BC	Stone Dust	100	-	D (1100%)	D (10%)	D (76%)	Same	High clay content in BD worsened the moisture sensitivity of its mixes.	Kuity et al., (2014)
	BC	Stone Dust	100	I (12%)	I (6%)	I (95%)	-	Same	Higher physic-chemical interactions with bitumen improve mix performance.	Arabani et al., (2017)
Concrete Dust (CD)	BC	Limestone Dust	100	-	I (25%)	I (31%)	I (4%)	I (1%)	CD has higher volume in mix which caused higher bitumen absorption & improved its moisture resistance.	Chen et al., (2011)(b)
	BC	Stone Dust	100	-	D (10%)	D (34%)	I (3%)	Same	Alkaline nature of CD leads to better moisture resistance of its mixes.	Kuity et al., (2014)
Plaster Board Waste	BC	Limestone Dust	0-50	-	D (18-54%)	-	-	-	Mixes with 40% replacement delivered most superior rutting resistance.	Kuttah et al., (2015)

Type of waste	Type of mix	Type of standard filler used	Substitution rate of standard filler (%)	Comparison of performance of waste filler incorporated mixes with standard mixes					Other remarks	References
				Marshall stability	Rutting resistance	Cracking resistance	Moisture resistance	OBC		
Municipal Solid Waste Incineration Ash (MSWIA)	SMA	Limestone Dust	100	D (1%)	I (5%)	D (11%)	D (7%)	I (1%)	MSWIA mixes had higher moisture sensitivity due to lower CaO and higher SiO_2 content.	Xue et al., (2009)
Sewage Sludge Ash (SSA)	SMA	Limestone Dust	100	-	I (2%)	I (5%)	D (2%) - I (2%)	Same	Resilient modulus of SSA mixes was found to be marginally lower than control mixes.	Tenza-Abril et al., (2014)
Coal Mine Waste (CMW)	BC	Limestone Dust	0-100	I (2-17%)	-	D (1%) – I (3%)	I (3-33%)	Same	Mixes having CMW & limestone in equal proportion formed most moisture resistant mixes, due to formation of cementitious components by lime and pozzolanic minerals of CMW.	Modarres and Rahmanzadeh (2014)
	BC	Limestone Dust	100	I (17%)	I (22%)	I (94%)	I (3%)	Same	Bitumen also found to stabilize the heavy metals present in the CMW & prevent their leaching	Modarres et al., (2015a)
Boron Wastes	BC	Limestone Dust	100	D (16%)	-	-	-	D (4%)	Si mineral in the borogypsum decreases aggregate-bitumen adhesion which reduced stability of the mix.	Kutuk-Sert and Kutuk (2013)
	BC	Limestone Dust	100	D (28%)	D (51%)	-	D (10%)	I (2%)	Boron waste consisted of relatively similar amount of SiO_2, CaO, and MgO in its composition.	Gurer and Selman (2016)
Oil Shale Waste	BC	Limestone Dust	100	D (9-27%)	D (1%) - I (25%)	-	-	I (5-6%)	Stability & rutting resistance in the mix decreased with increase in oil content.	Katamine (2000b)
Oil Shale Waste Ash	BC	Limestone Dust	0-100	D (10) - I (24)	D (40%) – I (200%)	D (6%) – I (4%)	I (12-21%)	Same	Oil shale ash exhibited pozzolanic properties and thus improve the moisture resistance of the mixes.	Asi and Assa’ad (2005)

Abbreviations: **I-**Increase; **D-**Decrease; **BC-**Bituminous Concrete; **SMA-**Stone Matrix Asphalt; **DBM-**Dense Bituminous Macadam**; PA-**Porous Asphalt.

2.4.2.1 Discussions and Insights

This section summarizes the performances of some various major waste filler modified mixes in primary aspects (Stability, rutting resistance, fatigue resistance, moisture resistance, and OBC) to mixes prepared with conventional fillers (stone dust, cement, hydrated lime, etc.). These results are shown in Table 2.14, which describe the range of performance these wastes show in comparison to conventional filler mixes.

Effect on Stability: All waste incorporated mixes provided satisfactory stability as demanded by their respective paving standards. It can be seen that out of 19 types of waste into consideration, nine types of waste mixes always have higher stabilities than conventional mixes, five always had lower stabilities while remaining wastes displayed mixed results. The improvement in Marshall stabilities in waste modified mixes were attributed to several factors such as improvement in stiffness and cohesion due to the reinforcing effect of non-spherical filler particles to the bitumen (Al-Hdabi, 2016), good physico-chemical interaction of filler with bitumen due to fineness of filler (Arabani et al., 2017; Modarres et al., 2015), high pozzolanic properties of CMW (Modarres et al., 2015), microcrystalline structure of filler (Katamine, 2000) and angular shape of filler (Arabani et al., 2017). Since all waste modified mixes delivered satisfactory stability, there should be no concern regarding the low stability on the field.

Effect on Rutting Resistance: Analysis of rutting resistance of mixes made from 24 different wastes is done in Table 2.14. Out of 24 wastes, 14 of them always showed higher rutting resistance than conventional mixes, whereas, 2 of them have shown lower results. The improvements in rutting resistance of waste modified mixes were attributed to factors such as higher stiffening of mastic due to the fine size of filler (Arabani et al., 2017), high physicochemical interaction between bitumen and filler due to angular shape and sharp particle edges (Arabani et al., 2017; Chandra and Choudhary, 2013), enhanced stiffness of mastic and mastic aggregate adhesion due to absorption of light components of bitumen by fillers (Mistry et al., 2017), chemical composition of filler (Zhang et al., 2018); high bitumen absorption due to large porosity (Chen et al., 2011b), and larger contact area of filler in mix due to its lower specific gravity (Chen et al., 2011b). MSWIA and SSA modified mixes have displayed lower rutting resistance than conventional mixes which may be attributed to their irregular particle shape and predominance of silica in their composition. It must be noted that phosphogypsum modified mixes displayed superior rutting resistance than conventional mixes in the absence of water as determined from Marshall quotient analysis. However, these mixes also found to be highly susceptible to water as they were collapsed under the water exposure in immersed wheel rut testing. This was attributed to the microcrystalline structure of phosphogypsum which loosens its mechanical interlock and softens in the presence of water. Brick dust mixes displayed the higher variability in results, they displayed not only superior rutting resistance due to their rough texture and angular particle shape (Arabani et al., 2017) but also displayed poor rutting resistance (higher permanent deformation in static creep test) (Kuity et al., 2014). Hence these materials should be utilized judiciously on the field.

Effect on Cracking Resistance: There are no well-defined provisions specified in paving specifications to design fatigue-resistant mixes. Out of 20 different types of waste filler modified mixes, 13 were found to have fatigue resistance higher than conventional mixes and MSWIA mixes displayed relatively inferior cracking resistance. The improvements in fatigue resistance in majority of waste modified mixes were attributed to factors such as: enhanced stiffness of mixes due to higher porosity of filler (Chandra and Choudhary, 2013), stiffening of mixes due to fine gradation of filler (Modarres and Bengar, 2017), improvement in cohesive strength of mixes (Muniandy and Aburkaba, 2010), hardening effect of $Ca(OH)_2$ present in composition of filler (Bocci, 2018).

Effect on Moisture Susceptibility: Out of 20 wastes, 8 types of mixes always had higher moisture resistance than conventional mixes, whereas 7 of them have shown mixed results. Seven types of materials (boron waste, MSWIA, recycled tyre rubber, red mud, glass powder, granite dust, and copper waste) have always shown relatively poor performance than conventional filler. The primary reasons behind this improved performance are: enhanced adhesion between aggregate and bitumen due to even distribution of fine filler particles (Mistry and Roy, 2018), low clay content in filler (Chandra and Choudhary, 2013; Kandhal and Parker, 1998; Sharma et al., 2010), pozzolanic properties of filler (Modarres et al., 2015), hydrophobic nature of filler (Chen et al., 2011b; Modarres et al., 2015), presence of adhesion promoters like calcite (Chandra and Choudhary, 2013) or free calcium etc. Brick dust mixes have shown high variability in their performance; in some studies, they have shown good performance which was attributed to the hydrophobic nature of brick dust (Chen et al., 2011b). Whereas, its poor performance was due to its high clay content and acidic nature (Kuity et al., 2014). Phosphogypsum and glass mixes have shown substantially poor performance in the presence of water. The poor performance of phosphogypsum mix is due to the microcrystalline structure of phosphogypsum which loosens its mechanical interlock and softens in the presence of water (Katamine, 2000). On the other hand, glass modified mixes have poor adhesion with bitumen due to high silica in its composition. High silica content was also the primary reason for the poor performance of MSWI and blast furnace slag mixes (Xue et al., 2009).

Effect on OBC: Physical and chemical properties of fillers have direct influence over volumetric properties of bituminous mix and thus affect the OBC of the mix. Majority of wastes modified mixes were found to have higher OBC than conventional filler mixes. Out of 29 types of mixes in consideration, five always have higher OBC than conventional mixes, five always have lower OBC. Higher OBC of waste modified mixes was due to higher bitumen absorption due to filler's: high porosity (Arabani et al., 2017; Chandra and Choudhary, 2013; Mistry and Roy, 2018), high specific surface area (Mistry and Roy, 2018; Taha et al., 2002), high surface roughness (Bocci, 2018; Chen et al., 2011a; Chen et al., 2011b) and particle shape and homogenous particle size distribution (Chen et al., 2011a; Sharma et al., 2010). Hence, in general observation, it can be said that inclusion of waste as filler increases the initial material cost of the mix. However, in many cases waste fillers with particle sizes smaller than bitumen film thickness exhibited bitumen extender function by which they can provide partial substitution of bitumen which also can economize the mix (Chandra and Choudhary, 2013; Sharma et al., 2010; Sobolev et al., 2014).

2.5 Summary

This literature review provided a brief introduction of filler, various standard tests for its physical and chemical characterization, the influence of its physical, chemical properties and proportion on performance of mastic and mixes, and suitability of various wastes from different sources as fillers. This literature review is covered in three sections. The first section discussed the need for the filler and the physical and chemical requirements of good filler as per the Indian specification. It delved into the influence of characteristics of filler and its quantity on the rheological properties of mastics and mechanical properties of the mixes. It also briefly discussed various standard test methods stated in different specifications to determine these properties. It was observed that filler plays an intricate role in influencing the performance of bituminous mix, which is dictated by its physical and chemical properties. However, the current Indian specification, (MoRTH, 2013) characterized fillers based on very limited criteria which might not be sufficient for reliable prediction for its performance.

The second section of this review discussed the primary distresses (rutting, cracking, and moisture sensitivity) occurring in bituminous pavements in Indian conditions. Ageing of bituminous mastic and mixes is not itself a distress, but since it catalyzes the occurrences of aforesaid distresses, it also has been included in the discussion. The section covers in detail about the various mechanisms of distresses, various standard and non-standard test methods for evaluation of these distresses in bituminous mastics and mixes, and how fillers affect these distresses. It has been helpful in identifying the effect of different types of fillers, their volume, and their physicochemical interaction with bitumen and aggregates on the various major distresses of bituminous mixes. The review has also been helpful for selecting appropriate test procedures for evaluating the rheological properties of mastics and performance of the mixes. There were only a few studies in which the influence of fillers on the long-term ageing performance of mixes was discussed.

The final section reviews conventional fillers and 30 wastes from different origins which could be utilized as alternative fillers. The advantages of utilizing hydrated lime as an anti-stripping agent as per different mechanism is explained in detail. In the case of waste fillers, the emphasis was given on their net production, their process of generation, their physical and chemical properties, and how they influenced the performance of bituminous mixes.

2.5.1 Identified Gaps

(a) The performance of bituminous mastics and mixes are dependent on several physical and chemical properties of fillers. However, in Indian specification (MoRTH, 2013), fillers are characterized by only the particle size distribution and plasticity index tests. There is a need to include simple, reliable, and economical test methods to determine properties like porosity and chemical composition of fillers in the specifications.

(b) The majority of the studies on the fillers are limited to their performance against primary distresses like rutting, fatigue, and moisture sensitivity. There are very limited studies which investigated the effectiveness of fillers on the performance of bituminous mixes in aspects like ravelling, active-passive adhesion, and long-term ageing. The author was unable to find any peer-reviewed Indian study in this context.

(c) The majority of the studies investigating the influence of waste fillers on the performance of bituminous mixes were restricted to a particular filler quantity and didn't cover the entire permissible range of fillers. Hence the effect of filler quantity on the performance of bituminous mixes is not well explored.

(d) There are very limited studies on several wastes that could be viably utilized as alternative fillers. In this study, the potential of Kota stone dust, and glass powder from dimensional stone and glass factory as fillers were explored. These wastes were seldom explored in the past as fillers, although they have high local availability and also fulfilled the requirements of fillers as per Indian specifications.

(e) There are also very limited studies which have done physical and chemical modification of waste fillers to improve their performance in the bituminous mastics and mixes. The performance of glass powder (which is expected to form moisture susceptible mixes due to its high silica content) can be improved by utilizing it along with the small quantity of hydrated lime as composite filler.

(f) Majority of the studies on fillers in the bituminous mixes are limited to exploring their potential against particular distress. Filler which delivers satisfactory against particular distress is not necessarily performed similarly against the other. However, there is a no well-defined methodology to rank various fillers based on the overall performance of their mixes in various aspects.

With these views, this study is taken up, to broadly analyse the effect of various waste fillers on the performance of bituminous mastics and mixes in various aspects. This will provide an insight into pavement engineers and contractors about the choice of different fillers depending on practical field conditions. The individual literature, its lacuna and the adopted study methodology on different aspects will be discussed in each chapter to make the need for the study more clear.