Laboratory test of physical, rheological, and chemical characteristics of aging binder modified with ZycoTherm and Evotherm

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The research aims to comprehensively assess the impact of anti-stripping agents ZycoTherm and Evotherm M1 on Warm Mix Asphalt (WMA). The study employs a range of laboratory tests to investigate the rheological and chemical properties of modified asphalt. Conventional tests such as penetration and ring & ball softening points are employed alongside advanced techniques including Dynamic Shear Rheometer (DSR), and Linear Amplitude Sweep (LAS) analyses to delve into the rheological properties. Chemical properties (SARA) were analyzed using thin-layer chromatography. Both short-term aging (STA) using The Rolling Thin-Film Oven (RTFO) and long-term aging (LTA) based on the Universal Simple Aging Test are simulated under controlled laboratory conditions. It was found in conventional tests that the value changed, but not significantly. In terms of rutting and fatigue performance, Zyc 0.05% showed positive results. Fatigue behavior shows Zyc has better resistance. The asphalt fractional composition test showed changes in each age condition.

Keywords: Asphalt aging, chemical additives, Chemical properties, Rheological.

1. Introduction

Hot Mix Asphalt (HMA) has been the dominant choice for road due to its excellent performance and durability. However, HMA production involves heating aggregates and bitumen to high temperatures, leading to significant energy consumption and greenhouse gas emissions. This environmental impact has spurred the need for a greener alternative - and that's where Warm Mix Asphalt (WMA) comes into play. Several studies have been conducted on WMA, including its performance, durability, and long-term effects on the environment [1], [2]. Overall, WMA has shown promise as a sustainable alternative to traditional hot mix asphalt, and its use is expected to continue to grow in the future.

The concept of using lower temperatures to produce asphalt mixes dates back to the 1950s [3]. Since then, several technologies have been developed to achieve this goal, including foaming nozzles, synthetic zeolites, and chemical additives [4]. The use of WMA has become increasingly popular in recent years due to its environmental benefits and potential cost savings [2]. WMA technology can be considered a green road construction method. Through this technique, asphalt mixtures can be compacted under lower temperatures by adding warm mixes [5]–[8]. WMA can be divided into three major categories: foaming technology, organic additive technology, and chemical agent technology. Two types of foaming technologies can be distinguished: direct foaming and indirect foaming [8]–[10]. For the former method, water is injected through a manual nozzle to create foam in asphalt binders, whereas for the indirect method, finely crushed synthetic zeolite is directly added to asphalt mixes. The use of organic additives has the advantage that since they contain wax, they reduce asphalt binder viscosity [12]–[15]. Nevertheless, wax lowers warm mix road performance to a certain extent [16], [17]. It has been shown that chemical agents improve the coating of aggregates in warm mixes without reducing their viscosity. Moreover, chemical agents can enhance warm mix asphalt properties, such as workability, adhesion, and compaction resistance [18]–[20]. It is possible to develop high-performance warm mixes by using chemical agents. Moreover, these agents reduce surface tension and facilitate a smooth interaction between asphalt binders and aggregates, thereby lowering production temperatures without affecting asphalt properties [21]. Hence, chemical agents are a good candidate for warm mix technology.

Evotherm M1 is a third-generation warm mix asphalt additive manufactured by Ingevity as one of the recent products used in WMA technology by MeadWestvaco Company's. This is surfactant *based* contains of two different groups: a hydrophilic group rich at the end of the cation, and a lipophilic group at the end of the nonpolar alkane [22]. The use of Evotherm M1 in paving applications has been shown to offset greenhouse gases generated in its manufacture by up to 23 times [23]. It is a water-free additive that can reduce the mixing temperature by 33°C to 45°C [23], improves adhesion by acting as both a liquid antistrip WMA [1], [24], [25]. Evotherm M1's rheological properties are generally unknown due to a limited amount of available information. However, a little information can be written as follows: M1 improved the penetration and ductility of high viscosity modified asphalt and decreased its softening point. The surface-active warm mixes increased the deformation resistance of high-viscosity bitumen and also enhanced the deformation resistance of the warm mixes [8]. With Evotherm M1, the mixing and compaction temperatures of the asphalt mixture can be effectively reduced by approximately 13 and 14 ºC, respectively, by the selected Styrene-Butadiene-Styrene (SBS) - modified asphalt [22]. Based on Brookfield rotational viscosity tests, Evotherm modifiers can reduce bitumen's high-temperature viscosity and improve its construction. In an analysis of dynamic shear rheology, Evotherm found that adding modifiers increased bitumen's elastic component, which increased its resistance to rutting [26]. Evotherm M1 has a very slight improvement in rutting resistance when aged by RTFO and unaged. But with the recycling binder, M1 improved fatigue performance [27]. The JNR values for MSCR were lower when Crum Rubber was added; these values increased when M1 was added. Conversely, asphalt binder recovery rates increased with CR but decreased with M1 [28].

ZycoTherm as an additive for warm mix asphalt is a promising alternative technology to decrease asphalt production temperature to 120-135 ºC and compaction temperature to 105-120 ºC [29]. ZycoTherm SP is an organosilane-based liquid anti-strip (LAS) additive used as a surfactant additive with the distinct advantage of traditional WMA [30] manufactured by Zydex Industries from Gujarat India. Ziari et al. [31] found that ZycoTherm did not significantly influence the elastic responses, fatigue, or rutting properties of the binder. The MSCR test was performed on asphalt binders of 85/100 grade by Mirzababaei et al., [32]. As a result, ZycoTherm appeared to reduce non-recoverable compliance values (J_{nr}), which is indicative of lower rutting distress susceptibility. A study by Raufi et al., [33] examined the performance of ZycoTherm modified binder after RTFO (short-term aging) showed improved rutting characteristics, and led to increasing $G^*/\sin\delta$ rates in comparison to a control binder. Ameli et al. (2023) ZycoTherm increased the softening point of the binder and J_{nr} but decreased its penetration degree. LAS tests showed that 0.3% increased binder performance at intermediate temperatures. The dosage range (0.08–0.12%) of

ZycoTherm at high, intermediate, and low temperatures does not affect rheological properties either positively or negatively studied by [24], [31], [32].

Within the context of this laboratory study, it was initially aimed to assess the influence of the Evotherm and ZycoTherm on the physical, rheological, and chemical properties of asphalt binder at the un-age and aged condition.

2. MATERIALS AND EXPERIMENTAL DESIGN

2.1 Binder

The control asphalt binder (neat) used in this study was a 60/70 penetration grade base bitumen from a Taiwan company. It had physical properties as Table 1.

Table 1. Reference properties of neat.

Property (Unit)	Standard	Result
Unage		
Penetration, 25°C, 100 g, 5s (0.1 mm)	ASTM D5	72.90
Penetration index	-	-0.60
Softening point (°C)	ASTM D6090	48.74
Specific gravity G_b	ASTM D70	1.035
Viscosity, 60°C (cP), 135°C (cP)	ASTM D4402	208000, 521
G*/sin δ, kPa (64˚C)	AASHTO T315	1.5437
Short term aging	ASTM D1754	
Penetration, 25°C, 100 g, 5s (0.1 mm)	ASTM D5	44.58
Penetration index	-	-1.34
Softening point (°C)	ASTM D6090	50.45
G*/sin δ, kPa (64˚C)	AASHTO T315	3.07
Long term aging	USAT	
Softening point (°C)	ASTM D6090	60.88

2.2 Additives

Table 2. Additive properties

Properties	ZycoTherm	Evotherm M1
Physical state	Liquid	Liquid
Color	Pale Yellow to brownish yellow	Dark amber liquid
Density gram/cc	1.03 ±0.01	0.99
Viscosity	<150 CPS (at 30 ±2 °C)	150-300 at 100°F (38°C)
Dosing range (by binder)	0.05-0.1 %	0.25-1%
Odour	Slight aromatic	Amine like

Company	Zydex, India	MWV Specialty Chemicals. Virginia Ave.

Evotherm M1 (Evo) and ZycoTherm (Zyc) in warm mix asphalt has been shown to have potential benefits in terms of reduced mixing temperature, improved adhesion, and environmental impact [23], [35]–[37]. EM 1 0.5, 0.75, and 0.1% (by weight of binder) were used based on supplier recommendations and previous studies [28], [38], [39]. Zyc in this study were 0.05, 0.07, and 0.1% (by weight of the binder) based on supplier recommendations and previous studies [27], [35]. The Additive properties can be seen in the Table 2 above:

2.3 Binder preparation and ageing procedure

Asphalt is heated to 160°C and placed in a container. Chemical additives are dripped according to dosage. To achieve homogeneity, the mixture is blended using shear mixers and stirrers with a 4-blade at 2000 rpm at a temperature of 135 to 140 °C for 15 minutes.

Short-term aging (STA) was performed using the RTFO (Rolling Thin Film Oven) method, specified in ASTM Standard D2872-12 (2013). This method simulates the aging process during asphalt production, distribution, and compaction. In the RTFO test, approximately 35 grams of unaged asphalt binder are placed in a rotating oven for 85 minutes at a temperature of 163°C, with a flow rate of 4 liters per minute.

For long-term aging (LTA), the Universal Simple Aging Test (USAT) method was used. This method was developed by the Western Research Institute (WRI) under the guidance and funding of the Federal Highway Administration (FHWA). In the USAT, asphalt binders are aged in a forced draft oven for 40 hours at 100°C. The aging process is carried out on a plate with three slots, allowing for the aging of three different films. Each slot requires approximately one gram of asphalt binder, resulting in a film thickness of about 300 μm. At a temperature of 120°C, the asphalt covers about two-thirds of the plate, and any remaining uncovered surface is manually covered using a spatula.

3. Experimental design

Several tests were carried out to assess the impact of chemical additives in warm mix asphalt on binder performance.

3.1 Conventional properties

The binder's conventional characteristics tests, such as penetration and softening point, were evaluated using ASTM D5 [40] and ASTM D36 [41] respectively. These tests were conducted under un-aged, short-term aging conditions and long term aged for softening point.

3.2 The rheological characteristics of asphalt binders

The asphalt samples were subjected to rheological testing using an Anton Paar MCR 102e Dynamic Shear Rheometer (DSR) according to AASHTO T 315 [42]. Parallel geometry was applied, with a sample diameter of 8 mm and a height of 2 mm. DSR is fundamental to analyzing and understanding viscoelastic material properties. Using this method provides valuable insights into the development of new materials by providing key parameters such as the complex shear modulus (G*) and phase angle (δ). The complex shear modulus (G*) indicates the material's elasticity and ability to endure strain before failure. On the other hand, the phase angle (δ) measures the material's energy absorption capacity and predicts its cyclic performance.

Rutting resistance factor (G*/sinδ)

Rutting resistance factor (G*/sinδ) is a measure of asphalt binders' ability to resist deformation and rutting at high temperatures [43]. It is calculated using the complex shear modulus of the elastic portion, G*, and the phase angle, δ, which is the angle between the stress and strain waves in the material [44]. The rutting factor, G*/sinδ, varies with temperature and is regarded as a rutting resistance indicator that evaluates high-temperature asphalt performance [45] around 46, 52, 58, 64 and 70 ºC at un-aged and STA condition according to AASHTO T 315 [42].

Linier Amplitude Sweep (LAS)

The LAS test is performance-based and measures damage resistance. It is an oscillatory strain sweep test that applies cyclic loading at increasing amplitudes. This is done to assess asphalt binders' fatigue resistance [46]. DSR device equipped with a parallel plate of 8 mm diameter and a 2 mm gap between the plates was utilized with LTA condition. In the LAS test, failure is defined as a reduction of 35% in the initial modulus [47]. During the testing process, peak shear strain and peak shear stress are measured every 10 load cycles (1 second). The phase angle (δ) and complex shear modulus (|G*|) are also recorded. To determine the fatigue life of the material at different strain amplitudes, the viscoelastic continuum damage (VECD) method is employed. Equation (1) calculates the number of cycles until failure. By utilizing the VECD model, the relationship between the number of cycles to failure (Nf) or fatigue life and the applied strain can be determined [48].

$$N_f = A\,(\gamma_{max})^B \tag{1}$$

In the equation, Nf represents the bitumen fatigue performance parameter, while γmax corresponds to the maximum strain endured by the bitumen in a specific pavement structure (expressed as a percentage). The coefficients A and B in the VECD model are influenced by the material's properties. It is commonly observed that binders with better fatigue resistance tend to have higher A values but lower absolute B values.

3.3 Chemical properties

Saturates, Asphaltenes, Resins, Aromatics (SARA)

5.2 Fatigue Behavior (LAS)

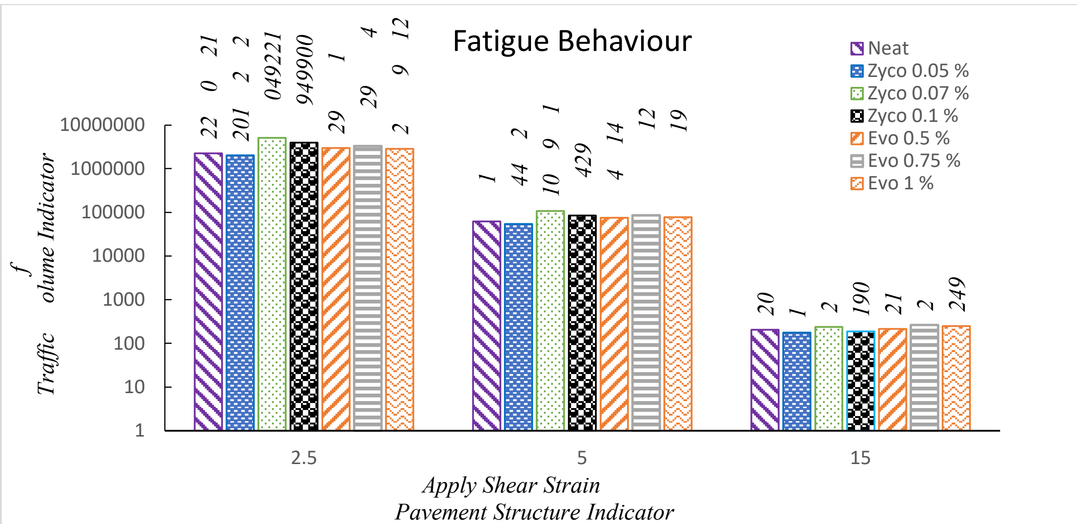

Fig. Effect of binder and strain levels on cycles to failure (N modified binders.

Fig. displays the N *modifier binders for the different strain levels 2. , 1 respectively. o strain levels usually experience an increase in fatigue life as viscosity increases . At 2. strain, yco 0.0 and 0.1 sho values greater than neat. The percentage difference bet een the t o is uite significant about 1 0 and 4 . In contrast, 0.0 indicates that the asphalt modifier is susceptible to fatigue. The Evo modifier sho ed positive results ith percentage levels of 4 , 2, and 2 respectively at 0. , 0. , and 1 , respectively. At strain, yco 0.0 and 0. sho ed significant results ith a percentage increase in value of and hile 0.0 eak against fatigue. Evo illustrates a trend here 0. , 1, and 0. have relatively higher values than neat, about 9, 24, and 21 respectively. Finally, at 1 sectional strain, 0.0 yco displayed a value 1 greater than neat, hile the other dosage forms ere eak against fatigue. It is interesting to note that Evo exhibited positive results ith a difference of 29, 21, and for the modifier, respectively 0. , 1, and 0. .*

It can be concluded that yco 0.0 and Evo 0. have better fatigue resistance than other doses. This indicates that these t o dosage forms can be used to improve the fatigue resistance of the material. It is also important to note that different dosage forms may be more beneficial in certain applications and therefore should be considered according to the specific re uirements.

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