Rheological Properties of Bituminous Binders with Synthetic Wax

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Abstract: Warm Mix Asphalt (WMA) has been introduced to reduce the environmental impact and improve energy efficiency of bituminous mixture production and application. With this purpose, synthetic waxes are effectively used to reduce temperature during mixing and compaction. Given these advantages, the basic WMA challenge is the production of a pavement mixture having at least the same performance as traditional HMA. This study describes the effect of a synthetic wax additive on the rheological properties of a paving-grade bitumen in a wide range of temperatures. The effect of wax at mixing and compaction temperatures (T > 100°C) was evaluated using viscosity tests. A Dynamic Shear Rheometer (DSR) and a Bending Beam Rheometer (BBR) were used to investigate the rheological properties of the base and the WMA binder at midrange and low service temperatures (from 18° C to 37° C). The time-temperature superposition principle was applied to analyze the complex shear modulus and the creep stiffness modulus through the construction of master curves. Particular attention was devoted to the study of the wax's effect on low temperature physical hardening.

Key words: Physical hardening; Rheological characterization; Synthetic wax; Warm asphalt binder.

Introduction

Background

In the last decade, a number of processes and products have been introduced to reduce the environmental impact and improve energy efficiency of bituminous mixture production and application. These technologies have been categorized based on the temperature reduction achieved during mixing and paving operations [1]. According to this criterion, Warm Mix Asphalts (WMA) are characterized by production temperatures 15°C to 40°C below traditional Hot Mix Asphalts (HMA). Lower production temperatures reduce fuel consumption and emissions such as bitumen fumes, carbon dioxide, and other greenhouse gases [2-4]. Lower placement and compaction temperatures also allow better working conditions, longer haul distances, and shorter construction operations. Given these advantages, the basic WMA challenge is the production of a pavement mixture having, at least, the same performance as traditional HMA.

A family of WMA technologies is based on the reduction of bitumen viscosity during the mixing phase by means of foaming processes, thus enhancing aggregate coating and mixture workability. Some of these technologies use the traditional foaming process directly (WAM-Foam, LT-Asphalt, LEAB [5]). In other applications, a controlled amount of water is brought into the mixture by means of synthetic zeolite or aggregates (Aspha-min[®], LEA [5]). In these cases moisture produces a foaming process in the mixing phase.

Chemical additives, such as polyphosphoric acid [6, 7] and surface active agents [8] are also used to reduce asphalt viscosity or

increase lubricity at mixing temperatures. Today, bitumen modification with fluidifying agents is achieved mostly by the addition of commercial functionalized waxes [9, 10]. The term "wax" is generally used to indicate an organic compound that is solid at ambient temperature and melts at higher temperatures, producing a low viscosity liquid.

Natural wax, present in almost all paving-grade bitumens (paraffinic components), has been considered a negative factor for bitumen quality. However, contents below 3% generally are not considered harmful for paving applications [11]. Synthetic waxes are currently used to improve flow.

In their state of the art review on wax in bitumen, Edwards et al. [12] summarized many aspects related to the influence of wax on physical and mechanical properties of bitumen. At mixing temperatures (i.e., above its melting point), wax reduces bitumen viscosity, enhancing aggregate coating and mixture workability. In the melting temperature range, as a consequence of the crystallization process, waxy additives can give rise to sudden changes in bitumen viscosity. At high pavement service temperatures, the stiffening effect produced by wax crystallization, has been used to improve resistance to permanent deformation. At mid-range and low service temperatures, wax may increase fatigue and thermal cracking susceptibility.

A link between wax content and low-temperature physical hardening of bitumens has also been suggested. Even if a cause and effect connection was not proved [13], the relationship between these two variables should be investigated, especially when wax is added to the binder.

Research Objectives and Description

This study evaluated the effect of a synthetic wax additive on the rheological properties of a paving grade bitumen. A wide range of temperatures was investigated to obtain a global picture of wax-binder interaction. The wax effect on flow properties of the binder was measured at mixing and compaction temperatures (T > 100° C). At midrange service temperatures, complex modulus tests were carried out using a Dynamic Shear Rheometer (DSR). Low

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temperature properties, stiffness, and physical hardening were investigated using a Bending Beam Rheometer (BBR). Long-term aged binders were tested with BBR and DSR. The time-temperature superposition principle (TTSP) was applied to analyze the rheological parameters through the construction of master curves. A similar principle was used to evaluate low temperature physical hardening using a hardening shift factor [13, 14].

Materials and Experimental Program

Materials

A penetration grade bitumen 70/100, regularly used in central and southern Europe for pavement construction, was selected as the base binder for this study (code "T"). This bitumen was modified by adding a commercial WMA additive called Sasobit[®] [15] (code "W"). The additive, typically used as flow improver, is a synthetic paraffin wax obtained from the Fischer-Tropsch process (F-T). The F-T process is used to convert synthesis gas containing hydrogen and carbon monoxide to hydrocarbon products that are mostly liquid at ambient temperature, such as alcohols, diesel fuel, kerosene, and waxes [16]. The F-T wax is in the carbon chain length ranging from C40 to C120, with a melting point generally above 90°C [9, 10]. On the other hand, paraffins naturally included in bituminous binders, have a carbon chain length ranging from C25 to C50 and a melting point ranging from 45°C to 70°C. Longer carbon chains in the F-T wax lead to a higher melting point and the smaller structure reduces brittleness at low temperatures [12].

A WMA additive dosage of 3% by weight of binder was selected, as recommended by the producer and confirmed in previous experimental studies on mixtures [17, 18]. Quantities as high as 6%, tested by other researchers, showed good performances with very soft binders (160/220 pen) [6, 7, 19]. Indeed, with harder bitumen (30/50, 50/70 pen and Pmb), problems may arise at midrange and low temperatures. In particular, the brittle behavior of the crystallized wax can have a negative impact on cracking susceptibility [20, 21].

The binder modification was obtained in the laboratory, where the prefixed amount of wax pellets was added to the hot binder (150 °C), with a portable mixer operating at high stirring rates.

Testing Protocols and Equipment

Preliminary Classification Tests

The base and the wax-modified binders were graded according to European empirical specifications (EN 12591) and Superpave performance specifications (AASTHO M320). Empirical test methods performed on unaged binder included: penetration at 25° C (EN 1426), softening point (EN 1427), Fraass breaking point (EN 12593), and dynamic viscosity (EN 12596). As far as the PG characterization is concerned, unaged, short-term aged with Rolling Thin Film Oven Test (RTFOT, AASTHO T 240), and long term aged with Pressure Aging Vessel (PAV, AASTHO R 28) binders were tested with the Dynamic Shear Rheometer (AASHTO T 313) and the Bending Beam Rheometer (AASHTO T 315).

Viscosity Tests

The influence of wax on flow properties of the base binder was studied with dynamic viscosity tests, carried out according to EN 13302. A constant shear rate (10 s^{-1}) was applied using a coaxial viscometer, at temperatures ranging from 100 °C to 160 °C, performing three replicates for each test temperature.

Dynamic Mechanical Analysis

Linear viscoelastic behavior of the binders at midrange service temperatures was investigated using a Dynamic Shear Rheometer (DSR). Long term aged binders, obtained after RTFOT and PAV procedures, were tested with frequency sweeps (f = 0.1, 1, 3, 10 and 30 Hz) at 5, 13, 25, and 37 °C, performing three replicates for each temperature and frequency. The norm of the complex shear modulus $|G^*|$ and the phase angle δ were evaluated applying a strain level of 1%. Hereafter in this paper, the norm of the complex shear modulus will be called simply complex modulus, and denoted as G^{*}.

BBR Tests

Low temperature performance of binders was investigated using a Bending Beam Rheometer (BBR). Creep tests were carried out on long term aged binders, obtained after RTFOT and PAV procedures, at -6, -12, and -18°C, performing three replicates for each temperature. The stiffness modulus S(t) was evaluated at t = 8, 15, 30, 60, 120, and 240 s. At each temperature, tests were carried out after 1-hour and 24-hour conditioning in order to evaluate the wax's influence on low temperature physical hardening.

Data Analysis

The time-temperature superposition principle was applied to describe the rheological behavior of the binders. Isothermal curves of complex modulus G*, phase angle δ , and stiffness modulus S(t) obtained from DSR and BBR tests, have been shifted using temperature-dependent shift factors, in order to obtain master curves. This allows the estimation of mechanical properties at ranges of temperature and frequencies that were not investigated in the laboratory.

The master curve model specifically developed by Bahia et al. [22, 23] to characterize bituminous mixtures and modified binders was used to fit DSR test results. For complex modulus of binders the equation reduces to:

$$G(f') = \frac{G_g}{\left[l + (f_c/f')^k\right]^{m_e/k}}$$
(1)

where: G_g is the glass complex modulus $(f' \rightarrow \infty)$, f_c is a frequency location parameter, and k, m_e are dimensionless shape parameters. At the frequency f_c , the distance between G(f') and G_g , on a logarithmic scale, is given by the parameter $R = m_e \log 2/k$.

The model equation for phase angle is:

Table 1. Test Results on Binders T and W for Empirical Classification, with Comparison to Previous Studies' Results [26, 27].

Binder Code	Penetration @ 25°C	Softening Point	Viscosity @135°C	Fraass Breaking Point	Penetration Grade
	[mm/10]	[°C]	[Pa•s]	[°C]	
Т	71	44	0.188	-10	70/100
W	64	49	0.163	-8	50/70
Base Bitumen C	71	49.6	0.460	-	-
C+3% Sasobit	50	79.9	0.320	-	-
Base Bitumen S	76	45.2	0.400	-	-
Base+3% Sasobit	44	71.5	0.323	-	-

Note: C denotes bitumen from study [26], S denotes bitumen from study [27]

Table 2. Test Results for PG Binder Characterization (Standard Values/Requirements Showed in Brackets).

UNAGED – Binders	Binder Code	$G^*/sin\delta$ [kPa]	Failure Temperature [°C]	η @ 135°C [Pa·s]	
	Т	1.24 (≥ 1)	59.75 (58)	0.1875 (≤3)	
	W	1.47 (≥ 1)	66.8 (64)	0.1625 (≤ 3)	
RTFOT - Residue	Binder Code	$G^*/sin\delta$ [kPa]	Failure Temperature [°C]	Mass Loss [%]	
	Т	4.47 (≥ 2.20)	62.9 (58)	0.143 (≤ 1)	
	W	2.30 (≥ 2.20)	70.45 (70)	0.110 (≤ 1)	
RTFOT+PAV - Residue	Binder Code	$G^* \sin \delta$ [kPa]	Failure Temperature [°C]	S @ -6℃ [MPa]	<i>m</i> @ −6°C
	Т	3420 (≤ 5000)	25.1 (25)	147.5 (≤ 300)	0.348 (≥ 0.3)
	W	4581 (≤ 5000)	30.15 (30)	182.5 (≤ 300)	0.291 (≥ 0.3)
	Binder T: PG 58-16		Performance Grade (PG)	Binder W: PG 64-10	

$$\delta = 90I - (90I - \delta_m) \left\{ 1 + \left[\frac{\log(f_d / f')}{R_d} \right]^2 \right\}^{-m_d/2}$$
(2)

where: δ_m is a phase constant, f_d is a frequency location parameter, R_d , m_e are shape parameters and I = 0 if $f' > f_d$ or I = 1 if $f' \le f_d$.

In Eqs. (1) and (2), f' is the reduced frequency obtained by the product of the test frequency and the temperature shift factors (f' = a(T)f).

Regarding the creep stiffness modulus S(t) obtained from BBR, test results are usually modeled adopting the equation:

$$S(t) = A + B\log(t) + [C\log(t)]^2$$
 (3)

These isothermal curves were shifted to obtain master curves and, for this purpose, the same analytical expression was used to fit the data:

$$S(t_r) = A_m + B_m \log(t_r) + [C_m \log(t_r)]^2$$
(4)

in this case, t_r is the reduced time obtained by the product of the test time and the temperature shift factors ($t_r = a_t(T)t$), while A_m , B_m and C_m , are experimental constants.

For rheologically simple materials, the effect of isothermal hardening on viscoelastic properties is similar to the effect of temperature. This behavior was also observed for physical hardening of bituminous binders about which a hardening shift factor $a_p(t_s)$ can be defined [13] to account for superposition between loading time and isothermal storage time (t_s) . Consequently, the creep stiffness curves obtained after isothermal storage can be shifted along the time axis without changing their shape [14]. In this study, hardening shift factors were calculated for each storage

temperature.

Results and Discussion

Preliminary Classification Tests

Results of the grading tests are summarized in Table 1 and Table 2. The stiffening effect due to the addition of 3% synthetic wax was confirmed by both empirical and Superpave tests. According to European specifications (EN 12591), the binder grade changed from 70/100 to 50/70. Correspondingly, the performance grade changed from PG 58-16 to PG 64-10. Therefore, from a specification point of view, the WMA binder showed improved performance at high service temperatures (rutting resistance), and a lower resistance to low temperature cracking. Both these effects can be explained by the synthetic wax crystallization and are well documented in the literature [7, 24, 25].

The lower part of Table 1 summarizes results of two previous studies [26, 27] on similar pure binders modified with 3% FT wax. Although such comparisons must be considered with care because of the potential differences in sample preparation and test procedures, the similarities with the results of the present study are clear.

It must be also noted that the changes in performance of the warm-mix binder could be altered by the reduced binder aging due to the lower production temperatures of WMA mixtures. In fact, a recent study confirmed [28] the significant influence of short term aging temperatures on the high temperature performance of the asphalt binder measured by $G^*/\sin \delta$, while the effect of the same parameter is negligible after long term aging. Reduced aging could lower the positive impact on rutting resistance as the values of $G^*/\sin \delta$ will be lower, while the negative impact of the low temperature grade remains the same.



Fig. 1. Viscosity Test Results.



Fig. 2. Synthetic Wax Effect on BBR Master Curves at Reference Temperature -12°C (1h Conditioning).

Flow Properties

The reduction of binder viscosity at mixing and compaction temperatures is considered to be the primary function of WMA additives. Dynamic viscosity data for binders T and W are shown in Fig. 1. Above the wax melting point, from 100°C to 160°C, the viscosity reduction ranges from 25% to 35%. These results are in agreement with those published by other authors [25, 27] and can

also be reported as a reduction of mixing and compaction temperature from 6°C to 8°C. These changes are limited and are not expected to cause major savings in emissions and energy required for construction.

Low Temperature Properties

In order to investigate wax influence on low temperature binder properties, BBR creep tests were carried out at -6, -12 and -18°C on long term aged binders, obtained after the RTFOT and PAV procedures. The PG determination only focused on the SHRP parameters S(60) and m, while the low temperature rheological behavior was analyzed measuring the stiffness modulus evolution during the test and applying the time-temperature superposition principle to obtain stiffness master curves.

For samples subjected to 1 h isothermal conditioning, the master curves at the reference temperature of -12°C are shown in Fig. 2. The shift factors were calculated using a numerical optimization procedure, and are shown in Table 3, together with the parameters of Eq. (4). The W-binder master curve indicated an increase of stiffness at all reduced loading times in comparison with the base binder. Moreover, the shape of the master curve became flatter, indicating that the addition of synthetic wax decreased the stress relaxation rate of the binder. Hence, the influence of wax crystallization on low temperature behavior cannot be described with a simple horizontal translation of the base binder master curve.

The effect of wax on low temperature physical hardening was described using the superposition principle between loading time and isothermal conditioning time. This is illustrated in Fig. 3, where the isothermal creep curves obtained after 24 h conditioning were shifted along the loading time axis, towards the reference 1 h conditioning curve. Initially, the 1 h isothermal curves were fitted using Eq. (3). Then, the 24 h curves were horizontally shifted to match the corresponding isothermal equation. The hardening shift factors $a_p(t_s)$ found at each test temperature (Fig. 4) represent the equivalency between loading time and isothermal storage time (t_s) : the higher the shift factor, the higher the hardening effect. For both binders, physical hardening significantly increased the creep stiffness. For the W-binder, the shift factors showed an increasing tendency of hardening as temperature decreased, while the same

Table 3. Estimated Shift Factors and Parameters for BBR Master Curv	ves
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Conditioning Time	Binder Code	T [℃]	$a_t(T)$	A_m	B_m	C_m
1 h		-6	1.1900			
	Т	-12	0	876.6	-378.0	44.2
		-18	-0.9827			
		-6	1.2211			
	W	-12	0	909.9	-369.5	41.9
		-18	-0.9359			
24 h		-6	1.4482			
	Т	-12	0	1019.6	-371.1	36.3
		-18	-1.0514			
		-6	1.4253			
	W	-12	0	1014.0	-365.3	37.3
		-18	-1.1939			



Fig. 3. Horizontal Creep Curves Shifting for (a) the Base Binder and (b) the Modified WMA Binder.



Fig. 4. Hardening Shift Factors.



Fig. 5. DSR Results: Master Curves at Reference Temperature 25 °C and Measured Data for Complex Modulus (G^*) and Phase Angle (δ).

Table 4. Estimated Parameters for DSR Master Curves.

Binder	D	***	1-	f
Code	Λ	m _e	ĸ	JC
Т	1.180	0.661	0.169	239.0
W	1.117	0.553	0.149	239.0
Binder	2	f,	DJ	122
Code	o_m	Ja	Ка	m_d
Т	39.8	3.521	1.029	0.576
W	29.7	9.250	0.810	0.310
	Binder Code T W Binder Code T W	Binder Code R T 1.180 W 1.117 Binder Code δm T 39.8 W 29.7	$\begin{array}{c c} {\rm Binder} & R & m_e \\ {\rm Code} & R & 0.661 \\ {\rm T} & 1.180 & 0.661 \\ {\rm W} & 1.117 & 0.553 \\ \\ {\rm Binder} & & & & \\ {\rm Code} & & & & & \\ {\rm Code} & & & & & & \\ {\rm T} & 39.8 & 3.521 \\ {\rm W} & 29.7 & 9.250 \end{array}$	$\begin{array}{c c} {\rm Binder} \\ {\rm Code} \end{array} & R & m_e & k \\ \hline {\rm T} & 1.180 & 0.661 & 0.169 \\ {\rm W} & 1.117 & 0.553 & 0.149 \\ \hline {\rm W} & 1.117 & 0.553 & 0.149 \\ \hline {\rm Binder} & & & & \\ {\rm Code} & & & & f_d & Rd \\ \hline {\rm T} & 39.8 & 3.521 & 1.029 \\ {\rm W} & 29.7 & 9.250 & 0.810 \\ \hline \end{array}$

trend was not observed for the base binder. This particular behavior was also found for polymer modified binders [14]. At -6 and -12°C, the effect of wax seemed to be a reduction of the hardening effect. Considering that the W-binder had a higher initial stiffness, this suggests that the stiffening effect produced by the addition of wax was simply added to that produced by isothermal storage. At -18°C the W-binder showed a higher hardening shift factor, reflecting a different interaction mechanism between wax crystallization and the reorganization of the bitumen microstructural systems that is involved in physical hardening [14].

Midrange Service Temperature Properties

DSR frequency sweeps were performed on long-term aged bitumen samples obtained after RTFOT and PAV procedures. Four temperatures (5, 13, 25 and 37 °C), five frequencies (0.1, 1, 3, 10 and 30 Hz), and a 1% strain level were used to elicit the viscoelastic behavior of the base and the modified binder. Results are presented in Fig. 5 in terms of complex modulus G* and phase angle δ master curves, at the reference temperature of 25 °C. The model parameters described in Eqs. (1) and (2) are reported in Table 4. For both binders, the glass complex modulus G_g was fixed at 106 MPa [22, 29].

The G* master curves (Fig. 5) showed that addition of wax resulted in a stiffness increase at midrange service temperatures. The stiffness ratio of the base to the W-binder is clearly frequency-dependent, as the difference between the two binders tends to vanish as reduced frequency increases. This trend is similar to that found for BBR creep stiffness master curves (Fig. 2), with reducing loading time. Again, the wax effect could not be described with a simple horizontal translation of the master curve. Other researchers found a similar trend for isochronal (10 rad/s) G* curves, as temperature decreases [7] in accordance with the time-temperature superposition principle.

The phase angle master curves (Fig. 5) show that, in the range of reduced frequencies covered by the DSR test results, the G* increase produced by the WMA additive was accompanied by a decrease of δ . The reduction of sin δ partially balances the effect of the G* increase and consequently mitigates the negative effect of the WMA additive on the value of the fatigue parameter G*sin δ [30] (Fig. 6).





Fig. 6. Storage (*G*') and Loss (*G*'') Components of the Complex Modulus.

A comparison between the stiffness master curves can also be done in terms of the rheological parameters R and f_c . A higher R value indicates a more gradual transition from the elastic to the viscous behavior, whereas a higher f_c value indicates a greater viscous component in the behavior [22, 23]. Results in Table 4 show that the wax's effect on these parameters was negligible. This suggests that, besides the higher stiffness, the addition of wax did not lead to significant modifications of the relaxation spectrum and did not increase the viscous response in the linear visco-elastic (LVE) domain.

Conclusions

The effects of synthetic wax addition to a traditional asphalt binder were measured using both conventional and rheological tests. The range of temperatures investigated ranged from 160°C for viscosity to -18°C for low temperature stiffness and isothermal hardening. The following conclusions can be drawn from the analysis of the experimental data:

- According to European specifications (EN 12591), binder grade changed from 70/100 to 50/70 and the Superpave PG changed from 58-16 to 64-10. This indicates that the addition of the WMA additive used in this study could improve performance at high service temperatures (rutting resistance), but resulted in lower resistance to low temperature cracking as measured by the stiffness and logarithmic creep rate.
- At mixing and compaction temperatures (T = 100°C 160°C) the viscosity reduction due to wax addition ranged from 25% to 35%. This can also be reported as a reduction of mixing and compaction temperature from 6°C to 8°C. These changes are limited and are not expected to cause major savings in emissions and energy required for construction.
- At midrange service temperatures (T = 5° C 37° C), a frequency-dependent increase of stiffness was observed: the complex shear modulus increase produced by wax addition tends to vanish at higher reduced frequency values. Therefore, the wax's effect could not be described with a simple horizontal shift

of the master curve.

- The phase angle master curves showed that, in the range of reduced frequencies covered by the test results, there is reduction of phase angle that partially balances the effect of the G^* increase and mitigates the negative effect of the WMA additive on the fatigue parameter $G^* \sin \delta$.
- At low service temperatures (T < -6°C), wax addition increased the creep stiffness modulus, and decreased the stress relaxation rate of the binder. Similar to the effects at intermediate temperatures, the influence of wax on low temperature behavior could not be described with a simple horizontal translation of the master curve.
- Low temperature physical hardening significantly increased creep stiffness for both the traditional and the wax-modified binder. At -6°C and -12°C, the effect of wax induced a reduction of the hardening rate effect, which is probably because the wax-modified binder had a higher initial stiffness. This suggests that the stiffening effect produced by the addition of wax was simply added to that produced by isothermal storage.
- The overall effects of the wax on bitumen performance parameters raise some concern regarding inferior resistance to thermal cracking. These effects could negatively impact pavement cracking resistance in low temperature climates. The assumption that this WMA additive will result in equal performance levels is thus not valid and consideration of such effects should be considered carefully before similar additives are used.

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