

WAX ADDITIVES IN WARM-MIX ASPHALT BINDERS AND PERFORMANCE IN THE MULTI-STRESS CREEP AND RECOVERY TEST (MSCR)

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ABSTRACT

Addition of select waxes (Fischer-Tropsch and Fatty Acid Amides) to asphalt is an accepted practice in warm-mix asphalt production. Additionally Fischer-Tropsch, Montan wax and Montan wax blends have been used in Europe for several years as compaction aids for bituminous mixtures. Addition of waxes to binders prompts concern as to detrimental effects they may have on asphalt binder performance, especially fatigue and low temperature performance. In this study one base asphalt and nine wax additives, for possible use in warm-mix asphalt binders, were used to evaluate the effect of wax additives on asphalt binder properties and a limited evaluation of mix performance.

Binder performance grade testing revealed considerable differences exist in the different products evaluated. Most of the products reduced the low temperature grade by a few degrees. The G^ mixture mastercurves for the Control binder and Montan wax exhibited the highest moduli at high frequencies and the Sasobit mix exhibited the lowest modulus of all mixes.*

All the wax products evaluated can be classified as non-elastic materials when tested in accordance with the multi-stress creep and recovery test (MSCR). This finding contradicts linear visco-elastic behavior at small strains levels which is suggestive of elastic network formation. It can be concluded for the wax type additives that the repetitive creep test must be conducted with an evaluation of J_{nr} rather than reliance of $1/J''$ ($G^/\sin \delta$) as per the current Superpave specifications.*

Keywords: Wax additives, rheology, J_{nr} , stress sensitivity, non-linear behavior

1. INTRODUCTION

Wax-like additives which melt in a temperature range between the highest pavement temperature and the desired compaction temperature have been used as an asphalt additive for warm mix and asphalt compaction aide applications (Brule et al., 1990). Waxes have long been viewed as a problematic component within an asphalt binder, largely due to their negative impact on bitumen temperature susceptibility. With this in consideration, the primary concern in this study was how addition of wax to asphalt to reduce construction temperatures can be beneficial with respect to overall binder performance. More importantly, can current specifications distinguish between beneficial additives versus those that might have a negative impact on the performance of hot mix asphalt (HMA).

Typical waxes melt within the pavement service temperature range. When even a small fraction of the asphalt undergoes a phase change from solid to liquid over a short temperature range, the Shell bitumen test data charts exhibit a unique behavior as defined by “W” type asphalts. With added wax, the resulting binder is both harder at low pavement temperatures and softer at high pavement temperatures. Both of these characteristics are considered as detrimental performance characteristics. When hot candle-wax is poured on a surface, it quickly solidifies to a soft, pliable mass. Over time it crystallizes into to a hard, non-ductile chip which occupies significantly less volume. This volume change also causes the well- known indentation of the candle wax around the wick as the ductile amorphous wax continues to crystallize.

More recent asphalt research studies suggest that waxes also exist in bitumen as two different physical states corresponding to amorphous and microcrystalline wax. As pavements cool to low temperatures, the solid-solid phase transition between the two states is accompanied by a significant decrease in volume, which yields a corresponding increase in binder density. This phenomenon, called reversible physical hardening (RPH), was first identified by Bahia and Anderson during Strategic Highway Research Program (SHRP) studies of the Bending Beam Rheometer (Anderson et al., 1994). They noted continuous stiffening of certain asphalt beams as they were held at -15°C for up to four days. Dilatometric studies confirmed that an increase in stiffness was directly correlated to an increase in density under the corresponding storage conditions. Brule et al. (1990) used analytical tools such as Differential Scanning Calorimetry (DSC), Phase Contrast Microscopy, Polarized Light Microscopy, Dilatometric measurements, Nuclear Magnetic Resonance (NMR), and Dynamic Shear rheology to conclusively tie RPH to the wax solid-solid phase transition from amorphous to microcrystalline states. The amount of hardening is significant, and detrimental to asphalt quality. Asphalt AAM, the SHRP core asphalt highest in wax, changes from a PG 64-22 to PG 64-10 after being stored at -15°C for four days. Upon reheating to 60°C , the wax crystals melt, and the binder is again PG 64-22. Two research teams led by Planche and Turner separately identified the crystallizable fraction as measured by DSC to be directly related to the physical hardening effect as measured by DSR (Planche et al., 1998; Robertson et al., 2005 and Michon et al., 1999).

For the purposes of the study of this paper, waxes were defined to be Paraffin and Non-paraffin wax. Paraffin waxes are those waxes which have molecular size less than C45 and have melting points less than 70°C (158°F). Non-paraffin waxes are those waxes that have molecular size greater than C45 and have melting points greater than 70°C (158°F). Paraffin waxes are, or are related to, refined/de-oiled microcrystalline waxes derived from crude oil. Non-paraffin waxes include, but are not limited to, natural waxes (animal and vegetable waxes), modified natural waxes (brown coal derived wax), partial synthetic waxes (ester and amid waxes) and synthetic waxes (Fischer Tropsch (FT) and polyethylene (PE) waxes) (Edwards, 2005; Radenburg, 2007).

The objective of this work was to evaluate the effect of non-paraffin wax additives on physical properties and characteristics of asphalt binders. Testing to include binder master curve development, binder true-grading, rotational viscosity profile, bending beam rheometer (BBR), direct tension (DTT), was used to evaluate changes in mechanical properties, other analytical methods were employed to offer effective means to evaluate the potential for waxy materials as warm-mix additives such as modulated differential scanning calorimetry (MDSC) to provide the glass transition temperature, change in heat capacity on melting, amount of crystallizable fraction, and melting point range of the wax in asphalt (ref). Further characterization of wax stereochemistry, Infrared Spectroscopy (IR) and/or Nuclear Magnetic Resonance (NMR) were used to determine the relative degree of branching in the wax molecules. Atomic Force Microscopy (AFM) was also used to evaluate the degree of crystallization of wax additives in asphalt (Baumgardner et al., 2009).

This paper reports on the evaluation of master curves and MSCR results and makes comments upon the use of data from these two types of testing.

2. MATERIALS

2.1 Asphalt

A single source of asphalt binder was used which was selected as a PG64-22 Lion Oil produced at El Dorado, Arkansas.

2.2 Waxes

Nine waxes were selected for the study. The products selected cover the range of waxes discussed earlier to include; paraffin, natural, partial synthetic and synthetic materials. In addition, the selection considered specific synthetic materials in common usage for asphalt modification (for example Sasobit). A paraffin wax (Astra Wax) that was

anticipated to give properties resulting in inferior performance was also selected. Materials selected are presented in Table 1.

Table 1: Waxes selected for study

Ref.	Category	Material	Notes
1	Natural	Romanta Normal Montan	
2	Natural/Synthetic	Romanta Asphaltan A	Blend of Montan normal and amide wax
3	Natural	Romanta Asphaltan B	Refined normal Montan
4	Partial synthetic	Licomont BS 100	N,N'-bisstearamide, stearic acid pitch
5	Synthetic	Sasobit	Fischer-Tropsch Wax
6	Partial synthetic	Luxco Pitch # 2	N,N'-bisstearamide, stearic acid pitch
7	Synthetic	Alphamin GHP	Also referenced as THP
8	Wax Ester	Strohmeyer and Arpe Montan LGE	
9	Paraffin	Astra Wax 3816D Microcrystalline	Refined microcrystalline wax

2.3 Asphalt Binders

Asphalt binders were prepared which consisted of the one (1) neat binder and twelve (12) wax modified binders. The wax modified binders were made with 3% wax additive and (for three additional modified binder blends) with 1% wax additive.

The control binder is referenced by a "0" in the various tables and figures of this report whereas the wax modified binders are represented by the modifier number – 1 to 9. The blends made with 1% wax modification follow the same naming convention as that for the 3% blends with an additional comment to clearly identify them as a 1% blend rather than a 3% blend.

3. BINDER TESTING

All testing other than true grading and master curve development was performed on pressure aging vessel (PAV) aged binders.

3.1 Superpave® True Grade

Superpave true grade was performed in accordance with AASHTO M320 Tables 1. One of the noted issues with the Superpave specifications has been that the high temperature specification parameter in Table 1 of AASHTO M320 ($G^*/\sin\phi$) has been shown to relate poorly to rutting for many "premium grade," modified asphalt binders. This has led to the development of the multiple stress creep-recovery (MSCR) (AASHTO TP70) test as the replacement for the conventional $G^*/\sin\phi$ parameter in the specification. From the MSCR test, the new high temperature specification parameter is determined by dividing the non-recoverable (or permanent) shear strain by the applied shear stress. The result is called the non-recoverable creep compliance, or J_{nr} . In addition, the percent recovery (% recovery MSCR) is also computed which provides more efficient method of characterizing the elasticity of a binder than that currently done with the elastic recovery test (AASHTO T301). These parameters were determined for the materials considered in this project.

4. BINDER TEST RESULTS AND DISCUSSION

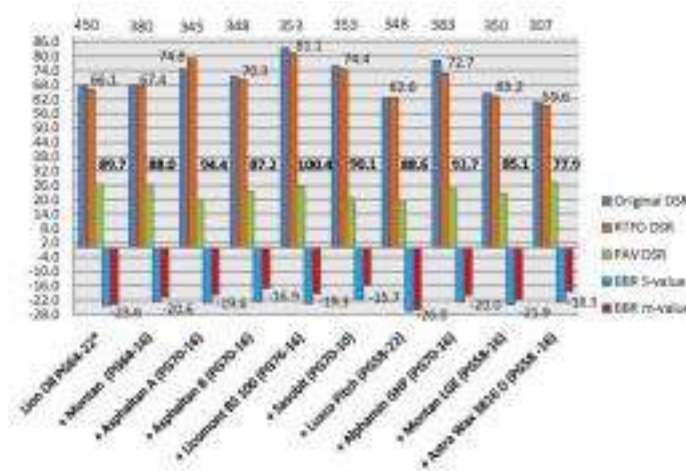
4.1 PG grading

Asphalt binder PG grades can be considered within the AASHTO M320 specification using either Table 1 or 2. In this work we have used an evaluation in accordance with AASHTO M320 Table 1. In addition a new table (originally referenced as AASHTO M320 Table 3 in the 2010 publication but then changed to AASHTO MP19 for the 2011 and subsequent year publications) has been introduced which evaluates the performance by the Multi-Stress Creep and Recovery (MSCR) test.

This method evaluates the non-recoverable creep compliance (Jnr) and has been proposed as a replacement test to the high temperature DSR evaluation of $G^*/\sin \phi$ in the existing tables of the AASHTO M320 specification. In addition to grade evaluation, the data from testing can also be shown as “true grades” by evaluating the pass/fail temperature for any given criteria. Data of this format has been evaluated for the various products and this is illustrated in Figure 1 and Figure 2 for the AASHTO M320 Table 1 requirements; and as Figure 3 for AASHTO MP19 (formally AASHTO M320 Table 3) requirements.

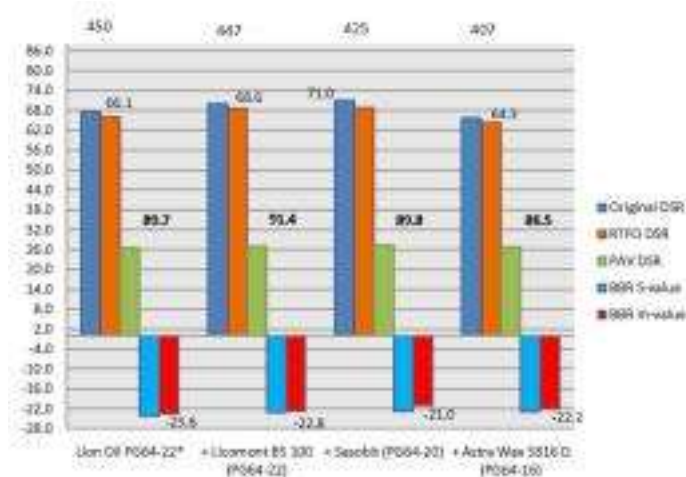
It can be observed from this data that considerable differences exist in the different products. The Astra Wax which was selected as a product unlikely to perform well has the poorest performance with a temperature spread of a mere 60.9°C. Six of the other waxes improved the performance range while two had reduced ranges. Most of the products reduced the low temperature grade by a few degrees but with careful design of modified products with the possible selection of softer products this aspect can be considered in the formulation stage of an asphalt binder.

Brookfield viscosity data obtained from the M320 specification evaluation is presented in Figure 4 which illustrates that all of the waxes reduce the viscosity within a range of 15 to 32%. However, it should be noted that the largest viscosity reduction was with the Astra wax which was selected as the “poor” performing product. This means that the range of viscosity production for the 3% wax addition is in the 15 to 23% range for possible effective products. The data with 1% wax showed smaller changes but an overall comment that could be applied is that the viscosity reduction appears to be linearly related to the percentage of wax used. It should be noted that 1% data was only obtained with 3-waxes so this comment is based on a limited data set.



Notes:
1 Number at top of figure is Brookfield viscosity. Numbers at top of bars indicates high grade passing temperature.
2 Numbers at bottom of bars indicates low grade temperature.
3 Numbers in middle of figure indicates PG grade range using M320 Table 1 criterion.

Figure 1: PG true grades (M320 Table 1) developed for control and 3%wax modified products



Notes:
1 Number at top of figure is Brookfield viscosity. Numbers at top of bars indicates high grade passing temperature.
2 Numbers at bottom of bars indicates low grade temperature.

temperature.
 3 Numbers in
 middle of figure
 indicates PG
 grade range
 using M320 Table
 1
 criterion.

Figure 2: PG true grades (M320 Table 1) developed for 1% wax modified products

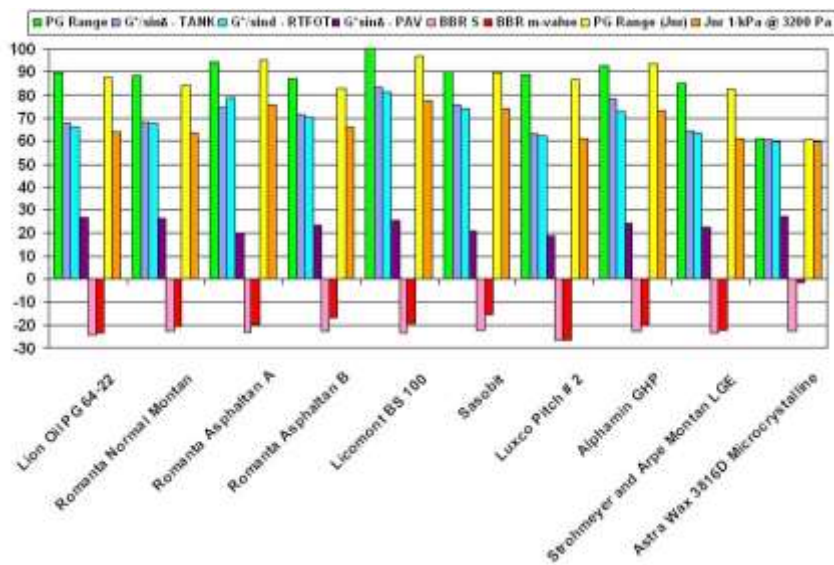


Figure 3: PG grade information showing the Jnr grade information

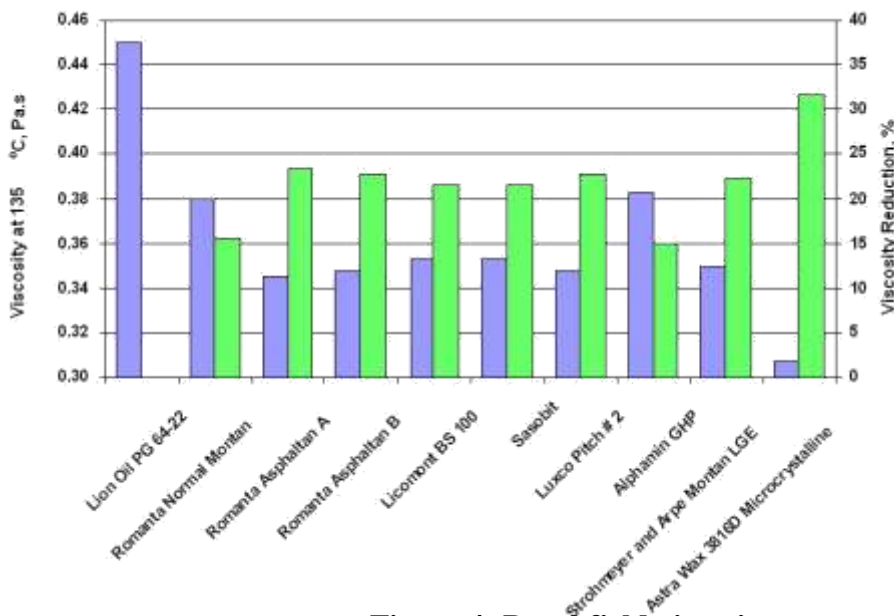


Figure 4: Brookfield viscosity

4.3 Non-recoverable creep compliance, J_{nr}

The results determined from the evaluation in accordance with AASHTO TP70 are summarized in Table 2 with the data presented in Table 3 whereas Table 4 contains the computed percentage recovery from the same test data. This data was used to compute the various grading parameters given earlier in Figure 3.

The data is presented further in Figure 5 and 6 which shows that the wax systems are more susceptible to stress level when classified in accordance with this method compare to the conventional binders. It should be noted that performance ranking for wax modified systems will change as the stress increases from 1 to 10 kPa. A typical example of this is seen in the data of material 5 – Sasobit – which is one of the best performing materials at the low stress levels but the poorest performer at a stress level of 10 kPa. When considering the elastic versus non-elastic type behavior of binders in this it should be noted that all of the products evaluated would be classified as non-elastic with all of the data points tending to fall on a line representing a single relationship, as shown in Figure 9.

Table 2: PG grading using J_{nr} data

Material	Ref.	PG Grade	PG High J_{nr} based 4 (1/kPa) for standard grade
Lion Oil PG 64-22	0	64S/58H	64.0
Romanta Normal Montan	1	64S/58H	63.4
Romanta Asphaltan A	2	70S/64H/58V	75.5
Romanta Asphaltan B	3	64S/58V	66.1
Licomont BS 100	4	70S/64H/58V	77.3
Sasobit	5	70S/64V/58V	74.1
Luxco Pitch # 2	6	58S	60.9
Alphamin GHP	7	70S/64H/58V	73.4
Strohmeier and Arpe Montan LGE	8	58S	60.9
Astra Wax 3816D Microcrystalline	9	58S	59.4

Note: The high PG grades given in this table have been based only on the J_{nr} data. In some cases these materials may fail the intermediate temperature criteria effecting the grade determination.

Table 3: Values of J_{nr} determined from the MSCR test (AASHTO TP70)

Ref.	T, °C	J_{nr} (1/kPa) determined at various stress levels (Pa)										
		25	50	100	200	400	800	1600	3200	6400	12800	25600
0	58	1.24	1.23	1.23	1.23	1.24	1.24	1.26	1.28	1.32	1.38	1.90
	64	2.95	2.96	2.97	2.99	3.01	3.03	3.06	3.12	3.22	3.44	6.44
	70	6.90	6.93	6.96	6.99	7.02	7.07	7.15	7.29	7.51	8.30	Failed
1	58	0.85	0.83	0.83	0.84	0.86	0.90	0.95	1.08	1.34	1.57	2.24
	64	2.10	2.07	2.10	2.16	2.24	2.37	2.62	3.17	3.74	4.23	9.86
	70	5.49	5.43	5.53	5.71	5.99	6.54	7.55	8.66	9.48	Failed	Failed

2	58	0.02	0.02	0.03	0.05	0.07	0.10	0.16	0.33	0.62	0.84	1.09
	64	0.01	0.02	0.03	0.05	0.09	0.18	0.45	1.10	1.77	2.23	3.56
	70	0.01	0.02	0.04	0.08	0.17	0.43	1.36	2.98	4.20	5.10	8.53
3	58	0.07	0.08	0.11	0.16	0.21	0.26	0.36	0.58	0.91	1.22	1.75
	64	0.23	0.30	0.43	0.56	0.69	0.97	1.40	2.06	2.77	3.34	Failed
	70	0.35	0.51	0.80	1.19	1.93	3.00	4.46	6.06	7.28	8.74	Failed
4	58	0.11	0.13	0.15	0.18	0.19	0.22	0.29	0.42	0.58	0.78	1.20
	64	0.12	0.14	0.20	0.27	0.33	0.49	0.75	1.11	1.60	2.18	5.60
	70	0.25	0.32	0.47	0.59	0.86	1.32	1.92	2.78	3.89	5.21	Failed
5	58	0.03	0.03	0.04	0.06	0.08	0.11	0.14	0.22	0.44	0.83	1.24
	64	0.03	0.03	0.04	0.07	0.11	0.19	0.34	0.78	1.73	2.53	4.56
	70	0.07	0.09	0.13	0.22	0.37	0.65	1.37	3.13	5.06	6.40	Failed
6	58	2.20	2.21	2.21	2.22	2.23	2.24	2.27	2.32	2.40	2.57	4.40
	64	4.82	4.85	4.95	5.00	5.04	5.08	5.15	5.27	5.45	6.06	Failed
	70	Failed										
7	58	0.08	0.10	0.13	0.16	0.18	0.20	0.26	0.37	0.55	0.77	1.06
	64	0.13	0.17	0.23	0.29	0.35	0.49	0.74	1.16	1.74	2.25	3.89
	70	0.20	0.31	0.45	0.61	0.92	1.43	2.19	3.34	4.50	5.54	Failed
8	58	2.25	2.26	2.26	2.28	2.30	2.31	2.34	2.40	2.51	2.70	4.74
	64	4.64	4.64	4.68	4.74	4.78	4.84	4.95	5.18	5.53	6.26	Failed
	70	Failed										
9	58	3.13	3.14	3.15	3.16	3.18	3.21	3.27	3.37	3.52	3.95	Failed
	64	7.24	7.28	7.32	7.38	7.41	7.49	7.62	7.83	8.17	9.52	Failed
	70	Failed										

Notes: Values have been reported as failures where the computed number exceeds 10 (1/kPa) or the number has decreased from the previous stress level evaluated.

Table 4: Values of Recovery (%) determined from the MSCR test (AASHTO TP70)

Ref.	T, °C	Recovery Percent MSCR (%) determined at various stress levels (Pa)										
		25	50	100	200	400	800	1600	3200	6400	12800	25600
0	58	3	3	3	3	3	3	2	2	1	1	0
	64	2	2	2	1	1	1	1	0	0	0	0
	70	1	1	0	0	0	0	0	0	0	0	Failed
1	58	16	17	17	16	15	13	10	6	2	1	0
	64	14	14	14	12	10	8	5	1	0	0	Failed
	70	9	10	9	8	6	3	1	0	0	Failed	Failed
2	90	90	88	84	78	71	55	27	9	3	1	Failed
	95	94	92	88	81	68	38	12	3	1	0	Failed
	95	95	92	87	78	55	19	4	1	0	Failed	Failed
3	75	75	68	59	50	42	29	14	4	1	0	Failed
	69	64	54	46	37	23	12	3	1	0	Failed	Failed
	75	69	58	45	27	12	3	0	0	0	Failed	Failed
4	58	70	68	62	57	54	48	32	17	7	2	Failed
	64	78	77	70	62	54	38	19	8	2	0	Failed
	70	75	71	63	54	38	20	9	2	0	Failed	Failed
5	58	78	78	74	67	61	54	45	30	11	2	1
	64	84	84	81	76	69	57	38	14	2	0	0
	70	82	80	74	68	57	39	16	3	0	0	Failed

6	58	2	2	2	2	1	1	1	1	0	0	0
	64	2	2	1	1	0	0	0	0	0	0	Failed
	70	Failed										
7	58	70	68	60	53	48	43	32	18	7	2	1
	64	74	70	63	57	50	37	21	8	2	0	0
	70	77	71	62	54	39	22	9	2	0	0	Failed
8	58	3	3	2	2	2	2	1	1	0	0	0
	64	1	1	1	1	1	1	0	0	0	0	Failed
	70	Failed										
9	58	1	1	1	1	1	1	0	0	Failed		
	64	Failed										
	70	Failed										

Note: Values have been reported as failures where the computed number is negative (indicative of flow/negative recovery after stress has been removed) or the value produced is exceed that obtained by previous values evaluated.

Figure 5: Jnr versus stress level for PG64-22 and materials modified with 3% wax at 64°C

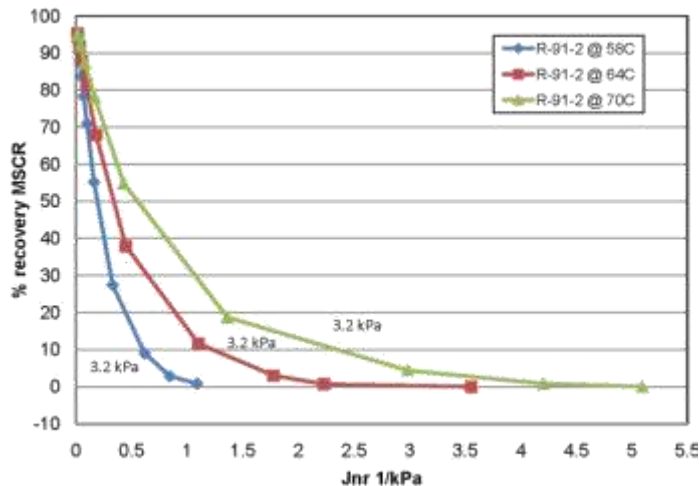


Figure 6: Jnr versus stress level for PG64-22 and materials modified with 3% wax passing temperature for a standard material (Jnr 4 □ (1/kPa))

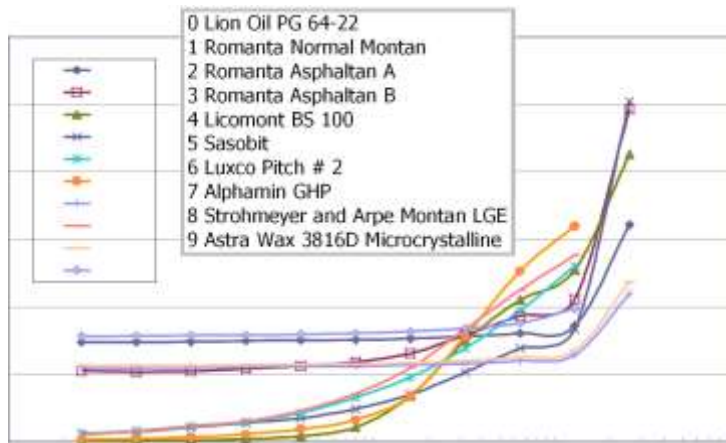


Figure 7: Percent recovery from MSCR versus Jnr for three temperatures evaluated

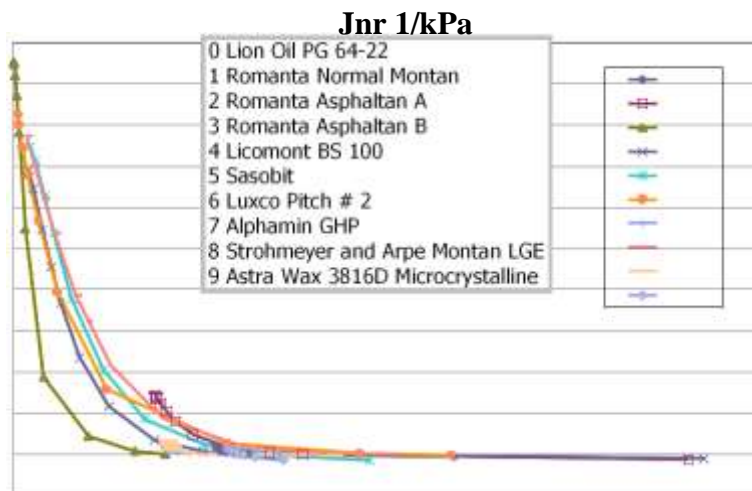


Figure 8: Percent recovery from MSCR versus Jnr for all materials at evaluation temperature which met the 4 (1/kPa) criteria

5. 5. MASTER CURVE DEVELOPMENT

Rheological properties for master curve development were obtained from two different types of tests (3% wax content), dynamic shear rheometer (DSR) and bending beam rheometer (BBR). DSR frequency sweep evaluations of original binder samples were performed from 5 - 90°C in 15°C increments using parallel plate geometry and measured directly G^* and phase angle (δ). Data from BBR testing for the Superpave true grade testing yielded measured stiffness values of $S(t)$ which were converted to G^* and phase angle (δ) and then combined with the data from DSR frequency sweep testing to produce master curves of rheological properties (Baumgaertel and Winter, 1989; Gordon and Shaw, 1994). Master curves were created using a software implementation of the shifting techniques de-scribed by Gordon and Shaw (1994). In this method algorithms are applied to successive pairs of isotherms to develop a shift factor which is independent of any underlying model constraints. The master curves were all shifted to a standard reference temperature of 25°C and the resulting shift factors, as reported in Table 2, were fitted to the WLF equation as follows:

$$\log a_T = \frac{-C_1(T - T_r)}{C_2 + T - T_r}$$

where:

- T_r is the reference temperature
- C_1 and C_2 are constants
- T is the temperature of interest

In addition shift parameters have been calculated in accordance with the modified Kaelble shift method developed by Rowe and Sharrock (2011), which expresses the shift factors in a sigmoid format, as follows: of a transition in behavior of the material occurring at a higher temperature. This would be consistent with expectations of these materials.

6. CONCLUSION

Addition of select waxes (Fischer-Tropsch and Fatty Acid Amides) to asphalt is an accepted practice in warm-mix asphalt production. Additionally Fischer-Tropsch, Montan wax and Montan wax blends have been used in Europe for several years as compaction aids for bituminous mixtures. Addition of waxes to binders prompts concern as to detrimental effects they may have on asphalt binder performance, especially fatigue and low temperature performance. In this study one (1) base asphalt and nine (9) wax additives, for possible use in warm-mix asphalt binders, were used to evaluate the effect of wax additives on asphalt binder properties and a limited evaluation of mix performance. Chemical analysis and physical testing was performed on wax additives, with physical testing also being performed on wax modified asphalt and mixtures containing wax modified asphalt. This paper reports on rheological testing in accordance with the Superpave (AASHTO M320), Multi-stress creep and recovery (MSCR) (ASSHTO TP70) and master curve testing methods. With regard to this testing work, we can make conclusions as follows:

- Binder performance grade (true grade or continuous grade) testing revealed considerable differences exist in the different products evaluated. The Astra Wax which was selected as a product unlikely to perform well has the poorest performance with a temperature spread of a mere 60.9°C. Six of the other waxes improved the performance range while two had reduced ranges. Most of the products reduced the low temperature grade by a few degrees but with careful design of modified products with the possible selection of softer products this aspect can be considered in the formulation stage of an asphalt binder.
- Except for the Luxco Pitch (#6), which was a highly oiled wax, the G^* modulus master curves for all bituminous mixtures were very similar at low frequencies (i.e. corresponding to high temperatures).
- All the wax products evaluated can be classified as non-elastic materials when tested in accordance with the repeated creep and recovery test. This finding contradicts linear visco-elastic behavior at small strains levels which is suggestive of elastic network formation. It can be concluded for the wax type additives that the repetitive creep test must be conducted with an evaluation of J_{nr} rather than reliance of $1/J^*$ ($G^*/\sin \delta$) as per the current Superpave specifications.

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