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PHYSICAL AND RHEOLOGICAL CHARACTERISTICS OF UNAGED AND AGED BINDERS

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Abstract: The properties of four binder types with the help of accelerated simulations of aging have been investigated as per European Norms (EN). Both short- and long-term aged binders showed lower penetration value, higher softening point and stiffer characteristics. Among the binders, 70/100 grade of binder showed relatively higher changes in the penetration ratio (62.58%) and softening point increment (5.85°C) for short-term aging and 41.20% and 12.60°C corresponding values for long-term aging. For polymer modified binders (PmB), the corresponding values were 74.32% and 2.25°C for short term aging and 49.42% and 8.30% for long term aging, respectively. The creep recovery results showed an increased shear modulus (G) and the viscous parameter (η) with aging. At 20°C, the initial shear modulus values for unaged binders of 30/45, 50/70, 70/100 and PmB were $4.77 \times 10^5 \text{N}$, $2.70 \times 10^5 \text{N}$, $1.83 \times 10^4 \text{N}$ and $3.70 \times 10^5 \text{N}$ and corresponding values after long-term aging were $1.11 \times 10^6 \text{N}$, $9.46 \times 10^5 \text{N}$, $5.57 \times 10^5 \text{N}$ and $1.42 \times 10^6 \text{N}$, respectively. With the same temperature, the viscous parameter (η) for unaged binders were $8.52 \times 10^5 \text{Pas}$, $4.93 \times 10^5 \text{Pas}$, $2.62 \times 10^5 \text{Pas}$ and $8.79 \times 10^5 \text{Pas}$ and that of long-term aged binders of $3.48 \times 10^6 \text{Pas}$, $2.77 \times 10^6 \text{Pas}$, $2.04 \times 10^6 \text{Pas}$ and $6.12 \times 10^6 \text{Pas}$ for binders of 30/45, 50/70, 70/100 and PmB, respectively. Further more, for both unaged and aged binders, the minimum modulus was recorded for 70/100 followed by 50/70, 30/45 and PmB binders except the 30/45 binder showed relatively higher modulus than the corresponding PmB at lower temperature ranges in both unaged and short-term aged cases. Among the samples, PmB had shown comparatively better results and performance characteristics.

1 BACKGROUND

Binders have been in use as an essential construction material for many of years due to their binding nature as well as meeting the demands of the road industry. They are the most determinant component of the Asphalt concrete (AC) pavement, which play major role in the performance, and at the same time taking the lion share, and responsible for most failures. While exposed to combinations of irregular traffic loadings and cyclic environmental conditions, binders undergo changes in their physical and rheological properties with time. A particular interest where the binder is extremely sensitive is the temperature which can directly be associated with the physical and rheological properties. Lower temperature harden the bitumen gives it stiff characteristics, on the other hand, higher temperature softens the binder makes less resistant to applied load. Furthermore the physical and rheological properties of asphalt binder and mixes change with time due to complicated set of physicochemical events involving oxidation and structuring. Early volatilization of light weight binder components as well as oxidation during mixing and lying lead the binder to short term aging.

The aging of binder further progressively increased during service time, termed as long term aging, due to active solar radiations and atmospheric air. In both cases there exist increased in viscosities of the asphalt, stiffening of the mixture and making it hard, brittle and susceptible to disintegration and cracking failures.

Accelerated aging simulation of asphalt binder is a step forward in order to address the temperature and other environmental sensitivity of asphalt concrete pavement during its service time. Among several investigation techniques adopted worldwide, the binder advanced testing techniques are getting more attention. For this particular research, the properties of four different binders were investigated and their physical and rheological characteristics were evaluated using standard testing methods as well as with pressure aging vessel (PAV) and advanced dynamic shear rheometer (DSR).

2 LITERATURE REVIEW

The concept of rheology emerged long ago to describe the relationship between stresses and deformations due to external action. The theory described and formulated constitutive equations in order to model the mechanical behaviors of a material which do not obey the simplest classical materials equations, phenomena which are not described by the concept of either a Newtonian fluid or a Hookean solid body (Malkin 1994).

The mechanical properties of binders were improved either by adding modifiers (Kumar et al. 2009, Ye and Wu 2009) or softer and harder binders were blended to produce a multigrade binder without adding any additives and chemicals (Jain et al. 2008) in such a way that the new product possesses the characteristic advantage of both softer and harder grades of bitumen. In all cases, the moves have been aimed at improving the performance and durability of the binders as well as reduce early development of distresses either due to overloading of commercial vehicles, increasing traffic density or due significant fluctuations of daily and seasonal variation of the environmental conditions. Among which, Kumar et al. (2009) had studied the rheological properties of 60/70 and 80/100 grades of binders before and after the binders are modified by crumb rubber (CR). It had been indicated in their conclusion that the binders modified with CR showed improved physical properties, increased the complex modulus and decreased the phase angle as well as lowered temperature susceptibility when compared with their corresponding unmodified binders. It had also been indicated that multigrade binders (Jain et al. 2008) behaved better than their counterparts. Practical experiences revealed that various types of polymer modified bitumen (PmB) have improved the performance of pavements (Schmalz et al. 1990, Sybilski 1996).

3 LABORATORY INVESTIGATIONS

A total of 4 binder grades, namely of 30/45, 50/70, 70/100 and polymer modified binders (PmB 25/55-55A) from the same source were used in this research. Aged and unaged samples were prepared and standard physical and rheological properties were evaluated.

3.1 Standard Tests

The conventional standard tests were carried out according to the norm and the reliabilities of test results were checked. Conducted tests included density (EN 3838), penetration (at 25°C, EN 1426), softening point (EN 1427) and force-ductility (EN 13589) tests.

3.2 Aging Test

In order to simulate the short term aging of asphalt during the plant hot mixing and the lay down process a rolling thin-film oven aging test RTFOT was conducted as per EN 12607-1. In this test, the 35±0.5g asphalt sample was placed in a glass bottle and cooled for about 1hr. At least two sample-glasses were weighed before putting to oven in which gain/loss in weight was later measured. Then the glass containers were placed in a carriage such that the axis of revolution was horizontal and the container

opening was facing a jet of air. The oven was kept at $163\pm 1^\circ\text{C}$ and the carriage was rotated in the oven at a rate of 15 rpm for 75 ± 1 min. on the other hand, accelerated long term aging was carried out to evaluate the susceptibility of binder during the service period. The laboratory investigation involved primarily subjecting the binder with short term aging (with RTFOT) and then the samples were placed in a pressure aging vessel (PAV) as per EN 14769 under a given pressure (usually 2.1 ± 0.1 MPa) and a temperature in between 80 - 115°C for specified period of time (20 hr).

3.2.1 Dynamic Shear Rheometer (DSR)

The dynamic shear rheometer had been used to characterize the creep and viscoelastic behaviors of asphalt binders at intermediate and high temperatures (as per EN 14770). Dynamic shear properties were carried out using RS6000 (RheoStress RS6000) dynamic shear rheometer. Samples of 2 mm and 25 mm in thickness and diameter, respectively, had been used throughout the investigation. Prepared binder sample was sandwiched between the spindle and the fixed steel plate where it was subjected to either creep or oscillatory test. Measurements were conducted at different loading conditions, temperatures and frequencies. The loading value was chosen in such a way that the binder properties remain in the linear viscoelastic range. The two main test categories are discussed below.

3.2.2 Creep – Recovery Test

The viscoelastic properties of binders under specified temperatures had been determined by creep–recovery tests. A constant shear stress was applied instantaneously on the prepared sample for a given period of loading time resulting in the deformation of the sample as a function of time and then the shear stress was set to zero in order to measure the recoverable portion of the deformation. A constant shear stress of 1000 Pa and duration of 180s of loading and unloading were adopted for all tests.

3.2.3 Oscillation Testing

The binders were subjected to non-destructive sinusoidal shear stresses of 1000Pa and the two basic tests conducted were stress-sweep and frequency-sweep tests. The former was conducted to determine the range of shear stress values such that the materials' stress-strain relationships remained in the linear viscoelastic range. The frequency sweep was used to evaluate the viscoelastic characteristics of the binder. The stress value, which was chosen to be in the linear viscoelastic range, had been applied in a frequency range of 0.1 – 10.0 Hz.

4 RESULT ANALYSIS AND DISCUSSION

4.1 Standard Test Results

The conventional penetration and ring and ball tests were used for initial characterization of asphalt binders. Test results indicated that the physical properties of all binders have been found in conformity with the expected and recommended boundary values and shown in Table 1 below.

Table 1: Standard binder test results

Bitumen Grade	Density (gm/cm^3)	Penetration (1/10 mm)		Softening Point ($^\circ\text{C}$)	
	Result	Recommended	Test Result	Recommended	Test Result
30/45	1.0248	30 – 45	43.13	52 – 60	52.55
50/70	1.0281	50 – 70	56.97	46 – 54	48.65
70/100	1.0209	70 – 100	82.13	43 – 51	45.50
PmB 45A	1.0156	25 – 55	40.27	≥ 55	61.20

The physical tests were extended to aged binders. In general, all aged binders showed lower penetration value, higher softening point and stiffer binder characteristics as illustrated in Figure 1.

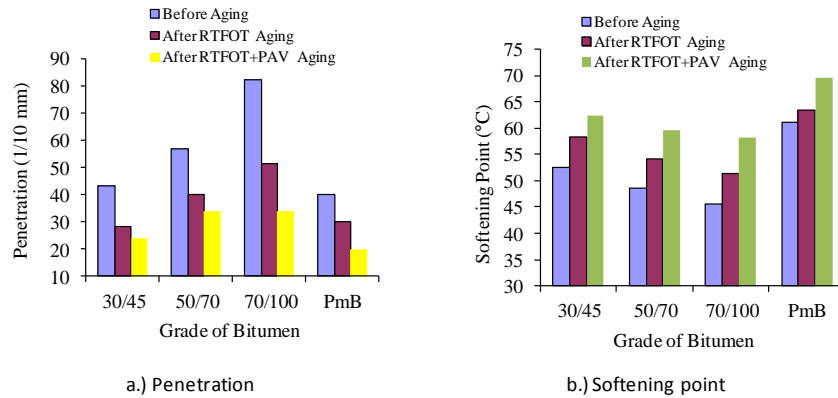


Figure 1: Physical Properties of unaged, short- and long-term aged binders

The physical changes of binders due to aging were addressed in terms of the penetration ratio and the change in the softening point. Penetration Ratio (PR) of the aged asphalt with their unaged asphalt could be used to reflect the change of properties of asphalt binder during aging and it is expressed as,

$$[1] \text{ PR} = \frac{P_{\text{aged}}}{P_{\text{unaged}}} \times 100\%$$

Softening point increment after aging can reflect the susceptible degree of aging. It can be expressed as ΔS , and it is calculated as follow.

$$[2] \Delta S = S_{\text{aged}} - S_{\text{unaged}}$$

Where ΔS is the softening point increment after aging, S_{aged} and S_{unaged} , are softening point asphalt binder before and after aging, respectively.

Table 2: Standard binder test results after aging

Bitumen Grade	After short term Aging		After long term Aging	
	PR (%)	ΔS (°C)	PR (%)	ΔS (°C)
30/45	65.85	5.68	55.41	9.91
50/70	69.98	5.50	59.51	10.90
70/100	62.58	5.85	41.20	12.60
PmB22-55A	74.32	2.25	49.42	8.30

Long term aged binders showed relatively lower PR value, implying that binder grades had undergone harder during aging. The PR value of PmB binder was relatively higher during the short term aging as shown in Table 2, indicating that the penetration properties of the binder was less sensitive to aging compared to unmodified binders during the initial phase, however, the change was slightly higher during long-term aging, meaning that the physical properties of PmB binder were relative sensitive in the long term. A similar trend showed that the change in the softening point of both short and long term aged binders were higher when compared with corresponding unaged binders. Among unmodified binders, the change in softening point were comparable among each other, however, the difference was higher compared with PmB binder. The ΔS value for PmB was lower, however, the increment of change of the softening point was rather higher with aging compared with other binders, which had raised questions regarding sensitivity of the PmB binder while aging in long terms and needed in depth investigation.

The force-ductility measurements were conducted to estimate the maximum force required for all binders and their deformation characteristics while subjecting the binders for long term and short term aging.

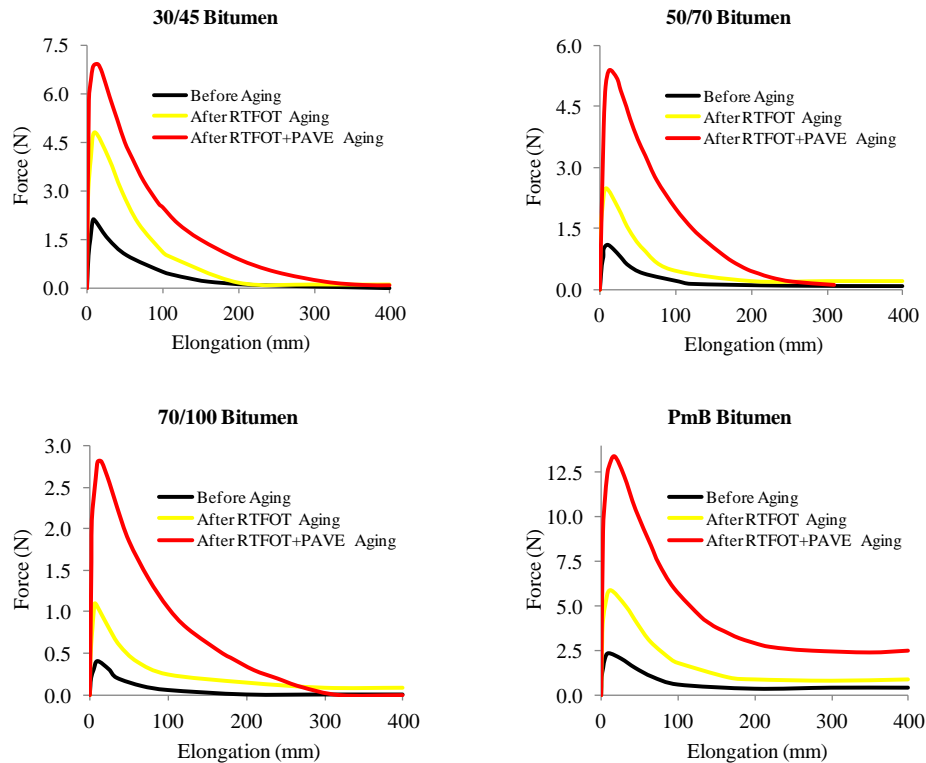


Figure 2: Force ductility test results

As illustrated in the Figure 2, long term aged binders required the highest force to undergo deformation due to the higher stiffness binders attained during the aging process. The force required to deform almost all unmodified binders significantly dropped or vanished after 200 mm deformation, however, both aged and unaged PmB relatively maintained a certain resistance beyond 400 mm deformation. A particular attention was given on the force-ductility behaviors of aged binders. As can be seen from Figure 2, the long-term aged binders showed the highest ductility-force of 6.8N, 5.3N, 2.8N and 13.7N for 30/45, 50/70, 70/100 and PmB binders, respectively.

4.2 Rheological Test Results

4.2.1 Creep-Recovery Test Results

The creep-recovery test conducted on four binder types indicated that at lower testing temperature the creep compliances were lower (i.e., higher shear modulus) to all samples compared with higher temperatures, as expected, due to the fact that the binder relatively hardened at lower temperature and softened at higher temperature.

$$[3] \quad J(t) = \frac{\gamma(t)}{\tau(t)} = \frac{1}{G(t)}$$

Where J, γ , τ and G are shear compliance, strain, stress and modulus of binders, respectively.

The recovery part also showed the same trend; at lower temperature the binders had relatively higher recovery part than the corresponding samples at higher temperature as the binder behaved relatively as

elastic or viscoelastic material when the temperature dropped. On the other hand, the viscous properties dominated as temperature raised and samples tended to behave as viscoelastic or viscous materials. By observing the shear modulus and the viscous parameter, the above arguments were further strengthened. The laboratory experiment indicated that at lower temperature the shear modulus (G) of all binders were higher than the corresponding samples at higher temperature. On the other hand, the viscous parameter (η) increased with increased in temperature. Figure 3 below shows the creep and recovery characteristics of both aged and unaged binders at temperature of 20°C.

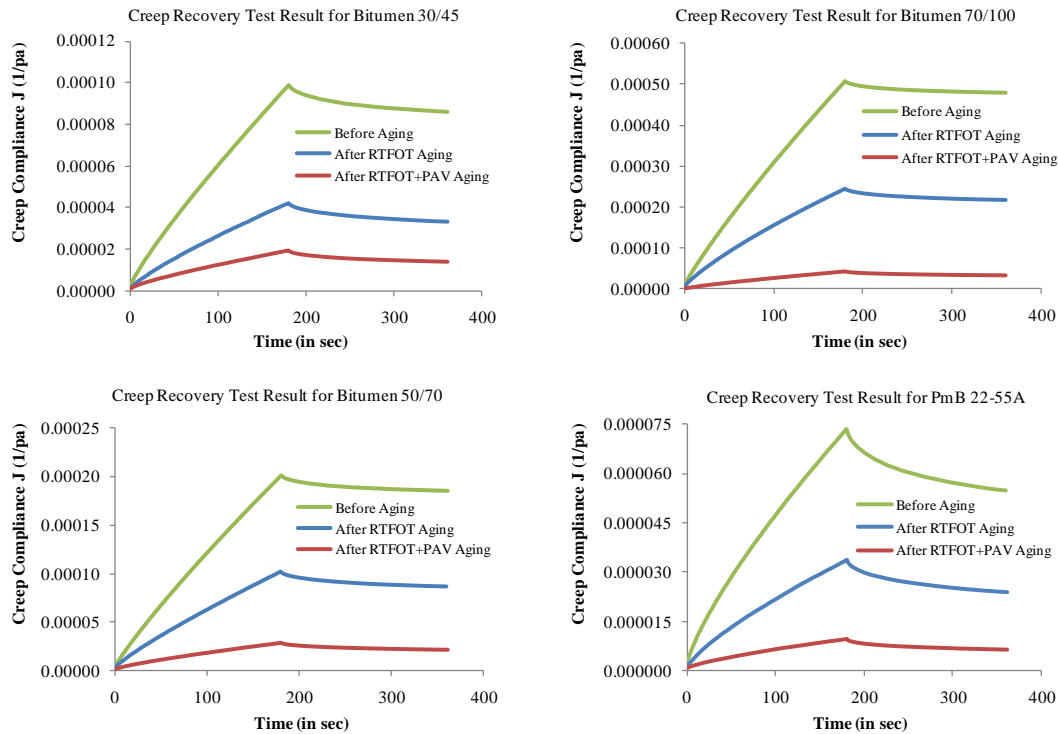


Figure 3: Creep-recovery test results at temperatures of 20 °C

Among binder types, hard grade bitumen (30/45) and polymer modified binder (PmB 45A) showed relatively higher shear modulus (G) and viscous parameter (η) due to their stiff properties they already inherited. The rate of deformation and recovery of both binders were relatively lower than softer grades (50/70 and 70/100). The deformation properties of the binders were related to the rheological properties, i.e., with higher shear modulus, higher viscous parameter and lower rate of creep-recovery, the total deformation decreased and at the same time the recovery parts were relatively lower. On the other hand, binder grades 50/70 and 70/100 showed relatively lower shear modulus and viscous parameter, in particular 70/100 grade of binder showed the least, as expected. They underwent higher deformation due to their softened properties (lower shear modulus and viscous parameter). Their higher rate of deformation/recovery properties hardly influenced to lower the total deformation.

The four binder types were aged for short (RTFOT) and long term (RTFOT+PAV) aging. The creep-recovery result indicated that all aged binders showed lower creep compliances (i.e., higher shear modulus and viscous parameter) compared with corresponding unaged ones due to the oxidation as well as the volatilization of light weight components of binders. Particularly binders subjected to long term aging showed relatively higher shear modulus and viscous parameters. At the same time the rate of deformation and recovery of long term aged binders were higher than the corresponding unaged and short term aged binders, particularly, 30/45 and PmB binders showed lower rate of deformation and recovery.

4.2.2 Frequency- Test Result

The test was carried out to characterize the elastic and viscous properties of the binders (for unaged and aged binders) while subjected to constant sinusoidal shear loading. The stress-sweep tests were primarily carried out to estimate the stress level in such a way that the binders remained in the linear viscoelastic ranges. The frequency-sweep tests were used to evaluate the complex shear modulus, phase angle and viscosity parameters of the binders as explained below.

4.2.3 Stress-Sweep Test Result

It was assumed that the binder remains in the linear range when change in the modulus is within 95% of the maximum observed /recorded value as shown in Figure 4 below.

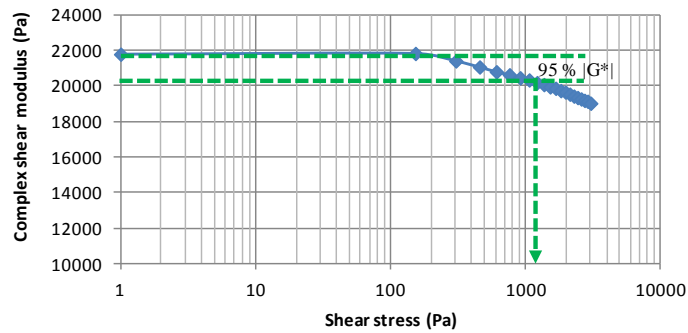


Figure 4: Stress-sweep result at frequency of 1.59 Hz

4.2.4 Frequency-Sweep Test Result

The frequency-sweep tests were conducted at different temperatures and corresponding results were superimposed by shifting all curves to produce a single master curve representing the rheological properties of a binder at selected temperature.

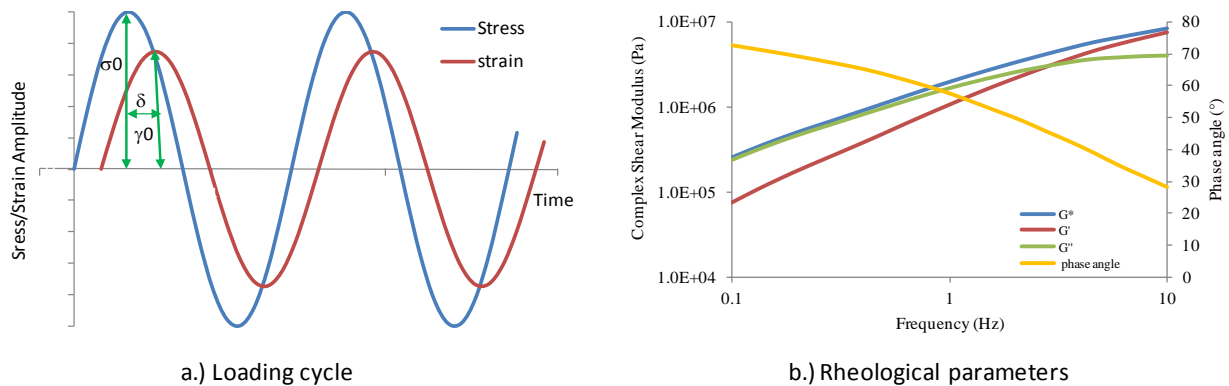


Figure 5: The relationship between cyclic loading and rheological properties

[4] $G^* = G' + iG''$, or

[5] $|G^*| = \frac{\sigma_0}{\gamma_0} = \sqrt{G'^2 + G''^2}$

Where G^* , G' and G'' are complex, storage and loss shear modulus, respectively.

σ_0, γ_0 are maximum shear stress and strain, respectively.

$$[6] \quad \tan(\delta) = \frac{G''}{G'}$$

Where δ - the phase angle defined (in radian) as time lag between the maximum shear stress and the maximum strain.

There was a general trend that the complex shear modulus (G^*) decreased when the temperature increased due to weakening of the bond strength within the binder with raised in temperature. Among the binders, 30/45 grade had maintained higher shear modulus at lower temperature indicating that it would be more brittle than other binder grades in the lower temperature ranges and on the other hand the complex shear modulus of PmB binder was relatively higher even at high temperature (60°C).

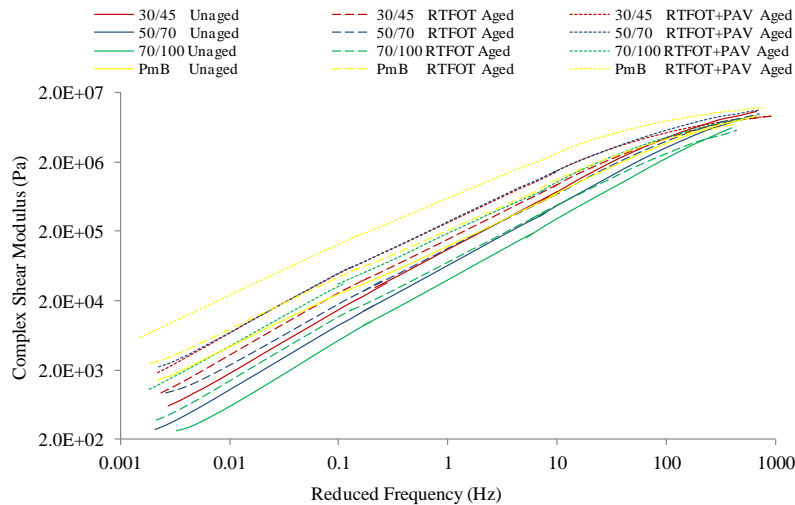


Figure 6: Master curve of frequency-sweep test result

There was a general trend that the complex shear modulus (G^*) decreased when the temperature increased as expected due to weakening of the bond strength within the binder with raised in temperature. Among the binders, 30/45 grade had maintained higher shear modulus at lower temperature indicating that it would be more brittle than other binder grades in the lower temperature ranges and on the other hand the complex shear modulus of PmB binder was relatively higher even at high temperature (60°C).

The investigation indicated that the complex shear modulus comparatively increased after short- and long-term aging compared to unaged binders. As shown in the Figure 6 above, the long-term aged binder showed the highest complex shear modulus. This simply showed that the PmB had reflected better results in wide ranges of temperatures and aging conditions compared with the rest of the binders.

At lower temperatures, 30/45 and PmB unaged binders showed lower phase angle and 70/100 binder showed the higher phase angle. In general, a decreased in phase angle was observed for aged binders, in particular short- and long-term aged PmB binders showed the lowest phase angle, due to the fact that aging had led an increased in storage modulus and a decreased in corresponding loss modulus. For the three unmodified binders presented, the phase angle generally exhibited higher values particularly at high temperatures, indicating a more viscous behaviour and a greater tendency to flow under pressure. For the polymer modified binders, however, the value of the phase angle was rather lower at higher temperature due to higher storage modulus value which was also evidenced during the force-ductility experiment.

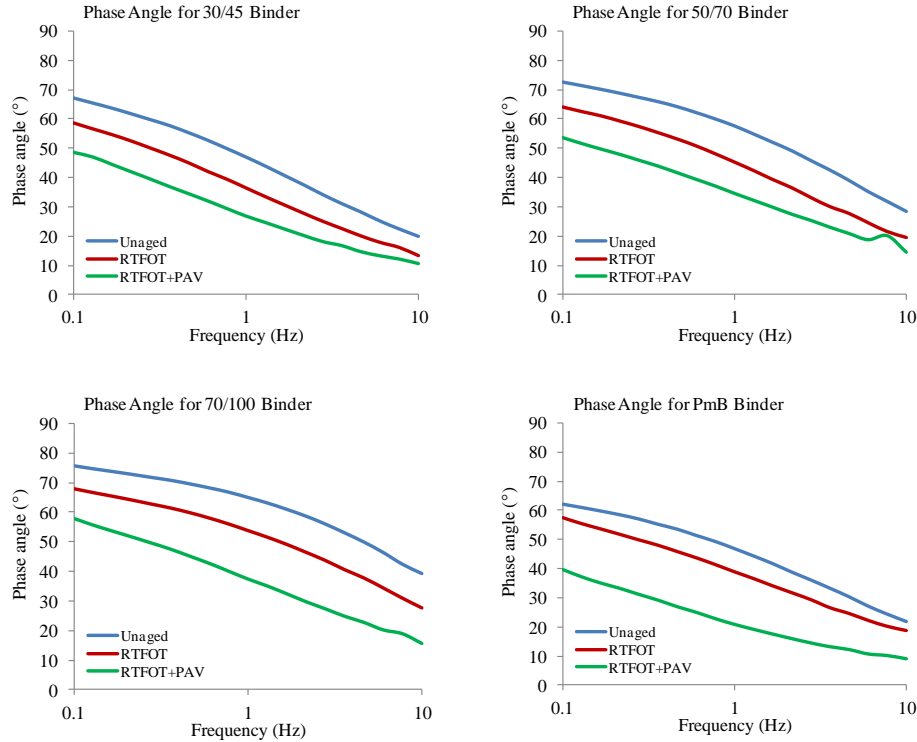


Figure 7: Phase angle at temperatures of 20°C

5 CONCLUSIONS AND RECOMMENDATIONS

Both short- and long-term aged binders showed lower penetration value, higher softening point and stiffer characteristics, which were also supported by the force-ductility test results. Among the binders, 70/100 binder grade showed relatively higher changes in the penetration ratio (62.58%) and softening point increment (5.85°C) for short-term aging and 41.20% and 12.60°C corresponding values for long-term aging. On the other hand, for the PmB binders, corresponding values are 74.32% and 2.25°C for short term aging and 49.42% and 8.30% for long term aging. These figures shows that the physical changes that PmB binder were relatively smaller compared to other binders for the case of short term aging implying that PmB binders would performing better than other binders during mixing and laying in practical situation, and at the same time, during long-term aging, the penetration ratio was rather higher than the remaining two binders (30/45 and 50/70), which gave the PmB binder an indisputable stiff characteristics. Such characteristics had also been evidenced during force ductility test with a maximum magnitude of 13.7N for short term aged PmB binder.

The creep recovery result showed that the shear modulus (G) and the viscous parameter (η) increased with aging, particularly highest values were recorded for long term aged binders. At 20 °C, the initial shear modulus values for unaged binders of 30/45, 50/70, 70/100 and PmB were 4.77×10^5 N, 2.70×10^5 N, 1.83×10^4 N and 3.70×10^5 N and corresponding values after long-term aging were 1.11×10^6 N, 9.46×10^5 N, 5.57×10^5 N and 1.42×10^6 N, respectively. With the same temperature, the viscous parameter (η) for unaged binders were 8.52×10^5 Pas, 4.93×10^5 Pas, 2.62×10^5 Pas and 8.79×10^5 Pas and that of long-term aged binders of 3.48×10^6 Pas, 2.77×10^6 Pas, 2.04×10^6 Pas and 6.12×10^6 Pas for binders of 30/45, 50/70, 70/100 and PmB, respectively.

Furthermore, the master curves revealed that the complex shear modulus of all binders had decreased and the phase angle increased with increased in temperature. At the same time, the modulus had increased and the phase angle had decreased significantly due to aging. In most circumstances, for both unaged and aged binders, the minimum modulus was recorded for 70/100 followed by 50/70, 30/45 and

PmB binders except the 30/45 binder showed relatively higher modulus than the corresponding PmB at lower temperature ranges in both unaged and short-term aged cases. The tests further indicated higher shear modulus and decreased stress relaxations and healing characteristics after aging. Among the samples, the PmB had shown comparatively better results and performance characteristics.

Further investigation is recommended that can possibly accommodate moisture, ultraviolet and other factors responsible for the reduction performance of binders. Besides, detailed investigations of the various components/composition of the binders are necessary to come across with the option to enhance their performance as well as to pin point important components which play an important role in the performance and properties of the binders.

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