Viscoelastic Response of Aged Asphalt Cements

KAMRAN MAJIDZADEH, Department of Civil Engineering, Ohio State University, and HERBERT E. SCHWEYER, Department of Chemical Engineering, University of Florida

The viscoelastic response of aged and unaged asphalt cements at 32 F was investigated. It has been known that aging of asphalt cements results in an increase in viscosity and degree of non-Newtonian behavior. However, the relative change in viscosity alone is not sufficient to characterize the durability of asphalt cements and the response of these materials under severe temperatures and loading conditions. To analyze aging phenomena from a viscoelastic point of view, cylindrical asphalt specimens were subjected to a creep test and strain-time data were obtained. Using curve-fitting techniques, the viscoelastic parameters characterizing aged and unaged asphalts at the selected temperature were calculated. The dynamic response of the asphalts was obtained by transforming the strain-time data into frequency domains.

The viscoelastic analysis indicates that aging results in the development of an initial elastic deformation mechanism and an increase in the coefficient of viscous traction. Similarly, it is shown that the storage modulus $E'(\omega)$ and phase angle are greatly affected by aging conditions. Due to the limitations of a standard aging index, to include the effect of aging on other deformation mechanisms, a dynamic aging index is proposed and its merits are discussed. The results of chemical analysis and rheological and durability tests on these asphalts are also presented.

•THE material characteristics responsible for the complex physicochemical changes occurring in bituminous mixtures have been subject to extensive studies ever since asphalt paving became useful. Because of the importance of aging phenomena on the life of a bituminous pavement, researchers have directed their studies toward the effects of aging on the properties of binder as well as the mixture itself. These durability studies, however, have been mostly confined to analyzing the changes in the properties of asphaltic binder, since it is this main constituent that contributes to the cohesiveness and adhesiveness of the mixture. The aging of the binder generally results in loss of adhesiveness, reduction of ductility, and increase in brittleness, and then eventually leads to a reduction in serviceability under induced traffic and climatic conditions.

In searching for a simple engineering tool for the analysis of aging phenomena, as well as to reveal the basic mechanisms involved in asphalt durability, three distinct approaches have been utilized by asphalt technologists encompassing physical, chemical, and rheological subject areas.

The simple physical tests such as penetration, softening point, and ductility, which are used by highway engineers as a measure of consistency, have yielded valuable information with respect to aging. The results of laboratory and field studies have indicated that the penetration of asphalt cement in the bituminous mixture drops significantly during mixing operations and the service life (1, 2, 3, 4). The occurrence of cracking of

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the bituminous surfaces has also been found to be related to the drop in the penetration of the bituminous binder. The decrease in the ductility and the increase in the softening point temperature due to aging also result in the brittle behavior of bituminous mixtures and a loss of load-carrying capacity (5, 6). Based on these field and laboratory observations, the thin film oven test and other tests have been developed to evaluate the durability of asphaltic materials (7, 8). However, it should be noted that these tests are largely empirical in nature, and it is rather difficult to establish a characteristic index relating the results of these tests, nor has it been possible to incorporate aging indices such as percent loss in penetration in any rational pavement design method.

In the physicochemical approach to the analysis of durability, extensive studies have been conducted to reveal the chemical reactions and mechanisms responsible for aging as well as to determine the quantitative changes in the chemical composition of the binder (9, 10, 11, 12). The controlled laboratory experiments conducted to elucidate the factors affecting durability have indicated that four basic mechanisms are involved in the aging phenomena: (a) evaporation of volatile components, (b) oxidation, (c) age hardening due to the development of internal structures, and (d) polymerization. It is obvious that other secondary mechanisms might also be involved. However, it is not the intent of this report to investigate the relative significance of these aging mechanisms and their methods of evaluation.

The physicochemical studies have conclusively indicated that aging results in changes in the colloidal structure of the binder and the proportions of chemical components and, possibly, in the formation of a complex internal structure. It has been frequently reported that aging in the bituminous binder has resulted in a substantial increase in the percent of asphaltene content (13, 14, 15, 16).

Investigations by Moavenzadeh and Stander have also shown that the increase in percent asphaltene content due to aging is related to the change in the flow characteristics as well as to the molecular weight distribution. At an earlier date Majidzadeh and Schweyer (17), investigating the non-Newtonian behavior of asphalts, had shown that structural changes occurring in the binder due to shear deformation might be related to the asphaltene content. These investigators, observing the variations in the magnitude of reaction rate equilibrium constant and the size of flow units among different asphalts, have also stated that the asphaltene content is one of the major parameters controlling the flow characteristics. However, other chemical components as well may contribute to the rheological properties of bituminous binder. Recently, Moavenzadeh (18), studying the fracture mechanics of asphalt cements at low temperatures, again has confirmed the significance of asphaltene content and the effect of chemical changing due to aging on the magnitude of critical strain energy release rate. It has been indicated that the asphalt, which showed a relatively large gain in the asphaltene content due to aging, also exhibited greater change in the magnitude to critical strain-energy release rate. In short, the physicochemical approach to the aging phenomenon emphasizes the effects of aging on the chemical components of the binder which might significantly alter the rheological properties of bituminous material. However, realizing the complex chemical structure of asphalt, greater effort is needed to understand all the micromechanics involved in aging phenomena.

In the rheological approach to the aging of asphalts, the basic objective is to relate the fundamental flow characteristics of the binder to the aging mechanism. In earlier attempts the change in viscosity and in the non-Newtonian constant due to aging have been investigated and indices for durability of asphalt have been established (19). In these analyses aging indices, AI, have been proposed as the ratio of aged and unaged viscosity at a selected reference temperature. Moavenzadeh and Stander (15), using the Arrhenius viscosity model, have related this index to the free energy of activation; that is,

$$\text{Aging Index, AI} = \frac{\eta \text{ aged}}{\eta \text{ unaged}} = \exp\left[\left(\Delta F_a - \Delta F_u\right)/RT\right] \quad (1)$$

where indices a and u refer respectively to the aged and unaged conditions, and the term ΔF is the free energy of activation. The other terms are defined in the list of

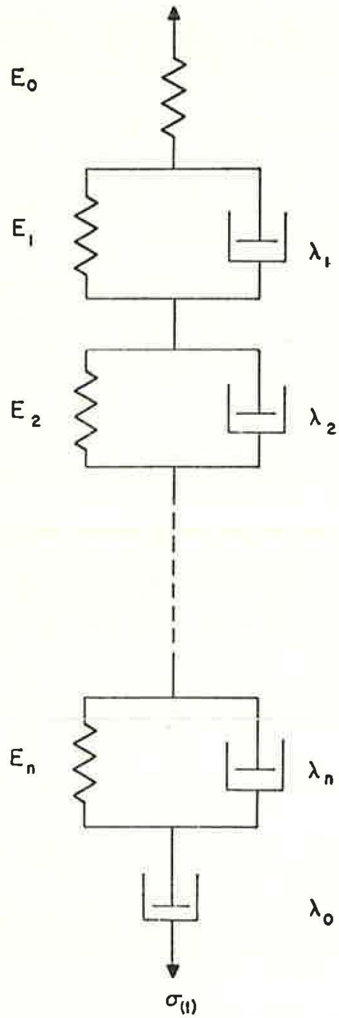

Figure 1. A rheological model.

nomenclature at the end of this section. It is unfortunate that the data presented by Moavenzadeh, due to the scatter of experimental points, did not result in a mathematical expression relating the aging index, temperature, aging condition, and thermodynamic variables. Nevertheless, it should be pointed out that the results show only a trend that could be approximated by a linear relation at certain aging conditions. A major limitation to the use of the aging index as a criterion for the durability of asphalts is the accuracy of the determination of viscosities at low temperatures. Since, as the environmental condition approaches the glass transition temperature and asphalts become more non-Newtonian and complex in nature, the significance of the aging index as a criterion for material selection becomes more apparent (16, 18). However, the determination of AI, which is based on viscosity measurement at these low temperatures, even with the most sophisticated viscometer is not considered possible with any degree of confidence.

To overcome the difficulty of obtaining an accurate measure of aging indices at low temperatures, the method of viscoelastic analysis has been utilized in this paper. Previously, Majidzadeh and Schweyer (20) had shown that asphalt cements at low temperatures, similar to polymeric systems, are characterized by linear viscoelastic equations of state represented in differential operators form given by

$$\left[a_n \frac{d^n}{dt^n} + \ldots + a_o\right]\sigma = \left[b_n \frac{d^n}{dt^n} + \ldots + b_o\right]\epsilon \qquad (2)$$

where $a_n \ldots a_o$ and $b_n \ldots b_o$ are material parameters related to the distinct deformation mechanism observed in viscoelastic materials. By proper determination of these constants, the viscoelastic response as well as the factors affecting these behaviors, such as aging, can be investigated. For the asphalt cements, it had been previously reported that the deformation mechanisms can be represented by one mechanical model, shown in Figure 1. Mathematically, this model corresponds to the operator equation

$$\epsilon(t) = \left[\frac{1}{E_o} + \frac{1}{\lambda_o \frac{d}{dt}} + \sum_{i=1}^{i=n} \frac{\frac{1}{E_i}}{\tau_i \left(\frac{d}{dt} + \frac{1}{\tau_i}\right)}\right]\sigma(t) \qquad (3)$$

where n refers to the number of Kelvin elements corresponding to the delayed elastic deformation mechanism and τ_i is the retardation time, λ_i/E_i. For the materials used in this study, it has been shown that two Kelvin elements will suffice, expressing the viscoelastic response of asphalts. As shown in an earlier paper (20), some asphalt cements, depending on the composition and test temperature, may not exhibit any initial elastic deformation, represented by modulus E_o.

To investigate the effect of aging on the response of asphalts, the material constants given in Eqs. 2 and 3 can be evaluated using a creep test $\sigma = \sigma_o$ = constant. The method

of successive approximations and curve fitting techniques (20, 21), as used in the previous study, has been used to determine these constants. To study the dynamic response of the materials, the observed strains are transformed into a frequency (ω) domain, written as

$$\epsilon^*(i\omega) = \frac{\sigma_o}{i\omega}\left[\frac{\frac{1}{\lambda_1}}{\frac{E_1}{\lambda_1} + i\omega} + \frac{\frac{1}{\lambda_2}}{\frac{E_2}{\lambda_2} + i\omega} + \frac{1}{i\omega\lambda_o} + \frac{1}{E_o}\right] \tag{4}$$

For a constant stress, σ_o, applied at t = 0, where $\sigma^*(\omega) = \frac{\sigma i}{i\omega}$, the complex dynamic modulus is obtained by

$$E^*(i\omega) = \frac{1}{\frac{1}{E_1 + i\omega\lambda_1} + \frac{1}{E_2 + i\omega\lambda_2} + \frac{1}{i\omega\lambda_o} + \frac{1}{E_o}} \tag{5}$$

This modulus in turn can be resolved into a real part or storage modulus $E'(\omega)$ corresponding to the stored energy and an imaginary part or loss modulus E'' representing the energy loss in the system. Similarly, the phase angle, ϕ, between stress and strain, which is a measure of the dissipation of the energy per cycle or internal damping, is given by

$$\phi = \tan^{-1}\frac{E''(\omega)}{E'(\omega)} \tag{6}$$

With respect to the derivation of an aging index criterion, the loss modulus $E''(\omega)$ is possibly of greater value than other viscoelastic parameters. In the field of polymer rheology a term designated as dynamic viscosity $\lambda'(\omega)$ has been defined as given by

$$\lambda'(\omega) = \frac{E''(\omega)}{\omega} \tag{7}$$

This parameter has been previously used by Sisko (23) to study the dynamic response of aged asphalt cements. It has also been shown that the relation existing between dynamic viscosity and frequency is very similar to that of steady state viscosity-rate of shear. This similarity is expected, since during the deformation process coiled molecules experience oscillatory forces with a frequency proportional to the rate of shear even though the material is only subject to a shear force. That is, the randomly coiled molecules in a viscous material rotate as they undergo a translatory motion. The dynamic viscosity at very low frequency approaches the ordinary steady state flow viscosity, λ. With increasing frequency λ falls monotonically, reaching values much smaller than steady state flow.

Then, in analogy with the aging index for the steady state flow, a dynamic aging index, $AI(\omega)$, can be defined as

$$AI(\omega) = \frac{\lambda'_a(\omega)}{\lambda'_u(\omega)} = \frac{\left(\frac{E''(\omega)}{\omega}\right)_a}{\left(\frac{E''(\omega)}{\omega}\right)_u} = \frac{E''_a(\omega)}{E''_u(\omega)} \tag{8}$$

where a and u refer to the aged and unaged conditions. At low frequencies, where the dynamic viscosity approaches that of steady state flow, $AI(\omega)$ equals the aging index (AI) of Eq. 1. However, with increasing frequency, the aged asphalt exhibits a more elastic response compared with that for the unaged specimens, and $AI(\omega)$

decreases substantially. Therefore, at a desired frequency, the AI(ω) might yield valuable information with respect to the effect of aging on the asphaltic materials.

Nomenclature

- AI = aging index, η_a/η_u
- AI (ω) = dynamic viscosity aging index $E_a''(\omega)/E_u''(\omega)$
- a, b = constants
- ϵ = strain response
- E = moduli; E' is real or the storage modulus and E'' is imaginary or the loss modulus
- ΔF = energy of activation, subscript a for aged sample, subscript u for original sample
- i = any ith item
- λ = viscosity
- $\lambda'(\omega)$ = dynamic viscosity
- n = number of Kelvin elements
- η = viscosity, subscript a for aged sample, subscript u for original sample
- o = initial condition
- ϕ = phase angle
- R = universal gas constant
- σ = stress
- T = absolute temperature
- t = time
- τ = retardation time λ/E
- ω = frequency
- * indicates transformed equation

MATERIALS AND TEST PROCEDURE

In this investigation four types of asphalts selected from different sources have been utilized. These asphalts were aged according to the standard procedure for thin film oven test at 325 F for a period of 5 hours. After aging, the penetration, ductility, and viscosity of these asphalts were determined and compared with the original test properties. The results of the thin film oven test and the relative changes in consistency are given in Tables 1 and 2.

The effect of aging on the chemical composition of asphalts was also studied. The aged and unaged asphalts were separated into four components using the Schweyer-Chipley method of analysis (22). The results of this chemical analysis (Table 3) indicate that aging causes an increase in the percent asphaltene content, as well as changes in the proportions of other components. The asphalt cement designated S63-13 is an air-blown asphalt with a high percentage of paraffinic-naphthenic (PN) component. The aging, as shown in Table 3, results in a small increase in percent PN and the light aromatic fraction (LA) of this asphalt. On the other hand, asphalts S63-6 and S63-9 exhibit a substantial gain in their percent of LA fraction and asphaltene content. Asphalt S63-4, in contrast to the other asphalts, showed some reduction in the LA fraction due to aging. It is also observed that the PN component of asphalts S63-4, S63-6, and S63-9, in contrast to S63-13, was slightly reduced. It should be pointed out that work is in progress at present to relate these chemical changes with the flow characteristics of asphalts. Since the results of these correlations have not yet been completed, no attempt will be made in this paper to relate composition to the aging phenomena.

For rheological studies, cylindrical specimens with diameter of 1.3125 in. and a height of 2.816 in. were prepared, using the Harvard miniature molds. Asphalt cements first were heated to a liquid state and poured into the molds, which were then cooled down gradually over a period of three hours to 32 F temperature. The specimens were cured at this temperature for an additional two hours. The solidified specimens were then removed from the 32 F water bath and the excess asphalt was removed to obtain a cylinder with the specified height. These samples were then transferred to a

TABLE 1

PROPERTIES OF ASPHALT CEMENTS

Property	Smackover S63-4	Florida AC-8 S63-6	Steam Refined Intermediate S63-9	Air Blown, Low Sulfur Naphthenic S63-13
Penetration, 77 F 100 g/5 sec	77	91	84	91
Softening point, deg F	116	118	115	119
Ductility, 77 F, cm	125	140+	200+	170
Specific gravity, 60 F/60 F	1.021	1.037	1.033	0.988
Sulfur, %	3.56	5.83	4.24	0.69
Hexasphaltene, %	12.9	19.4	16.8	12.8
Glass transition Temperature, deg F	-11.8	-12.2	-13.9	-10.8

TABLE 2

PROPERTIES OF ASPHALT CEMENTS BEFORE AND AFTER THIN FILM OVEN TEST

Identification	Penetration at 77 F			Ductility at 77 F		Viscosity at 140 F		
	Original	Residual	% Ret.	Original	Residual	Original	Residual	Ratio
S63-4	77	56	72.7	125+	125+	1912	3778	1.98
S63-6	91	54	59.3	140+	145	2579	6632	2.57
S63-9	84	54	64.3	150+	150+	1704	4318	2.53
S63-13	91	64	70.3	170	105	1726	4002	2.32

TABLE 3

COMPOSITION ANALYSIS OF AGED AND UNAGED ASPHALTS, PERCENT

Component	S63-4		S63-6		S63-9		S63-13	
	Unaged	Aged	Unaged	Aged	Unaged	Aged	Unaged	Aged
Paraffinic-Naphthenic	9.8	8.6	8.0	7.5	13.1	11.1	23.9	24.9
Light aromatic	34.6	31.5	26.4	32.7	28.3	35.7	23.6	23.9
Heavy aromatic	43.9	42.8	46.0	37.4	43.7	33.4	37.1	32.2
Hexasphaltene	10.9	14.1	17.8	22.0	15.8	19.2	12.1	15.1

TABLE 4

VISCOELASTIC PARAMETERS OF AGED AND UNAGED ASPHALTS AT 32 F
(All numbers should be multiplied by 10^8)

Model Parameters	Condition	S63-4	S63-6	S63-9	S63-13
E_0, dynes/cm^2	Aged	1.204	0.895	2.023	1.340
	Original	(–)	(–)	(–)	(–)
λ_0, poises	Aged	144.6	83.63	92.99	56.66
	Original	113.3	73.68	50.84	110.6
E_1, dynes/cm^2	Aged	0.4959	0.3043	0.3699	0.2727
	Original	0.4688	0.2658	0.3725	0.2510
λ_1, poises	Aged	94.07	47.38	48.88	24.68
	Original	66.14	53.16	32.34	41.60
E_2, dynes/cm^2	Aged	0.9146	1.098	0.9410	0.6466
	Original	1.735	0.7081	0.9292	0.5944
λ_2, poises	Aged	11.56	19.14	6.413	7.609
	Original	24.47	14.75	9.308	7.753

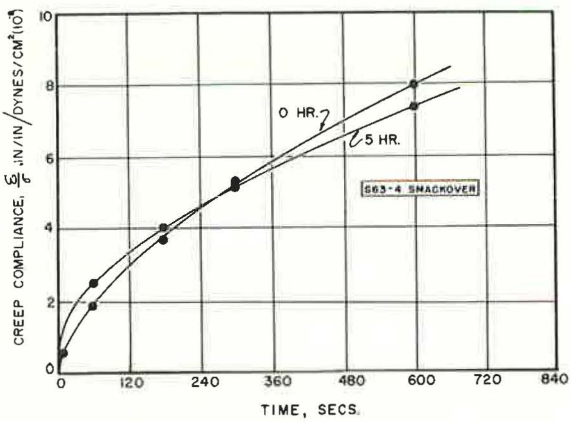

Figure 2. Effect of aging on creep response at 32 F of certain asphalt cements.

controlled-temperature water bath kept at 32 F and were cured there for three hours before testing. For viscoelastic characterization, prepared specimens were subjected to a creep test, constant stress σ_0 at $t = 0$, and appropriate strain-time data were analyzed as previously (20).

DISCUSSION AND ANALYSIS OF RESULTS

Creep Response

The viscoelastic analysis of creep data for both aged and unaged asphalts indicates that parameters describing the material characteristics are significantly affected by aging (Table 4). The analyses reveal that aging results in development of an instantaneous elastic response as well as other changes in the flow mechanism (Fig. 2). The presence of an elastic deformation mechanism in aged specimens might be attributed to the formation of certain molecular bonds similar to crosslinking phenomena observed in rubber-like materials. It has been recognized that aging results in structural changes in asphalts, classically known as sol-gel transformation. These structural effects are often associated with changes in the composition, such as the increase in percent asphaltene and changes in other components shown in Table 3.

The other effect of aging is on the viscous flow characteristics of asphalts, which is evident from the increase in the coefficient of viscous traction as measured by the slope of the steady-state flow portion of the creep curves. An increase in viscosity is often taken as an indication of aging susceptibility of asphalts and is expressed by the aging index as previously discussed. Among all the asphalts studied, only one has exhibited a decrease in viscosity. This asphalt (S63-13) had previously exhibited certain peculiar rheological behavior (20).

Dynamic Response

As discussed earlier, the dynamic response of asphalts can be calculated using the viscoelastic parameters obtained from a creep test. In Figures 3, 4, 5, and 6 the variations of the dynamic moduli and phase angle with frequency and aging condition are shown. Similar relations have also been obtained for other asphalt cements.

The comparison of these dynamic moduli indicates that aging results in significant changes in the dynamic response of asphalt cements. The storage modulus $E'(\omega)$, representing the energy stored in the system per cycle of deformation, increases with aging as well as frequency as evaluated by the proposed constitutive equation. The increase of $E'(\omega)$ with aging is due to the presence of the instantaneous elastic deformation and stiffer dashpot flow mechanisms which contribute to the behavior of asphalts. At higher frequencies the storage modulus approaches an ultimate value of the order of 10^8 dynes/cm^2. Values of a similar order of magnitude had been previously reported for asphalt cements (23). Similarly, the loss modulus $E''(\omega)$, representing the energy loss per cycle of deformation, is greatly affected by aging and frequency. Aged asphalt cements which exhibit initial elastic response and stiffer dashpot responses (λ_0 in Table 4) dissipate less energy per cycle than unaged materials. This difference becomes more pronounced at the higher frequencies, where aged asphalts approach a perfectly elastic body. At low frequencies, where an ample time is available for the molecular motions, and the energy loss $E''(\omega)$ becomes proportional to frequency, the behavior of viscoelastic materials approaches that of steady-state flow conditions. In these frequency ranges the aged asphalts exhibit greater amounts of energy loss compared with unaged specimens.

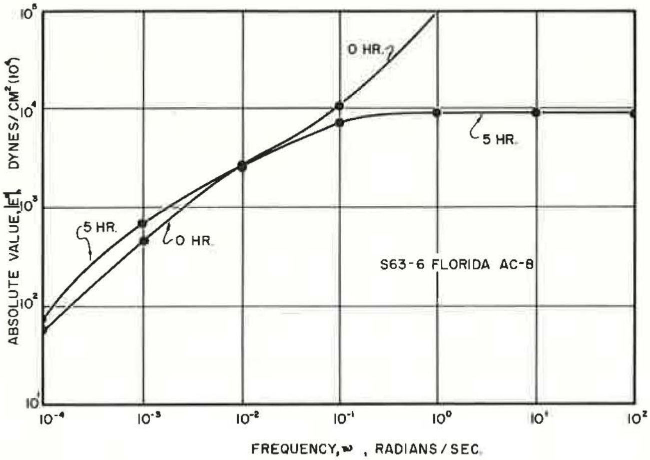

Figure 3. The complex modulus of original and aged (TFOT) Florida asphalt cement.

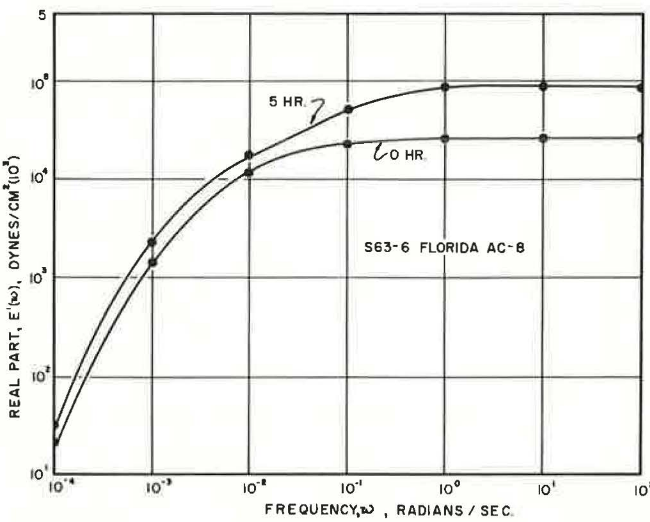

Figure 4. The storage modulus of original and aged (TFOT) Florida asphalt cement.

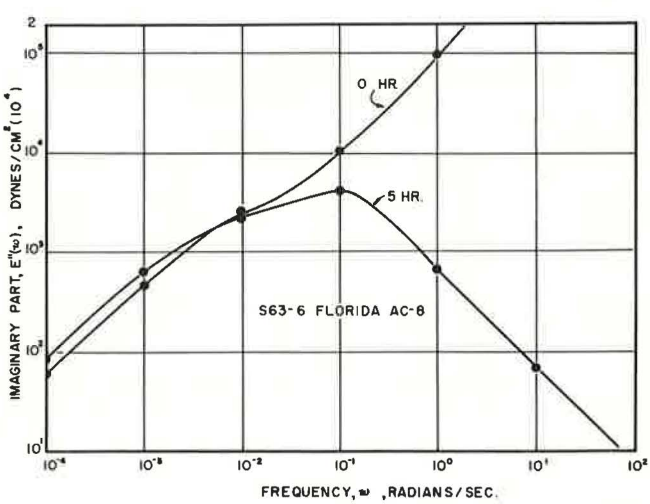

Figure 5. The loss modulus of original and aged (TFOT) Florida asphalt cement.

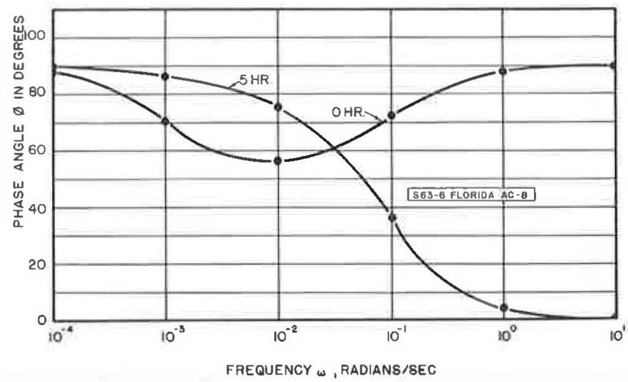

Figure 6. The phase angle of original and aged (TFOT) Florida asphalt cement.

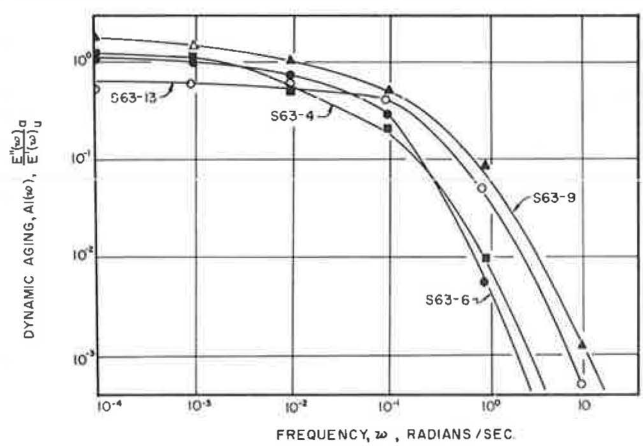

Figure 7. Variations of dynamic aging index with frequency.

TABLE 5

DYNAMIC AGING INDEX OF FOUR ASPHALT CEMENTS AT VARIOUS FREQUENCIES

Frequency, Radians/Sec	AI(ω)			
	S63-4	S63-6	S63-9	S63-13
1×10^{-4}	1.219	1.134	1.826	0.514
1×10^{-0}	1.05	1.096	1.621	0.646
1×10^{-2}	0.56	0.768	1.062	0.629
1×10^{-1}	0.20	0.293	0.502	0.519
1×10^{0}	0.900×10^{-2}	0.675×10^{-2}	9.6×10^{-2}	5.0×10^{-2}
10	0.98×10^{-4}	0.685×10^{-4}	12.14×10^{-4}	5.5×10^{-4}
10^{2}	0.98×10^{-6}	0.685×10^{-6}	121.7×10^{-6}	5.5×10^{-6}

The variation of phase angle with frequency also explains in a similar manner the differences in the storage and dissipation of energy per cycle of deformation. At low frequencies the stress and strains in both aged and unaged asphalts are 90 deg out of phase, corresponding to the presence of a dashpot flow mechanism. However, at higher frequencies, the phase angle of aged asphalt approaches zero, indicating a perfectly elastic response. The unaged asphalt, on the other hand, due to the immobilization of a retarded deformation mechanism, still exhibits a viscous flow behavior which is represented by a 90 deg out-of-phase angle. At intermediate frequencies, the variations of dynamic moduli and phase angle depend on the differences in the viscoelastic responses and the relative contribution of different flow mechanisms.

Dynamic Aging Index

In the discussion of the aging phenomena, it was pointed out that aging susceptibility of asphalts is commonly measured by an aging index, which is the ratio of aged and unaged viscosities at a standard temperature. It has also been shown that asphaltic binders during their service life become harder and may eventually result in severe road failures. The aging index, then, should reveal these detrimental hardening tendencies and predict the possible fatigue of bituminous mixtures under adverse service conditions. However, this aging index has certain limitations. First, it has been shown in this paper that the aging not only changes the viscosity of the binders, but also alters the initial elastic (related to E_o) and delayed elastic (related to E_n) responses of asphalts; therefore, the relative viscosity change is not a sufficient criterion for the aging phenomena. Second, the fatigue type failures of road surfaces which might be due to asphalt hardening never occur under steady-state deformation. Rather, they are always associated with the repeated nature of wheel loads. Thus, the aging index should be related to the frequency of load applications as well as to the increase in the resistance to steady-state deformation. Third, the loss of flexibility and the subsequent road cracking are more pronounced at low temperatures where the present aging index cannot be evaluated accurately using conventional rheometers.

Considering the foregoing discussions, a dynamic aging index as given by Eq. 8 has been utilized in this paper. In Figure 7, the dynamic aging index calculated for the asphalts studied has been plotted as a function of frequency (Table 5). As observed at low frequencies, when the asphalt response approaches that of steady-state flow, the dynamic aging index reaches a limiting value that is a function of the steady-state viscosity. At this range of frequency, there is little difference in the aging susceptibility of the four asphalts. However, at higher frequency ranges, the dynamic aging index is reduced considerably and the effect of aging and the type of asphalt on this index becomes more apparent. The smaller values of the dynamic aging index in this range indicate changes to a more elastic behavior of asphalt cements. Therefore, the magnitude of the aging index at high frequencies can be taken as a measure of the increase in the stiffness of asphalts due to aging and might have many applications in the pavement performance analysis.

SUMMARY AND CONCLUSIONS

In this investigation the effect of aging on the viscoelastic response of four different asphalt cements tested at 32 F was studied. Cylindrical specimens were subjected to a constant stress level and the strain-time data were obtained. These data were analyzed and the viscoelastic responses describing the deformation mechanism of asphalts were determined. The time responses of the asphalts were then transformed into a frequency domain and appropriate dynamic moduli were calculated. The following conclusions can be drawn from this study:

1. Aging phenomena result in the development of instantaneous elastic response as well as other changes in the deformation mechanisms of asphalts such as steady-state flow.
2. The dynamic responses of asphalts are significantly affected by aging. At high ranges of frequency, due to the presence of instantaneous elastic response, the dynamic behavior of aged asphalts approached that of perfectly elastic bodies.

3. The limitations of the steady-state aging index are discussed and a dynamic index is utilized to include the effect of repeated loading.

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