

# INFLUENCE OF SHORT-TERM AGEING AND LONG-TERM AGEING ON THE THERMAL EQUILIBRIUM TIME FOR TWO DIFFERENT VISCOSITY GRADES OF BITUMEN USED FOR ROAD CONSTRUCTION

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**Abstract:** The field performance of bituminous roads depends highly upon the rheological properties of the bitumen used. Therefore, it is paramount that the determination of the rheological properties of bitumen binders is done accurately. Determining the time it takes for a specimen to reach the desired test temperature before starting any rheological examination is one such important aspect. This time is known as the time for thermal equilibrium. This study focuses on determining the time it takes for a specimen to achieve the thermal equilibrium. Here, the unaged, rolling thin film oven (RTFO) aged and pressure aging vessel (PAV) aged bitumen binders were subjected to an oscillatory test at 10 rad/s using a Dynamic Shear Rheometer (DSR) and the variation of the complex modulus with time was analyzed. The observed results clearly showed that the test temperature and aging significantly affects the time it takes for a sample to reach the thermal equilibrium.

**Keywords:** complex modulus, dynamic shear rheometer, long-term ageing, short-term ageing, thermal equilibrium.

## 1. Introduction

Almost 94% of the paved roads in the US are flexible pavements (WAPA, 2019) whereas in Europe, more than 90% of the paved roads and highways are flexible pavements (NAPA and EAPA, 2011). A similar scenario exists in India wherein more than 90% of the road network consists of flexible pavements (IAT, 2000; CRRI, 2019). A flexible pavement is composed of compacted bituminous surfacing course/courses over a compacted base and sub-base course. A bituminous surfacing course is generally composed of coarse and fine aggregates mixed with

bitumen acting as a binding agent. The smooth performance of the roads is crucial for the economic growth of any country. Studies have shown that the performance of bituminous mixtures especially at higher pavement temperatures is highly dependent upon various rheological properties of bitumen binders wherein parameters like  $G^*/\sin\delta$  (Chen and Lin, 2007; Planche *et al.*, 1996), Zero shear viscosity (ZSV) (Sybilski, 1996; Biro *et al.*, 2009), Jnr (Hafeez and Imran, 2014; Radhakrishnan *et al.*, 2018) have provided good correlation with the performance of bituminous mixtures. It is also known that bitumen is a material

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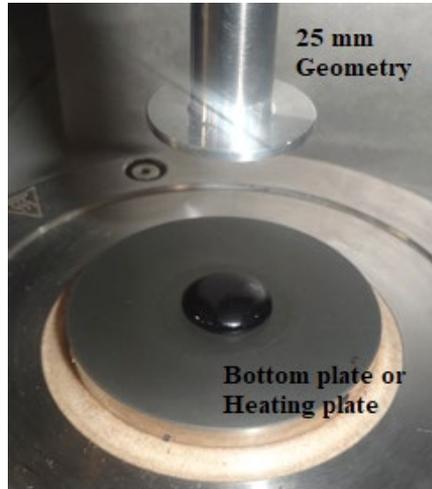
that is highly susceptible to temperature (Poulikakos *et al.*, 2014). Moreover, with time, there is an increase in the stiffness of bitumen binder because of volatilization and oxidation (Yin *et al.*, 2017). This process of stiffening of the bitumen is known as 'ageing' of the bitumen binder. Ageing of bitumen binder occurs in two distinct phases. In the first phase, the ageing of the bitumen is due to mixing and compaction of bituminous mixtures which is termed as 'short-term ageing' (Sirin *et al.*, 2018). The ageing of bitumen binders due to oxidation which happens during its service life is known as 'long-term ageing' of bitumen (Sirin *et al.*, 2018). The short-term ageing and the long-term ageing in bitumen binders are simulated in the laboratory by conducting a RTFO test and PAV test, respectively. A comprehensive rheological characterization of bitumen binders consists of determining the rheological properties of unaged, RTFO aged and the PAV aged binders.

The determination of various rheological properties of the bituminous binders is done using sophisticated instruments such as a DSR. The DSR is used for the rheological characterization of the bitumen binders using precise measurements. Therefore, it is important to accurately determine the rheological properties of the bitumen binders. The DSR measures the rheological properties of the bitumen binders by shearing the required amount of the bitumen sample between two parallel plates. The two parallel plate setup of a DSR is shown in Figure 1. The sample is placed on the bottom plate whereas the top plate applies shear which in turn induces a resultant torque through which the rheological properties are measured. The accuracy of a measurement made at the desired test temperature will be truly representative of the said temperature only if

the sample has attained the said temperature. That is, the various tests conducted to determine the rheological properties of the bitumen binders can be started only after the sample has attained the thermal equilibrium. The scientific definition of thermal equilibrium (AHD, 2019) is: "*the condition under which two substances in physical contact with each other exchange no heat energy, i.e., two substances in thermal equilibrium are said to be at the same temperature*". That is, the transfer of heat takes place from one system to another system until there is an equilibrium in the two systems. Here, the bottom plate of the DSR is connected to a heating element which heats the plate to the desired test temperature. The bitumen sample is then placed on the bottom plate as shown in Figure 1. The bitumen sample will be at a different temperature than the bottom plate. When the bitumen sample is placed on the bottom plate, heat from the bottom plate is gradually transferred to the bitumen sample over a period of time until both the bottom plate and the bitumen sample are at equal temperatures. The determination of the time it takes for a specimen to reach the thermal equilibrium is an essential part of the rheological investigations as it is very much important to determine the response characteristics of the bitumen binders at a precise test temperature. Further, the influence of test temperature and the ageing condition of the binder on the thermal equilibrium time is not clearly established in the literature. This study is part of a larger research work concerned with the comprehensive rheological investigations on the unaged, RTFO aged and PAV aged bitumen binders. The main focus of the current study is to determine the thermal equilibrium time for the unaged, RTFO aged and the PAV aged bitumen binders at different temperatures. It

is important to recall here that the bitumen binders are highly susceptible to temperature and the response characteristics of the

bituminous binders very much depend on the test temperature apart from the ageing conditions.



**Fig. 1.**  
*Bitumen Sample Placed on the Bottom Heating Plate of a DSR Undergoing Thermal Equilibrium*

## 2. Materials

VG20 and VG40 binders are used in this study. The physical properties of both the binders satisfied the relevant test specifications of IS: 73 (2013).

## 3. Experimental Work

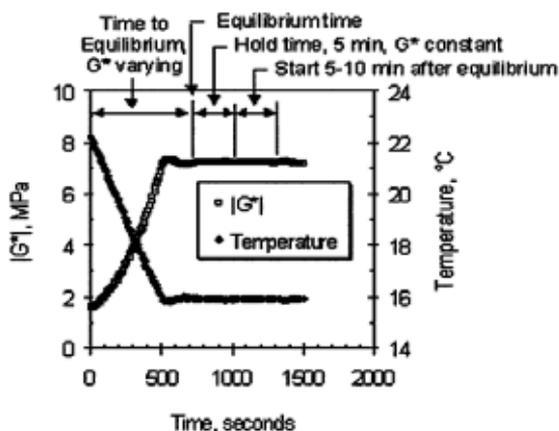
ASTM D7175 (2015) provides the standard test procedure for determining the rheological properties of bitumen binders using a DSR. Determining the time for thermal equilibrium is one of the preliminary investigations on the bitumen binders before rheological properties can be determined. ASTM D7175 (2015), in its appendix section X4, describes the procedure to determine the time it takes for a sample of bitumen to reach the thermal equilibrium. Here, the test sample is subjected to an oscillatory test at a fixed frequency

of 10 rad/s using a small strain amplitude which gives a meaningful data resolution. The protocol was adopted such that the DSR records the complex modulus ( $G^*$ ) at every 30 seconds. Figure 2 extracted from ASTM D7175 (2015) gives a visual illustration for determining the time for thermal equilibrium. The VG20 and the VG40 bitumen binders were RTFO aged and PAV aged by following the protocols specified in ASTM D2872-12e1 (2012) and ASTM D6521 (2018), respectively. The testing was conducted using a 25 mm parallel plate geometry as the test temperatures were equal to or above 40°C.

The main objective of the study is to determine the thermal equilibrium times for the unaged, RTFO aged and PAV aged VG20 and VG40 binders at 40, 45, 50, 55, 60 and 65°C. However, the testing was carried out only at three selected temperatures as

shown in Table 1 that includes the lowest temperature (40°C or 50°C) and the highest temperature (65°C) apart from an intermediate temperature (50°C or 55°C). For the thermal equilibrium data obtained at the above three temperatures, using a linear curve fit, the thermal equilibrium temperatures are determined for the remaining intermediate temperatures.

The DSR was set to the desired test temperature and the sample was placed on the bottom plate. The test was started after trimming the sample. Table 2 shows the time taken by the respective bitumen samples to spread and to trim as per the test geometry at different test temperatures and the aging conditions. This time was eventually added to the thermal equilibrium time.



**Fig. 2.**  
Variation of Complex Modulus with Time  
Source: ASTM D7175 (2015)

**Table 1**  
Test Temperatures

Binder grade	Temperature, °C		
	Unaged	RTFO aged	PAV aged
VG20	40, 50, 65	40, 50, 65	40, 50, 65
VG40	40, 50, 65	40, 50, 65	50, 55, 65

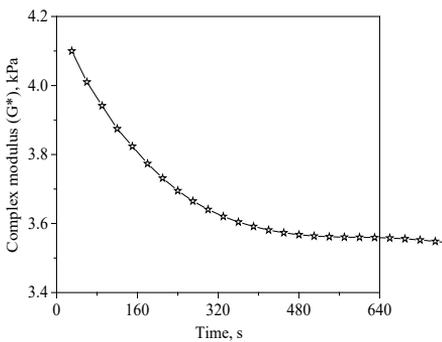
**Table 2**  
Sample Spread and Trimming Time

Temperature, °C	Time, s					
	VG20			VG40		
	Unaged	RTFO aged	PAV aged	Unaged	RTFO aged	PAV aged
40	120	120	300	120	360	-
50	10	60	120	60	120	360
55	-	-	-	-	-	360
65	10	60	120	60	120	120

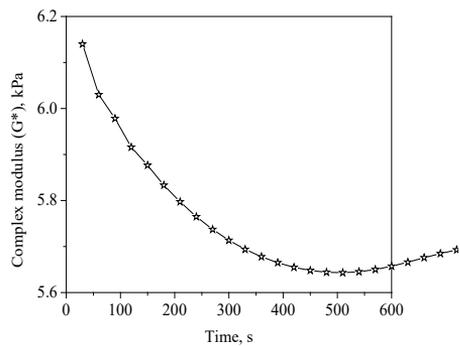
### 4. Results and Discussion

Figure 3 shows the variation of the complex modulus ( $G^*$ ) with respect to time for the unaged, RTFO aged and PAV aged VG20 and VG40 binders at 65 °C. Similar plots were generated at all the remaining test temperatures for the unaged, RTFO aged as well as PAV aged VG20 and VG40 binders. The ASTM D7175 (2015) states that “the time to reach thermal equilibrium is the time required to reach a constant modulus. Typically, this time will be greater than the time required to reach a constant reading on the DSR thermometer”. In order to determine the time for thermal equilibrium in a more rational manner the variation of complex modulus with time, i.e.,  $dG^*/dt$  is analyzed. The complex modulus obtained at 30 second intervals was smoothened using a five point moving average method. The percentage difference between the complex modulus at every 30 second interval was determined. The time where the percentage difference between the successive complex modulus decreases below 0.5% is chosen as the thermal equilibrium time. Table 3 shows the time where the percentage change in complex modulus for

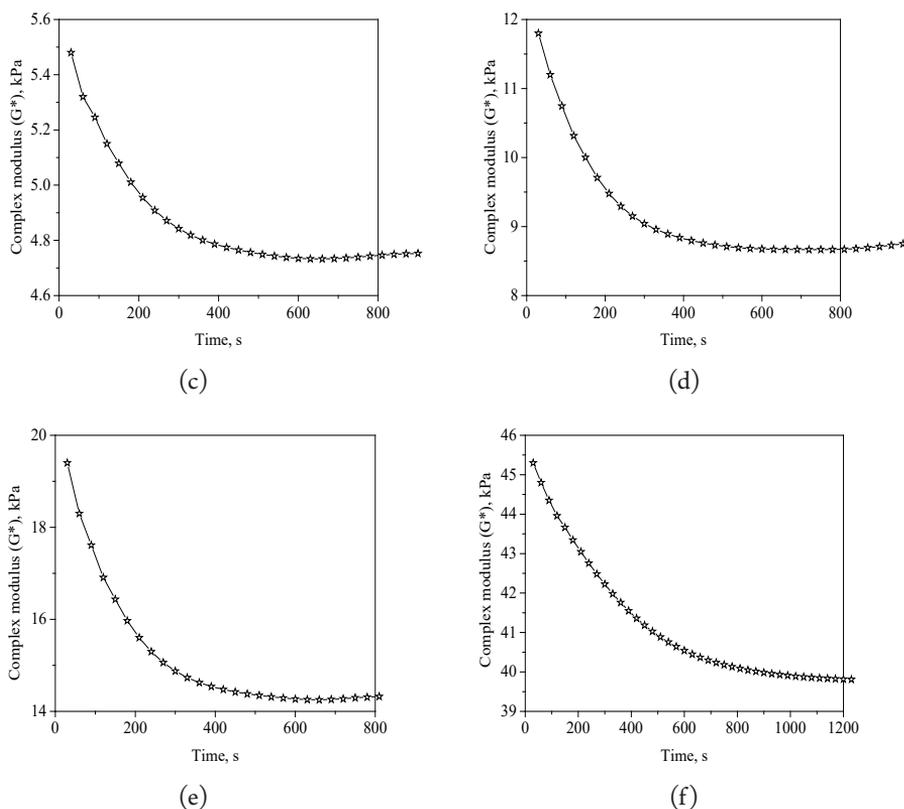
the unaged, RTFO aged and PAV aged VG20 and VG40 binders is less than 0.5%. It can be observed from Table 3 that the time when the percentage change in complex modulus is less than 0.5% decreases with increase in temperature, increases with the binder ageing as the binder becomes more stiffer, and the time is higher for VG40 binder when compared to the VG20 binder at a given test temperature and aging condition. It is to be noted here that the times shown in Table 3 very much depends on the sample spread and trimming times as shown in Table 2. For instance, for RTFO aged VG40 binder the combined sample spread and trimming time at 40°C is 360 s whereas this time is only 120 s at 50°C. This resulted in a drop in the time (for the percentage change in complex modulus to reach less than 0.5%) to 630 s for the RTFO aged VG40 binder at 40°C when compared to 660 s for the RTFO aged VG40 binder at 50°C as can be observed from Table 3. Similar observation can also be made from Table 3 if the respective times at: (i) 50°C for the RTFO aged VG40 binder and the PAV aged VG40 binder are compared, (ii) 65°C for the unaged VG20 binder and the unaged VG40 binder are compared.



(a)



(b)



**Fig. 3.** Variation of the Complex Modulus with time at 65°C for: (a) Unaged VG20 Binder, (b) Unaged VG40 Binder, (c) RTFO Aged VG20 Binder, (d) RTFO Aged VG40 Binder, (e) PAV Aged VG20 Binder, and (f) PAV Aged VG40 Binder

**Table 3**  
Time when the Percentage Change in Complex Modulus is less than 0.5%

Temperature, °C	Time, s					
	VG20			VG40		
	Unaged	RTFO aged	PAV aged	Unaged	RTFO aged	PAV aged
40	480	600	720	600	630	-
50	450	540	660	540	660	600
55	-	-	-	-	-	480
65	330	330	420	300	420	480

As stated in the ASTM D7175 (2015), the thermal equilibrium time determined based upon the stabilization of complex modulus will be slightly lower than the actual time for the thermal equilibrium. Therefore, for a conservative practice an additional 300 s is added to the determined thermal equilibrium time intervals. The new thermal equilibrium times are shown in Table 4. Now it can be clearly observed from Table 4 that the thermal equilibrium time decreases with increase in temperature, increases with the binder ageing as the binder becomes more stiffer, and the time is higher for VG40 binder when compared to the VG20 binder at a given test temperature and aging condition. This data is further used to generate best fits where a linear curve as shown by Eq. (1) is found to be ideal for all the binders and aging conditions

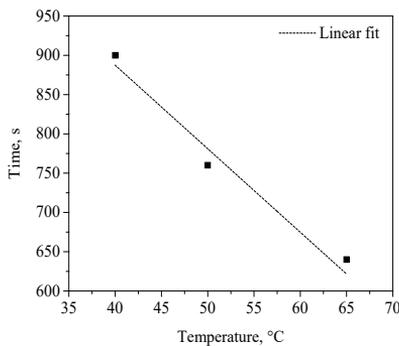
considered in this study. The linear fits to determine the thermal equilibrium times for the unaged, RTFO aged and PAV aged VG20 and VG40 binders are shown respectively in Figures 4 and 5. The linear fit parameters are shown in Table 5. The parameters obtained from the linear fit are then used to determine the times for thermal equilibrium for all the test temperatures. Table 6 shows the final thermal equilibrium times for all the desired test temperatures, i.e., 40, 45, 50, 55, 60 and 65°C for both the binders and all for all the three ageing conditions.

$$y = a + b \times x \tag{1}$$

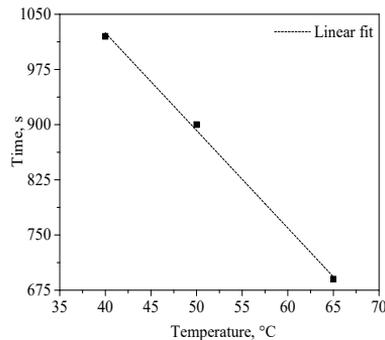
Where, x is an independent variable, y is a dependent variable, and a, b are linear fit parameters.

**Table 4**  
Time After Adding the Spread and Trim Time and an Additional 300 s

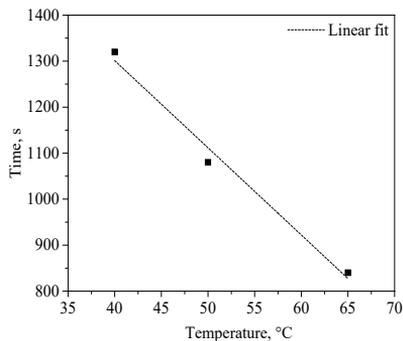
Temperature, °C	Time, seconds					
	VG20			VG40		
	Unaged	RTFO aged	PAV aged	Unaged	RTFO aged	PAV aged
40	900	1020	1320	1020	1290	-
50	760	900	1080	900	1080	1260
55	-	-	-	-	-	1140
65	640	690	840	660	840	900



(a)



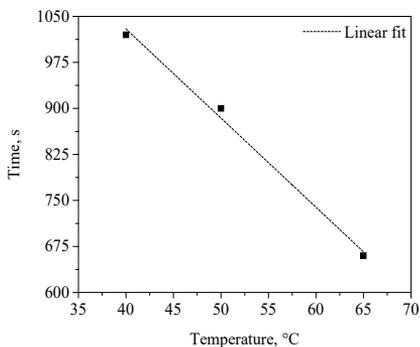
(b)



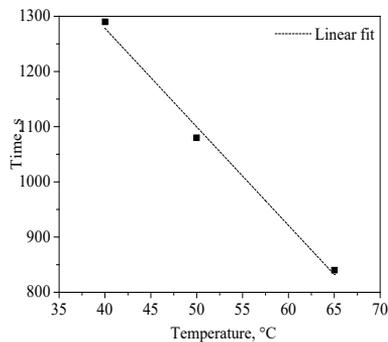
(c)

**Fig. 4.**

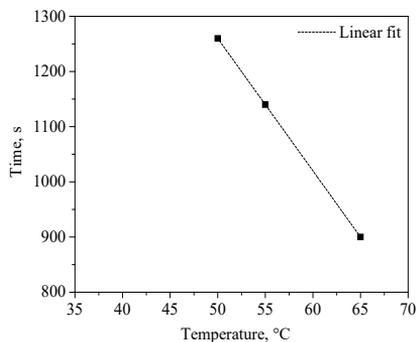
Linear Fit for the VG20 Binder: (a) Unaged, (b) RTFO Aged, and (c) PAV Aged



(a)



(b)



(c)

**Fig. 5.**

Linear Fit for the VG40 Binder: (a) Unaged, (b) RTFO Aged, and (c) PAV Aged

**Table 5**

Parameters of the Linear Fit

Linear fit parameters	VG20			VG40		
	Unaged	RTFO aged	PAV aged	Unaged	RTFO aged	PAV aged
a	1312.63	1555.26	2058.95	1610.53	1991.84	2460.00
b	-10.63	-13.26	-18.95	-14.53	-17.84	-24.00

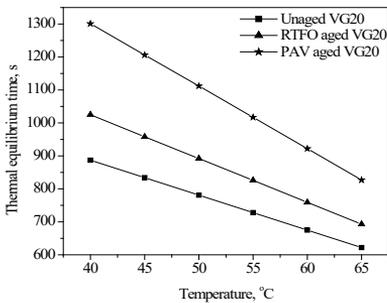
**Table 6**

Final Thermal Equilibrium Time

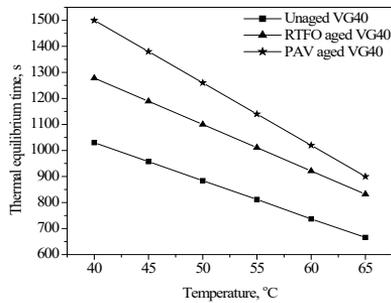
Temperature, °C	Time for Thermal Equilibrium, s					
	VG20			VG40		
	Unaged	RTFO aged	PAV aged	Unaged	RTFO aged	PAV aged
40	887	1025	1301	1030	1278	1500
45	834	958	1206	957	1189	1380
50	781	892	1112	884	1100	1260
55	728	826	1017	812	1011	1140
60	675	759	922	737	921	1020
65	622	693	827	666	832	900

It can be observed from Figure 6 that even though the variation in the thermal equilibrium time is significantly higher at lower test temperatures, the thermal equilibrium times tend to be converging with increase in test temperature for all the variables considered in this study signifying a lower variation in the thermal equilibrium time at higher test temperatures. Similarly,

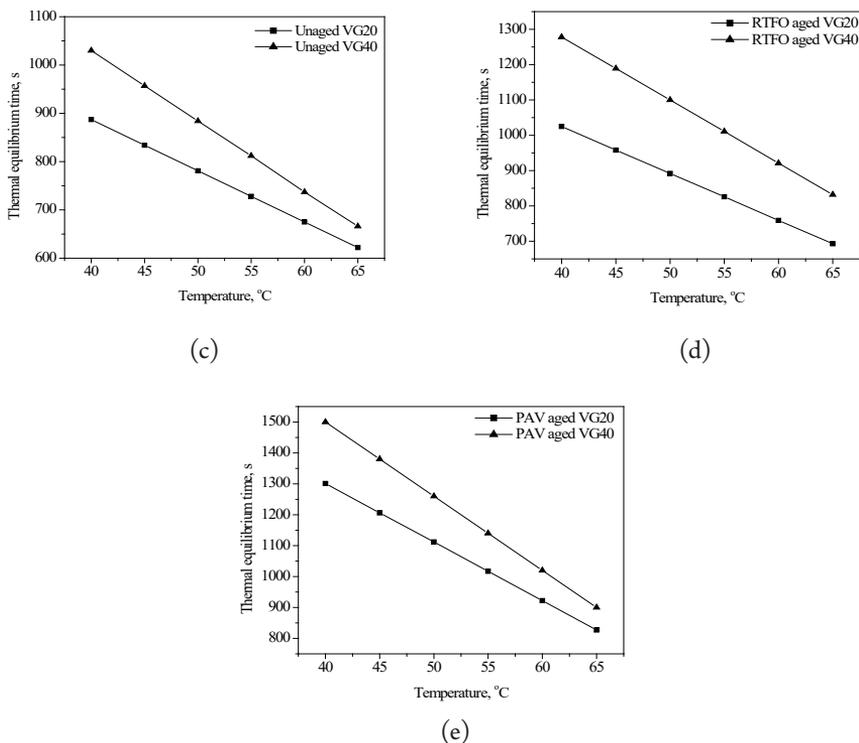
the influence of binder ageing condition is observed to be higher at lower test temperatures as can be seen clearly for both the VG20 and VG40 binders from Figure 6(a) and 6(b). In the similar lines, the influence of binder grade is observed to be higher at lower test temperatures as can be seen from Figures 6(c) to 6(e).



(a)



(b)



**Fig. 6.**

Variation of Thermal Equilibrium Time as a Function of Temperature for: (a) VG20 Binder with Different Ageing Conditions, (b) VG40 Binder with Different Ageing Conditions, (c) Unaged VG20 and VG40 Binders, (d) RTFO Aged VG20 and VG40 Binders, and (e) PAV Aged VG20 and VG40 Binders

### 5. Conclusions

The unaged, RTFO aged and PAV aged VG20 and VG40 binders were subjected to oscillatory tests at 10 rad/s. The complex modulus plotted against time was analyzed and the time for thermal equilibrium was determined. From the analysis of the thermal equilibrium time determined for the unaged, RTFO aged, and PAV aged VG20 and VG40 binders, the following conclusions are drawn:

- The time for thermal equilibrium varies with the test temperature. It is observed that with an increase in test temperature, the time for thermal equilibrium decreased. Such trend is observed for both the VG20 and VG40 binders for different ageing conditions. This clearly shows that it is very much essential to determine the thermal equilibrium time at each test temperature and also for each binder type.

- The PAV aged binder exhibited higher thermal equilibrium time followed by RTFO aged binder and the unaged binder. This clearly shows that there is an increase in the time for thermal equilibrium when the binder age hardens. This kind of response is observed for both the VG20 and the VG40 binders.
- When the comparison between the two binders is made, the VG40 binder required a longer duration of time to achieve thermal equilibrium than the VG20 binder at a particular ageing condition and at a specific test temperature. This clearly shows that binders with a higher viscosity require a relatively longer duration of time to achieve the desired test temperature than the soft binders.
- The variation in equilibrium times is observed to be higher at lower test temperatures and the values converged at higher test temperatures for both the binder grades and for all the three ageing conditions.
- The results clearly highlight the need to determine the time for thermal equilibrium for individual temperatures and in order to get precise characterisation of the rheological properties of the bitumen binders.
- When the samples are allowed to thermally equilibrate more than the required time, steric hardening in the bitumen samples could lead to undesirable results.
- The thermal equilibrium times determined in this study are specific to the dynamic shear rheometer used in this study. The thermal equilibrium

times may vary depending upon the type of temperature controller and the size of bottom plate assembly used in the dynamic shear rheometer.

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