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Evaluation of Effects of Extended Short-Term Aging on the Rheological Properties of Asphalt Binders at Intermediate Temperatures Using Respond Surface Method

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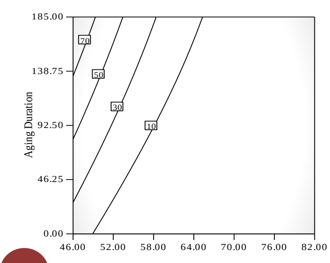
Graphical abstract



Abstract

Predicting the effects of short term aging on asphalt binders' rheological properties can be a complicated task. This is due to the exposure of different binders to different conditions. Hence, the utilization of a Respond Surface Method (RSM) is a practical way to predict these effects. An experimental matrix was planned to predict asphalt binders behavior at intermediate temperatures based on the central composite design for aging duration and test temperature. The test results showed that prolonging aging increased the binder complex modulus, but decreased the phase angle, while increasing the test temperature decreased the complex modulus but increased the phase angle. However, the trends in aging differ and depend on the binder type, test temperature and aging conditions. It was also found that the RSM method is a fast, effective and reliable tool to predict the effects of aging on binders' rheological behavior.

Keywords: Asphalt binder, rheological properties, aging, response surface method

1.0 INTRODUCTION

Asphalt binder aging is one of the major causes of short pavement service life. Researchers [1-3] stated that aging increment directly reduces the Pavement Condition Index (PCI). Short term aging changes the binders' rheological properties during mixing, transportation and construction. Aging varies due to variations in binder and aggregate type, mixing and construction duration and environmental temperature [4]. Each factor exerts its own effects on mixture performance. For instance, exposing the mixture to unforeseen conditions such as construction delays or traffic jams during mixture transportation can result in higher short term aging. Therefore, evaluation of aging effects prior to construction may allow the researchers to enhance the pavement long term performance by resolving some construction concerns such as attaining the required pavement density. Mirza and Witczak (1995) employed a simple performance tester and developed the Global Aging System (GAS) to predict asphalt viscosity based on time and pavement depth [5]. Glover et al. (2014) developed a model to predict the oxidation rate with the input of binder kinetics data, temperature profile of the pavement, and mixture characteristics [6]. Caro et al. (2014) utilized a micromechanical model and proposed an acceptable model to estimate air voids influence on asphalt mixtures oxidation [7]. Ashrafi and Farid (2010) used Boundary Element Method (BEM) to predict and treat visco-elastic problems [8]. Although different models have been proposed to evaluate aging effects on different aspects of pavement engineering, there is still a gap in knowledge on its complex effects on binder rheological properties. Hence, this study aims to propose a new approach to predict the effects of aging on the rheological properties of binders using response surface method (RSM).

RSM is a set of techniques that are used to develop a series of experiment designs, determining relationships between experimental factors and responses, and using these relationships to determine the optimum conditions [9]. Kavussi et al. (2014) evaluated the effects of aggregate gradation, hydrated lime and Sasobit content on the indirect tensile strength of warm mix asphalt (WMA) using RSM [10]. Hamzah et al. (2013) applied RSM for determining the optimum binder content of WMA incorporating Rediset [11]. Khodaii et al. (2012) used RSM to evaluate the effects of aggregate gradation and lime content on the tensile strength ratio of dry and saturated hot mix asphalt [12]. Jamshidi et al. (2013) evaluated the rheological properties of Sasobit modified asphalt binder at high temperatures using RSM [13]. RSM has been effectively used in several other research undertakings. For instance, Chakchouk et al. (2009) used RSM to study the effects of calcination time of clay, calcination temperature and its percentage in the blended cement on the compressive strength of blended cements [14]. Khan et al. (2012) applied RSM to study the influence of welding current, welding time and welding pressure and their interaction on the tensile shear strength of weld-bonds [15]. Goh et al. (2011) used central composite design and RSM as a systematic experimental method to optimize the ethanol organosolv pretreatment of empty palm fruit bunch (EPFB) [16].

In this study, a central composite method was applied to design experiment plans. The dynamic modulus and phase angle were selected as test responses, while aging durations, test temperatures and binder types were selected as design factors. The significance of design factors and their interactions on response values were evaluated using analysis of variance (ANOVA). Mathematical equations were then generated and actual and predicted values of test responses were compared.

2.0 MATERIAL AND METHODS

2.1 Methods

Two types of binders from two sources were used. For ease of reference, binders are designated according to their source and type. Thus, binders A1 and A2 refer to the conventional penetration grade 80/100 and 60/70 binders from Petronas Sdn Bhd (source A), respectively. Binders B1 and B2 refer to the 80/100 and 60/70 penetration grade binders from Shell Sdn Bhd (source B), respectively. The basic properties of binders determined in the laboratory are summarized in Table 1.

Table 1 Conventional binder properties

Aging State	Binder Type	Penetration at 25 °C (dmm)	Softening point (°C)	G*Sinδ at 64 °C (Pa)	G*Sinδ at 70°C (Pa)	Viscosity at 135 °C (Cp)
Standards		ASTM D5 [17]	ASTM D36 [18]	ASTM D7175 [19]	ASTM D7175 [19]	ASTM D4402 [20]
Un-Aged	80/100 (A1)	80	46	1342	------	300
	60/70 (A2)	63	49	------	1935	500
	80/100 (B1)	81	47	1581	------	390
	60/70 (B2)	62	50	------	1734	510
85 min Aged	80/100 (A1)	------	------	3090	------	475
	60/70 (A2)	------	------	------	3291	720
	80/100 (B1)	------	------	2471	------	500
	60/70 (B2)	------	------	------	3083	690

2.2 Samples Preparation

The effects of aging on a binder were evaluated from the differences between their un-aged and aged rheological properties. The Rolling Thin Film Oven (RTFO) was used to produce a homogenous artificial short-term age asphalt binder in accordance with the procedures outlined in ASTM D2872 [21] except the durations were varied between 0 to 185 minutes at 92.5 minutes intervals based on RSM design. Subsequently, the sufficiently fluid un-aged and aged binders were poured into a mould and then ready for testing using the dynamic shear rheometer (DSR) in accordance to ASTM D7175 [19] procedures.

2.3 Experimental Designs

One set of experiments were designed using the central composite method to characterize the rheological properties of asphalt binders at intermediate temperatures as shown in Table 2. Test temperature, binder type and aging time were selected as the independent variables, while the complex modulus and phase angle were defined as the response.

2.4 Laboratory Tests

The effects of extended aging duration on the rheological properties of binders were investigated using the complex modulus and phase angle values obtained from the DSR test. The tests were conducted using a 25 mm diameter spindle rotating at the rate of 1.59 Hz frequency of loading. Consequently, temperature sweeps were applied from 46°C to 82°C at 6°C increments for the un-aged and short-term aged samples in accordance with ASTM D7175 [19] procedures.

2.5 Analysis Methods

RSM is the application of the regression models as well as experiment design methods, and other techniques to understand the behavior of the responses of the system. For developing a regression model for each response, linear, quadratic and two-factor interaction regression models were evaluated using sequential F-tests, lack-of-fit tests, and R-square value. For the selected regression model, the significance of each factor (linear, quadratic and interaction terms) was examined by analysis of variance (ANOVA). Insignificant factors were then discarded and proposed models were used for predicting the responses. These analyses were conducted using the Design-Expert 6.0.6 software.

3.0 RESULT AND DISCUSSION

3.1 Model Development

Based on the experimental results shown in Table 2, increasing aging duration increases the complex modulus and decreases the phase angle. On the contrary, increasing test temperature decreases the complex modulus but increases the phase angle. In order to formulate aging duration and test temperature effects on binders' rheological properties, Table 3 presents the ANOVA analysis results obtained from RSM. The Values of "Prob > F" less than 0.05 indicate model terms are significant. In the complex modulus case, test temperature (A), aging duration (B), binder type (C), square of test temperature (A2) and interaction between test temperature and binder type (AC) are significant model terms since "Prob > F" is less than 0.05, while the square of aging duration (B2) and interaction between aging duration and binder type (BC) are not significant by corresponding values 0.4630 and 0.2757, respectively. Hence, B2 and BC are excluded from the aging prediction model based on the complex modulus scenario. In the phase angle case, all terms are significant based on the "Prob > F" less than 0.05 except B2 where the corresponding value is greater than 0.05. Therefore, B2 is discarded from the aging prediction model.

Table 2 Matrix of experiment design

No.	Binder Type	Test Temperature (°C)	Aging Duration (min)	Complex Modulus (kPa)	Phase Angle (°)	No.	Binder Type	Test Temperature (°C)	Aging Duration (min)	Complex Modulus (kPa)	Phase Angle (°)
1	A1	82	92.5	0.42	89.24	23	B1	46	0.0	26.16	84.43
2	A1	46	0.0	15.53	84.14	24	B1	82	0.0	0.20	89.43
3	A1	82	185.0	0.79	89.37	25	B1	46	92.5	34.50	81.33
4	A1	46	185.0	86.87	74.88	26	B1	64	92.5	2.24	88.53
5	A1	64	92.5	3.53	86.63	27	B1	46	185.0	69.12	79.67
6	A1	64	92.5	3.53	86.60	28	B1	82	92.5	0.24	89.03
7	A1	46	92.5	47.79	78.28	29	B1	64	92.5	2.35	88.64
8	A1	64	0.0	1.27	89.33	30	B1	64	185.0	4.08	87.41
9	A1	64	185.0	6.92	83.69	31	B1	64	0.0	1.67	89.54
10	A1	82	0.0	0.18	87.78	32	B1	64	92.5	2.89	88.48
11	A1	64	92.5	3.60	86.51	33	B1	82	185.0	0.41	89.08
12	A2	64	92.5	7.47	82.25	34	B2	64	92.5	7.04	84.31
13	A2	64	185.0	12.35	79.30	35	B2	46	185.0	161.34	74.80
14	A2	64	0.0	3.81	86.55	36	B2	82	92.5	0.81	89.69
15	A2	64	92.5	7.04	82.12	37	B2	82	0.0	0.44	88.58
16	A2	82	0.0	0.50	89.34	38	B2	82	185.0	1.20	89.19
17	A2	82	92.5	0.90	88.42	39	B2	46	92.5	97.09	77.55
18	A2	46	92.5	84.25	72.34	40	B2	64	0.0	3.64	88.36
19	A2	46	0.0	50.32	77.44	41	B2	64	92.5	7.22	84.56
20	A2	46	185.0	142.95	69.01	42	B2	64	92.5	7.62	85.77
21	A2	82	185.0	1.44	87.01	43	B2	46	0.0	48.20	82.72
22	A2	64	92.5	6.55	84.31	44	B2	64	185.0	11.61	84.24

Table 3 Analysis of variance for response surface reduced quadratic model of complex modulus and phase angle

Response	Factor	Sum of Squares	DF[a]	F value	Prob > F
Complex Modulus	A[b]	30573.37	1	229.74	< 0.0001
	B[c]	5021.94	1	37.74	< 0.0001
	C[d]	2845.49	3	7.13	0.0010
	A^2	9289.86	1	69.81	< 0.0001
	B^2	73.62	1	0.55	0.4630
	AB	6302.33	1	47.36	< 0.0001
	AC	3883.88	3	9.73	0.0001
	BC	541.44	3	1.36	0.2757
Phase Angle	A	699.56	1	1004.57	< 0.0001
	B	104.10	1	149.48	< 0.0001
	C	155.86	3	74.61	< 0.0001
	A^2	63.07	1	90.57	< 0.0001
	B^2	1.06	1	1.52	02272
	AB	55.91	1	80.28	< 0.0001
	AC	50.24	3	24.05	< 0.0001
	BC	9.98	3	4.78	0.0080

Note1: a is Degree of Freedom, b is Test Temperature, c is Aging Duration, d is Binder Type.
Note 2: In order to propose accurate regressions, factors B^2 and BC for complex modulus and factor B^2 for phase angle were eliminated due to "Prob > F" greater than 5%

In order to develop models which can characterize and predict the rheological properties of asphalt binder, the statistical analysis was conducted using RSM and the results are shown in Table 4. The results indicate that the models for both complex modulus and phase angle are significant since the "Prob > F" is less than 0.05. Based on the lack of fit, the corresponding values obtained for complex modulus and phase angle are 0.01% and 30.39%, respectively. Since, this study attempts to fit the model, the significant value for lack of fit is not desirable. Hence, it can imply that the proposed model for complex modulus might be affected due to the unexpected variation in a sample or test results since the "Prob > F" is less than 0.05. However, Table 4 shows that the models are in reasonable agreement since the R-squared are 93% and 98% for complex modulus and phase angle, respectively. The developed quadratic mathematical equations to predict complex modulus and phase angle of asphalt binders based on RSM are shown in Table 5. From the complex modulus equations, test temperatures exhibit negative impact which indicates that temperature increment causes reduction in complex modulus, while aging duration exhibits positive impact on the related responses which shows that additional aging duration increases the corresponding value. However, the interaction effects between test temperature and aging duration exhibits negative impact. This implies that interaction between these values causes reduction in the complex modulus. From the phase angle equations, test temperatures have positive impact which shows that temperature increment increases the phase angle, while aging duration exhibits negative impact, which indicates that an increase in aging duration causes a reduction on the related response. However, the interaction between test temperature and aging duration exhibits positive impact, which implies that the interaction between temperature and aging leads to an increase in phase angle.

Table 4 Models proposed for complex modulus and phase angle

Title		Sum of Squares	DF[a]	Mean Square	F Value	Prob > F	Model Type
Complex Modulus	Model	59108.52	10	5910.85	43.59	< 0.0001	Quadratic (Sig)
	Residual error	4474.36	33	135.59			
	Lack of fit	4473.51	25	178.94	1677.49	< 0.0001	(Sig)
	R-squared	0.93					
Phase Angle	Model	1138.93	13	87.61	123.65	< 0.0001	Quadratic (Sig)
	Residual error	21.26	30	0.71			
	Lack of fit	16.99	22	0.77	1.45	0.3039	(Not Sig)
	R-squared	0.98					

Note: a is Degree of Freedom

Table 5 Equations obtained from the response surface method

Title	Binder Type	Equations
Complex Modulus	A1	y = +393.59482 - 12.52088 * A + 0.91926 * B + 0.095669 * A^2 - 0.011920 * A * B
	A2	y = +481.57055 – 13.68645 * A + 0.91926 * B + 0.095669 * A^2 – 0.011.920 * A * B
	B1	y = +379.40260 – 12.33686 * A + 0.91926 * B + 0.095669 * A^2 – 0.011.920 * A * B
	B2	y = +501.65104 – 13.95958 * A + 0.91926 * B + 0.095669 * A^2 – 0.011.920 * A * B
Phase Angle	A1	z = +47.61913 + 1.11708 * A - 0.095817 * B - 7.43394E-003 * A^2 + 1.12267E-003 * A * B
	A2	z = +34.90668 + 1.27347 * A - 0.10431 * B - 7.43394E-003 * A^2 + 1.12267E-003 * A * B
	B1	z = +52.48709 + 1.05237 * A - 0.084896 * B - 7.43394E-003 * A^2 + 1.12267E-003 * A * B
	B2	z = +44.74276 + 1.14764 * A - 0.092440 * B - 7.43394E-003 * A^2 + 1.12267E-003 * A * B

Note: y is Complex Modulus, A is Test Temperature, B is Aging Duration, Z is Phase Angle

3.2 Data Characterization

Figure 1 illustrates the contour plot of relationship between aging duration and test temperature effects on the complex modulus of binders A1, A2, B1 and B2. From the results, increase in test temperature decreases the complex modulus, while extended aging duration increases the corresponding value. For instance, the complex modulus of binder A2 at 95.5 minutes aging duration, decreases by approximately 400% when temperature increases from 52°C to 64°C. Conversely, the complex modulus of binder A2 increases by approximately 28% when aging duration is extended from 70 minutes to 138.8 minutes and temperature is maintained at 52°C. These binders' behaviors are consistent with earlier results obtained by Hamzah et al. (2012) [22]. Complex modulus increment due to extended aging duration is related to the volatilization and oxidation of the binders during conditioning in the RTFO, while the corresponding value decrement due to temperature increment relates to the binder tendency to behave as a viscous material at higher temperature. However, changes in complex modulus at different conditions clearly testify that test temperature effects on complex modulus are higher compared to the aging duration effects. This can also be inferred from the complex modulus equations where the interaction between test temperature and aging duration decreases the complex modulus. The magnitude of binders' complex modulus is also dependent on binder type due to their different chemical composition. Hence, the effects of source are also determined based on the comparison between binders complex modulus obtained at 52°C and 70°C. For example, the results show that complex modulus of binder B2 at 52°C is approximately 10% higher compared to the corresponding value for binder A2 for 92.5 minutes aging duration, while the complex modulus of both A2 and B2 binders exhibit no significant differences at 70°C. Thus, it can be concluded that binders with the same penetration grade exhibit different rheological behavior at lower temperature but exhibit same trend at higher temperatures. The binders' temperature susceptibility phenomenon can be attributed to its viscous fluid behavior at higher temperature and lower resistance to deformation when repeatedly sheared.

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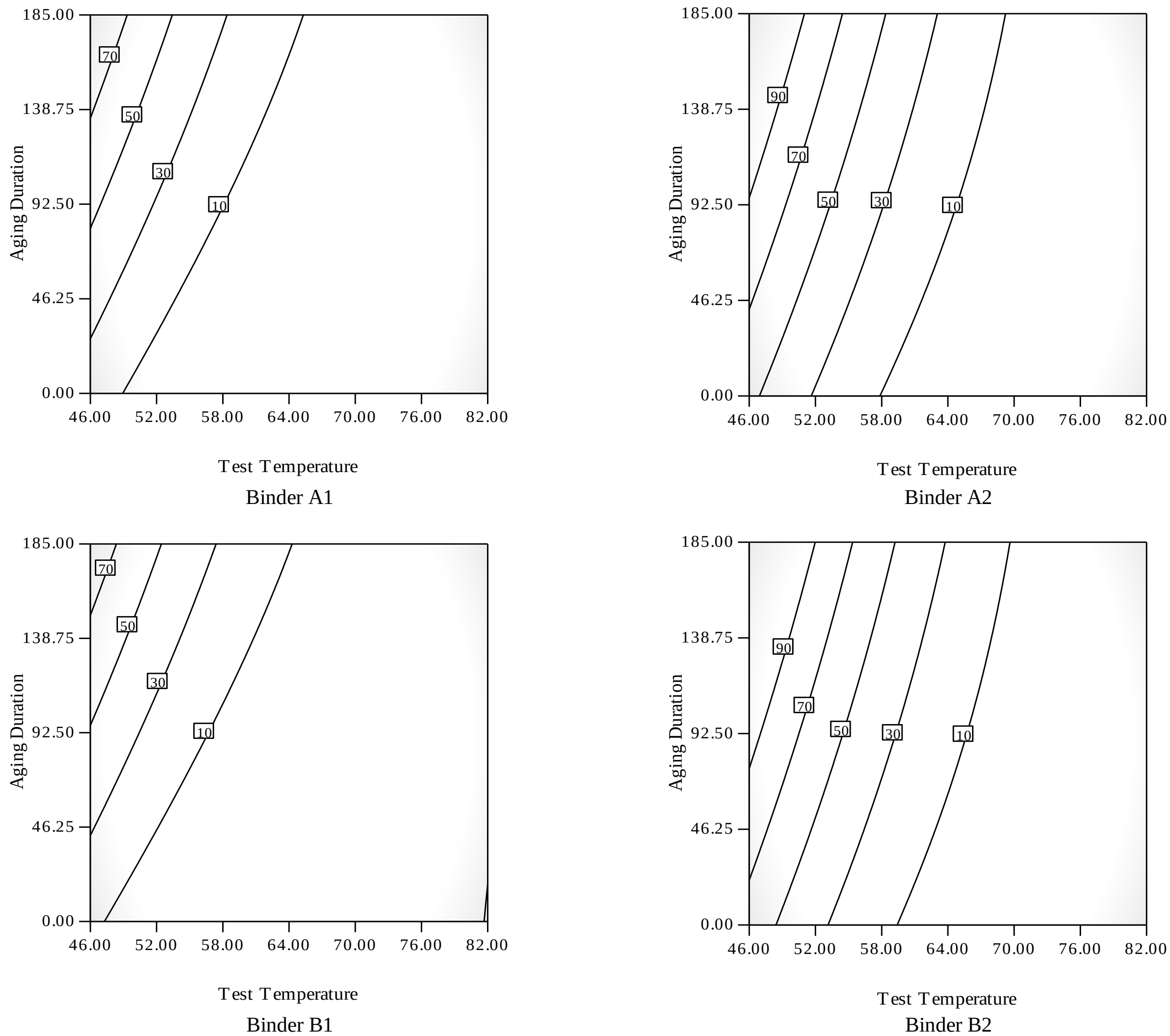

Figure 1 Binder A1, A2, B1 and B2 complex modulus pattern (kPa)

Figure 2 illustrates the contour plot showing the relationship between aging duration and test temperature effects on the phase angle of binders A1, A2, B1 and B2. From the results, increment of test temperature increases the phase angle, while extended aging duration decreases the corresponding values. For instance, the phase angle of binder A1 subjected to 95.5 minutes aging duration increases from 82° to 86.5° when the test temperature increases from 52°C to 64°C. On the contrary, the phase angle of binder A1 decreases from 82° to 80.5° when aging duration is extended from 70 minutes to 138.75 minutes and temperature is maintained at 52°C. These discrepancies show that test temperature effects on phase angle are higher compared to the aging duration effects. The same conclusion can also be drawn from the phase angle equations where the interaction between test temperature and aging duration increases the phase angle. Since, phase angle is an indicator of the relative amount of recoverable and non-recoverable deformation, its decrement due to extended aging duration shows that conditioning the binder in the RTFO increases the binder elastic component while temperature increment reduces the binder elastic component based on phase angle increment. The binder source effects are also evaluated based on the comparison between the phase angle obtained at 52°C and 70°C. For example, the results show that for 92.5 minutes aging duration, the phase angle of binder A1 are approximately 82° and 88° at 52°C and 70°C, respectively, while the corresponding values of binder B1 are approximately 84.5° and 89° when tested at 52°C and 70°C, respectively. From the results, it can be seen that binders with the same penetration grade exhibit higher phase angle discrepancy at lower temperature compared to the corresponding values obtained at higher temperatures. These binders' characteristics are consistent with the results obtained by other researchers [23-27]. Hence, it can be concluded that aging duration increment increases the binder relative amount of recoverable deformation, while increasing test temperature increases the binder relative amount of non-recoverable deformation.

To verify the accuracy of the complex modulus and phase angle regressions, the actual and predicted values are compared and shown in Figure 3. The results show that the phase angle mathematical equation fits the experimental observation with higher accuracy compared to the complex modulus regression. This greater deviation for complex modulus might be related to the higher lack of fit or unexpected variation in samples and test results as explained earlier.

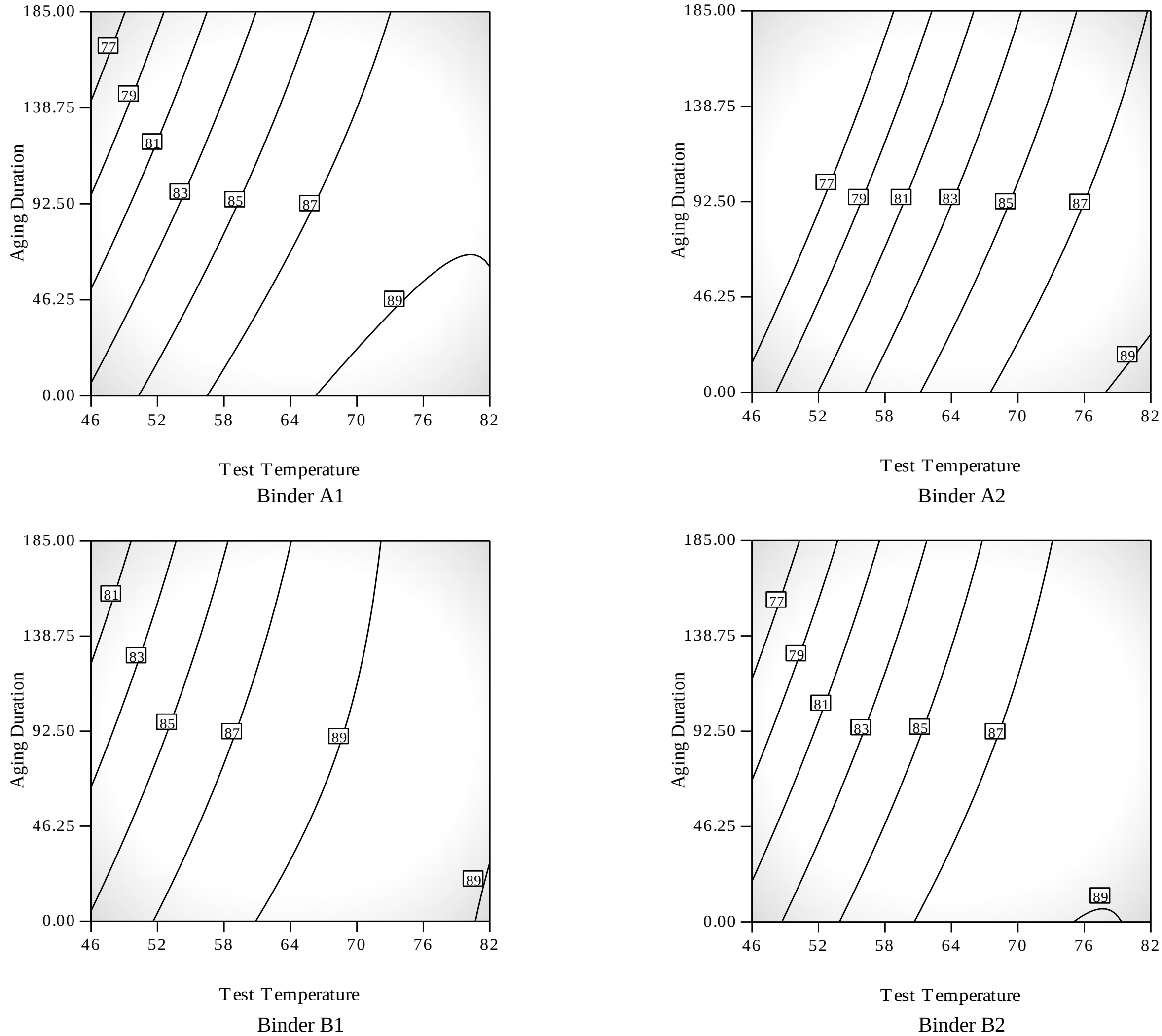

Figure 2 Binder A1, A2, B1 and B2 phase angle pattern (°)

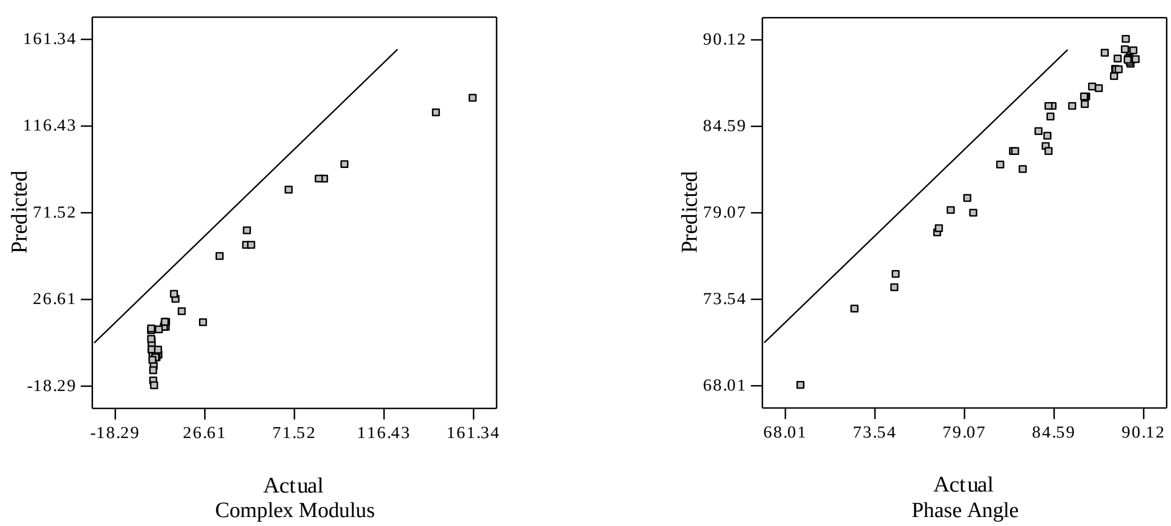

Figure 3 Predicted complex modulus and phase angle versus actual results

4.0 CONCLUSION

Extended aging duration and test temperature effects on binders were quantified in terms of their complex modulus and phase angle. The RSM technique employed exhibits the ability to quickly determine the rheological behavior of binders at various conditions. The results indicate that extending the aging duration increases the binder complex modulus but decreases its phase angle. However, increasing test temperature leads to reduction in complex modulus but increases the phase angle. The binders' complex modulus exhibit different behavior at lower temperature but exhibits similar trend at higher temperatures and this is attributed to the binders' viscous fluid behavior at higher temperature and lower resistance to deformation when repeatedly sheared. It is also found that the magnitudes of these changes vary based on the variation in binder sources and types. The final results show that the mathematical equation fits the experimental observation with a good accuracy.

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