RHEOLOGICAL PROPERTIES OF CRUMB RUBBER-MODIFIED BITUMEN CONTAINING ANTIOXIDANT

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الخلاصــة:

لقد أصبحت الخصائص الفيزيائية أداة قيمة في تصنيف أداء الإسفلت في الطرق . وقد تم قياس خصائص اللدونه لفتات المطاط المحسّن باستخدام مضادات الأكسدة (CR30) لقياس الخصائص الفيزيائية ، وقد أعطت هذه القياسات معلومات أفضل لأداء الأسفلت عند تعرضه لظروف مختلفة من الحرارة والأحمال، كما يشاهد عند إنشاء الطرق، وخلال الخدمة. وقد استخدم جهاز القص الديناميكي لتصنيف رابط الأسفلت قبل التعتيق بالفرن وبعده، حيث تم تعتيق الرابط الإسفلتي لثلاثة وتسعة أيام. وقد بينت نتائج اختبارات المطابقة أن إضافة الإسفلت المحسن (CR30) مساوية للأسفلت الحر ، كما بينت النتائج أن الإسفلت غير المعتق بإضافة 1% (CR30) و 5% (CR30) نتج عنه زيادة في معامل القص (G^*) نتيجة للتغيرات الفيزيائية ، كما بينت النتائج بأن التعتيق قد أثر على الخصائص الفيزيائية للإسفلت بشكل كبير، وذلك بزيادة معامل القص وإنقاص زاوية الحالة.

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ABSTRACT

Rheology has become a useful tool in the characterization of the bitumen performance on the pavement. Visco−elastic properties of crumb rubber modified bitumen with antioxidants (CR30) were determined by the means of rheological measurement. This measurement led to a better knowledge of bitumen behavior that occurs when subjected to different thermal and mechanical conditions, as seen during road construction and services in the field. Dynamic Shear Rheometer (DSR) was used to characterize the rheology of the binders before and after oven aging. The binders were aged for 3 and 9 days. Results of a compatibility test showed that the addition of CR30 modified bitumen is compatible with the base bitumen. The results of unaged samples indicated that the addition of 1% CR30 and 5% CR30 modified binders caused an increase in $G*$ value as a result of the rheological changes. Results showed that aging has significant influence on bitumen rheology, by increasing complex modulus and decreasing phase angle.

Key words: bitumen, rubberized bitumen, aging, rheological characterization

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1. INTRODUCTION

Conventional bitumen 80/100 penetration grade is widely used in most countries, including Malaysia, where it tends to harden at the early stage of handling in storage, during mixing, and in service [1]. The level of performance of service life has an intimate relationship with the properties of bitumen used in the asphalt concrete [2]. This rheological weakness of conventional bitumen has generated an increasing interest in the use of polymer-modified binders to enhance conventional bitumen properties. The development of modified asphalt materials to improve the overall performance of pavements has been the focus of several research efforts made over the past few decades. A limited number of polymers have been used as modifying agents due to their high cost. Because of the high cost of these polymers compared to bitumen, the amount needed to improve pavement performance should be as small as possible. The use of discarded vehicle tires in pavement construction was one of the steps that were taken in this direction. Research on crumb rubber has been going on for the last three decades [3]. The characteristics of crumb rubber depend on the rubber type, asphalt composition, and size of rubber crumbs, as well as time and temperature of reaction. These factors have considerable affect on pavement performance [4–6]. Researchers [7] carried out experiments to study the effects of three different rubber concentrations (3%, 9%, and 15%). According to this study, after a rolling thin film oven test (RTFOT), the unmodified bitumen showed an improvement of about 1.5 times in $G*$ value, and in the case of rubberized bitumen, the samples with 3% and 9% rubber showed an increase of about 2.5 times, the sample with 15% rubber showed an increase of about 1.5 times compared to their original unaged values. After a pressure aging vessel (PAV) test, the $G*$ of unmodified bitumen increased by about 2 times its unaged values and by about 2 to 3 times in the case of rubberized bitumen. Devulcanization and depolymerization of rubber in the presence of bitumen and the application of temperatures and shear is discussed in reference [8], while [9] found that extending the blending time from 1 hour and increasing blending temperature from 177°C can significantly reduce the high-temperature viscosity. Other research indicated that with concentration of rubber below 12% the binder viscosity declines with time and stabilizes after approximately 45 and 60 minutes of mixing [10]. According to [11], the elastic recovery time increases with a decrease in crumb rubber particle size. Similarly, an investigation to observe the effect of crumb rubber particle size on the stability of asphaltic concrete was performed by [12]. Four different concentrations and three different crumb rubber particle sizes were used simultaneously. It was concluded that stability of asphaltic concrete mixtures decreases as the crumb rubber concentration in bitumen of 20% and particle size of 0.300 mm to 0.075 decreases.

The durability of asphaltic concrete is greatly influenced by the environmental changes during the year, between hot and cold temperatures and between day and night. High temperatures can soften the bitumen and consequently reduce the stiffness of asphaltic concrete, making the mix more susceptible to rutting [13]. On the other hand, low temperature, can increase the stiffness of bitumen and reduce the flexibility of the asphaltic concrete, inducing fatigue failure as a result. Cracking of the pavement surface may develop, which adversely affects the performance of the asphaltic concrete. Thus, high temperature stiffness and low temperature flexibility are important properties in bituminous mixtures respectively to avert rutting and cracking [14]. Bitumen is a viscoelastic material that is characterized by a certain level of rigidity of an elastic solid, but at the same time, flows and dissipates energy by frictional losses as a viscous fluid. As with any viscoelastic material, asphalt's response to an imposed stress or strain is dependent upon both temperature and loading time. In order to predict the engineering performance of bitumen, under the wide spectrum of temperatures and loading conditions encountered in the field, the bitumen rheologists have repeatedly tried to describe its viscoelastic behavior using dynamic shear rheoemter. DSR was used to characterize the rheology of the binders before and after oven aging. Rheology has become a useful tool in the characterization of the bitumen performance on the pavement [15, 16], above all since the SHRP (Strategic Highway Research Program) protocol brought up a set of tests and methods in a very wide range of temperatures, which allowed obtaining information about the suitability of given bitumen in a further application. An enhanced behavior of the modified bitumen is commonly demanded in a wide range of temperatures and loading [17]. The effects of modified and unmodified bitumen on viscoelastic properties are discussed in this paper

2. METHODS AND MATERIALS

2.1. Materials

Several standard grades of bitumen are available commercially, but conventional 80/100-pen grade bitumen is used as bituminous binder in road pavements in Malaysia. The properties are as given in Table 1. Crumb rubber used for this study was supplied by Ara Jaya Teguh Enterprise and passed through No. 40 (0.425 mm) mesh sieve. The properties of the crumb rubber used are as shown in Table 1.

Table 1. Material Properties Used in this Investigation

Material	Parameter Measured	Value	Standard
80/100	Specific Gravity	1.026	ASTM D70
	Softening Point (°C)	42	ASTM D36
	Penetration at 25°C (d-mm)	83	ASTM D5
	Ductility (cm) at 25°C	> 100	ASTM D 113
Crumb Rubber	Rubber Size	40 mesh	-
	Specific gravity of Crumb rubber	1.029	-

2.2. Production of CR30 Through Wet Process

There are two types of mixers used to blend base bitumen with modifiers; namely, propeller mixer and high shear type mixer. In practice, it is easier to control the propeller type mixer. A study reported that the binder produced using high shear type mixer appears to have superior properties compared to that produced using propeller type mixer [18]. The high shear type mixer used for the digestion process is shown in Figure 1. CR30, Drain Asphalt Modified Additive (DAMA) modified binders were blended for 1.0 hour at 180°C and 2200 rpm. These parameters, adopted in this study, are similar to those conducted by other authors [19–22]. The 80/100 conventional binder was accurately weighed using an electronic balance. The base 80/100 bitumen was preheated at 180°C in a 4 liter container. 1% CR30, by total weight of mix, was added. The mixing was done at a temperature of 180°C and 2200 rpm. Mixing of the binder continued for a total of 1 hour while the temperature was maintained at 180°C.

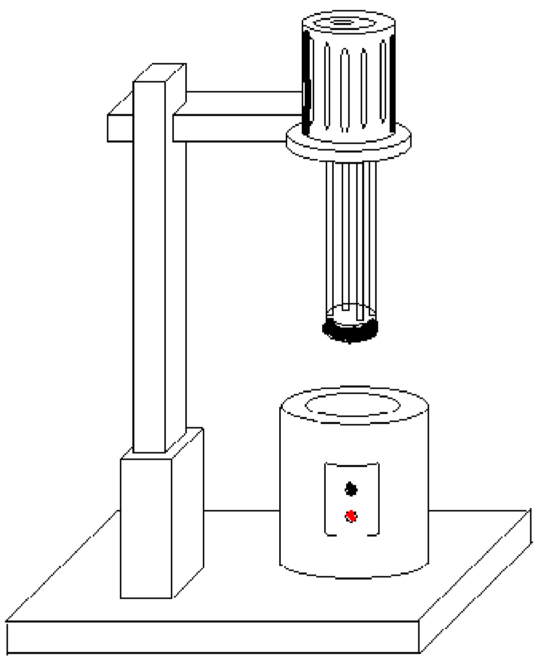

Figure 1. Blender specially fabricated for this study

2.3. Compatibility Test

The separation of polymer and bitumen during hot storage was evaluated by comparing the softening point of the top and bottom samples taken from a conditioned, sealed tube of polymer-modified bitumen. The test was conducted according to [22]. The tubes made of aluminum (25.4 mm diameter and 139.7 mm height) were used.

2.3.1. Procedure of Compatibility Test

Two samples from the modified bitumen CR30 were poured into the tubes immediately after blending. Binders were kept in containers in an oven at constant temperatures of 163°C ± 5°. The tubes were allowed to stand undisturbed in the oven for a period of 48 ± 1 hour, after that the tubes were removed from the oven. Immediately after 48 hours, the samples were transferred to a freezer, at −6.7 °C ± 5°C for 4 hours to solidify. The samples were cut into three pieces upon removal from the freezer. The middle section was discarded while the top and bottom portions were put into separate beakers and heated again at 163°C ± 5°C until the bitumen was sufficiently thin and tested for its softening point.

2.4. Procedure of Bitumen Aging

Procedures of bitumen aging that we adopted consist of pouring bitumen in a tray to form a film thickness of 3 mm. The trays were designed in such a way that four samples of dimensions 180 mm × 180 mm and 15 mm deep could be placed in each tray, to be tested simultaneously. The conditioning procedures involved the placing of bitumen in the tray inside a forced-draft oven at 60°C for 3 days and 9 days. These were similar to those conducted by [23], but the oven aging was conducted between two and ten days. After conditioning, the samples were removed, and the aged samples were cooled to room temperature prior to subsequent testing. The samples placed in the oven were maintained at 60°C.

2.5. Dynamic Shear Rheometer (DSR)

SHRP has developed concepts for a new bitumen specification. This specification is included in the AASHTO bitumen specification [24]. The new specification is based on the rheological properties of the binders measured over a wide range of temperatures. Generally, dynamic shear rheometer is associated with two testing geometries, namely a 8 mm diameter, spindle with a 2 mm testing gaps and a 25 mm diameter spindle with a 1 mm testing gap. The DSR tests were performed under the test conditions shown in Table 2. The DSR test used a thin asphalt binder sample sandwiched between two plates. The lower plate is fixed, while the upper plate oscillates back and forth across the sample at 1.59 Hz (10 radians/second) to create a shearing action. This magnitude of oscillation simulates the shearing action corresponding to that of a traffic speed of about 90 km/hr [25].

Table 2. DSR Tests Parameters Used in the Study

Parameter	Value
Mode of Loading	Control Strain
Strain amplitude (%)	12
Temperature (°C)	10 to 50
Spindle geometries (mm)	25 ϕ 1 gap
Frequency of loading (rad/sec)	10
CR30 and CR50 (%)	1 to 5
Aging Condition of Binder (day)	3 and 9
Number of replicate measurements	2

3. RESULTS AND DISCUSSION

3.1. Discussion of Results of Compatibility Test

The softening point temperature between top and bottom sections is used to evaluate compatibility of base and modified bitumen. Test results presented in Table 3 show a low temperature difference to the addition of CR30 mixed with bitumen 80/100, when tested top and bottom, although in Malaysia, there are no set standard tests. Other researchers [26] reported that highway engineers in Taiwan select 2°C as the maximum temperature difference to monitor PMB's stability. Therefore, it can be concluded that the addition of CR30 modified bitumen is compatible with the base bitumen.

Table 3. Results of Compatibility Test of CR30

Section Side	Softening Point (°C)	
	SAMPLE (1)	SAMPLE (2)
Top	47.0	50.0
Middle	48.5	49.0
Bottom	48.0	48.0
Average	**47.8**	**49.0**

3.2. CR30 Binder Complex Shear Modulus (G^*)

Table 4 shows the average values of G^* for all binders tested over temperatures ranging from 10°C to 50°C. With all binders, the values of G^* decrease with the temperatures increased. It can be noted that the addition of DAMA, 1% CR30, and 5% CR30 increased G^* as compared to base bitumen 80/100. This is particularly clear because at temperature (30°C−40°C) due to the presence of crumb rubber, which provides a significant elastic capacity. However, at low temperature (10°C) the effect of 1% CR30 and 5% CR30 modification on G^* values appears to be insignificant; the behavior of the modified binders remains close to that of base bitumen. These results are similar to the research findings [27] that claim there was no significant difference between the base and modified bitumen when tested at 10°C. As temperature increases, the G^* values from 20°C to 40°C showed higher than modified bitumen. For instance, 1% CR30 and 5% CR30 also showed an increase of 39.5% and 34.80% respectively. This indicates a degree of change in the structures and compositions of bitumen and may be attributed to the fact that the viscosity of the components is too low to allow the elastic network of the rubber to influence the mechanical properties of the modified bitumen [28].

Table 4. Comparison of Complex Modulus of CR30 and 80/100 at Different Temperatures

T (°C)	Bitumen Type			% Increase Compared to $G^*$of 80/100	
	80/100	1% CR30	5% CR30	1% CR30	5% CR30
10	6784000	6822000	6708000	0.56	1.67
20	2283000	2418000	2455000	5.90	7.53
30	245100	341800	370400	39.45	51.122
40	36710	45130	53370	22.93	45.38
50	5106	5423	5605	6.21	9.77

3.3. Effect of Aging on Complex Modulus

Base and modified bitumen have been subjected to high temperatures with ultraviolet exposure in an oven for 3 and 9 day aging. The changes occurred were assessed in terms of complex modulus. The effects of 3 and 9 day aging on the rheological characteristics of the 1% CR30 modified binders and base bitumen are shown in Figure 2. The increase in temperature is followed by a decrease in the complex modulus. This is observed for the aged and non-aged binders. Results in Figure 2 indicate an increase in the value of $G*$ for the 1% CR30 aged bitumen as compared to the unaged one from (20°C – 50°C) both 3 and 9-day. The results shows an increase in $G*$ with aging, although the changes tend to be lower for the temperatures above 50°C compared to the unaged bitumen.

The results in Figure 3 indicate an increase in the value of $G*$ for 1% CR30 with aging. The 3-day aging produced an increase of 18% to 71% when tested at 20°C to 50°C respectively. The increase is more pronounced in 9-day

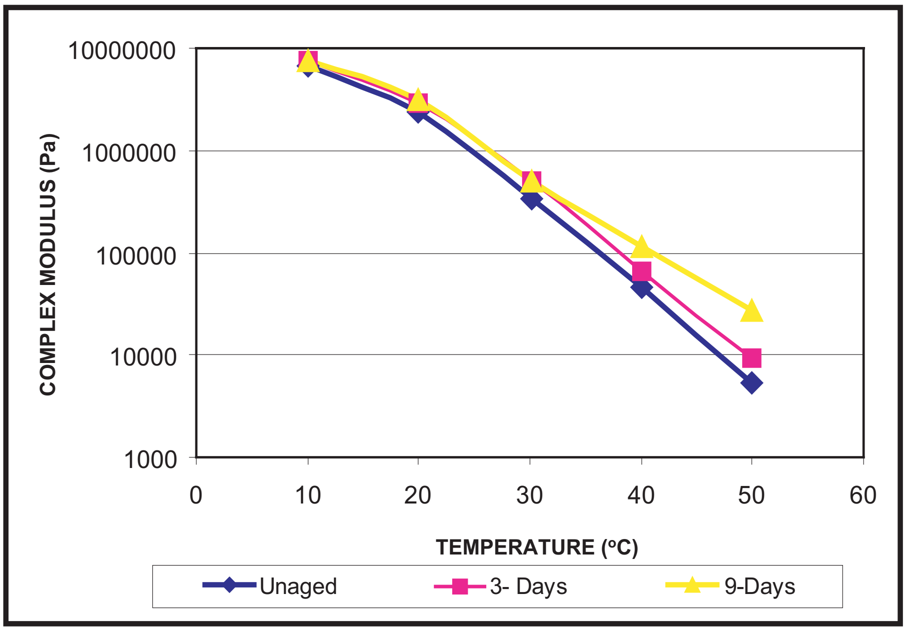

Figure 2. Effect of aging on complex modulus at varying temperature using 1% CR30 modified binder

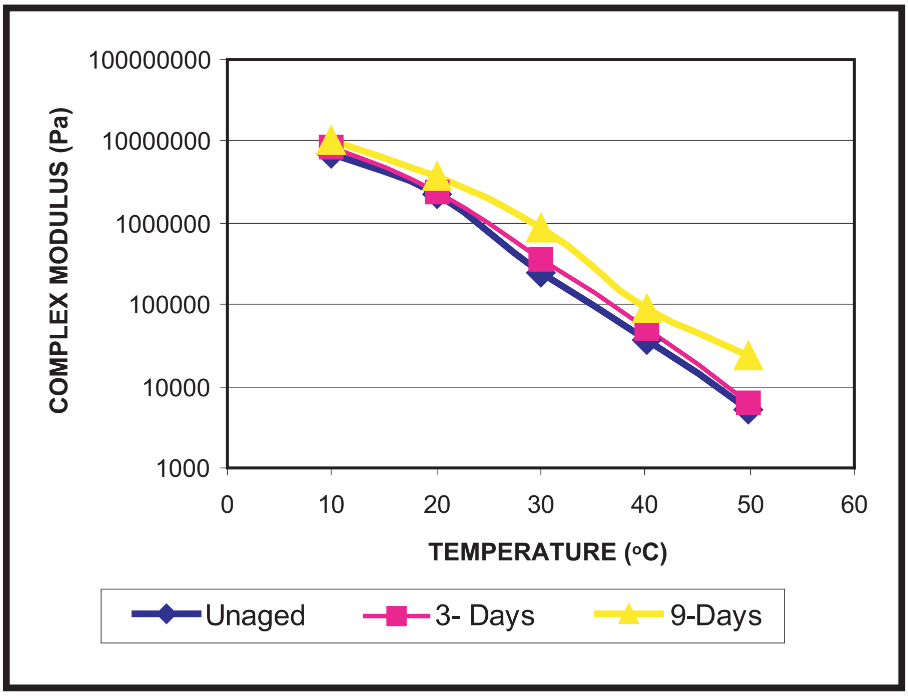

Figure 3. Effect of aging on complex modulus at varying temperature using 80/100 base bitumen

aging, which is about 9% to 398% for temperatures ranging from 10°C to 50°C. This indicates that the aged binder becomes harder as compared with the unaged one. These changes are mainly attributed to oxidative age hardening of bitumen. The effect of oxidation appears to reduce the difference in temperature susceptibility between modified and unmodified bitumen. It was also reported that substantial changes can be expected in the behavior of polymer-modified bitumen after aging [29]. Aging at a temperature of 10°C shows little influence on $G*$. This means that the aged binders become harder as compared to their unaged state.

3.4. Effect of Modification on Phase Angle

Measurements of δ are generally considered to be more sensitive to the chemical structures. The results illustrate the improved elastic response (reduced phase angles) of the modified binders, compared to base bitumen. The study shows decreases in phase angle were observed after adding modified bitumen to the base in the temperature range of 20°C to 30°C. For instance, at 10°C 1% and 5% CR30 showed a decrease in δ of 7.85% and 6.72% respectively. At 20°C 1% CR30 and 5% CR30 also showed a decrease in δ of 5.54% and 11.75% respectively, as compared to base bitumen. These results indicate that at 40°C and 50°C the phase angle of both the base and modified binders approach 90°. It can be interpreted from this behavior that the stored energy per cycle of deformation becomes negligible as compared to the dissipated energy.

3.5. Effect of Aging on Phase Angle

The major effect of aging on bitumen is the reduction of phase angle across the range of temperature studies. The results in Figures 4 and 5 reflect these behaviors. The changes in phase angle after 3 and 9-day aging are the same as those shown for unmodified bitumen at 10°C. This region corresponds to conditions where the base bitumen is more or less the same as mentioned by [30]. From the results of modified bitumen, tested at temperatures ranging from 20°C to 40°C, the decrease by 55% to 7% after 3-day aging can be observed, while the corresponding value of unmodified bitumen is 7% to 3%. The influence of the modification is more noticeable for the 1% CR30 where the elastic response is greater, particularly at the 9-day aging. The decrease is in a range of 55% to 10% from 20°C to 40°C. Phase angle decrease demonstrates the tendency of the polymer to form a continuous elastic network when dissolved or dispersed in the bitumen [21]. As a result, it can be observed that the value of phase angle at 40°C is the threshold for modification of the rheological characteristics of materials in this study.

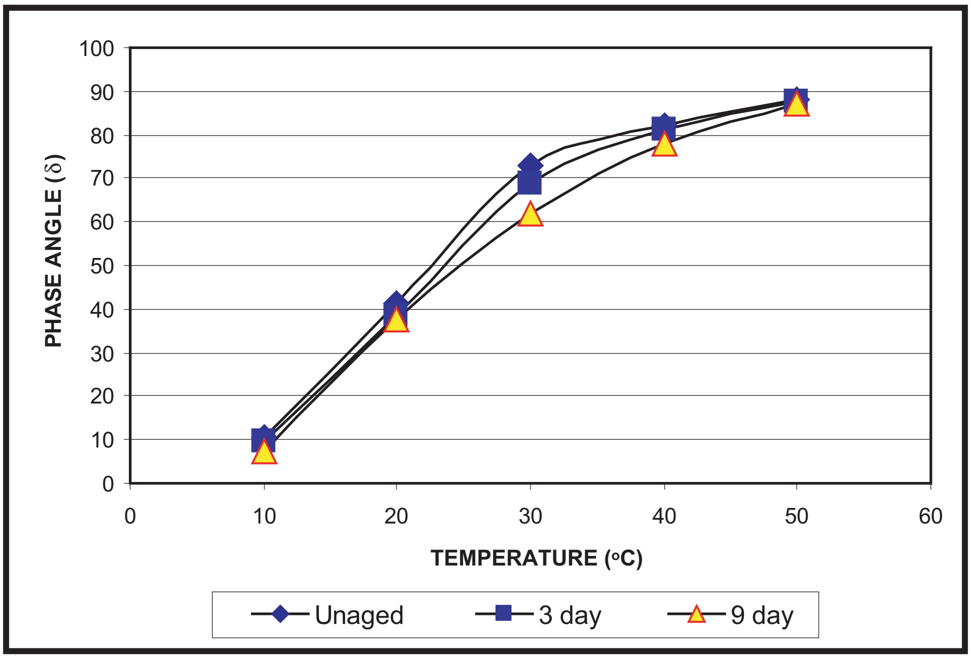

Figure 4. Effect of aging on phase angle of 80/100 base bitumen subjected to varying temperature

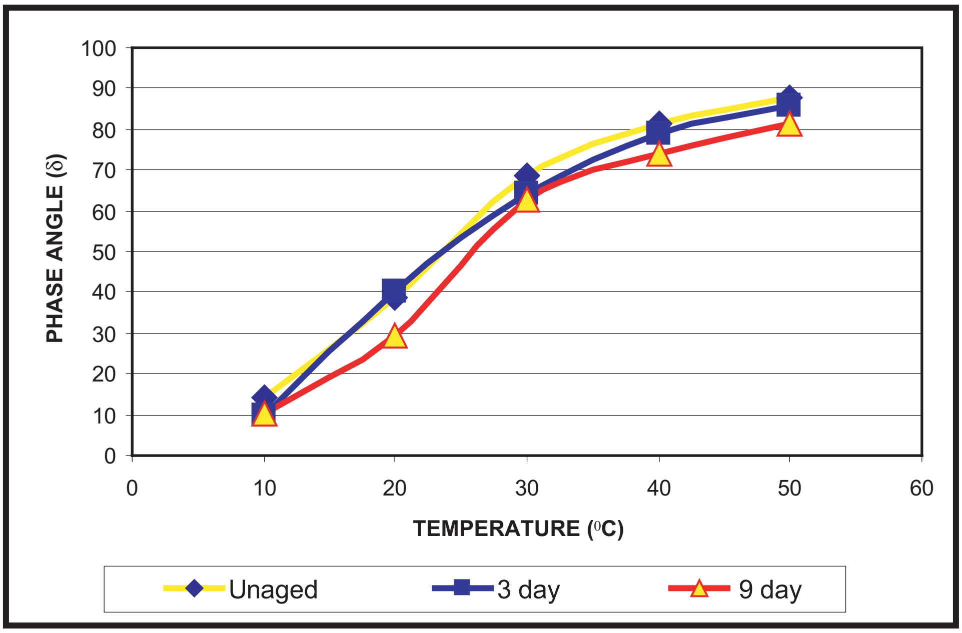

Figure 5: Effect of aging on phase angle of 1% CR30 modified bitumen subjected to varying temperature

4. CONCLUSION

The effects of modified and unmodified binders on viscoelastic properties are discussed in this paper. The dynamic shear rheometer was used to characterize the rheological properties of the modified and unmodified binders. The binders were aged for 3 and 9 days in an oven. The results of unaged samples indicated that the addition of 1% CR30 and 5% CR30 modified binders caused an increase in G^* value. As a result of the rheological changes, the mechanical properties of aged 1% CR30 and binders show improvement, as indicated by increased complex modulus and decreased phase angle. These results showed an increase in G^* with aging, although the changes tend to be lower for the temperatures 50°C compared to the unaged bitumen. Results showed that at 50 °C the phase angle of both the base and modified binders approach 90°.

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