

Creep-Recovery behaviour of Crumb Rubber Modified Bitumen (CRMB) Containing Trans-Polyoctenamer

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ABSTRACT:

The main objective of this study is to analyse the creep-recovery behaviour of crumb rubber modified bitumen (CRMB) and crumb rubber modified bitumen containing Trans-polyoctenamer (CRMB-TOR). Moreover, the effect of different stress levels to creep parameters, namely, non-recoverable compliance (J_{nr}) and per cent recoveries (% R) are also studied. The Multiple Stress Creep Recovery (MSCR) test performed by Dynamic Shear Rheometer (DSR) was executed at 1 s creep loading with 9 s recovery

over the multiple stress levels ranged from 25 to 25,600 Pa. In this study, CRMB was prepared with different percentages of crumb rubber namely 4%, 8% and 12%. The CRMB-TOR was prepared by adding 4.5% of TOR (by weight of crumb rubber) to each CRMB blend. Adding crumb rubber in the bitumen shows desirable change in creep-recovery behaviour. Moreover, application of TOR to the CRMB seems to further improve the creep parameters. An increase in creep-recovery parameters (low Jnr and high % R) can significantly improve the properties of bituminous mixtures. Thus, pavement constructed with good creep-recovery parameters would increase the service life with better rutting resistance.

Keywords: Crumb Rubber, Dynamic Shear Rheometer, Multiple Stress Creep Recovery, Rubberized Bitumen, Trans-Polyoctenamer,

1. INTRODUCTION

Many research works have been devoted to binder properties in order to formulate a method that can produce bituminous pavements that has a longer service life, lower maintenance, less expensive and environmentally friendly. Therefore, the search for suitable methods wherever possible to re-use any locally waste materials becomes a challenge to engineers and authorities. Use of waste materials such as crumb rubber has shown promising benefits not only as an additive in the bitumen, but in many engineering fields such as in mortars [1], concretes [2], railways [3] etc.

Due to the benefits offered by crumb rubber, attempts have been carried out by researchers on the implementation of chemical additives. For instance, chemical stabilizers, activation agents and polymers were incorporated in order to activate the chemical reaction between crumb rubber and bitumen. Introducing trans-polyoctenamer (TOR) to bitumen as a crosslinking agent might be one of the options [4, 5]. TOR can be utilized effectively for bitumen modification, especially with a combination of crumb rubber to produce thermodynamically stable blends, reduce phase separation when the modified bitumen is stored and reduced tackiness [6].

Studies have been conducted recently by many researchers by adding TOR to the bitumen where it was revealed that TOR can increase the high temperature performance of rubberized binders [7,8,9]. Solaimanian *et al.* (2003) [7] for instance, studied the influence of TOR on rubberized bitumen with a virgin bitumen of PG 58-28 and CRM of 14 and 30 mesh size. The study revealed that by incorporating 4.5% TOR (by weight of CRM) with 5% of 14 mesh CRM increased the high temperature binder by one grade. Moreover, the study observed three grades increased for mixes with 10%, 14 mesh CRM. However, the study also determined that by adding the TOR to 30 mesh CRM did not improve the high binder performance nearly as much as the 14 mesh CRM. The study conducted by Plemons (2013) [8] using a combination of 0.5% TOR by weight of bitumen and 10% of crumb rubber (sized 30 and 40 mesh) increased the high temperature grade of PG 67-22 bitumen.

It was reported that use of higher strain levels are required to determine the high

temperature performance of modified binder including crumb rubber modified bitumen [6]. Therefore, the Multiple Stress Creep Recovery (MSCR) test was developed to overcome the shortage of the creep and recovery tests (CS Recovery-Test) that uses low strain level. According to Federal Highway Administration report entitled *The Multiple Stress Creep Recovery (MSCR) Procedure, 2011* [10]; the low strain level used in the CS Recovery-Test may not be enough to activate the modifier thus the performance of the modified bitumen are not fully accessed. In addition, the results obtained from the CS Recovery-Test also shows lack of correlation with rutting measurements on bituminous mixtures, loading type and loading level [11,12].

Recent studies also show that MSCR test has better correlation with rutting performance of bituminous pavement as compared to the CS Recovery-Test [13]. In addition, the parameters obtained from MSCR test is recognized in the Superpave specification to filter the binders that are too stress sensitive [14]. For this reason, the Multiple Stress Creep Recovery (MSCR) test has been used by many researches to evaluate the high temperature performance of bitumen [15-20]. The MSCR test was performed by a Dynamic Shear Rheometer (DSR) at stress level of 0.1 and 3.2 kPa [21]. Started with 0.1 kPa, the stress level was applied for 1-s loading time, followed by a 9-s recovery time in which no load is applied. At each stress level, the creep-recovery procedure was repeated for 10 times. From the MSCR test loading results; the creep parameters, namely, non-recoverable compliance (J_{nr}) and per cent recoveries (% R) are used to evaluate the creep-recovery behaviour. Higher R values and lower J_{nr} values are favourable to the resistance of the binder to rutting.

It was acknowledged that the TOR improves properties of rubberized bitumen and increase the workability of the mixture, but the influence of TOR on other properties of rubberized bitumen, such as creep-recovery, is not clear. In this paper, the influence of TOR at different concentration of crumb rubber modified bitumen (CRMB) on the creep-recovery behavior is studied. The Multiple Stress Creep Recovery (MSCR) test performed by Dynamic Shear Rheometer (DSR) was used to evaluate the effects of TOR on the CRMB.

2. MATERIALS AND METHODS

2.1 Bitumen

Bitumen grade 80/100 penetration obtained from the vacuum distillation residue derived from crude oil is widely used in Malaysian road construction. In this study, bitumen 80/100 was obtained from the Asphalt Technology Sdn. Bhd. located at Port Klang, Malaysia. Table 1 shows the specification of the bitumen 80/100 penetration used in this study.

2.2 Crumb Rubber

Crumb rubber sized 0.4 mm (40#) obtained through the ambient grinding process of truck tyres was used to produce CRMB for this study (see Figure 1). The specification

of the crumb rubber is presented in Table 2. One batch of crumb rubber bought from RUBPLAST Sdn. Bhd. was used throughout this study to ensure the similar properties of crumb rubber utilised.

2.3 Trans-Polyoctenamer

Trans-polyoctenamer (TOR) with trademark Vestenamer® 8012 (Figure 2) bought from Evonik Degussa GmbH, Germany was chosen as the crosslink dispersant agent. The specification of Tran-polyoctenamer is shown in Table 3.

Table 1. Specification of Bitumen 80/100 Penetration used in this Study

Property	Unit	Test Method	Value	
			Min	Max
Penetration at 25°C	0.1 mm	ASTM D5	80	100
Softening Point (Ring & Ball)	°C	ASTM D36	45	52
Flash Point (Cleveland Open Cup)	°C	ASTM D92	225	-
Relative Density at 25°C	g/cm ³	ASTM D71	1.00	1.05
Ductility at 25°C	cm	ASTM D113	100	-
Loss on heating, wt.	%.	ASTM D6	-	0.5
Solubility in trichloroethylene, wt., min.	%	ASTM D2042	99	-
Drop in penetration after heating, max.	%	ASTM D5	-	20
Application temperatures, mixing at	°C	-	140	165

Table 2. Specification of Crumb Rubber used in this Study

Property	Unit	Test Method	Value
Acetone Extract	%	ISO 1407	10 ± 3
Ash Content	%	ISO	8 ± 3
Carbon Black	%	ISO 1408	30 ± 5
Rubber Hydrocarbon	%	RHC	52 ± 5
Passing	%	ASTM D5644	>90
Heat Loss	%	ASTM D1509	<1
Metal Content	%	ASTM D56),	<1
Fiber Content	%	ASTM D5603	<3

Table 3. Specification of Trans-polyoctenamer used in this Study

Property		Test method	Unit	Value
Molecular weight M_w	GPC	DIN 55672-1	-	90,000
Glass transition temperature	T _g	ISO 11357	°C	-65
Crystallinity	23°C	ISO 11357	%	30
Melting point	DSC	ISO 11357	°C	54
	2 nd heating			
Apparent density	23°C	ISO 60	g/l	560
Density	23°C	ISO 1183	g/cm ³	0.91
Tensile test		ISO 527-1/-2		
Stress at yield			MPa	7.5
Strain at yield			%	25
Strain at break			%	400
Tensile impact strength		ISO 8256		
	23°C		kJ/m ²	165
	0°C		kJ/m ²	190
	-20°C		kJ/m ²	240

**Fig.1.** Rubber Crumb used in this Study



Fig.2. Trans-polyoctenamer used in this Study

2.4 Preparation of Modified Binder

The bitumen 80/100 penetration was heated in the oven until fully melted at temperature 180°C for about 3 hours. Three different percentages of crumb rubber content, namely, 4, 8 and 12% were utilized and blended with the bitumen 80/100 penetration to produce different concentrations of CRMB. The crumb rubber modified binder with TOR (CRMB-TOR) was prepared by adding 4.5% TOR to each CRMB blend. Both modified bitumen's (CRMB and CRMB-TOR) were prepared using a propeller mixer at a speed of 200 rpm for 1 hour and the mixing temperature was maintained at 180°C. Table 4 presents the binder abbreviation with the matrix for the seven types of binder that were developed and evaluated in this study.

Table 4. Matrix of Binders Developed

Base Bitumen 80/100 Penetration Binder Abbreviation	Rubber Content, %	Trans- polyoctenamer (TOR), %		Binder group		
		0	4.5	Control	CRMB ^a	CRMB- TOR ^b
OR	0	✓		✓		
C4R	4	✓			✓	
C8R	8	✓			✓	
C12R	12	✓			✓	
C4RT	4		✓			✓
C8RT	8		✓			✓
C12RT	12		✓			✓

^a Crumb rubber modified binder

^b Crumb rubber modified binder added with Transpolyoctenamer

2.5 Multiple Stress Creep Recovery (MSCR) Test

The multiple stress creep recovery (MSCR) was measured using a dynamic shear rheometer (see Figure 3) under controlled strain conditions. The plate used for MSCR was 25 mm in diameter and in order to improve the repeatability of testing the gap between the parallel plates was selected at 2 mm [22, 23]. The test was conducted by applying 1 s creep loading with 9 s recovery over the various stress levels (25, 50, 100, 200, 400, 800, 1600, 3200, 6400, 12,800 and 25,600 Pa) [24]. Each stress level was repeated for 10 cycles. In this study, the test was performed at 40°C.

The creep parameters, namely, non-recoverable compliance (J_{nr}) and per cent recoveries (% R) were then calculated and analysed. The non-recoverable compliance (J_{nr}) was calculated by dividing the average non-recovered strain for the 10 creep and recovery cycles with the applied stress for those cycles. The J_{nr} equation is shown in Equation 3.1:

$$J_{nr} (Pa^{-1}, kPa^{-1}) = \frac{\epsilon_R - \epsilon_0}{\sigma (Pa, kPa)} \quad (1)$$

The per cent recoveries, R , were calculated by means of the ratio of the per cent recovery of the binder at 1/9 s to the one at 2/18 s. Equation 3.2 was used for calculation of the per cent recoveries, % R :

$$\% R = \frac{[(\epsilon_c - \epsilon_0) - (\epsilon_R - \epsilon_0)]}{\epsilon_c - \epsilon_0} \times 100 \quad (2)$$

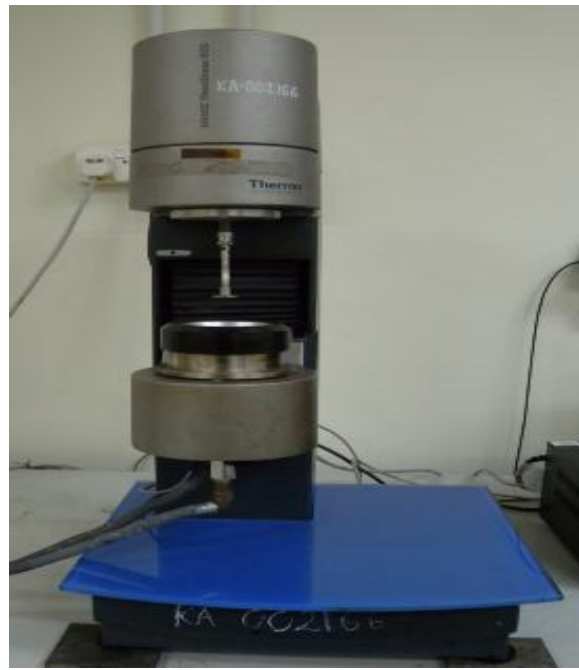


Fig.3. Dynamic Shear Rheometer

3. RESULTS AND DISCUSSION

3.1 Effects of Shear Stress on Non-Recoverable Creep Compliance (J_{nr})

Figure 4 shows a plot of the J_{nr} versus the applied stress for all the binders at the test temperature of 40°C. All the binders show consistent compliance at stress levels between 25 Pa and 6,000 Pa. However, when the stress level was raised higher than 6,000 Pa, the J_{nr} for all binders except 0R, C12R and C12RT, increased significantly and differences in the bitumen response (inflection point) were obvious. This indicates that at severe stress levels (>6,000 Pa), the binders show a wider spectrum of behaviour, as indicated by the apparent difference of J_{nr} . Therefore, by using the higher levels of stress in the MSCR test, the response of the binder captures the stiffening effects.

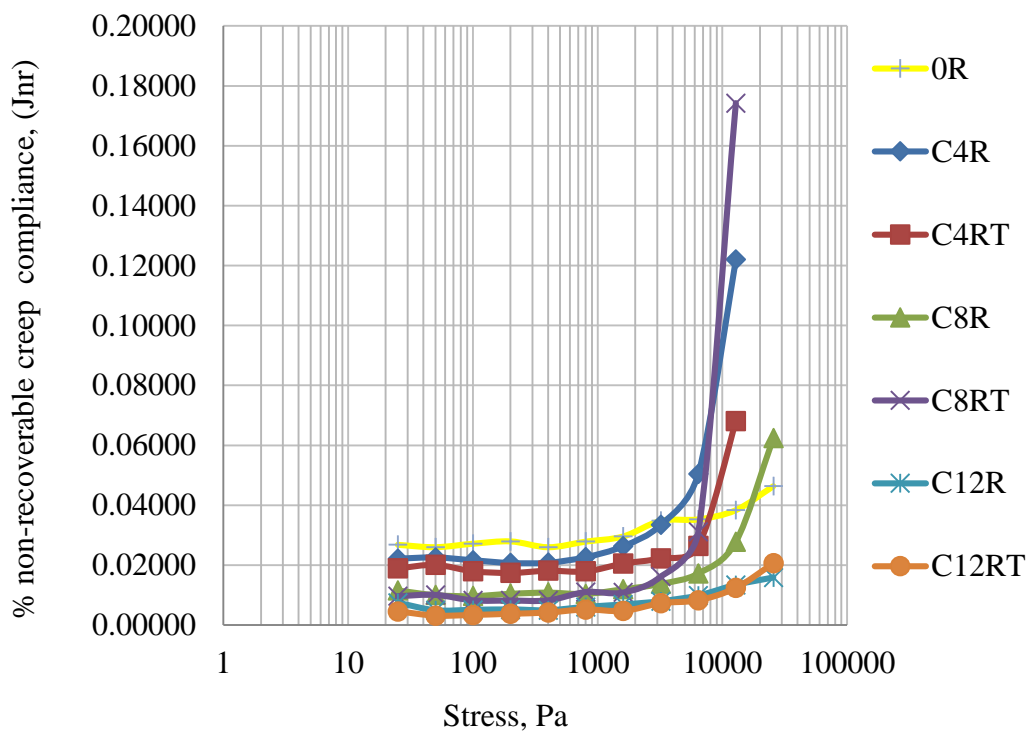


Fig.4. Comparisons of J_{nr} Values for Binders at 40°C

Moreover, binders that show a significant increase in J_{nr} as the stress level increases to 6,000 Pa, indicate that such binders are highly susceptible to rutting when high traffic loadings are applied to the pavement surface. Similar results observed by Lemarchand *et al.* (2015) [25]. These results suggest that at low shear rates until a critical shear rate, the molecular order parameter is roughly constant and non-zero; however, as the shear rate increases above the critical value, the molecular order parameter begins to increase with the shear rate [25]. Nevertheless, 0R, 12R and 12RT violated this pattern, i.e. they

observed consistent J_{nr} at all stress levels. It is possible to say that these effects on the J_{nr} values are, to some extent, related to the amount of the crumb rubber and TOR in the binder formulation. In other words, the recoverable strain of the binder is affected by the stiffness of the base binder, the volume of the crumb rubber and TOR, and the extent of the chemical reaction in the binder.

The use of different stress levels in the MSCR test is to perform the MSCR test with many stress levels (from low to severe) to capture the nonlinear behaviour of the binder and relate that behaviour to rutting in the bituminous mixtures. From Figure 5, it can be seen that in order to study the behaviour of the binders in the non-linear range, stress levels higher than 6,000 Pa seem to be needed for crumb rubber modified binder. It can be seen that at a stress level of 6,000 Pa, the J_{nr} increased sharply; indicates that the resistance to deformation of the binder starts to decrease. This sharp change in non-recoverable creep compliance can be an indicator that the binder is in the nonlinear region.

Another objective to use the different stress levels in the MSCR test is to better simulate the actual traffic conditions that can be observed in the field, as we know that the vehicles do not travel at the same speed and do not apply equivalent loads on the pavement structure. As has been noted in Figure 4, the results support the concern of many highway authorities that overloaded vehicles can rapidly accelerate the rutting potential. [24].

Moreover, the test can also be used to rank the binders based on the stress levels. For instance, 12RT is the most resistant to deformation followed by 12R, as an increase in the stress levels has a relatively minor effect on the J_{nr} value. From the MSCR results, the rank of the binder from most resistant to deformation, to the worst at stress levels below 6,000 Pa is as follows: 12RT, 12R, 8RT, 8R, C4RT, C4R and 0R. However, increasing the stress level to 6,000 Pa affects the ranking of the binders (C12RT is still the best followed by C12R at all stress levels). This indicates that a high rubber concentration offers better rutting resistance. Moreover, it can also be mentioned that crumb rubber and TOR are sensitive to the severe stress level as the ranking of the binder was different, as observed by the intermediate (<6,000 Pa) and high (>6,000 Pa) stress level. This higher nonlinearity in this CRMB and CRMB-TOR binder might lead to poor performance at high stress levels in the pavement; therefore, stress sensitivity should be taken into account in selecting the amount of crumb rubber in the modification of CRMB and CRMB-TOR.

3.2 MSCR Test Results at 100 Pa and 3200 Pa

The MSCR test procedure was specified as in *ASTM D7405 Standard Test Method for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binder Using a Dynamic Shear Rheometer* [21]. The standard specifies the test conditions: 1-s creep time, 9-s recovery time and 10 creep-recovery cycles at stress levels 100 Pa and 3200Pa. Therefore, it is necessary to discuss the MSCR results at 100 Pa and 3200 Pa.

3.2.1 Actual Strain Curve

Figures 4.8 (a) – 4.8 (b) show the actual strain for all the binders at a shear stress of 100 Pa and 3200 Pa, respectively. Both figures depict the data results at a 1 s creep loading with 9 s recovery phase. During loading, the results show that the actual strain level increases. However, the strain decreases in the recovery phase. The overall test results display a reflection of the visco-elastic-plastic property of the binders [20].

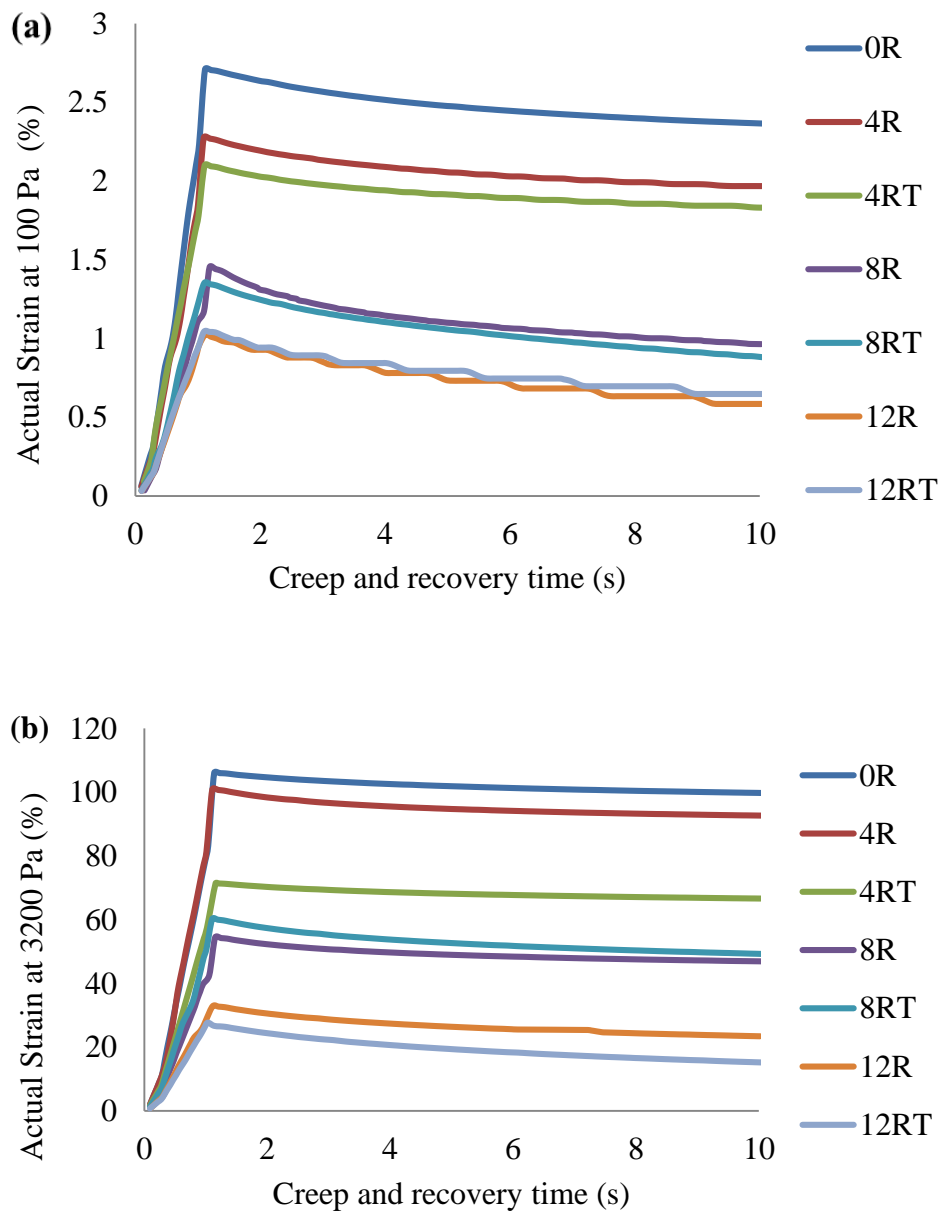


Fig.5. Actual Strains of the Binders at 40°C: (a) 100 Pa and (b) 3200 Pa

Comparing Figure 5(a) with Figure 5(b), the strain was recovered immediately at low stress level (100 Pa); whereas at the high stress level (3200 Pa) the strain was recovered gradually. This was as expected, as the strain recovered faster at the low stress level (100 Pa) compared to the higher stress level (3200 Pa). At the low stress level, the sample was creeping less than that of the sample creeping at the high stress level; thus, when stress is removed during the recovery phase the strain at low stress level (100 Pa) recovered faster than that at the high stress level (3200 Pa). Analysis of the per cent recovery (Figure 7) justifies the above explanation.

Moreover, it was as expected that the maximum strain level of the sample creeping at 3200 Pa was much higher than that of the samples creeping at 100 Pa. Taking the control binder (OR), for instance, the maximum strain levels were 2.71% and 106.1% at 100 Pa and 3200 Pa, respectively; an increase of about 39 times (see Figure 5). This shows that the maximum strain level increases with an increase in the creep level. This finding is similar to other research studies [20].

3.2.2 Non-Recoverable Compliance (J_{nr}) and per cent Recovery (%R)

Figure 6 and Figure 7 show the non-recoverable compliance (J_{nr}) and per cent recovery (% R), respectively. Both figures were plotted based on the MSCR results at 100 Pa and 3200 Pa. The results indicate that a higher creep loading level (3200 Pa) resulted in a higher non-recoverable compliance and lower per cent recovery. This means that high stress level leads to higher permanent deformation to the binder with low ability for recovery. Take control sample (OR), for instance, the increase in the stress level from 100 Pa to 3200 Pa caused the J_{nr} to increase 1.28 times (from 0.027% to 0.035%) and the % R to decrease 2.51 times (12.920% to 5.151%).

Moreover, a comparison of Figure 6 with Figure 7 shows the close relationship between the non-recoverable compliance and the per cent recovery: high non-recoverable compliance pairs with low per cent recovery. This finding is in agreement with Adorjányi, K., and Füleki, P. (2011) [26] who found good correlation ($R^2 = 0.934-0.941$) between non-recoverable compliance and recoverable strain for polymer modified bitumen. This indicates that the binder elasticity has an enormous effect on binder rut resistance behaviour. It was expected that the binder with higher elasticity shows less non-recoverable deformation. As the elasticity of the binder is increasing, the binder can recover a greater amount of deformation, which will result in less permanent deformation. Therefore, a binder with higher elasticity is more visco-elastic, and, thus, shows a lower non-recoverable compliance value (J_{nr}) and more rut resistance.

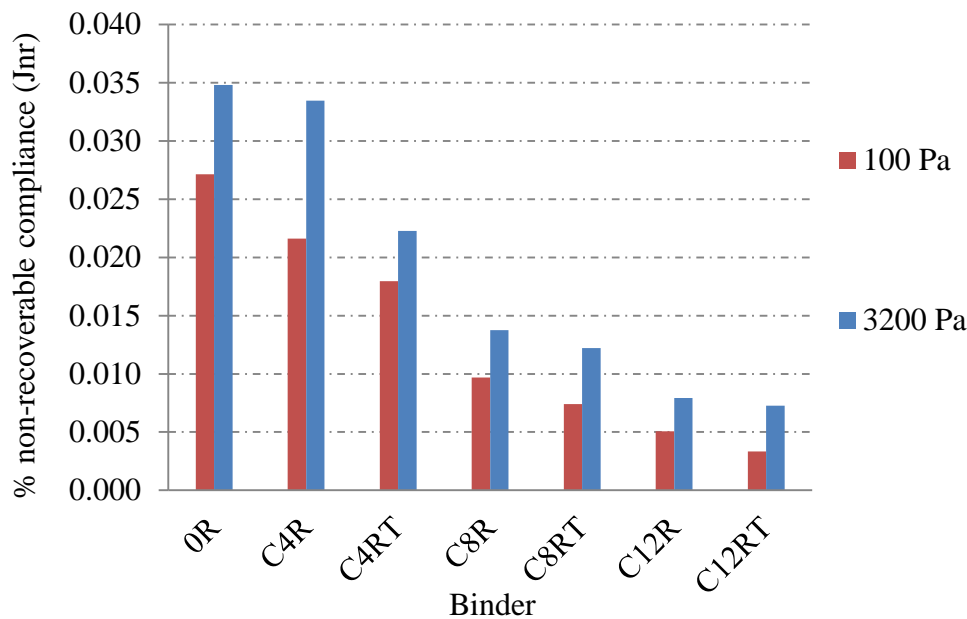


Fig.6. Non-Recoverable Compliance of Binders at 100 Pa and 3200 Pa

In terms of the effect of crumb rubber on the MSCR test results, it was determined that all of the CRMB binders had a lower J_{nr} compared to the control binder (0R) (see Figure 6). Moreover, adding TOR to CRMB (CRMB-TOR) seems to further decrease the J_{nr} compared with CRMB binders. For instance at a creep loading of 100 Pa, the J_{nr} of 4R, 8R and 12R were 20.37%, 64.30% and 81.38% lower than that of the control binder, respectively. For the crumb rubber binder with TOR (CRMB-TOR) the J_{nr} for 4RT, 8RT and 12RT decreased 33.88%, 69.6% and 87.70% compared to the control binder, respectively. Overall, an increase in the rubber content causes the J_{nr} to show a decreasing trend.

Moreover, adding TOR to the crumb rubber binder seems to decrease the J_{nr} more significantly compared to the control binder. A similar pattern was observed for both stress levels (100 Pa and 3200 Pa). It is possible to attribute these results to the chemical reaction among the base binder, crumb rubber and TOR. Therefore, it can be concluded that the crumb rubber and TOR improve the high temperature stability of the modified binders and that it would be expected that these binders would offer better rutting resistance in the field.

In terms of the per cent recovery ($\%R$), the addition of crumb rubber and TOR led to an increase in the elastic response (Figure 7). This indicates that the presence of crumb rubber and TOR was responsible for the reduction in the amount of unrecovered strain of the binder at typical high pavement temperature (40°C).

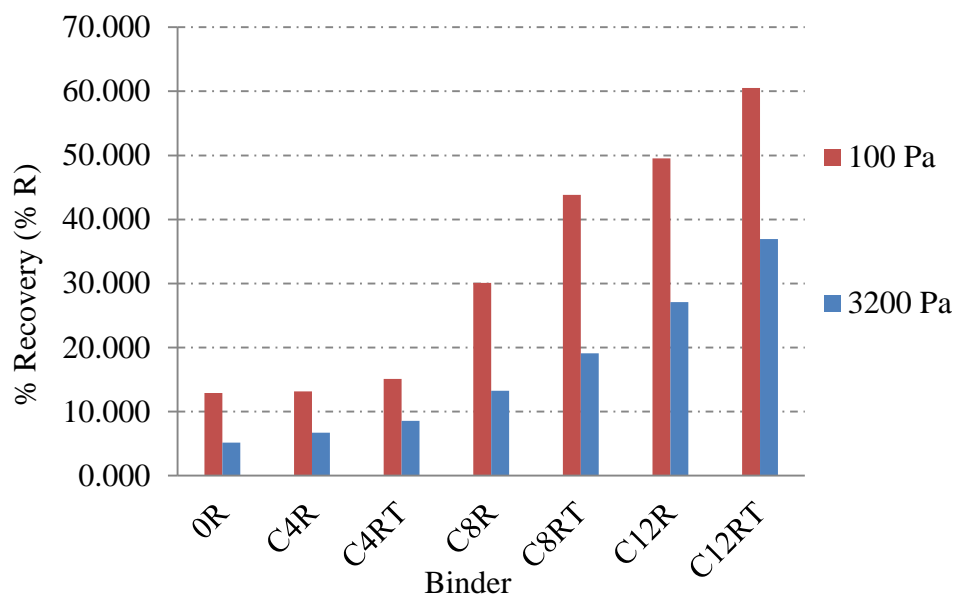


Fig.7. per cent Recovery of Binders at 100 Pa and 3200 Pa

5. CONCLUSIONS

Based on the analysis of results on the creep-recovery behaviour of CRMB and CRMB-TOR the following conclusions are drawn:

1. The addition of CR to the base binder (bitumen 80/100 penetration) reduced the non-recoverable creep compliance (J_{nr}) and increased the per cent recovery (%R). Furthermore, the creep-recovery behaviour of CRMB was significantly affected by the addition of TOR. The results obtained showed that the rheology of CRMB-TOR was improved by adding TOR to all percentages of crumb rubber, as shown by the low non-recoverable compliance (J_{nr}) and high the per cent recovery (%R). This indicated that the TOR improved the high temperature stability of CRMB. Therefore, it can be concluded that the bituminous mixtures prepared with CRMB-TOR would be less susceptible to the appearance of rutting compared with the mixtures prepared with CRMB without TOR. However, the amount of crumb rubber and TOR should be studied intensively since it's significantly influence the chemical reaction between them.
2. MSCR test performed with different stress levels can be used as a new method to evaluate the high temperature performance of modified binder. The test is relevant to study the nonlinear response of the binder and relate that response to rutting in the bituminous mixtures. As shown in this study, at stress level less than 6,000 Pa, the J_{nr} for all binders are consistent. On the other hand, at severe stress level (>6,000 Pa), the J_{nr} for all binders except 0R, C12R and C12RT, increased significantly. If only binder is considered, obtained results indicate that at stress level higher than 6,000 Pa would cause severe rutting deformation of bituminous mixtures.

Moreover, different stress levels performed in the MSCR test can be used to present the actual traffic conditions in the field. The study observed that high stress levels increase the J_{nr} value significantly; therefore this study confirmed that overloaded vehicles can rapidly accelerate the rutting potential. In addition, the test can also be used to rank the binders based on the stress levels.

3. The actual strain of the binder was recovered immediately at low stress level; whereas at the high stress level the strain was recovered gradually. The results indicate that at low stress level causes the binder to creep less and recovered faster compared to high stress level. This phenomenon results in close relationship between the non-recoverable compliance and the per cent recovery: high non-recoverable compliance pairs with low per cent recovery.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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