

Dynamic and Creep Properties of Binders Modified with Waste Tyre Rubber

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ABSTRACT: A dynamic shear rheometer was used to investigate the effect of Crumb Rubber Modifier (CRM) from used tyres on the dynamic and creep properties of bituminous binders. Modified binders were produced by blending CRM from two different origins, car and truck tyre, with bitumen under different conditions of time and temperature using a high shear mixer. Rubber-bitumen composites are considered heterogeneous (2-phase) systems of rubber particles dispersed within a bitumen matrix. Absorption of bitumen components by the rubber (swelling) inevitably depletes the bitumen of the absorbed components and modifies the properties of both the rubber and the bitumen phase. To assess the consequences, changes in dynamic properties of the composite material as well as those of the bitumen phase were investigated. Furthermore, a technique was developed to separate the two phases by filtering through a polyester cloth. Dynamic tests showed that CRM increases the stiffness and reduces the phase angle of the composite material but the extent of these changes is affected by mixing (interaction) time and temperature. They also indicated that the bitumen phase had increased stiffness and lower phase angle. The evolution of these properties with mixing time at 180 °C, however, followed the same pattern as the unmodified bitumen, which aged when heated at this temperature. Creep tests also confirmed that creep and creep recovery properties of rubber-modified binders are affected by the origin of the rubber as well as by the mixing conditions.

KEY WORDS: Bitumen, CRM, rubber-bitumen interaction, dynamic-recovery properties

1 INTRODUCTION

For many years researches and engineers have developed and used a wide range of modifying materials for bitumen to improve the mechanical properties of asphalt mixtures and, consequently, to improve pavement performance. Polymers are the main modifiers that have been shown to lead to improved properties. Four generic classes of polymers have been widely used as asphalt modifiers namely, rubbers, thermoplastic elastomers, thermoplastic polymers and thermosetting resins (Collins et al., 1991).

Rubbers can be incorporated in bitumen in either unvulcanized or vulcanized state. Vulcanization is the process of chemical crosslinking of the rubber polymer chains. Waste tyres are the main source of vulcanized rubber for bitumen modification. To incorporate tyre rubber into bitumen, the tyres are processed into crumb rubber by means of grinding and/or mechanical shearing. The use of crumb rubber as an asphalt modifier has the added environmental benefit in that it could help to solve the problem of the disposal of used tyres that are currently stockpiled or landfilled.

Two generic methods have been developed for incorporating crumb rubber in asphalt mixtures, namely the wet process and the dry process. The main difference between these two methods is that in the wet process, crumb rubber is mixed with bitumen at elevated temperatures to produce a modified binder, which is then incorporated into the aggregate blend, whereas in the dry process, ground and/or crumb rubber is blended with the aggregates before incorporating the bitumen into the mixture (Epps, 1994).

When crumb rubber is blended with bitumen at high temperatures to produce a modified binder, i.e. wet process, the materials interact. Initially, the interaction between CRM and bitumen is a non-chemical reaction where the rubber particles are swollen by absorption of the bitumen's aromatic oils to form a gel-like material (Heitzman, 1992). This interaction not only increases the effective volume of the rubber particles (swelling) but also changes the nature of the liquid phase, thus, the bitumen. As a consequence, the primary effect of CRM is to increase the viscosity of the binder at high temperature (Bahia and Davies, 1994).

The rubber particles may also suffer some form of degradation when mixing with bitumen at high temperatures. The main mechanisms of rubber degradation are devulcanization and depolymerization. Billiter et al. (1997a and 1997b) showed that by utilizing high mixing temperatures and high shear, along with extended interaction times, the rubber particles could be devulcanised and depolymerised into the bitumen to produce a binder that is both homogeneous and truly elastic. Zanzotto and Kennepohl (1996) also indicated that by applying high temperatures and shear rates, CRM could be partially devulcanised and depolymerised in the bitumen medium.

Moreover, the extent of swelling and degradation depends on the nature of the rubbers, the bitumen's chemical composition and the mixing conditions of time, temperature and degree of agitation. In addition, these processes will govern the final mechanical properties of the rubber-modified binders (Abdelrahman and Carpenter, 1999).

The aim of this study is to investigate the influence of the process variables, mixing time and mixing temperature on the dynamic and creep properties of a bitumen modified with CRM from two different origins: car and truck tyre. Furthermore, dynamic properties of binders recovered (filtered) after interaction with the rubber at 180 °C for mixing times of up to 24 h were also evaluated and compared with the unmodified and CRM-modified binders.

2 CRUMB RUBBER MODIFIED BINDERS

2.1 Materials considerations

Crumb rubber from used tyres is primarily a composite of natural and synthetic rubber. Natural rubber (NR) is obtained from the tree *Hevea Braziliensis* and it is essentially a hydrocarbon. Chemically, the rubber hydrocarbon is a polymer of isoprene built up in the form of a continuous chain. Synthetic rubbers, on the other hand, are produced by a chemical process known as polymerization. The most common synthetic rubber used in tyre manufacturing is styrene-butadiene statistical copolymer (SBR). To achieve elastomeric properties most rubbers are vulcanized. The process most commonly used is sulphur vulcanization. During tyre-rubber manufacture various additives are also incorporated. They include carbon black, pigments, e.g. zinc oxide, softeners, e.g. petroleum oils or waxes, and others (Stern, 1967). In general, truck-tyre rubber contains larger percentages of natural rubber compared to that from car tyres (Hicks et al., 1995).

Bitumen is a complex mixture of organic molecules of predominantly hydrocarbon nature that vary widely in composition according to the origin of the crude oil from which it is extracted. These molecular structures also contain varying amounts of functional groups,

predominantly, oxygen, nitrogen and sulphur, together with trace quantities of metals (Petersen, 1984). Because the number of molecules in bitumen with different chemical structures is extremely large, a complete analysis of bitumen is generally considered impractical. However, it is possible to study bitumen composition by separation into four chemical groups, namely saturates, aromatics, resins and asphaltenes (SARA). Asphaltenes are considered as highly polar and complex aromatic compounds. They are colloiddally dispersed in a continuous medium of oils (saturates and aromatics) and this dispersion is stabilized by the absorption of resins on the peripheries of the asphaltenes (Read and Whiteoak, 2003). Moreover, molecular weights of the bitumen fractions increase gradually from saturates to asphaltenes (Peramanu and Pruden, 1999; Bukka et al., 1991).

When polymer networks such as rubbers are exposed to low-molecular liquids, the liquid molecules might be absorbed within the polymer network causing it to swell. The extent of swelling depends on factors like the compatibility of the solvent and the polymer, the cross-link density in the polymer and the temperature (Treloar, 1975). The swelling of rubbers by organic solvents can be regarded as a diffusion process in which the two components are chemically neutral. The solvent molecules diffuse into the rubber network forcing the macromolecules to move apart such that the rubber swells or expands until the concentration of solvent is uniform and equilibrium swelling is reached. For rubber-bitumen systems, although most components of bitumen and rubber are hydrocarbons, low-molecular-weight components of the bitumen, i.e. aromatic oils, will diffuse into the rubber preferentially (Airey et al., 2003).

2.2 Production of crumb rubber modified binders

Crumb rubber modified binders were produced using a high-shear Silverson mixer under controlled conditions of temperature, time and shear rate. Approximate amounts of 600 g of bitumen contained in 1-litre flasks were preheated in an oven to the selected mixing temperature for about two hours. The flasks then were placed on an iso-mantle heater and covered with an insulation mantle also preheated to the required temperature. The hot bitumen was then stirred in the mixer for about 10 minutes at 1000 rpm after which the temperature of the bitumen remained constant as measured by a thermometer. Specific quantities of CRM were added gradually to the hot bitumen. Once all the CRM was added to the bitumen, the shear rate was increased up to 2000 rpm and mixing was continued for various times, during which the temperature of the binder was monitored.

The bitumen used in this study was a 100 penetration grade from the Middle East (100 Pen KSR). This bitumen was modified by adding 10 % CRM by weight of binder, from either truck or car tyres. CRM had a maximum nominal size of 600 μm (mesh 30) and was produced by ambient grinding. Mixing temperatures selected were 150, 180 and 210 $^{\circ}\text{C}$. Mixing time varied from 1 up to 6 hours, and the shear rate applied during mixing process was 2000 rpm.

2.3 Isolation of components from rubber-bitumen blends

A procedure was developed to separate the bitumen and the rubber particles from rubber-bitumen blends produced as above. The procedure consisted of filtering 50 ml of rubber-modified bitumen through a polyester cloth filter of 50 μm sieve size while hot (180 $^{\circ}\text{C}$). Following this method, it was possible to obtain samples of bitumen, after interaction with the rubber crumb, from the rubber-bitumen blends. Furthermore, bitumen samples isolated (filtered) from the rubber-bitumen blends were obtained after 1, 6, 12 and 24 hours interaction with CRM at 180 $^{\circ}\text{C}$. The base (unmodified) binder was also stirred at a rate of 1000 rpm in the Silverson mixer and at a temperature of 180 $^{\circ}\text{C}$ for up to 24 hours with the absence of any rubber modifier.

3 EXPERIMENTAL PROCEDURE

3.1 Dynamic mechanical tests

A dynamic shear rheometer (DSR) was used to determine the dynamic viscoelastic properties of unmodified and crumb rubber modified binders. Controlled-strain mode of testing was performed in the DSR by applying a sinusoidal, oscillatory strain to a bitumen sample placed between the two parallel plates of the DSR. The device measured the magnitude and phase of the resulting stress under a wide variety of temperatures and loading frequencies. The principle of a dynamic test is shown in Figure 1. Furthermore, dynamic tests were conducted within the linear viscoelastic region (LVER) of response. The fundamental viscoelastic parameters determined from dynamic tests in the DSR were the complex shear modulus, G^* , and the phase angle, δ . The complex modulus is defined as the ratio of the stress amplitude to the strain amplitude and is a measure of the overall resistance to deformation. The phase angle, on the other hand, is the phase difference between the stress and strain in an oscillatory deformation and is a measure of the viscoelastic character of a material.

Dynamic tests were performed at temperatures of 45 and 55 °C, using a 25 mm plate diameter and 1 mm gap width. Furthermore, at each temperature a frequency sweep was applied over the range 0.1 to 10 Hz, at fixed strain amplitude of 0.1 %. For brevity, however, only the data at 45 °C and 10 Hz is presented in the subsequent sections.

3.2 Creep and creep recovery tests

The DSR was also used to perform creep and creep recovery tests on selected binders. In a creep test a sample is subjected to a step in shear stress, σ , which is held constant for a specific time, t_c . The resulting shear strain, $\gamma(t)$, is then monitored by the DSR as a function of time. The material's creep behaviour is characterised in terms of its compliance, $J(t)$, which is defined as the ratio of the strain to the imposed stress. The behaviour of typical viscoelastic materials in a creep test is described by three components representing elastic (J_g), delayed elastic (J_d) and viscous response. In general, the short time behaviour is governed by the elastic terms whereas at long times the material exhibits steady viscous flow. For long times, where the steady-state viscous flow prevails, the viscosity can be estimated from the slope of the compliance curve.

Moreover, when the applied stress, σ , is removed at some time t_c elastic recoil or creep recovery takes place. The recoverable compliance, after the applied load is removed, is a measure of the elasticity in a material. This recoverable compliance can be obtained from creep or from creep recovery tests. The recoverable compliance in a creep test is calculated by extrapolating back along the creep curve when in steady viscous flow and it is denoted as J_{0C} . This method is fine for estimating the recoverable compliance but is prone to error since it generally involves extrapolating back from a large number to obtain a small number. A far better method is to measure it directly by the creep recovery test. After the steady-state viscous flow is reached and the stress is removed, the material will recoil due only to elasticity. By directly measuring this recoil, the recoverable compliance, J_{0R} , can be determined. Ideally, J_{0C} and J_{0R} should be the same but J_{0R} is, in general, more accurate. The principle of a creep and creep recovery test is shown in Figure 1.

As for dynamic tests, creep tests have to be conducted within the linear region. At a test temperature of 40 °C, a shear stress of 50 Pa was found to satisfy the requirements of linearity for all the materials investigated. Using the test geometry of 25 mm parallel plates and 1 mm gap width, creep loading was applied for 600 seconds after which the load was removed and creep recovery was measured for another 600 seconds. The following parameters were

recorded: creep compliance at 600 seconds loading (J_C) and creep recovery compliance at 600 seconds unloading (J_R), creep shear rate, creep viscosity (η), the approach to steady flow ($d\ln J/d\ln t$), the recoverable compliance measured in creep (J_{0C}) and the recoverable compliance measure in creep recovery (J_{0R}).

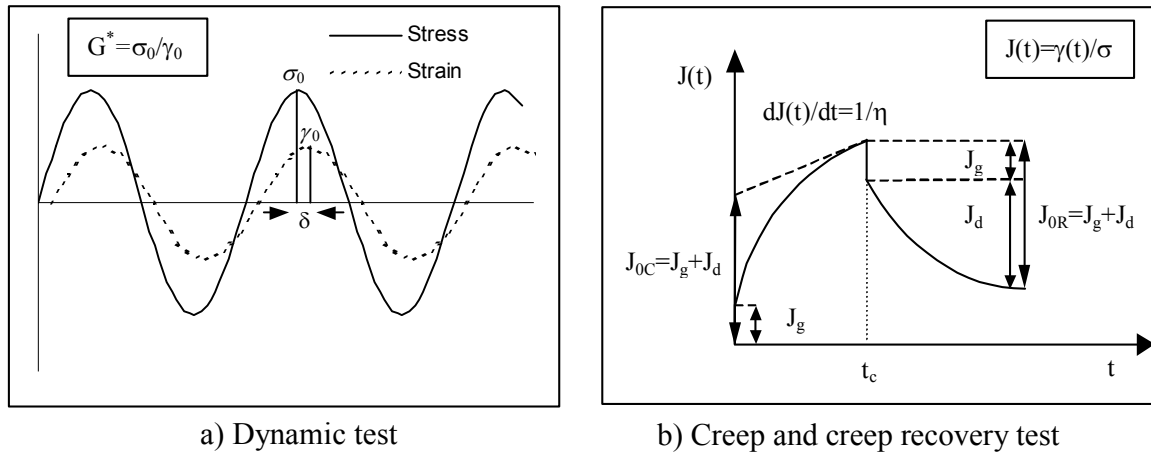


Figure 1: Dynamic and creep/creep recovery tests principles

4 RESULTS AND DISCUSSIONS

4.1 Dynamic parameters

The changes in binder properties, complex modulus, G^* , and phase angle, δ , with mixing (interaction) time from 1 up to 24 hours at 180 °C are presented in Figure 2. Data showed that CRM-modified binders exhibited higher G^* and lower δ than the base (unmodified) bitumen at all interaction times. Thus, the binders became stiffer and more elastic by the addition of CRM under the mixing conditions of time and temperature investigated.

When heating the base bitumen at 180 °C for prolonged times, G^* increased and δ decreased with time. This effect was indicative of bitumen oxidation (ageing) at high temperatures. The evolution of G^* and δ with interaction time for the CRM-modified binders was, however, more complex and depended on the source of CRM.

Bitumen modified with CRM from truck tyres attained their highest G^* after one hour mixing at 180 °C. This initial high stiffness was attributed to swelling of rubber particles, reducing the inter-particle distance, and the stiffening of the bitumen matrix by diffusion of light components into the rubber particles. Starting at a high value, G^* then decreased continuously up to about 6 hours interaction time. This decrease in G^* might suggest some degree of degradation by devulcanization and/or depolymerization of the rubber particles. For longer interaction times, G^* increased at a similar rate to the unmodified binder suggesting, thus, hardening as a result of binder oxidation. The effect of interaction time on the modification of phase angle was seen as a slight increase in δ during the first 6 hours. For longer interaction times, δ stabilised, indicating no phase angle modification.

Bitumen modified with CRM from car tyres showed different behaviour. Initially, G^* after 1 hour interaction time increased with respect to the base bitumen, as a result of CRM modification. Then, G^* continued to increase with interaction time up to about 6 hours, probably as a result of binder oxidation and/or some residual swelling. Between 6 and 12 hours a small decrease in G^* was observed, suggesting some degree of degradation of the

CRM particles from car tyres. For longer interaction times, G^* increased indicating again hardening by oxidation. The effect of interaction time on phase angle was seen as a slight decrease in δ during the first 6 hours, confirming the ageing of the modified binders. For longer interaction times, δ stabilised, indicating no phase angle modification.

Comparison of data for the binders modified by CRM from truck and car tyres showed that after one-hour mixing time, binders modified with CRM from truck tyres were stiffer than the ones modified with CRM from car tyres. This was attributed to differences in the chemical composition of the two types of rubbers.

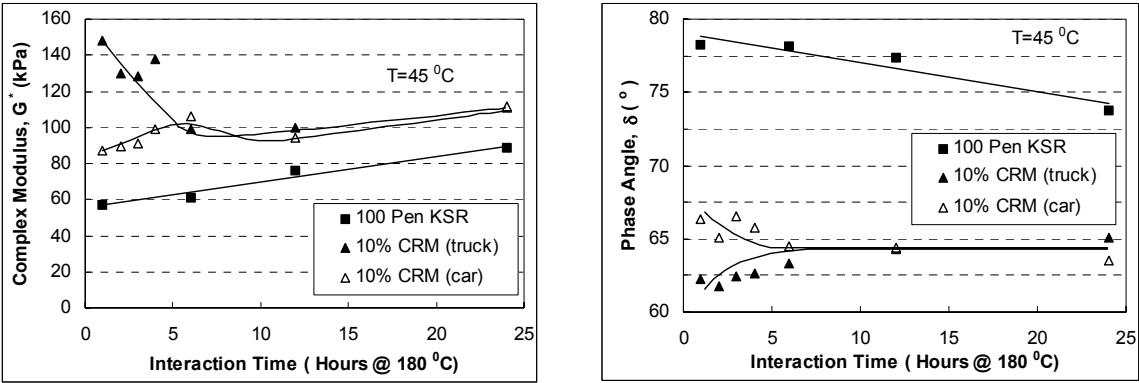


Figure 2: Evolution of G^* and δ with interaction time for 100 Pen KSR modified with 10% CRM from truck and car tyres

G^* and δ data corresponding to the unmodified bitumen and the residual binders isolated (filtered) from the rubber-bitumen blends are presented in Figure 3. Data showed that the residual binders, after interaction with the rubber particles, had increased stiffness, as seen by the higher values of G^* . Thus, it is concluded that CRM and bitumen interaction at high temperature hardens the bitumen matrix. Furthermore, this increase in stiffness is consistent with diffusion of mobile components from the bitumen into the rubber particles and responsible for the swelling of the rubber.

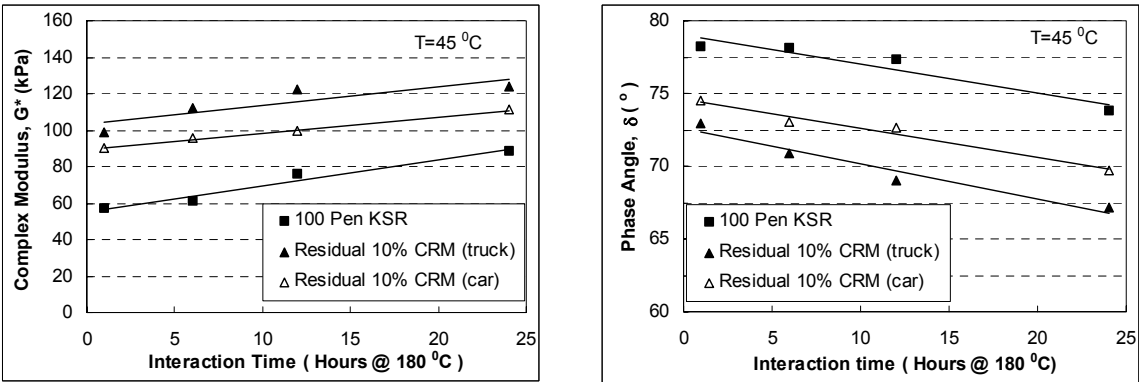


Figure 3: Evolution of G^* and δ with interaction time for residual 100 Pen KSR modified with 10% CRM from truck and car tyres

Comparison between G^* for the residual binders showed that, the stiffness of the residual binders after interaction with truck-tyre rubber was higher than those after interaction with car-tyre rubber. This was attributed to higher swelling of the truck rubber (NR) particles. Also, the increase in G^* of the residual binders followed a similar trend to that of the base

bitumen. This suggests that diffusion of bitumen components into the rubber and the consequent swelling occurred during the early stages of the interaction process, i.e. the first hour of interaction at 180 °C. The residual binders had lower phase angles than the unmodified bitumen, indicating a more elastic response of the former binders. Also, the phase angles for the residual binders after interaction with truck-tyre rubber were lower than those after interaction with car-tyre rubber. This was attributed, as for G^* , to differences in the absorption of bitumen into the different types of rubber.

The changes in G^* and δ , with interaction time from 1 to 6 hours and at interaction temperatures of 150, 180 and 210 °C are presented in Figure 4. It can be seen that for 150 °C interaction temperature, G^* practically did not change, though a small increase in G^* was observed after 6 hours. The slight increase in G^* was accompanied by a reduction in δ , suggesting a small degree of oxidation of the binders. For the 180 °C interaction temperature, G^* and δ showed differences between the binders modified with CRM from truck and from car tyres, as explained earlier. This different behaviour might be attributed to differences in the oxidative stabilities of the two types of rubber at high temperatures, car-tyre rubber (SBR) being more stable than truck-tyre rubber (NR). At the 210 °C interaction temperature, G^* decreased while δ increased with interaction time, indicating continuous partial degradation of both types of rubber during mixing with bitumen at this high temperature.

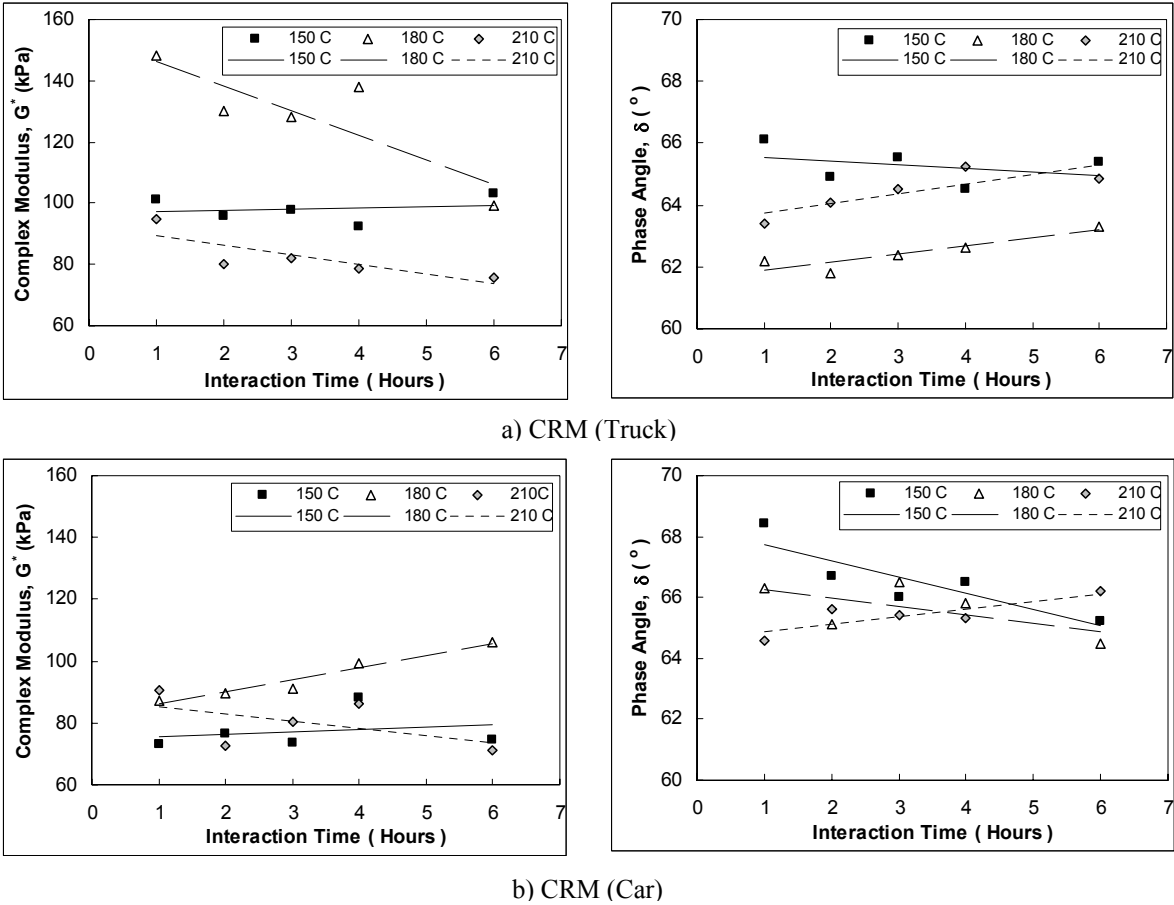


Figure 4: Evolution of G^* and δ with interaction time at different interaction temperatures for 100 Pen KSR modified with 10 % CRM from truck and car tyres

4.2 Creep and creep recovery parameters

Creep and creep recovery results for the 100 Pen KSR and the CRM-modified binders are presented in Table 1. Creep and creep recovery curves are also given in Figure 5. It can be seen first that the steady-state viscous flow was reached within 600 seconds, considering a 5 % tolerance on the steady state condition, as seen by the values of $d\ln(J)/d\ln(t)$ which varied between 0.95 and 1 for all the materials investigated; see Table 1. Viscosity values obtained from the slopes of the creep curves, once the steady state was reached, showed that the addition of CRM increased the viscosity of the binders. Furthermore, shear rates decreased, as expected, as the viscosity of the binders increased on addition of CRM.

Table 1: Creep and recovery data for 100 Pen KSR modified with 10 % CRM from truck and car tyres

CRM source	Mixing temp. ($^{\circ}\text{C}$)	Mixing time (h)	Steady flow ($d\ln J/d\ln t$)	Viscosity (kPa.s)	Shear rate $\times 10^{-3}$ (s^{-1})	J_C (1/kPa)	J_{0C} (1/kPa)	J_R (1/kPa)	J_{OR} (1/kPa)	Recovery (%)
Neat binder			1.03	6.13	8.16	94.86		94.07	0.79	0.83
Truck	150	1	0.95	11.94	4.19	52.89	0.27	50.37	2.51	4.75
Truck	150	6	0.94	14.26	3.57	44.96	0.28	42.88	2.09	4.64
Truck	180	1	0.96	39.27	1.27	15.85	0.64	14.24	1.60	10.12
Truck	180	6				20.46		18.30		10.57
Truck	210	1	0.95	37.88	1.32	16.68	0.92	14.66	2.01	12.07
Truck	210	6	0.97	15.63	3.20	39.55	0.13	37.05	2.50	6.32
Car	150	1	0.95	13.89	3.80	48.46	0.26	45.97	2.49	5.14
Car	150	6	0.97	13.33	3.84	47.52	0.16	45.30	2.22	4.66
Car	180	1	0.98	18.42	2.07	33.15	0.51	30.75	2.40	7.25
Car	180	6	0.98	18.94	2.64	32.44	0.69	30.24	2.20	6.79
Car	210	1	0.95	23.31	2.14	27.15	0.14	24.69	2.45	9.03
Car	210	6	0.96	20.89	2.39	29.99	0.14	27.83	2.16	7.21

Creep and creep recovery curves showed that creep compliances at 600 seconds loading (J_C) for the CRM-modified binders produced at 150 $^{\circ}\text{C}$ interaction temperature were the highest. Furthermore, J_C decreased when the mixing time was increased from 1 to 6 hours, as was also observed during oscillatory testing. CRM-modified binders produced at 180 $^{\circ}\text{C}$ showed different behaviour depending on the type of CRM. The binder modified with CRM from truck tyres exhibited an increase in J_C with increasing mixing time from 1 to 6 hours, whilst the same binder modified with CRM from car tyres did not show any notable differences. Modified binders produced at 210 $^{\circ}\text{C}$ showed that J_C increased with mixing time when the base binder was modified by CRM from both truck and car tyres, thus, indicating a decrease in stiffness for longer interaction times at this high temperature

Recoverable compliances calculated from creep tests, J_{0C} , from recovery tests, J_{OR} , and creep recovery values (%) obtained from creep recovery tests are presented in Table 1. The first point to notice is that the unmodified bitumen showed practically no recovery. CRM-modified binders, however, showed between 4 and 12 % recovery depending on CRM origin and production conditions of time and temperature.

In general, binders modified with CRM from car tyres showed less recovery than those modified with CRM from truck tyres, when produced under the same conditions of temperature and time. This indicates the greater elasticity of the latter binders. The effect of interaction temperature on recovery is seen as an increase in recovery with increasing interaction temperature when comparing binders after mixing for one hour. The effect of interaction time on recovery is, however, dependent on the interaction temperature. For the

binders investigated, recovery decreased after 6 hours interaction at the highest interaction temperature, 210 °C. At interaction temperatures of 150 and 180 °C, the modified binders did not show much change in recovery with interaction time.

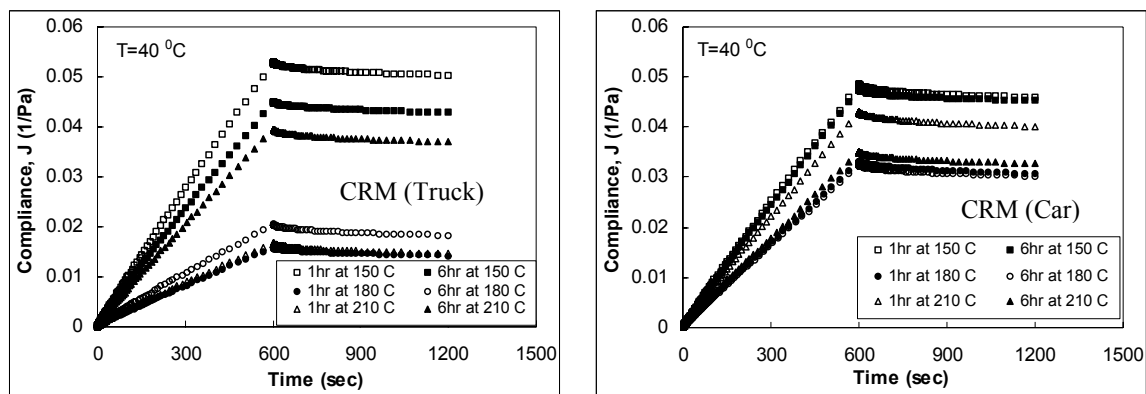


Figure 5: Creep and recovery curves for 100 Pen KSR modified with 10 % CRM from truck and car tyres

5 CONCLUSIONS

From dynamic and creep/creep recovery tests performed in the DSR, the following conclusions can be drawn:

- Interaction of CRM with bitumen stiffened the bitumen phase, i.e. increased the complex stiffness modulus, G^* , and lowered and phase angle, δ . Furthermore, truck-tyre rubber hardened the bitumen matrix appreciably more than car-tyre rubber. This hardening effect, due to absorption of bitumen components by the rubbers, occurred during the early stages of interaction (within an hour).
- The evolution of the viscoelastic properties with interaction time at different interaction temperatures depended on the origin of the CRM, thus, on the chemical composition of the rubbers.
- Creep and creep recovery tests showed that steady-state viscous flow was attained within the applied loading time of 600 seconds, with a 5 % tolerance.
- Creep recovery values indicated that the unmodified bitumen showed practically no recovery. CRM-modified binders, however, showed between 4 and 12 % recovery depending on CRM origin and mixing conditions of time and temperature.
- Binders modified with CRM from truck tyres exhibited more recovery than those modified with CRM from car tyres, when they were produced at 180 and 210 °C
- For one hour interaction time, recovery increased when the interaction temperature was increased. The effect of interaction time on recovery is, however, dependent on interaction temperature.

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