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Modelling rheological characteristics of rejuvenated aged bitumen

Lidija Ržek^a, Marjan Tušar^{a,b} and Lidija Slemenik Perše^c

^aSlovenian National Building and Civil Engineering Institute, Ljubljana, Slovenia; ^bNational Institute of Chemistry, Ljubljana, Slovenia; ^cFaculty of Mechanical Engineering, Ljubljana, Slovenia

ABSTRACT

The present study reports on rheological properties of rejuvenated non-aged and aged bitumen samples with a pyrolytic product (PP) as a rejuvenator. Various experimental and numerical methods were used in order to investigate the addition of various concentrations of PP to aged and non-aged bitumen samples. It was confirmed that the application of PP as a rejuvenator to the aged bitumen has been successful. The main focus of the study was on the use of several mathematical and mechanical models, which would successfully describe and predict experimental results. Precise calculations for the complex modulus were obtained by generalised five-parameter Sigmoidal model, while the Al-Qadi model enabled good predictions for the phase angle estimations. However, better predictions of rheological parameters were obtained for non-aged bitumen. The results confirmed the possibility of using the prediction models for the estimation of various rheological properties of aged and non-aged bitumen samples with the addition of rejuvenator.

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Introduction

The most common surface layer on European roads is asphalt, which consists of two main components: a binder (usually a bitumen) and an aggregate. Since both components can be reused, recycling of the asphalt presents one of the most important environmental and resource saving possibilities (Kandhal *et al.* 1995, Shah *et al.* 2007). Recycling preserves nature because reclaimed asphalt (RA), which would otherwise become useless waste, is reused.

Complexity of asphalt recycling process should not be underestimated. Even though the bitumen represents only a small proportion of asphalt (around 5%), it plays a crucial role in the behaviour of asphalt. The ageing of the bitumen during asphalt application is divided in two stages: (i) short-term ageing, which begins already during mixing and asphalt paving procedure, and (ii) long-term ageing, which progresses through the lifetime of the roads (Lu and Isacsson 2001, Saoula *et al.* 2013, Hofko *et al.* 2015). The result of the ageing process is increased hardness and brittleness of the bitumen, which leads to asphalt damages. In order to enable the reuse of the RA, the properties of the aged bitumen have to be restored by using special additives called rejuvenators, also referred to as recycling agents. Rejuvenators or recycling agents have the ability to soften and revive the aged bitumen of RA; however, at the same time, they have to be completely compatible with the bitumen. The positive effects of the addition of rejuvenators have been already reported in several studies, which confirmed that after the addition of rejuvenator, the aged bitumen becomes less viscous, more ductile and its coating properties are restored (Shen *et al.* 2007, Brownridge 2010, Grilli *et al.* 2013, Kaseer *et al.* 2018). The rejuvenators improve the performance of the asphalt and increase the recycling ratio (Kaseer

et al. 2018, Ržek *et al.* 2020). Moreover, they can partially restore or reduce the stiffness and embrittlement of the recycled materials and enable equivalent or even better rheological properties and cracking resistance (Yin *et al.* 2017). Years of experiences with RA applications enabled high recycling ratios; up to 100% (Zaumanis *et al.* 2014), but usually the ratios between 25% and 40% are used (Kaseer *et al.* 2019). In the present study, a new rejuvenator, prepared from pyrolytic product, which was obtained from the slow pyrolysis of scrap tires (Avsenik *et al.* 2016), was used in order to improve the rheological properties of RA.

Traditional concept of the bitumen as a colloidal system describes its rheological properties from Newtonian to non-Newtonian. Presently, the best approach to describe rheological behaviour of the bitumen is to use oscillatory, rotational, creep and recovery tests. However, these tests require expensive laboratory equipment and are time-consuming. Hence, for representing linear viscoelastic behaviour of the bitumen, mathematical and mechanical models could be used. In mathematical (phenomenological or constitutive) approach, the mathematical formulation is fitted to experimental data. The quality of adjustment is the sole criterion of the choice of formulation. So-called predictive models are valuable alternative for evaluating rheological behaviour of the bitumen. Rheological parameters are estimated by constitutive equations to an accuracy that is acceptable for most purposes. The mechanical (or analogical) approach is based on the fact that linear viscoelastic behaviour could be described by a combination of spring and dashpot mechanical models, resulting in particular mathematical equations. A good rheological model should be able to describe linear viscoelastic functions of studied materials as completely as possible (Yusoff *et al.* 2011, Yusoff 2012, Behzadfar and Hatzikiriakos 2013).

In the present study, the mathematical models were used to predict the results of oscillatory and rotational rheological tests. Creep and recovery tests were simulated by using the mechanical approach. The purpose of the study was to demonstrate that mathematical and mechanical modelling can be used to predict the behaviour of rejuvenated non-aged and aged bitumen samples. Existing studies assess the possibility of using rheological modelling for bitumen (non-aged and aged) from different origins. However, in our study the modelling was used also to evaluate the impact of different concentrations of rejuvenator on bitumen.

In addition to complex modulus and phase shift angle, zero shear viscosity (ZSV) of the bitumen was investigated. ZSV or the viscosity at zero shear rate is an indicator of the bitumen performance (Vissscher *et al.* 2004) and can be determined using various rheological tests like rotational tests (flow curve), oscillatory tests (frequency sweeps) and creep tests.

Materials and methods

Material samples

50/70 penetration grade bitumen (B50/70) from Hungarian manufacturer (MOL) was used as a reference material and as a matrix for all the blends, used in the study.

Pyrolytic product (designated as PP) was obtained by slow pyrolysis of scrap car tires as high temperature condensate, which was afterwards modified by tire crumbs. The PP from a tire pyrolysis may contain high concentrations of polycyclic aromatic hydrocarbons (PAH) (Martinez *et al.* 2013). In order to ensure minimum PAH concentration, pyrolysis process was performed at relative low temperature, i.e. the highest treatment temperature did not exceed 500°C. More details about PP production were explained in the previous study (Avsenik *et al.* 2016).

The main purpose of the PP studied was rejuvenation of bitumen. Blends containing different concentrations of PP (3%, 5%, 10% and 20%) were therefore prepared in the laboratory by adding a controlled quantity of PP to non-aged and aged bitumen samples (Table 1). The upper value of added rejuvenator was limited to 20% due to possible excess of PAH in rejuvenator. Blending process involved heating of both components to 130°C for 60 min. Two components were afterwards stirred for approximately 5 min in order to produce uniform bitumen blend. After blending and mixing, no visual evidence of any non-homogeneity or separation of the components, i.e. bitumen and rejuvenator, was

observed, therefore, the obtained mixtures were considered uniform.

The same tests were conducted on non-aged bitumen and also on the aged bitumen samples. All bitumen samples were RTFOT aged for 75 min at temperature 163°C, as determined in EN 12607-1. PAV aging of the samples was performed for 20 h under the pressure of 2.1 MPa at 100°C, as described in EN 14769.

Test methods

Samples were characterised according to the standard test methods: penetration at 25°C according to EN 1426, softening point according to EN 1427, Fraass breaking point according to EN 12593 and storage stability according to EN 13399.

Viscoelastic measurements were conducted in the temperature range from 20°C to 80°C by using dynamic shear rheometer (MCR 302, Anton Paar) with parallel plate geometry sensor systems (PP08/2 mm at 20°C and 30°C; and PP25/1 mm at 40°C to 80°C (Anderson *et al.* 1994)). Frequency tests were performed in linear viscoelastic region (LVR), which was previously determined with stress sweep tests at constant frequency of oscillation 10 rad/s. During frequency tests, the frequency of oscillation varied from 100 to 0.1 rad/s at constant shear strain within LVR, depending on a sample. Creep and recovery tests were conducted within LVR only at the temperature of 40°C. Time-dependent deformation function, $\gamma(t)$, determined from the measurements was transformed to creep compliance time-dependent function, $J(t)$, using the equation:

$$J(t) = \gamma(t)/\tau_c, \quad (1)$$

where τ_c is the applied constant shear stress.

All measured data were fitted to mathematical or mechanical models. To evaluate the accuracy of fit, three statistical parameters were determined: sum of square residuals (SSR), normalised mean square error (NMSE) and discrepancy ratio (R_i) (Yusoff *et al.* 2010). Statistical parameters served as a measure of divergence between experimental data and calculated values using different models.

SSR is expressed as

$$SSR = \sum_{i=1}^n (y_i - f(x_i))^2 \quad (2)$$

Table 1. Samples used in the study.

	Label	Proportion of the bitumen (%)	Proportion of the rejuvenator (%)		Label	Proportion of the bitumen (%)	Proportion of the rejuvenator (%)
Non-aged bitumen	B50/70	100	0	Aged bitumen	B_PAV	100	0
	B50/70_3%	97	3		B_PAV_3%	97	3
	B50/70_5%	95	5		B_PAV_5%	95	5
	B50/70_10%	90	10		B_PAV_10%	90	10
	B50/70_20%	80	20		B_PAV_20%	80	20

NMSE is defined as

$$NMSE = \frac{1}{N} \sum_i \frac{(f(x_i) - y_i)^2}{f(x_i) \bar{y}_i} \quad (3)$$

$$\bar{f(x_i)} = \frac{1}{N} \sum_i f(x_i) \quad (4)$$

$$\bar{y}_i = \frac{1}{N} \sum_i y_i \quad (5)$$

Discrepancy ratio R_i is

$$R_i = \frac{f(x_i)}{y_i} \quad (6)$$

In Equations (2)–(6), the parameter y_i is the i th value of the variable to be predicted, x_i is the i th value of explanatory variable, $f(x_i)$ is the predicted value of y_i and N is total number of data set. A small SSR and NMSE indicate a tight fit of the model to experimental data, whereas the R_i should equal 1 for obtaining the best fit.

Results and discussion

Standard mechanical tests

The results of softening point, Fraass breaking point and penetration, determined for aged and non-aged bitumen samples with various concentrations of PP, are presented in Figure 1. The addition of the PP had an impact on all three standard mechanical properties. As expected, due to the ability of PP to soften the bitumen, the increasing concentration of PP increased the penetration and decreased the softening point for the samples with aged and non-aged bitumen samples. However, for non-aged bitumen the Fraass breaking point decreased with increasing concentration of PP with the lowest value for 5% of PP, while for the aged bitumen, the values of Fraass breaking points slightly increased with the addition of PP. The results of Fraass breaking point were unexpected, since the results of

low temperature bending beam rheometer (BBR) test showed an improved ability to resist low temperature cracking when rejuvenator was added (Avsenik *et al.* 2016). Aged bitumen with 20% of rejuvenator exhibited similar stiffness value as it was determined for non-aged bitumen. Moreover, for rejuvenator, high frequency rheological data (low temperature range) exhibited the prevalence of G'' over G' , indicating that glassy state in the studied range was not achieved. More details on low temperature rheology tests were explained in the previous study (Avsenik *et al.* 2016). Furthermore, the Fraass breaking point is an objectively fairly unreliable method, because the standard repeatability of the method is 3°C.

During the addition of various additives to bitumen, blending and homogeneity of a resulting blend are extremely important, as well as storage stability of the blend in the absence of mixing. Stability was studied according to the standard EN 13399 on a B50/70 bitumen sample with rejuvenator in ratio 1:1. Tested sample was heated for 3 days at an elevated temperature ($T = 180^\circ\text{C}$). During this time, the blend was not mixed. After heating, the blend was cooled and cut into three equal parts. On the basis of differences in mechanical properties measured on lower and upper parts of the samples, the stability level of the bitumen with rejuvenator was determined. Results of the test (Table 2) showed that the softening point and penetration only slightly differed at both parts of the samples, therefore the bitumen blend with rejuvenator was considered homogeneous. Moreover, there was no visual evidence of the separation of bitumen and rejuvenator.

Table 2. The results of storage stability test according to standard EN 13399.

	Condition	Softening point ($^\circ\text{C}$) EN 1427	Penetration (1/10 mm) EN 1426
B50/70_50%	Ambient temperature	42.6	115
B50/70_50% upper part	72 h at 180°C	43.1	118
B50/70_50% lower part	72 h at 180°C	42.8	117

Figure 1. Standard mechanical properties for the aged and non-aged bitumen samples with and without rejuvenator.

Prediction of complex modulus using the rheological models

The suitability of time-temperature superposition (TTS) for the presentation of linear viscoelastic behaviour of the samples has been confirmed by linear relationship between shift factor, a_T , and temperature. Consequently, WLF and Arrhenius equations were in linear relationship (Gabbott 2007):

$$(T - T_0)/\log a_T = -1/C_1(T - T_0) + (-C_2/C_1) \quad (7)$$

and

$$\log a_T = (E_a/2, 303 R)(1/T) + (-E_a/2, 303 R)(1/T_0) \quad (8)$$

Figure 2 presents temperature dependency of shift factor for non-aged bitumen. Similar was observed also for aged bitumen, but for the sake of clarity the results are not presented here. Dependency of the shift factor at reference temperature $T = 30^\circ\text{C}$ was completely linear with statistic fit $R^2 = 1$ for all the samples studied (i.e. non-aged and aged, with and without rejuvenator).

For the construction of master curves only horizontal shifts were used. Master curves $G^*(\omega)$ were fitted with different models: two Sigmoidal models (standard four-parameter model and generalised five-parameter model) and the model of Al-Qadi and co-workers (Elseifi *et al.* 2002, Pellinen and Witczak 2002, Pellinen *et al.* 2007). First two models can only be applied to the construction of G^* master curve, whereas Al-Qadi model considers also the behaviour of phase shift angle δ . Generalised Sigmoidal model is given as

$$\log|G^*| = \delta + \frac{\alpha}{[1 + \chi e^{\beta + \gamma(\log \omega_{red})}]^{1/\chi}} \quad (9)$$

where ω_{red} is the reduced frequency, δ lower asymptote, α the difference between the values of upper and lower asymptotes, β and γ define the shape between asymptotes and location of inflection point, respectively, while χ parameter allows the curve to take a non-symmetrical shape for master curve.

When χ is equal to 1, Equation (9) reduces to standard Sigmoidal model.

Al-Qadi model determines complex modulus as

$$|G^*| = G_g \left[1 - \frac{1}{\left[1 + \left(\frac{\omega_{red}}{\omega_0} \right)^v \right]^w} \right] \quad (10)$$

and the phase shift angle as

$$\delta = \frac{90}{\left[1 + \left(\frac{\omega_{red}}{\omega_0} \right)^v \right]^w} \quad (11)$$

where ω_0 is the scale parameter that defines the location of transition along frequency axis, while v and w are dimensionless parameters.

The values of statistical parameters, obtained for all the samples used in the study by using various rheological models, are shown in Table 3. In general, SSR parameters were lower for non-aged bitumen. According to SSR value, the best fit was obtained with five-parameter Sigmoidal model, while some discrepancies were observed with the Al-Qadi model. Values of NMSE for non-aged and aged bitumen samples were similar. According to NMSE minimal value, the best agreement between experimental and theoretical values was obtained by using standard four-parameter Sigmoidal model. However, the highest precision of the prediction, according to R_i , was accomplished when the five-parameter Sigmoidal model was used. Significant differences between non-aged and aged bitumen samples were observed for the Al-Qadi model. Although the Al-Qadi model did not provide the best results for the predictions of complex modulus, the predictions of phase angle with this model were in very good agreement with experimental results.

The results of master curves for complex modulus, obtained from experimental data and calculated by using various mathematical models for non-aged and aged bitumen samples with

Figure 2. Temperature dependency of shift factors for non-aged bitumen: (a) WLF equation and (b) Arrhenius equation.

Table 3. Statistical parameters for evaluation and parameters of mathematical models for the prediction of complex modulus and phase angle for the non-aged and aged bitumen samples.

		B50/70	B50/70_3%	B50/70_5%	B50/70_10%	B50/70_20%	B_PAV	B_PAV_3%	B_PAV_5%	B_PAV_10%	B_PAV_20%
Sigmoidal 4 parameter	SSR	3.9566	4.3653	3.8722	3.5773	2.7773	3.2655	5.1212	4.8658	5.2137	5.1871
	NMSE	0.2473	0.3222	0.3159	0.3303	0.3873	0.1425	0.1442	0.1705	0.1616	0.2056
	Ri	0.9665	0.9662	0.9707	0.9734	0.9790	0.9759	0.9620	0.9636	0.9617	0.9627
	α	7.80E+06	7.13E+06	6.37E+06	6.04E+06	4.22E+06	2.05E+07	1.77E+07	1.87E+07	1.41E+07	1.23E+07
	β	4.45	4.66	4.51	4.66	4.91	2.88	2.79	3.05	3.02	3.55
	γ	-1.91	-1.91	-1.94	-1.96	-2.00	-1.34	-1.37	-1.42	-1.47	-1.64
	δ	-4.94	-4.96	-4.20	-3.95	-2.32	-386.93	-228.03	-162.99	-112.06	-42.44
Sigmoidal 5 parameter	SSR	2.0391	2.3354	2.1104	1.8686	1.4486	2.4024	2.8090	2.8104	2.7702	2.5622
	NMSE	1.8123	2.8105	2.3730	2.3533	2.4420	0.9077	1.0321	1.1282	1.1650	1.4445
	Ri	0.9738	0.9744	0.9768	0.9792	0.9833	0.9730	0.9690	0.9694	0.9694	0.9718
	α	7.80E+06	7.13E+06	6.37E+06	6.04E+06	4.22E+06	2.05E+07	1.77E+07	1.87E+07	1.41E+07	1.23E+07
	β	1.69	1.68	1.78	1.90	2.18	0.88	0.73	0.97	0.84	1.10
	γ	-0.91	-0.87	-0.94	-0.96	-1.03	-0.68	-0.65	-0.69	-0.69	-0.75
	δ	-1.95	-1.94	-1.65	-1.76	-0.91	-150.46	-120.61	-91.07	-56.71	-20.42
Al-Qadi	χ	0.43	0.41	0.44	0.45	0.48	0.43	0.40	0.43	0.40	0.40
	SSR	4.0093	4.8186	4.0630	3.9802	2.8137	8.4141	9.2161	8.5154	8.2746	6.8379
	NMSE	1.6692	2.7092	2.1789	2.3258	2.2686	1.8104	2.0443	1.9589	1.9387	2.0332
	Ri	0.9586	0.9565	0.9637	0.9644	0.9746	0.9245	0.9174	0.9237	0.9259	0.9388
	Gg	1.00E+08	1.00E+08	1.00E+08	1.00E+08	1.00E+08	1.00E+08	1.00E+08	1.00E+08	1.00E+08	1.00E+08
	ω_0	12.37	13.89	13.44	14.82	22.77	0.79	0.57	1.20	1.06	2.80
	ν	0.88	0.88	0.89	0.90	0.91	0.69	0.70	0.70	0.73	0.79
Al-Qadi delta	w	0.01	0.01	0.01	0.01	0.01	0.02	0.01	0.02	0.01	0.01
	SSR	0.0221	0.0246	0.0222	0.0287	0.0383	0.0445	0.0531	0.0324	0.0583	0.0397
	NMSE	0.0002	0.0002	0.0002	0.0002	0.0003	0.0003	0.0004	0.0003	0.0005	0.0003
	Ri	0.9997	0.9994	0.9998	0.9996	0.9995	0.9994	0.9986	0.9992	0.9994	0.9994
	ω_0	0.01	0.01	0.01	0.01	0.01	0.00	0.00	0.00	0.00	0.00
	ν	0.57	0.50	0.47	0.52	0.60	0.58	0.61	0.67	0.73	0.55
	w	0.09	0.10	0.10	0.09	0.07	0.09	0.08	0.07	0.06	0.09

Figure 3. Mathematical predictions for complex modulus of (a) B50/70_5% and (b) B_PAV_5%.

the addition of 5% rejuvenator (B50/70_5% and B_PAV_5%) are presented in Figure 3. The divergences between the predictions and experimental data were for all models observable only at the highest frequencies. Similar was observed also for the samples with other concentrations of the rejuvenator, however, for the sake of clarity, only one concentration is presented.

The results of experimental data and mathematical predictions of phase angles are presented in Figure 4. In general, the predictions were in good agreement with the experiments, however, some deviations were observed in the frequency range between 0.001 and 1 rad/s. Nevertheless, compared to complex modulus, the statistical parameters showed better agreement for phase angle predictions.

Numerical values of all calculated parameters are shown in Table 3. The results showed that the values of parameter δ in Sigmoidal models were negative, indicating that the modulus at low frequencies/high temperature were small, which can be observed also in Figure 3. Low value of parameter δ , detected in the analysis for the non-aged bitumen demonstrates that these samples behaved like viscoelastic liquid (Yusoff *et al.* 2010). For the aged bitumen, on the other hand, the values of parameter δ increased significantly indicating that rheological properties of these samples approached to viscoelastic solids. The addition of PP decreased the parameter δ , which confirms

the favourable effect of PP as rejuvenator. The parameter β , which regulates the horizontal position of inflection point – a point at which the curve changes from being concave to convex – decreased for non-aged compared to the aged bitumen samples and increased with the addition of PP. This can be attributed to the fact that ageing increases the content of asphaltenes and subsequently produces bitumen with higher hardness and stiffness (Yusoff 2012). The parameter β decreased for the aged bitumen and increased when rejuvenator was added, which displayed in softer structure of the bitumen. The parameter γ was almost constant for non-aged and aged bitumen samples, respectively, regardless of the amount of rejuvenator added. This indicates that the addition of PP had no influence on the slope of the master curve from intermediate to high temperatures (20–80 °C). The greatest effect of PP addition was observed in the decrease of α parameter, indirectly indicating that the bitumen softened when the rejuvenator was added. In general, the higher the proportion of the added rejuvenator, the greater the reduction of α . These results are also in good agreement with other researches (Yusoff 2012, Yusoff *et al.* 2013). The difference between the two Sigmoidal models was observed for χ parameter, which allows the curve to take a non-symmetric shape. In the present study, the role of the parameter χ was less than 1, therefore it cannot be ignored.

Figure 4. Al-Qadi model prediction for phase angle of (a) B50/70_5% and (b) B_PAV_5%.

However, the parameter χ was similar for all the samples, regardless of the structure of the bitumen, as well as the addition of the rejuvenator. This indicates that the non-symmetry of the shape of master curves was similar for all the samples used.

The parameter ω_0 in Al-Qadi model can be associated with β parameter from previous two models. Its trend followed the trend of the parameter β : the parameter decreased when the bitumen was aged and increased when the rejuvenator was added.

The parameters of Al-Qadi model for predicting phase angle were very small: moreover, very similar values were observed for different samples.

Mechanical model for creep and recovery tests

Creep and recovery tests (within LVR) are non-destructive tests, performed in two shear stress steps. In the first step (creep phase), the sample is exposed to a constant shear stress, while in the second step (recovery phase) the applied shear stress is instantly removed (Mezger 2006). In the present work, experimentally determined time-dependent deformation function, $\gamma(t)$, was fitted to the six parameter model according to the literature (Collop *et al.* 1995, Šušteršič 2014). The six parameter model consists of two Kelvin-Voight models and a Maxwell model joined in series. Deformation function in the creep phase is written as

$$\gamma(t) = \frac{\tau_c \cdot t}{\eta_0} + \frac{\tau_c}{G_0} + \sum_{i=1}^2 \frac{\tau_c}{G_i} [1 - e^{-(t/\lambda_i)}] \quad (12)$$

and in the recovery phase as

$$\gamma(t) = \frac{\tau_c \cdot t_1}{\eta_0} + \sum_{i=1}^2 \frac{\tau_c}{G_i} e^{-(t-t_1)/\lambda_i} \quad (13)$$

Considering Equation (6) the creep compliance in creep phase

is written as

$$J(t) = \frac{t}{\eta_0} + J_0 + \sum_{i=1}^2 J_i [1 - e^{-(t/\lambda_i)}] \quad (14)$$

and in recovery phase as

$$J(t) = \frac{t_1}{\eta_0} + \sum_{i=1}^2 J_i e^{-(t-t_1)/\lambda_i} \quad (15)$$

The results of statistical evaluation of creep and recovery models are shown in Table 4. Excellent agreement was observed in the recovery phase, where SSR and NMSE were close to 0 and R_i was close to 1 for all the samples used in the study. In the creep phase, the divergence between experimental and predicted data was the highest for the samples with 10% or 20% of PP. However, in general also in the creep phase the experimental values were in very good agreement with the predicted values.

Creep and recovery tests of the samples were conducted within LVR. Although shear stress increased (as well as time-dependent function $\gamma(t)$), creep compliance response, $J(t)$ curves, overlapped with one another independently of preset stress τ_c (Figure 5). Furthermore, in Figure 5 (b) very good agreement of the six parameter model and experimental data could be observed for the sample with 5% of the PP. Similar results were obtained also for other samples, but for the sake of clarity only the results of one sample are presented.

The parameters of six parameter mechanical model (Equations 12–15) for all the samples used in the study are presented in Table 4. The instantaneous compliance, J_0 , is a coefficient that characterises the elastic deformability of a material. The materials with J_0 close to zero are considered as absolute rigid (Mezger 2006). The results show that at temperature 40° C the tested samples exhibited very low values of J_0 indicating solid-like behaviour. J_0 increased with the addition of the rejuvenator for non-aged bitumen, and decreased for the aged bitumen, indicating a less rigid structure of the non-aged samples. The dashpot in the Maxwell model demonstrates the viscosity response of the material, which is presented as η_0 . Compared to

Table 4. The statistical and mechanical parameters of creep and recovery model for the non-aged and aged samples at temperature $T = 40^\circ\text{C}$.

		B50/70	B50/70_3%	B50/70_5%	B50/70_10%	B50/70_20%	B_PAV	B_PAV_3%	B_PAV_5%	B_PAV_10%	B_PAV_20%
Creep	SSR	2.1486	0.5506	3.9928	14.2105	2.9581	0.1070	0.5713	0.6930	2.2165	4.6300
	NMSE	0.0007	0.0004	0.0005	0.0007	0.0008	0.0005	0.0022	0.0011	0.0091	0.0185
	Ri	1.0569	1.0115	0.9907	0.8745	0.9515	1.0026	1.0289	1.0482	0.9098	0.7577
	τ_c (1/Pa)	0.3	0.25	0.2	0.2	0.1	20	20	12	8	3
	J_0 (1/Pa)	8.74E-07	8.74E-07	1.09E-06	1.57E-06	1.99E-06	2.07E-06	1.61E-06	1.29E-06	1.26E-06	8.56E-07
Recovery	η_0 (Pas)	19843	15287	13355	10689	5195	1493661	1156101	891346	543369	243369
	λ_1 (s)	19.01	1.34	0.25	0.16	0.01	5.58	37.51	53.17	39.01	46.04
	λ_2 (s)	327.71	87.28	100.73	265.41	3.13	69.35	4.34	4.43	7.15	9.38
	SSR	0.0306	0.0071	0.0123	0.0202	0.0193	0.0178	0.0040	0.0004	0.0004	0.0008
	NMSE	0.0002	0.0000	0.0001	0.0001	0.0001	0.0001	0.0000	0.0000	0.0000	0.0000
	Ri	0.9860	0.9955	0.9949	1.0110	1.0107	1.0104	0.9953	1.0000	1.0000	1.0000
	η_0 (Pas)	20288	14800	13100	10503	5226	1623929	1371528	1147732	657256	311302
	λ_1 (s)	16.17	13.21	9.24	8.65	4.54	9.80	13.97	14.68	12.51	10.38
	λ_2 (s)	663.44	771.39	1218.74	1385.61	1700.36	221.11	283.33	300.94	276.09	256.92

non-aged bitumen, η_0 for the aged bitumen without rejuvenator was significantly higher, because the proportion of solid phase in bitumen increased due to oxidation and volatilisation. For non-aged bitumen, η_0 values were similar in the creep and recovery phases, whereas η_0 for the aged bitumen was higher in the recovery phase, regardless of the amount of rejuvenator added. The addition of PP decreased η_0 for the aged and non-aged bitumen samples, respectively.

A certain time delay of the elastic behaviour, which is the characteristic for viscoelastic materials, was evaluated with retardation and relaxation time. Retardation is a response of delay to an applied shear stress and can be described as a 'delay of elasticity'. Relaxation is a process in the state at rest after a forced deformation (Mezger 2006). The values of retardation or relaxation times, resulting from two Kelvin-Voight models (λ_1 and λ_2) for unmodified aged bitumen, were lower compared to the non-aged bitumen. Retardation time λ_1 decreased when higher amounts of PP were added to the non-aged bitumen. For the aged bitumen λ_1 increased at lower amounts of PP (B_PAV_3% and B_PAV_5%) while for the highest amounts of PP (i.e. B_PAV_10% and B_PAV_20%) the λ_1 decreased, but still exhibited higher values compared to B_PAV without the rejuvenator. Similarly, the values of retardation time λ_2 depended on the amount of the rejuvenator, but in general, the values of λ_2 for all modified samples were lower compared to unmodified samples. If we conclude, the addition of PP to non-aged bitumen significantly influenced the retardation and relaxation times, respectively. PP decreased the friction between the particles, leading to decreased viscosity and increased elastic contribution to viscoelastic behaviour. Consequently, the creep straining and stress relaxation of non-aged bitumen samples with added PP were more rapid. On the other hand, the addition of PP to the aged bitumen did not significantly decrease retardation and relaxation times, respectively. Moreover, retardation and relaxation times slightly increased although the viscosity decreased. This effect could be attributed to the degradation of the aged bitumen which led to decreased elastic contribution and consequently to slower creep straining and slower stress relaxation.

The viscosity models

For describing the viscosity behaviour five different models were used (Mezger 2006). All models were fitted to viscosity master curves which were obtained from complex modulus master curve as $\eta^* = G^*/\omega_{red}$. The experimental values of viscosity were compared to the predictions, obtained by using (i) the Cross model:

$$\frac{\eta^* - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{1 + (c \cdot \omega_{red})^p} \quad (16)$$

(ii) the Carreau model:

$$\frac{\eta^* - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{(1 + (c_1 \cdot \omega_{red})^2)^p} \quad (17)$$

(iii) the Carreau/Gahleitner model (modification of Carreau

Figure 5. Mechanical creep and recovery model for B_PAV_5%: (a) $\gamma(t)$ and (b) $J(t)$ response to increasing shear stress.

model):

$$\frac{\eta^* - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{(1 + (c_1 \cdot \omega_{red})^{p_1})^p} \quad (18)$$

(iv) the Carreau/Yasuda model (modification of Carreau model):

$$\frac{\eta^* - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{(1 + (\lambda \cdot \omega_{red})^{p_1})^p} \quad (19)$$

and (v) the Vinogradov/Malkin model:

$$\frac{\eta^* - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{1 + c_1 \cdot \omega_{red}^p + c_2 \cdot \omega_{red}^{2p}} \quad (20)$$

where parameters c and c_1 are time constants related to relaxation times, while parameters p and p_1 are dimensionless exponents. The results showed that the values of η_∞ were compared to η_0 very low and negligible. Therefore, we simplified the models and ignored the η_∞ , except for the last Vinogradov/Malkin model.

The values of the parameters for the predictions of viscosity with various models are presented in Table 5. SSR and NMSE values revealed substantial differences between the predictions of the viscosity for non-aged and aged bitumen samples, respectively. The results showed a tighter fit of the models to experimental values for the samples of non-aged bitumen. It was shown that according to SSR values the best model for these samples was the Carreau/Yasuda model, while for the aged bitumen the Vinogradov/Malkin model provided the best results. The cross model and the Carreau/Gahleitner model yielded similar SSR values. The poorest fits were obtained by means of the Carreau model. According to R_i values, the Carreau model exhibited significantly lower values than other models. However, the values of R_i were still higher than 0.97, indicating that also this model could be used for the prediction of the viscosity for the aged and non-aged samples, respectively.

Figure 6 presents the predictions and experimental values of viscosity for non-aged and aged bitumen samples with 20% of PP. Results indicate that all models, used for the predictions, excellently predicted the viscosity in the whole frequency range.

The predicted values of zero shear viscosity η_0 were very similar when the Cross model, the Carreau/Ghaleitnem model and the Vinogradov/Malkin model were used for the aged and non-aged samples, respectively. Some deviations from these values were observed with the use of other models. The highest discrepancies in the parameter η_0 were observed with the Carreau model, where η_0 decreased with increasing amount of added PP; however, even when the highest amount (20%) of PP was added to the aged bitumen, the value of η_0 did not come close to the value of initial non-aged bitumen.

The effect of PP was also observed for the other parameters. The parameters c , c_1 and λ decreased with increasing PP content, while the parameters p and p_1 for non-aged and aged samples, respectively, were similar, regardless of the amount of PP added.

The infinite viscosity was determined with the Vinogradov/Malkin model. As expected, the obtained values were very low compared to η_0 and very similar for all non-aged and aged samples, respectively, regardless of the amount of PP added. It can be concluded that rejuvenator exhibited a very minor impact on the viscosity in high shear rate range.

Zero shear viscosity – ZSV

The value of ZSV is at a certain temperature a material constant. It could be determined using rotational tests as a limiting value of shear viscosity function $\eta(\dot{\gamma})$ for $\dot{\gamma} \rightarrow 0$ or from oscillatory test, where η_0 is determined as a limiting value of complex viscosity function $\eta^*(\omega)$ for $\omega \rightarrow 0$. For many polymeric systems, the steady-state shear viscosity, η , plotted against shear rate, $\dot{\gamma}$, equals the magnitude of the complex viscosity, $|\eta^*|$, plotted against angular frequency, ω . The relation is called Cox–Merz relation: $\eta(\dot{\gamma}) = |\eta^*(\omega)|$, which was in the present study verified for all the samples. ZSV could also be determined by using creep test at the end of stress phase, when steady-state

Table 5. Statistical parameters and model parameters for predicting viscosity with various mathematical models for the non-aged and aged samples.

		B50/70	B50/70_3%	B50/70_5%	B50/70_10%	B50/70_20%	B_PAV	B_PAV_3%	B_PAV_5%	B_PAV_10%	B_PAV_20%
Cross model	SSR	0.5000	0.5844	0.5308	0.3592	0.2513	1.0298	0.9221	0.8386	0.7564	0.6168
	NMSE	0.0059	0.0072	0.0064	0.0045	0.0028	0.0268	0.0142	0.0093	0.0104	0.0075
	Ri	0.9948	0.9947	0.9953	0.9963	0.9977	0.9908	0.9918	0.9925	0.9932	0.9945
	c	6.63	6.10	5.32	2.95	1.35	5317.82	5589.71	4001.19	1820.88	257.45
	p	0.41	0.41	0.40	0.40	0.39	0.53	0.53	0.51	0.50	0.46
	η_0	3.25E+05	2.34E+05	2.25E+05	1.61E+05	7.42E+04	8.75E+07	8.32E+07	4.99E+07	2.54E+07	4.65E+06
Carreau model	SSR	0.7893	0.7168	0.8615	0.6930	0.6151	3.2779	2.7636	2.3004	2.5486	1.8637
	NMSE	1.2400	1.1943	1.1508	1.1419	1.0267	2.0691	2.0691	2.0304	1.8344	1.6231
	Ri	0.9919	0.9935	0.9923	0.9929	0.9945	0.9707	0.9753	0.9795	0.9772	0.9834
	c_1	34.49	33.38	34.27	22.41	13.06	6398.89	7362.59	5110.54	2653.85	528.76
	p	0.16	0.15	0.15	0.14	0.13	0.25	0.25	0.24	0.23	0.20
	η_0	2.81E+05	2.01E+05	1.94E+05	1.42E+05	6.50E+04	6.49E+07	6.36E+07	3.76E+07	1.93E+07	3.69E+06
Carreau/Gahleitner model	SSR	0.5000	0.5844	0.5308	0.3592	0.2513	1.0298	0.9221	0.8386	0.7564	0.6168
	NMSE	1.1115	1.1830	1.1440	1.0779	1.0235	2.1308	2.0811	2.0456	1.8643	1.6265
	Ri	0.9948	0.9947	0.9953	0.9963	0.9977	0.9908	0.9918	0.9925	0.9932	0.9945
	c_1	6.63	6.10	5.32	2.95	1.35	5318.05	5589.59	4001.32	1820.88	257.46
	p	0.08	0.08	0.08	0.08	0.08	0.24	0.24	0.23	0.23	0.22
	η_0	3.25E+05	2.34E+05	2.25E+05	1.61E+05	7.42E+04	8.75E+07	8.32E+07	4.99E+07	2.54E+07	4.65E+06
Carreau/Yasuda model	p_1	5.12	5.07	5.02	5.04	4.99	2.23	2.23	2.19	2.18	2.09
	SSR	0.4586	0.4571	0.4729	0.3243	0.2147	0.7173	0.7565	0.7569	0.6703	0.6083
	NMSE	1.1269	1.1879	1.1461	1.0793	1.0245	2.1912	2.1110	2.0640	1.8812	1.6281
	Ri	0.9953	0.9959	0.9958	0.9967	0.9981	0.9936	0.9932	0.9932	0.9940	0.9946
	p	0.63	0.66	0.65	0.65	0.66	0.43	0.45	0.47	0.48	0.53
	p_1	0.55	0.65	0.55	0.52	0.52	0.34	0.38	0.39	0.39	0.42
Vinogradov/Malkin model	η_0	3.08E+05	2.15E+05	2.12E+05	1.54E+05	7.08E+04	1.19E+08	1.02E+08	5.79E+07	2.90E+07	4.81E+06
	λ	14.25	18.56	13.73	7.12	3.81	3879.14	4434.53	3351.08	1422.15	222.98
	SSR	0.4864	0.5825	0.5265	0.3555	0.2513	0.6944	0.6353	0.6025	0.5380	0.4834
	NMSE	1.1099	1.1830	1.1441	1.0780	1.0235	2.1598	2.1022	2.0622	1.8790	1.6327
	Ri	0.9950	0.9948	0.9953	0.9963	0.9977	0.9938	0.9943	0.9946	0.9952	0.9957
	c_1	7.06	6.23	5.51	3.05	1.35	6496.00	6641.69	4729.05	2165.52	301.71
	c_2	0.01	0.00	0.01	0.00	0.00	-0.09	-0.10	-0.07	-0.06	-0.02
	η_0	3.27E+05	2.34E+05	2.26E+05	1.62E+05	7.42E+04	9.11E+07	8.61E+07	5.15E+07	2.62E+07	4.76E+06
	η_∞	0.30	0.30	0.30	0.30	0.30	0.16	0.16	0.16	0.16	0.16
	p	0.41	0.40	0.39	0.40	0.39	0.52	0.52	0.50	0.49	0.45

Figure 6. Experimental data and predicted values of the viscosity for (a) B50/70_20% and (b) B_PAV_20%.

flow behaviour is reached. First, deformation change in corresponding period of time, $\Delta\gamma/\Delta t$, is calculated, i.e. shear rate, $\dot{\gamma}_0$. In the second calculation step ZSV: $\eta_0 = \tau_c/\dot{\gamma}_0$ is determined (Mezger 2006). The time of creep test is converted in frequency scale as $\omega = 1/t_{creep}$. In the present study, the frequencies, obtained by above-mentioned procedure using creep tests, were lower (as low as 0.002 rad/s) than shear rate range of rotational and frequency range of oscillatory tests. Figure 7 presents the viscosity results obtained from the three above-mentioned tests. Oscillatory and rotational tests were performed at five temperatures (40–80°C), while creep tests were conducted only at 40°C. The results of oscillatory and rotational tests were in good agreement for non-aged and aged bitumen samples, respectively. On the other hand, the values of the viscosity, determined by using the creep test, were similar to the results of oscillatory and rotational tests for non-aged bitumen samples (Figure 7a), while some discrepancies were observed for the samples of the aged bitumen (Figure 7b).

ZSV was determined at different frequencies of oscillation, depending on the type of the test (rotational or oscillatory, respectively). ZSV was determined by oscillatory tests at $\omega = 0.1$ rad/s, by rotational tests at $\omega = 0.01$ rad/s and during creep tests at $\omega = 0.002$ rad/s. The results of ZSV, obtained by using all three methods, are presented in Table 6. For all the samples used the ZSV decreased with increasing temperature,

regardless of the determination method used. At the same time, the values of ZSV decreased with an increasing amount of PP. The results of oscillatory tests were in good agreement with the results, obtained by rotational tests, while ZSV values, determined during creep tests, were significantly higher, especially for the samples of the aged bitumen. The reason for such results could be attributed to the fact that during creep test the binder did not reach a steady state, or the frequency of oscillation during frequency test was not low enough to obtain a constant value of the viscosity (Biro *et al.* 2009). According to the Figure 7(b), it is reasonable to believe that in the present study both effects were present.

Conclusion

The results of the present study confirmed several positive effects of the addition of rejuvenator to the aged bitumen: decreasing of softening point, increasing penetration and decreasing consistency (viscosity) of the aged bitumen. Based on the results, the following conclusions could be established:

- The rheological behaviour of the bitumen could be successfully described with various models. For the predictions of complex modulus, the SSR and the R_i showed that the best results were obtained by using the standard four-parameter

Figure 7. Zero shear viscosity determination by using different tests for (a) B50/70_5% and (b) B_PAV_5%.

Sigmoidal model. According to the *NMSE* criterion, the best prediction was obtained by the generalised five-parameter Sigmoidal model, while the Al-Qadi model was very reliable for phase angle estimations. In general, the predictions for non-aged bitumen were in better agreement with experimental data than those obtained for the aged bitumen.

- The use of mathematical model for creep and recovery tests showed that the addition of PP to non-aged bitumen significantly decreased retardation and relaxation times, respectively. On the other hand, the addition of PP to the aged bitumen slightly increased retardation and relaxation times although the viscosity decreased.

Table 6. ZSV, determined with different tests.

	ZSV (Pa)		40°C	50°C	60°C	70°C	80°C
B50/70	Oscillatory	$\omega = 0.1 \text{ rad/s}$	13,200	2730	426	114	35
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	13,400	2280	425	114	34
	Creep	$\omega = 0.002 \text{ rad/s}$	19,127				
B50/70_3%	Oscillatory	$\omega = 0.1 \text{ rad/s}$	11,800	1870	410	104	32
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	12,600	1880	410	104	33
	Creep	$\omega = 0.002 \text{ rad/s}$	14,872				
B50/70_5%	Oscillatory	$\omega = 0.1 \text{ rad/s}$	10,500	1670	363	97	32
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	10,600	1670	364	97	31
	Creep	$\omega = 0.002 \text{ rad/s}$	13,261				
B50/70_10%	Oscillatory	$\omega = 0.1 \text{ rad/s}$	8490	1490	330	83	27
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	8590	1490	331	82	26
	Creep	$\omega = 0.002 \text{ rad/s}$	10,554				
B50/70_20%	Oscillatory	$\omega = 0.1 \text{ rad/s}$	5940	1010	237	63	21
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	5860	1010	237	63	21
	Creep	$\omega = 0.002 \text{ rad/s}$	7027				
B_PAV	Oscillatory	$\omega = 0.1 \text{ rad/s}$	236,000	65,600	16,000	3070	685
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	333,000	67,500	15,800	3100	689
	Creep	$\omega = 0.002 \text{ rad/s}$	1,411,658				
B_PAV_3%	Oscillatory	$\omega = 0.1 \text{ rad/s}$	179,000	51,700	12,500	2460	564
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	240,000	53,700	12,000	2480	566
	Creep	$\omega = 0.002 \text{ rad/s}$	1,099,945				
B_PAV_5%	Oscillatory	$\omega = 0.1 \text{ rad/s}$	160,000	43,400	9700	1990	453
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	206,000	46,200	9760	2060	455
	Creep	$\omega = 0.002 \text{ rad/s}$	874,231				
B_PAV_10%	Oscillatory	$\omega = 0.1 \text{ rad/s}$	101,000	24,500	6940	1400	326
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	153,000	29,700	6830	1410	327
	Creep	$\omega = 0.002 \text{ rad/s}$	540,994				
B_PAV_20%	Oscillatory	$\omega = 0.1 \text{ rad/s}$	67,400	15,400	2940	621	158
	Rotational	$\dot{\gamma} = 0.01 \text{ rad/s}$	84,300	16,200	2970	621	158
	Creep	$\omega = 0.002 \text{ rad/s}$	240,630				

- ZSV values were determined by the rotational, oscillatory and creep tests. It was observed that the addition of the rejuvenator decreased ZSV of the sample, irrespective of the test method and model used.

Nevertheless, the presented results confirmed beneficial trends of using PP as a rejuvenator resulting in effective recycling of the bitumen.

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