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EVALUATION OF THE ANTI-RUTTING POTENTIAL OF POLYMER-MODIFIED BINDERS BY MEANS OF CREEP-RECOVERY SHEAR TESTS

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ABSTRACT

In the experimental study reported in this paper a creep-recovery shear test method was used to evaluate the anti-rutting potential of different polymer-modified bituminous binders. The effects of several factors related to modification were investigated, such as polymer type (SBS, Styrene-Butadiene-Styrene vs. EVA, Ethylene-Vinyl-Acetate), composition (styrene content), structure (linear vs. radial SBS) and dosage (3-6% by weight of the base bitumen). The effect of short-term aging was also considered by comparing binder response before and after treatment with the Rolling Thin-Film Oven Test (RTFOT). Following previous work on the development of the test protocol, experimental results were analyzed by referring to shear modulus curves $G(t)$ and to permanent compliance (J_p), obtained by dividing residual strain at the end of the unloading phase by the stress applied during creep loading. Results indicated the effectiveness of the proposed method in discriminating between the behavior of the different polymer-modified binders and in capturing the effects caused by the factors considered in the investigation. Reliable rankings of the binders were established and were explained by referring to the specific behavior of employed modifiers.

KEY WORDS: polymer-modified bituminous binders, rutting, creep-recovery, permanent compliance, aging

INTRODUCTION

The modification of bitumen by means of polymers may provide a significant enhancement of its viscoelastic properties (King et al. 1986; Collins et al. 1991;

1 Wardlaw and Shuler 1991). For this reason, the use of polymer-modified
2 bituminous binders can be suitable for heavy-duty pavements subjected to high
3 traffic volumes with a significant percentage of heavy and slow-moving vehicles.
4 However, since addition of a modifier to a base bitumen can considerably increase
5 its cost, reliable laboratory test methods and ranking criteria are needed to fully
6 appreciate, before use in paving operations, their effective performance-related
7 value.
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12 The initial SUPERPAVE grading system (Harrigan et al. 1994) developed in the
13 early 90's, although supported by an experimental database mainly composed of
14 tests performed on un-modified bitumens, was conceived to accommodate binders
15 of all types, without limitations on the possible presence of additives and/or
16 modifiers. However, several investigations carried out after approval of
17 SUPERPAVE specifications showed that they lead to an underestimate of the real
18 field performance potential of polymer-modified binders, which cannot be clearly
19 discerned from unmodified products (Bahia et al. 2001; D'Angelo 2002). In the
20 specific case of rutting, this is due to the fact that the performance indicator
21 selected by SUPERPAVE, $G^*/\sin\delta$, derives from small-strain oscillatory loading.
22 As a result of such testing conditions, the contribution of polymer structures (e.g.
23 diffuse networks and/or entanglements) to rheological behavior cannot be fully
24 captured. Moreover, delayed elastic response cannot be clearly separated from
25 viscous flow which is truly responsible for the accumulation of permanent
26 deformation.
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31 A number of studies have been carried out by researchers worldwide to replace
32 the $G^*/\sin\delta$ rutting parameter used in SUPERPAVE. They either refer to the zero
33 shear viscosity concept, or to parameters extracted by modeling the response of
34 binders under repeated creep loading.
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Zero shear viscosity (ZSV) is the value of viscosity which is exhibited by binders
subjected to shear deformation as shear rate approaches zero. ZSV of binders can
be measured by means of different laboratory test methods (creep, frequency
sweep, shear rate sweep and multi-creep tests) and it has been documented that
results show a good correlation with rutting performance of corresponding asphalt
mixtures (Sybilski 1994; Sybilski 1996; Phillips and Robertus 1996). However,
coherency and reliability of testing protocols is not always ensured in the case of
polymer-modified binders. Morea et al. (2010) found that in the case of SBS-

1 modified binders, ZSV values derived from frequency sweeps and creep tests
2 were not in agreement with each other. Desmazes et al. (2002) observed that since
3 cross-linked and highly-modified binders behave as viscoelastic solids, they never
4 reach steady state flow conditions and therefore the ZSV concept is not applicable
5 for these materials. To solve this problem, De Visscher and Vanelstraete (2004;
6 2009) proposed the use of low shear viscosity (LSV) as an alternative parameter
7 to describe resistance to permanent deformation.
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11 Since permanent deformation in pavements develops as a result of repeated
12 loading, for the purpose of rutting prediction other studies have focused on
13 laboratory tests in which such an occurrence is adequately mimicked. In NCHRP
14 Project 9-10, Bahia et al. (2001) proposed the use of repeated creep recovery tests
15 (RCRT) at high temperatures, with the subsequent calculation, by means of a four
16 element Burger's model, of the viscous component of binder creep stiffness (G_v).
17 D'Angelo et al. (2007) improved the RCRT by developing a multiple stress creep
18 recovery test method (MSCR), which allows evaluation of stress dependency in a
19 single test run.
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22 Taking into account the findings of these and other studies on the subject of
23 permanent deformation, EN standards for the measurement of ZSV and LSV have
24 been developed (EN 15324 2008; EN 15325 2005). Moreover, SUPERPAVE
25 specifications have been updated with the inclusion of new criteria for the
26 determination of high critical temperatures based upon MSCR tests (AASHTO
27 MP 19-10 2010). Recent studies, however, indicate that both LSV and MSCR test
28 protocols do not seem adequate to characterize SBS-modified binders. In the case
29 of LSV, viscosity lines obtained for SBS binders show the absence of a plateau
30 value which implies that low shear viscosity cannot be determined as the viscosity
31 continues to increase as shear rate decreases (Zoorob et al. 2012a). In the case of
32 MSCR, the current protocol of 1 s loading followed by 9 s recovery may be not
33 sufficient to ensure a full delayed elastic recovery for cross-linked materials such
34 as SBS polymers (Zoorob et al. 2012b). On the basis of such limitations, Merusi
35 and Giuliani (2011) introduced the concept of "intrinsic resistance" to non-
36 reversible deformation, which however applies only to a restricted category of
37 modified binders.
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40 The authors of this paper in the past approached the subject of permanent
41 deformation of bituminous binders mainly by making use of simple creep-
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1 recovery shear tests (Santagata and Baglieri 2002; Santagata and Baglieri 2005;
2 Baglieri 2004). This was initially done for un-modified and modified binders,
3 trying to correlate test results with the corresponding response of bituminous
4 mixtures subjected to creep in simple compression (Santagata and Baglieri 2002).
5 Preliminary findings were quite encouraging, showing that by analysing the
6 recovery portion of the time-strain curve of binders a number of parameters can be
7 determined. Their relationships with mixture stress-strain behaviour can be then
8 used with the purpose of selecting and comparing binders on a performance-
9 related basis. Further studies consisted in the application of the same type of
10 approach for the prequalification of modified binders for heavy-duty motorway
11 pavements (Santagata and Baglieri 2005), focusing on the percent residual and
12 recovered strain. Finally, investigations carried out on a calibration fluid and on
13 selected binders, representative of those commercially employed in the paving
14 industry, led to the fine-tuning of the testing protocol (Baglieri 2004). Altogether,
15 the investigations showed that creep-recovery tests not only are simple and quick
16 to perform, but also allow the assessment of anti-rutting potential since they
17 induce high levels of strain, at which polymer structures are adequately stressed,
18 and clearly differentiate reversible and non-reversible strain components.
19 By means of the experimental study presented in this paper, the authors intend to
20 further validate this characterization approach by presenting results obtained in
21 tests carried out on several laboratory-prepared modified binders. In particular, by
22 considering the response of a single base bitumen as a reference, the effects of
23 several factors related to modification were investigated, such as polymer type
24 (SBS, Styrene-Butadiene-Styrene vs. EVA, Ethylene-Vinyl-Acetate), composition
25 (styrene content), structure (linear vs. radial SBS) and dosage (3-6% by weight of
26 the base bitumen). Moreover, the effects of other factors such as test temperature
27 (between 40 and 80°C) and short-term aging (by means of the Rolling Thin Film
28 Oven Test, RTFOT) were also considered.

52 **MATERIALS AND METHODS**

56 **Modified Binders**

59 Modified binders employed in the investigation were prepared in the laboratory
60 by adding different polymers to a single base bitumen (70/100 penetration grade)

1 which was preliminarily subjected to chemical analysis by means of the combined
2 use of Thin-Layer Chromatography (TLC) and Flame Ionization Detection (FID).
3 Such a technique has been often employed for the characterization of bitumens
4 (Leroy 1989; Brulé et al. 1988; Ecker 2001; Stangl et al. 2007; Santagata et al.
5 2009) since it leads to the quantification of the relative amounts of saturates,
6 aromatics, resins and asphaltenes (SARA analysis) as required by the simplified
7 colloidal model (Nellensteyn 1924).

8 Test solutions were prepared by using 0.1 ± 0.01 g of bitumen and 10 ml of
9 toluene. Capillary flow through quartz rods covered by silica gel was then induced
10 by successively employing n-esane, toluene and a solution of dichloromethane
11 (95%) and methilic alcohol (5%). Results obtained from such an analysis are
12 summarized in Table 1, which also contains the values of two synthetic
13 parameters calculated as a function of the four molecular fractions (i.e. the ratio
14 between the percentages of asphaltenes and resins, As/Re; the ratio between the
15 percentages of saturates and aromatics, Sa/Ar). These data show that the base
16 binder can be considered adequate for modification since it satisfies SARA
17 specifications adopted in the past for the Italian motorway network (Autostrade
18 SpA 2000). In particular, values of the abovementioned synthetic ratios are
19 comprised within ranges that ensure compatibility with SBS polymers
20 characterized by a radial structure (As/Re = $0 \div 0.7$; Sa/Ar = $0 \div 1.3$).

21 The polymer modifiers considered in the investigation were selected among those
22 commercially available for paving applications and included one plastomer
23 (Ethylene-Vinyl-Acetate, EVA) and three elastomers (Styrene-Butadiene-Styrene,
24 SBS). SBS modifiers differed in composition and molecular configuration since
25 one was of the linear type (with 30% styrene), while the remaining two were
26 characterized by a radial structure (with 31 and 22% styrene). By making use of a
27 bath-oil mixer, each polymer, available in powder form, was incorporated in the
28 base bitumen by adopting two dosages, equal to 3 and 6% (by weight of the base
29 bitumen). Blending was carried out at 180°C for 60 min with a mixing speed of
30 800 rpm, visually controlling the homogeneity of the final composite material.
31 Before testing the binders were stored at room temperature for a maximum time
32 of one week.

33 The complete set of binders prepared in the laboratory and subsequently subjected
34 to testing is described in Table 2. All of them were considered both in their

1 original state (unaged) and after short-term aging carried out with the Rolling
2 Thin-Film Oven Test (RFTOT) in accordance with the corresponding AASHTO
3 protocol (AASHTO T240-09 2009). No special care was required for aging of the
4 more heavily modified binders (with 6% polymer).
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7 8 **Creep-Recovery Tests** 9

10 Creep-recovery tests carried out in this investigation followed the protocol
11 previously developed by the authors (Baglieri 2004) and were based on the use of
12 a high-resolution shear rheometer ($8 \cdot 10^{-8}$ N·m torque and $6 \cdot 10^{-5}$ rad angle)
13 equipped with a temperature control system capable of guaranteeing an accuracy
14 of at least 0.1°C (HAAKE Rotovisco RT10).
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17 Tests consisted in single creep-recovery runs in which creep loading time and
18 recovery time were both set at 900 s. The duration of the loading phase is similar
19 to the one chosen by Delgadillo and Bahia (2010) in their study on nonlinearity of
20 bituminous binders and rutting of asphalt mixtures. Recovery time was observed
21 to be sufficient to monitor almost all delayed elastic strain for the materials
22 considered in the investigation and matches the practical need of limiting total
23 testing time.
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26 The cone and plate geometry (35 mm diameter, 4° angle) was adopted to induce a
27 uniform shear strain level in the tested materials. Samples were prepared by
28 slowly heating the blended binders and by thoroughly mixing them before pouring
29 a small quantity on the lower plate. The upper cone was then lowered to reach
30 measurement position and excess material was thereafter removed by trimming.
31 Conditioning was carried out until target test temperature was stable for at least 15
32 minutes. In order to avoid spurious effects due to changes in test geometry, each
33 sample was tested only at one temperature.
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36 Based on the results of previous investigations performed on similar materials, a
37 constant value of shear stress, equal to 100 Pa, was imposed to all binders
38 (Santagata and Baglieri 2005). In order to evaluate the temperature-dependency of
39 creep-recovery response, tests were carried out at 40, 60 and 80°C. Three
40 replicates were run for each binder-aging-temperature combination and the
41 average results were thereafter subjected to analysis.
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RESULTS AND DISCUSSION

Shear Modulus Curves

Experimental data recorded during the loading phase of creep-recovery tests can be represented in terms of the shear modulus $G(t)$ as a function of time. Figure 1 shows $G(t)$ curves obtained at different temperatures for binders modified with radial SBS (31% styrene content) and with EVA, at 3 and 6% polymer dosages. For comparison purposes, $G(t)$ curves of the base 70/100 bitumen are also reported. As expected, for both polymer types the stiffness of the materials increases as temperature decreases and for a given temperature it increases as the polymer dosage increases. However, substantial differences exist between SBS and EVA binders.

The inclusion of EVA in the base binder enhances the stiffness of the material with minor changes in creep rate, as shown by modulus curves that vertically shift in the log-log plane as the polymer concentration increases from 3 to 6%.

Moreover, the stiffening effect due to the high EVA content appears to be more significant at 40°C, whereas at 60 and 80°C the gap in response between 3 and 6% EVA becomes smaller. This indicates that when temperature is sufficiently low, the role played by the tough structure of the plastomeric skeleton created by EVA is predominant, while as temperature is raised, a reduction of bond strength between polymer chains may occur, causing the macromolecules to slip within the bituminous matrix, thus resulting in a limited stiffening contribution.

In the case of SBS-modified binders, the use of 3% polymer qualitatively produces effects which are similar to those observed for EVA. In fact, shear modulus curves obtained at different temperatures are almost parallel to those of the base bitumen, thus suggesting that the amount of added elastomer may not be sufficient to create a continuous network. Behavior of the binder containing 6% of the same modifier is significantly different: shear modulus is clearly increased and creep rate reduced, as indicated by the presence of an almost horizontal plateau over a loading time interval which depends upon temperature. This typical rubber-like response is governed by the cross-linked polymer structure created within the bituminous matrix by radial SBS when the higher dosage is employed. The response gap between 3 and 6% SBS increases with loading time and temperature, when occurrence of rutting is more relevant.

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Considerable differences in creep response can also be highlighted when comparing binders containing SBS polymers with different composition and structure (styrene content and molecular configuration). This is shown in Figure 2, which displays $G(t)$ master curves, plotted as a function of reduced time at 60°C, of heavily-modified binders (with 6% polymer). For comparison purposes, data obtained for the EVA binder are also included.

It can be observed that binders containing the two polymers with high styrene content (30 and 31%, linear and radial) exhibit the stiffest response, with a very similar behavior in the range of reduced loading times considered in this investigation (i.e. curves overlap almost perfectly). Three distinct stages in creep response can be identified for these two materials: a first stage characterized by a linear trend of $G(t)$ in the log-log scale, a second stage in which the slope of the curve is significantly reduced (the “plateau region”) and a third stage in which the slope of the curve increases again following a linear trend. The first stage reflects the elongation of poly-butadiene chains under constant applied stress; the plateau region highlights the response of the elongated polymer network for which strain rate is mostly governed by the stiff polystyrene domains; the third stage reveals that at high loading times the strength of bonds between styrene blocks diminishes and the material begins to flow.

Three distinct stages of creep response are observed also in the case of the binder modified with low styrene content SBS (22%, radial structure). However, slope changes of the $G(t)$ curve are less pronounced due to the lower effectiveness of styrene domains which lead to a less structured network.

In the case of the EVA binder, no plateau region is observed. Trend of the $G(t)$ curve is approximately bi-linear, since the stiffening effect of plastomer is partially lost when a certain threshold loading time (or test temperature) is reached. As reported in literature, this can be related to the crystallization temperature of EVA, which generally falls within the 50-80°C range (de Bondt and Rooijen 2004).

Permanent Compliance

As previously mentioned, one of the main advantages offered by creep-recovery tests is that they allow the permanent component of accumulated strain to be directly measured. Thus, data recorded in the recovery phase were analyzed by

1 focusing on permanent compliance (J_P), obtained by dividing residual strain (after
2 900 s) by the stress applied during creep loading. Such a parameter was used to
3 compare and rank the binders with respect to their rutting potential. Even though
4 in the investigation a single value of applied stress was considered, it should be
5 mentioned that for the purpose of analysis J_P is generally preferred to residual
6 strain since it can take into account possible stress level variations.
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10 The plot of J_P as a function of temperature for all the unaged modified binders
11 (and for the reference bitumen) is shown in Figure 3. As expected, in all cases
12 polymer modification improves the resistance to permanent deformation (i.e. J_P is
13 reduced). However, experimental results are extremely sensible to factors
14 considered in the investigation such as polymer type, composition, molecular
15 structure and dosage.
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18 The highly-modified SBS binders (with 6% dosage) are clearly distinguished from
19 all the other binders, exhibiting a higher resistance to permanent deformation (i.e.
20 lower J_P values) over the whole range of testing temperatures. Plotted data also
21 highlight differences in behavior due to composition, since binders containing
22 SBS with high styrene content (30 and 31%, linear and radial) show similar J_P
23 values (with a slight behavior improvement in the case of radial polymer),
24 definitely lower than those of the binder containing low styrene content SBS.
25 These observations are coherent with those already made when considering the
26 creep loading phase of performed tests.
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29 In the case of the binder with 6% EVA, rutting resistance as described by J_P is
30 comparable to that of highly-modified SBS binders only at 40°C, whereas at
31 higher temperatures, as expected, it is very similar to that of binders with the
32 lower polymer content (equal to 3%). For such binders no clear distinction can be
33 made between J_P curves, thus revealing a similar anti-rutting potential. Only in the
34 case of the linear SBS a slight performance improvement can be noticed in the
35 entire temperature range.
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38 Since J_P values can be significantly dependent upon the response of binders in the
39 creep phase of testing, for a complete comparison between different materials it is
40 important to consider also the ratio between permanent compliance and total
41 compliance (J_{TOT}) evaluated immediately before load removal. In the case of
42 constant stress applied to all binders, this coincides with the ratio between
43 measured strains.
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1 Values of J_P/J_{TOT} calculated for all unaged modified binders are listed in Table 3.
2 These data show that rankings established on the basis of J_P only (Figure 3) are
3 confirmed. Binders with a high dosage of high styrene content SBS are the most
4 resilient, exhibiting a capability to recover a relative amount of total deformation
5 of the order of 85-90% at 40°C and of 10-15% at 80°C. This capability is
6 gradually reduced when considering low styrene content SBS and EVA. Data
7 provided in Table 3 also indicate that materials with 3% polymer show a limited
8 elastic behavior only at 40°C, with negligible recovery at higher temperatures.
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15 **Effect of Aging**

17 It is well recognized that oxidative aging is a process which may have substantial
18 effects on the physical and mechanical properties of bituminous binders (Tuffour
19 1989; Lu and Isacsson 2002). In the case of polymer-modified materials, two
20 parallel phenomena occur: the base bitumen hardens mainly due to the increase in
21 asphaltenes, while the added polymer may reduce its effectiveness as a result of
22 degradation, which consists in a reduction of the length of the long-chain
23 molecules and in partial separation of existing domains (Lu and Isacsson 1998;
24 Zhang et al. 2010). Thus, it has been reported that aging may significantly reduce
25 the benefits due to polymer modification which are observed in the unaged state
26 (i.e. reduction of temperature susceptibility, complex modulus increase and phase
27 angle decrease at high temperatures or low loading frequencies) (Tuffour et al.
28 1989).
29

30 With respect to the assessment of rutting resistance, it is generally accepted that
31 laboratory investigations on binders should be carried out on samples both in the
32 unaged state and in the short-term aged state, which corresponds to conditions
33 reached as a result of hot mix asphalt production, transport and laying. This is also
34 the approach adopted by SUPERPAVE specifications, which require the
35 identification of the critical rutting temperature by referring to two different
36 threshold values of relevant rheological parameters (Harrigan et al. 1994,
37 AASHTO MP 19-10 2010). Thus, in the experimental investigation described in
38 this paper the same tests carried out on unaged binders were repeated on samples
39 subjected to simulative short-term aging by means of the Rolling Thin-Film Oven
40 Test (RTFOT).
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As shown in Table 4, the impact of aging on the anti-rutting potential of the considered polymer-modified binders was assessed by comparing permanent compliance calculated before ($J_{P,U}$) and after RTFOT ($J_{P,A}$) and by calculating the corresponding aging index ($AI_{Jp} = J_{P,U} / J_{P,A}$). With the exception of heavily-modified SBS binders, for all materials J_P decreases after RTFOT at all test temperatures (i.e. $AI_{Jp} > 1$). This is mainly due to the reduction of the total deformation at the end of the creep phase caused by hardening of the base bitumen. However, the effects of aging on the efficiency of the polymer structure are difficult to evaluate since J_P/J_{TOT} values (Table 5) do not change significantly and do not follow a clear pattern (i.e. they increase or decrease depending upon binder-temperature combination).

In the case of heavily-modified SBS binders, polymer damage is more likely to occur during RTFOT treatment given the higher amount of modifier incorporated within the bitumen and subjected to oxidation. Thus, observed J_P changes after RTFOT depend upon test temperature. At 40°C J_P always increases ($AI_{Jp} < 1$), thus revealing the fact that degradation of the polymer network, reflected by a significant increase of J_P/J_{TOT} , is not completely compensated by hardening of the base bitumen. At 80°C, where the components of viscous flow are predominant, the opposite phenomenon occurs: as a result of the combined effect of hardening and degradation, J_P values tend to decrease with aging ($AI_{Jp} > 1$). At 60°C the occurrence of either phenomenon depends upon polymer type (i.e. J_P either increases or decreases).

In conclusion, since the combination of the above described aging phenomena may differ depending upon polymer type and dosage, relative rankings of the considered binders may change as a result of RTFOT treatment and of the selected test temperature. However, the overall ranking of the polymer modified binders is not influenced by aging and temperature since heavily-modified SBS binders continue to show a superior anti-rutting potential, followed by binders containing 3% SBS and EVA.

CONCLUSIONS

The experimental investigation described in this paper showed that the proposed approach for the evaluation of the anti-rutting potential of polymer-modified binders, based on creep-recovery shear tests, is extremely valuable. In particular,

1 creep-recovery tests are not only simple and quick to perform, but yield results
2 which are sensible to factors of premium importance in binder modification such
3 as polymer type, composition, molecular configuration and dosage. Moreover, by
4 means of these tests, effects related to temperature-dependency and aging can also
5 be captured.
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9 Analysis of test results was carried out by referring to shear modulus $G(t)$ curves
10 derived from the loading phases of testing and to permanent compliance (J_P) and
11 compliance ratios J_P/J_{TOT} (permanent over total) calculated from data recorded in
12 the recovery portion of test runs. It was thus observed that for the specific
13 materials considered in the study, highly-modified SBS binders (with 6%
14 polymer) exhibited by far the greatest anti-rutting potential, especially at higher
15 temperatures and longer loading times. It was also found that styrene content
16 seems to have a prevalent effect on molecular structure and that binders
17 containing EVA have an acceptable resistance to permanent deformation only in
18 the case of the highest dosage. However, for this type of modification benefits
19 may be partly lost at higher test temperatures.
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23 Aging effects were discussed by comparing results obtained before and after
24 RTFOT treatment. It was observed that for the analyzed binders aging may
25 change relative rankings depending upon temperature as a result of the
26 phenomena which alter either the bituminous matrix or polymer structure.
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30 In order to further validate the proposed experimental approach and test method,
31 additional studies will be carried out to expand the available database and to
32 directly link binder evaluation to asphalt mixture testing. It is also felt that a major
33 contribution to future research may derive from supplementary investigations
34 carried out for the direct assessment of the microstructure of binders.
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36 REFERENCES

- 37
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- AASHTO T240-09 (2009) Effect of heat and air on a moving film of asphalt binder (Rolling Thin-Film Oven Test), Association of American State Highway and Transportation Officials, Washington, D.C.
- AASHTO MP 19-10 (2010) Performance-graded asphalt binder using multiple stress creep recovery (MSCR) test., Association of American State Highway and Transportation Officials, Washington, D.C.
- Autostrade SpA (2000) Performance-based technical specifications for pavements. Rome

1 de Bondt AH, van Rooijen RC (2004) Bitumen performance in pavements under extreme climatic
2 conditions. In: Proceedings of the 2nd Symposium of Bitumen and Asphalt, Tehran, Iran
3 Baglieri O (2004) Rheology of modified bituminous binders in the creep mode. Proposal of a new
4 experimental methodology for performance characterisation. Ph.D. thesis, University of Ancona,
5 Italy
6
7 Bahia HU, Hanson DI, Zeng M, Zhai H, Khatri MA, Anderson RM (2001) Characterization of
8 modified asphalt binders in Superpave mix design. NCHRP Report 459, National Cooperative
9 Highway Research Program, Transportation Research Board, National Research Council,
10 Washington, D.C.
11
12 Brulé B, Brion Y, Tanguy A (1988) Paving asphalt polymer blends: relationships between
13 composition, structure and properties. *J Ass Asphalt Paving Technol* 57: 41-64
14
15 Collins JH, BouldinMG, Gelles R, Berker A (1991) Improved performance of paving asphalts by
16 polymer modification. *J Ass Asphalt Paving Technol* 60: 43-79
17
18 D'Angelo J, Dongre R (2002) Superpave binder specifications and their performance relationship
19 to modified binders. In: Proceedings of the Canadian Technical Asphalt Association Annual
20 Meeting, Calgary, Canada
21
22 D'Angelo J, Kluttz R, Dongré R, Stephens K, Zanzotto L (2007) Revision of the Superpave high
23 temperature binder specification: the multiple stress creep recovery test. *J Ass Asphalt Paving*
24 *Technol* 76:123–162
25
26 Delgadillo R, Bahia HU (2010) The relationship between nonlinearity of asphalt binders and
27 asphalt mixture permanent deformation. *Road Mat Pav Des* 11(3): 653-680
28
29 Desmazes C, Lecomte M, Leseur D, Phillips MA (2002) A Protocol for reliable measurement of
30 zero shear viscosity in order to evaluate the anti rutting performance of binders. In: Proceeding of
31 2nd Euroasphalt and Eurobitume Congress, Barcelona, Spain
32
33 De Visscher J, Vanelstraete A (2004) Practical test methods for measuring the zero shear viscosity
34 of bituminous binders. *Mater Struct* 37: 360–364.
35
36 De Visscher J, Vanelstraete A (2009) Equiviscous temperature based on low shear viscosity:
37 evaluation as binder indicator for rutting and critical discussion of the test procedure. In:
38 Proceeding of 7th international RILEM symposium ATCBM09 on advance testing and
39 characterization of bituminous materials, Athens, Greece
40
41 Ecker A (2001) The application of Iatroscan-technique for analysis of bitumen. *Petrol Coal* 1(1):
42 51-53
43
44 EN 15324 (2008) Bitumen and bituminous binders - Determination of equiviscous temperature
45 based on low shear viscosity using dynamic shear rheometer in low frequency oscillation mode
46
47 EN 15325 (2005) Bitumen and bituminous binders. Determination of zero shear viscosity (ZSV)
48 using a shear stress rheometer in creep mode
49
50 Gahvari F (1997) Effects of thermoplastic block copolymers on rheology of asphalt. *J Mater Civil*
51 *Eng* 9(3): 111-116
52
53 Harrigan ET, Leahy LB, Youtcheff JS (1994) The Superpave mix design system. Manual of
54 specifications, test methods and practices. Report A-379, Strategic Highway Research Program,
55 National Research Council, Washington, D.C.
56
57
58
59
60
61
62
63
64
65

1 King GN, Muncy HW, Proudhomme JB (1986) Polymer Modification: Binder's Effect on Mix
2 Properties. *J Ass Asphalt Paving Technol* 55: 519-540
3 Leroy G (1989) Bitumen analysis by thin layer chromatography (Iatroscan). In: Proceedings of the
4 4th Eurasphalt&Eurobitume Congress, Madrid, Spain
5 Lu X, Isacson U (2002) Effect of ageing on bitumen chemistry and rheology. *Constr Build Mater*
6 16(1): 15-22
7 Lu X, Isacson U (1998) Chemical and rheological evaluation of ageing properties of SBS
8 polymer modified bitumens. *Fuel* 77: 961-972
9 Merusi F, Giuliani F (2011) Intrinsic resistance to non-reversible deformation in modified asphalt
10 binders and its relation with specification criteria. *Constr Build Mater* 25: 3356-3366.
11 Morea F, Agnusdei JO, Zerbino R (2010) Comparison of methods for measuring zero shear
12 viscosity in asphalts. *Mater Struct* 43: 499–507
13 Nellensteyn FJ (1924) The constitution of asphalt. *J Inst Petrol Technol* 10: 311-324
14 Phillips M, Robertus C (1996) Binder rheology and asphaltic pavement permanent deformation,
15 the zero shear viscosity concept. In: Proceeding of 1st Euroasphalt and Eurobitume Congress,
16 Strasbourg, France
17 Santagata E, Baglieri O (2002) Study of the effect of binder rheology on the resistance to
18 permanent deformation of bituminous mixtures. In: Proceedings of the 24th National PIARC
19 Congress, Saint Vincent, Italy (in Italian)
20 Santagata E, Baglieri O (2005) Experimental evaluation of modified bituminous binders for heavy
21 duty applications, In: Proceedings of the 3rd International SIIV Congress, Bari, Italy
22 Santagata E, Baglieri O, Dalmazzo D, Tsantilis L (2009) Rheological and chemical investigation
23 on the damage and healing properties of bituminous binders, *J Ass Asphalt Paving Technol* 78:
24 567-595
25 Stangl K, Jäger A, Lackner R (2007) The effect of styrene-butadiene-styrene modification on the
26 characteristics and performance of bitumen, *Chem Monthly* 138(4): 310-307
27 Sybilski D. (1994) Relationship between absolute viscosity of polymer-modified bitumen and
28 rutting resistance of pavement. *Mater Struct* 27: 110-120
29 Sybilski D. (1996) Zero-shear viscosity of bituminous binder and its relation to bituminous
30 mixtures rutting resistance. *Transport Res Rec* 1535: 15–21
31 Tuffour YW, Ishai I, Craus J (1989) Relating asphalt aging and durability to its compositional
32 changes. *J Ass Asphalt Paving Technol* 58: 163-181
33 Wardlaw RK, Shuler S (1991) Polymer modified asphalt binders. STP 1108, American Society for
34 Testing and Materials, Philadelphia, PA
35 Zhang F, Yu S, Wu S (2010) Effect of ageing on rheological properties of storage-stable SBS
36 sulphur-modified asphalts. *J Hazard Mater* 182: 507-517
37 Zoorob SE, Castro-Gomes JP, Pereira Oliveira LA (2012a) Assessing low shear viscosity as the
38 new bitumen softening point test. *Constr Build Mater* 27: 357-367
39 Zoorob SE, Castro-Gomes JP, Pereira Oliveira LA, O’Connel J (2012b) Investigating the multiple
40 stress creep recovery bitumen characterisation test. *Constr Build Mater* 30: 734-745
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Fig. 1 Shear modulus as a function of time at different test temperatures and polymer percentages:
a.1), a.2) and a.3) radial SBS with high styrene content (SBS-RH); b.1), b.2) and b.3) EVA

Fig. 2 Shear modulus as a function of reduced time of heavily-modified binders at 60°C (6% polymer)

Fig. 3 Permanent compliance as a function of test temperature for all binders

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Table 1 Results of SARA Analysis Performed on the Base Bitumen

Fraction or parameter	Test result
Saturates	8.2%
Aromatics	47.5%
Resins	30.3%
Asphaltenes	14.1%
Asphaltenes/Resins	0.464
Saturates/Aromatics	0.173

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Table 2 Binders Considered in the Investigation

Binder Code	Polymer	Dosage
70/100	-	-
SBS-RL-3	Radial SBS (22% Styrene)	3%
SBS-RL -6		6%
SBS-RH-3	Radial SBS (31% Styrene)	3%
SBS-RH-6		6%
SBS-LH-3	Linear SBS (30% Styrene)	3%
SBS-LH-6		6%
EVA-3	EVA	3%
EVA-6		6%

Table 3 Values of $(J_p/J_{TOT}) \cdot 100$ for the Unaged Modified Binders at Different Test Temperatures

T [°C]	SBS-RL-3	SBS-RL-6	SBS-RH-3	SBS-RH-6	SBS-LH-3	SBS-LH-6	EVA-3	EVA-6
40	89.5	29.2	94.7	13.5	70.0	17.3	99.0	51.0
60	100	65.6	99.9	35.8	98.7	44.1	100	100
80	100	100	100	85.5	100	93.3	100	100

Table 4 Comparison of Permanent Compliance and Aging Index Values Before and After RTFOT

Binder Code	40°C			60°C			80°C		
	$J_{p,U}$ [Pa ⁻¹]	$J_{p,A}$ [Pa ⁻¹]	AI_{jp}	$J_{p,U}$ [Pa ⁻¹]	$J_{p,A}$ [Pa ⁻¹]	AI_{jp}	$J_{p,U}$ [Pa ⁻¹]	$J_{p,A}$ [Pa ⁻¹]	AI_{jp}
70/100	2.09E-01	6.95E-02	3.01	7.70E+00	3.51E+00	2.19	6.64E+01	3.76E+01	1.77
SBS-RL-3	3.34E-02	1.58E-02	2.11	1.92E+00	8.69E-01	2.21	2.45E+01	1.35E+01	1.81
SBS-RL-6	1.22E-03	2.11E-03	0.58	1.69E-02	2.89E-02	0.58	4.19E+00	3.53E+00	1.19
SBS-RH-3	4.49E-02	1.03E-02	4.36	2.08E+00	8.64E-01	2.41	1.97E+01	1.10E+01	1.79
SBS-RH-6	3.08E-04	4.02E-04	0.77	1.71E-03	1.38E-03	1.24	3.19E-02	2.38E-02	1.34
SBS-LH-3	1.70E-02	1.14E-02	1.49	7.75E-01	5.18E-01	1.50	1.50E+01	9.30E+00	1.61
SBS-LH-6	3.52E-04	5.70E-04	0.62	2.77E-03	4.59E-03	0.60	9.48E-02	8.78E-02	1.08
EVA-3	3.88E-02	1.07E-02	3.63	2.78E+00	8.20E-01	3.39	2.40E+01	1.07E+01	2.24
EVA-6	1.84E-03	1.49E-03	1.23	1.85E+00	2.10E-01	8.81	1.79E+01	1.23E+01	1.46

Table 5 Values of $(J_p/J_{TOT}) \cdot 100$ for the RTFOT-Aged Modified Binders at Different Test Temperatures

T [°C]	SBS-RL-3	SBS-RL-6	SBS-RH-3	SBS-RH-6	SBS-LH-3	SBS-LH-6	EVA-3	EVA-6
40	95.1	49.0	91.5	20.2	89.4	24.9	94.8	59.4
60	99.8	71.4	99.8	32.0	98.2	39.9	100	98.6
80	100	100	100	77.5	100	92.3	100	100

Figure 1

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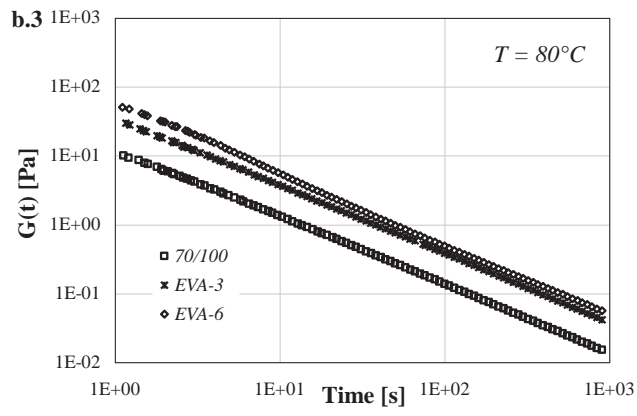
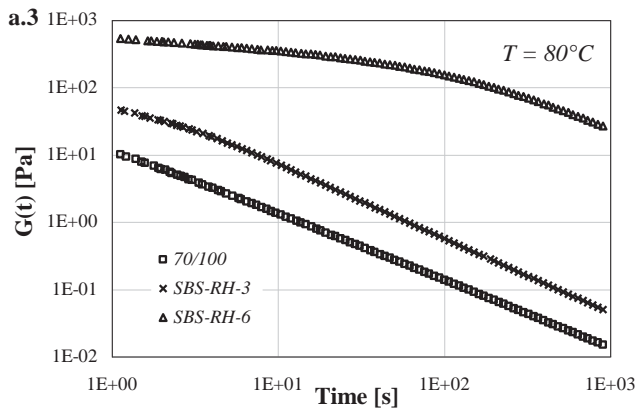
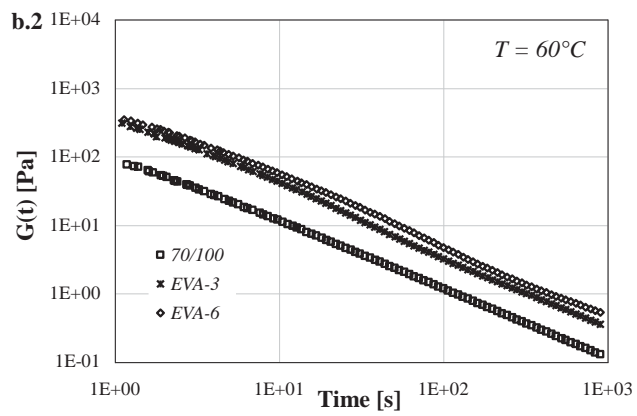
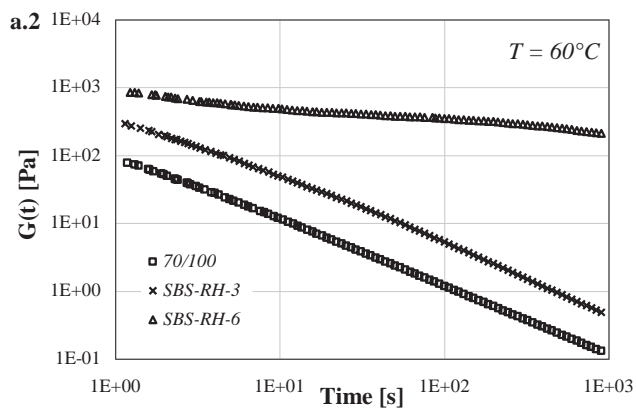
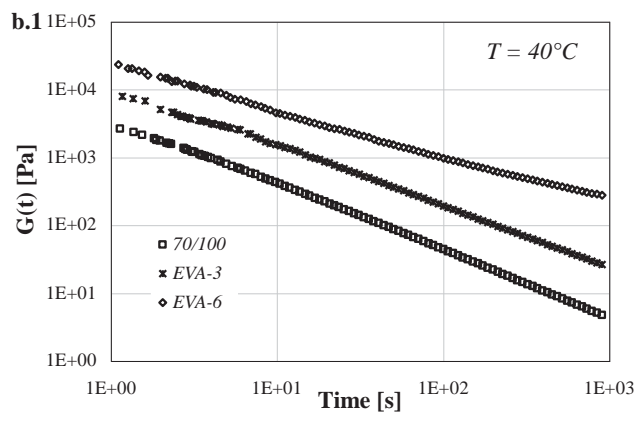
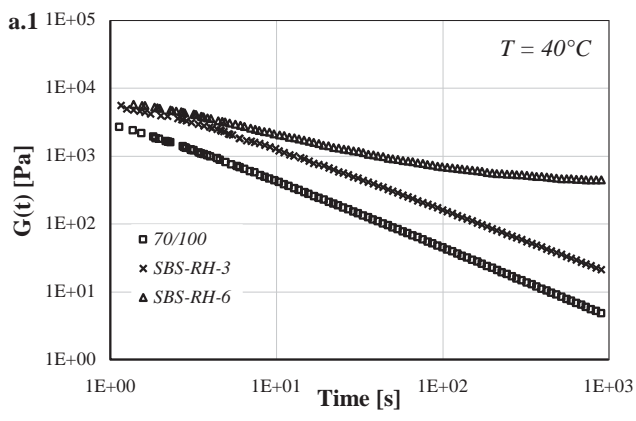
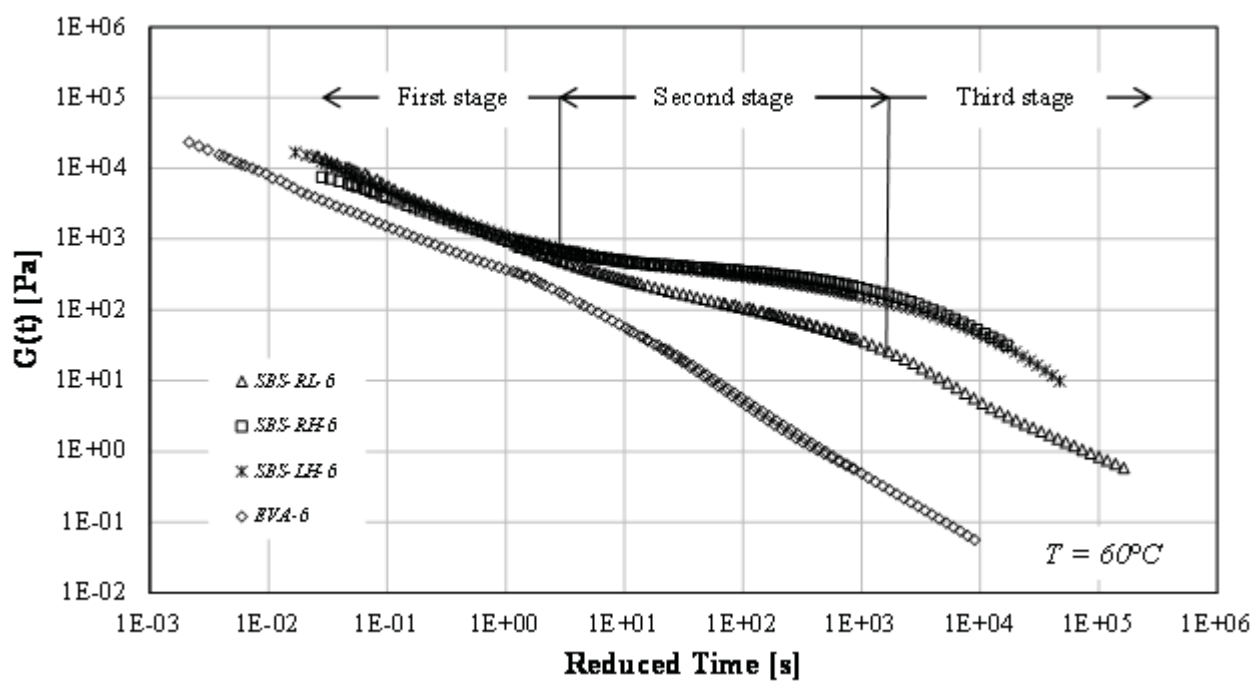


Figure 2

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