Experimental Evaluation of Modified Bituminous Binders for Heavy Duty Applications

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Synopsis

As a result of their superior performance-related properties, modified bituminous binders are increasingly being used in paving applications, especially in the case of pavements subjected to intense traffic flow with a high percentage of heavy vehicles. Experimental procedures and technical specifications used for such binders have to be necessarily based on a rheological approach by means of which their contribution to the overall pavement response can be assessed.

One of the most advanced methods currently adopted in Italy for the characterization and acceptance of modified bituminous binders for heavy duty applications has been developed by *Autostrade per l'Italia S.p.A.*, the Road Agency which manages and maintains most of the Italian motorway network. Technical specifications, used for quality control purposes during construction and maintenance, have recently been integrated by a preliminary qualification system which involves a more detailed evaluation of the binders by means of empirical tests, rheological measurements and tests carried out on a reference bituminous mixture.

In this paper the Authors present the results of an experimental investigation which was carried out with the purpose of evaluating the rheological properties of four different modified binders which were obtained from the use of three different SBS elastomers and one EVA plastomer. These were added to a single base bitumen in the same percentage (6%), typical of heavy duty paving applications. The experimental program included most of the laboratory tests indicated in the *S.A.* acceptance system: however, additional investigations were carried out in order to gather further information related to the expected performance of the binders.

Experimental results indicate that the rheological behaviour of modified binders is strongly dependent upon polymer type. This was observed by analysing the results obtained in all testing configurations (cone-plate shear and beam bending) and modes (oscillatory and creep) in a wide range of temperatures and loading times (or frequencies). The effects associated to the tough and rigid EVA matrix were highlighted at intermediate to low temperatures especially when loads were applied for short time durations and/or high frequencies: in such conditions these binders exhibited a stiffer and more elastic response than SBS-modified materials. This was observed both in rheological tests and in tests carried out on reference mixtures. As expected, plastomer stiffening effects were observed to be greatly reduced at higher temperatures and/or when approaching flow conditions, where binders containing SBS elastomers proved to be stiffer and more elastic, with a greater potential in contributing to permanent deformation resistance.

Further differences in the response of the plastomer and elastomer modified binders were detected by analysing Black diagrams and creep-recovery curves. It was shown that different shapes are associated to different mechanisms with which the polymer matrix contributes to the overall response of the binder. It was thus proven that the use of these graphical representations can be useful in the interpretation of visco-elastic data.

Test results were also analysed in order to evaluate the effects caused by variations of the composition (styrene content) and structure (radial or linear) of the SBS polymer. In most testing conditions increases of the stiffness and elasticity of the binder were associated, especially in the intermediate to high temperature range, to increases of the styrene content, which affects the volume of the corresponding rigid domains created within the rubbery polymer network.

The results obtained in the experimental investigation allowed the Authors to critically analyse the contents of the *S.A.* preliminary qualification system. It was concluded that the system requires to be carefully fine-tuned in order to make set requirements fully compatible with each other or, as an alternative, in order to establish a hierarchy among them. Improvements to the system may stem, in the future, from the introduction of additional rheological tests such as those described in this paper, from the analysis of a wide database of test results, from the harmonization of the specification approach with other validated systems (such as SUPERPAVE) and from the observation of actual field performance.

Experimental Evaluation of Modified Bituminous Binders for Heavy Duty Applications

Polymer-modified binders are becoming increasingly popular in paving applications due to their superior performance-related properties. The use of these innovative materials is one of the preferred design options in the case of heavy duty pavements, which are subjected to intense traffic flow, with a high percentage of heavy vehicles. In such conditions, by selecting appropriate polymer-bitumen combinations, the rheological properties of the binder may be tailored to suit specific needs, thus reducing the risk of pavement distress.

Experimental procedures and technical specifications used for the characterization of conventional unmodified binders are not suitable for modified binders which need to be selected and analysed by means of a rheological approach. Only by referring to visco-elastic properties, evaluated in defined temperature and loading time conditions, a complete description of the behaviour of these materials can be made and their contribution to pavement performance can be assessed. As an example, in the SUPERPAVE system, measurements are carried out with two different rheometers depending upon test temperature, and the resulting grading of bituminous binders, which may contain polymers, is based on fundamental quantities (complex modulus, phase angle, creep stiffness, m-value, strain at failure) [1].

In Italy, the use of modified binders dates back to the ‘70s, when the first polymers were employed as bitumen modifiers during maintenance works carried out on the major motorway network [2]. Since then, technological innovations have progressively taken place with the development of modifying techniques of increasing reliability. At the same time, technical specifications have evolved by gradually including test procedures specifically devised to take into account the peculiar properties of polymer-modified binders. Such technical improvements and commercial availability have spurred the widespread use of modifiers, which has increased in time and has been extended t infrastructures of various categories for the enhancement of pavement life and for the design of function-specific bituminous mixtures.

One of the most advanced methods currently adopted in Italy for the characterization and acceptance of modified bituminous binders for heavy duty applications has been developed by *Autostrade per l’Italia S.p.A.* (previously denominated *Società Autostrade* and therefore indicated as *S.A.* in the rest of this paper), the Road Agency which manages and maintains most of the Italian motorway network. Technical specifications [3] used for quality-control purposes during construction and maintenance are essentially based on requirements set on the results obtained from empirical tests, which are also used to address key issues such as storage stability and short-term ageing. Rheological measurements are carried out at a single test temperature (50°C) in the oscillatory mode with the purpose of evaluating fatigue resistance by means of the loss modulus $G^*\cdot\sin\delta$.

In order to select modified binders prior to their use, a further preliminary qualification system has been laid out [4]. Such a system, which may serve as a design guideline for candidate suppliers, involves a more detailed evaluation of the binders by means of empirical tests, rheological measurements and tests carried out on a reference bituminous mixture.

In this paper the Authors present the results of an experimental investigation which was carried out with the purpose of evaluating the rheological properties of different modified binders typically employed in heavy duty applications. The discussion is focused on the identification of the performance potential of each material and on the comparison between polymers which differ in type, composition and structure. Moreover, a critical analysis of the abovementioned qualification system is carried out in order to assess its reliability and its practical implications.

PRELIMINARY QUALIFICATION SYSTEM FOR HEAVY DUTY MODIFIED BINDERS

The preliminary qualification system adopted by *Autostrade per l’Italia S.p.A.* for the acceptance of heavy duty modified binders is based on a combination of laboratory tests, listed in Table 1, which are carried out both on the binder and on a reference bituminous mixture, appropriately designed in order to emphasize the role played by the binder in mix response [4]. It can be observed that, while empirical and viscosity tests refer to well-known standardized protocols, rheological measurements (Reodin), Indirect tensile strength measurements and the unconventional LaBer test have to be performed according to testing procedures which have been specifically devised by *S.A.* researchers and technicians with the purpose of highlighting the peculiar characteristics of polymer modifiers and their contribution to the improvement of the performance of bituminous binders. A brief description of these testing procedures is given in the following sections.

Table 1: Tests included in the *S.A.* preliminary qualification system

Test method	Test standard
Penetration @ 25°C	CNR 24/71 – EN 1426
Ring & Ball softening point	CNR 35/73 – EN 1427
Elastic recovery @ 25°C	CNR 44/74 mod.EN 13398-99
Tube test	EN 13399-99
Viscosity	EN 13702-2
LaBer test	Internal protocol
Reodin test	Internal protocol
Indirect tensile strength (on reference mixture)	Internal protocol

LaBer Test

As indicated in the *S.A.* qualification system, the purpose of the LaBer test is to evaluate, by means of a single procedure, three key properties of the binder: compliance, tensile strength and its adhesion to aggregates. The test is carried out by extracting, in controlled temperature conditions and with a prescribed rate of displacement, a metallic sphere of known characteristics from the binder sample in which it is enclosed.

The parameters determined from the load-displacement curve recorded during testing (Figure 1), are the following:

- peak load and corresponding displacement, which identify the failure conditions of the system;
- displacement corresponding to 1/3 of the peak load, evaluated on the descending portion of the load-displacement diagram.

At the time of the experimental investigation described in this paper, *S.A.* technical specifications did not indicate acceptance limits for LaBer test results since an adequate database on employed binders had not been created yet. For this reason and due to the unavailability of the testing equipment, these tests were not included in the testing program.

Based on the analysis of the *S.A.* protocol, the performance-related significance of the test appears to be questionable with respect to two fundamental issues. Binder-aggregate adhesion is a typical material-specific property which depends upon physical and chemical interactions: thus, it cannot be evaluated by using a metallic sphere which is not representative of the aggregate particles used in bituminous mixtures. Other criticisms which can be made to the test refer to the stress-strain conditions associated to the process of sphere extraction: they are not modelled to take into account the specific testing geometry and are certainly binder-dependent, thus leading to a comparison of different materials in different conditions. In such a context the test, even though yielding results expressed in fundamental units, should be considered totally empirical.

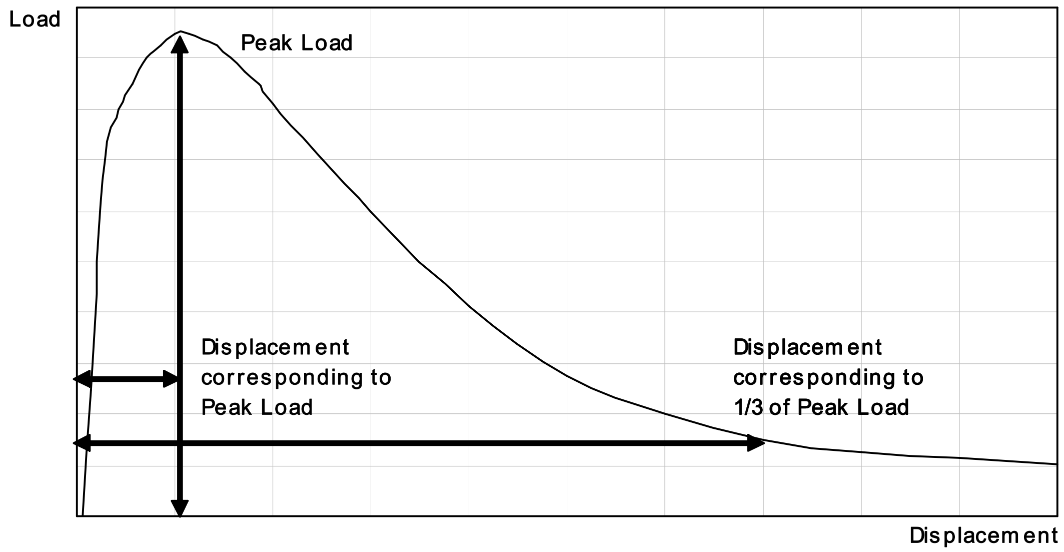

Figure 1: Typical results of a LaBer test

Reodin Test

In order to take into account the visco-elastic time- and temperature-dependent properties of highly modified bituminous binders, the *S.A.* system includes rheological measurements which require the use of a Dynamic Shear Rheometer (DSR). The testing protocol illustrated in the specifications, referred to as “Reodin”, consists in a sequence of measurements performed in the oscillatory mode at a single frequency (ω = 10 rad/s) and in a wide temperature range (from 5 to 85°C, in 5°C increments). No indications are given with respect to the stress (or strain) level to adopt during testing, nor on the preferred test geometry.

Acceptance criteria are defined by referring to the measured values of the complex modulus, G*, and of the phase angle, δ, for which admissible ranges are indicated at each test temperature. As shown in Figure 2, a control chart can thus be obtained by plotting the prescribed minimum and maximum values of G* and δ as a function of temperature.

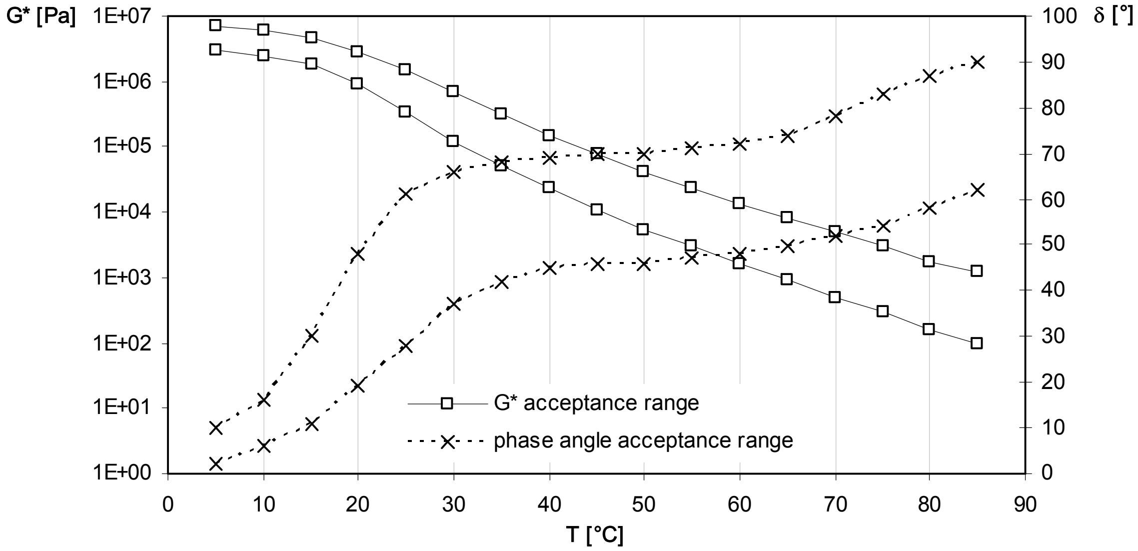

Figure 2: Control chart for the acceptance of modified binders according to the Reodin protocol

Indirect Tensile Strength Test

The indirect tensile strength test is commonly employed for the assessment of bituminous mixtures both in the context of mix design and of quality-control; however, in the *S.A.* system it is used as an indirect method for the evaluation of modified binders. Acceptance criteria are formulated by referring to the results that are obtained on samples of a reference open-graded mixture, the recipe of which is strictly defined by imposing not only binder content (equal to 5.2%), filler type (limestone) and aggregate size distribution (see Figure 3), but also aggregate type (basaltic) and source (2 quarries, indicated ad “Orvieto” and “Biagioli”).

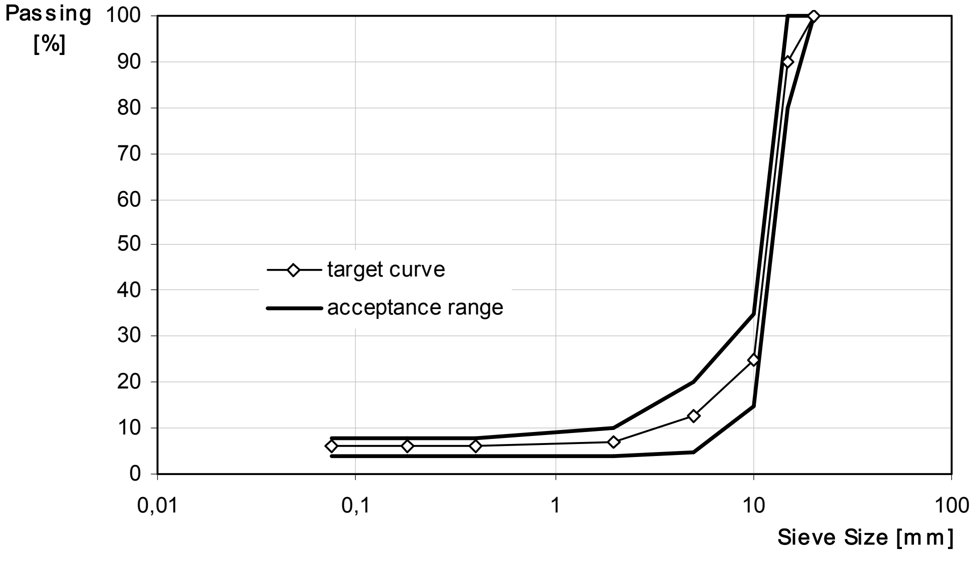

Figure 3: Aggregate size distribution of the reference mixture

For each binder subjected to characterization, cylindrical specimens (100 mm in diameter) of the reference mixture have to be prepared by means of a gyratory shear compactor operated in standard SUPERPAVE conditions (1.25° angle, 600 kPa vertical pressure, 30 gyrations/min rotation speed) with an imposed number of gyrations equal to 130. Regardless of the specific visco-elastic properties of the binder, *S.A.* specifications require the compaction to be always carried out at a temperature of 150°C.

ITS tests are carried out at 25 and 40°C by adopting a constant rate of deformation (0.85 mm/s) and by continuously measuring, until failure, vertical and horizontal deformation and vertical load. From the maximum vertical load P (expressed in N) and the corresponding vertical deformation ΔV_0 (in mm), the indirect tensile strength R_t and the stiffness parameter CTI can be calculated as a function of sample diameter D (in mm) and height h (in mm) by using the following equations:

$$R_t = \frac{2P}{\pi \cdot D \cdot h}$$

$$CTI = \frac{\pi}{2} \cdot R_t \cdot \frac{D}{\Delta V_o}$$

The obtained results, expressed at each temperature as the average of the data obtained from three specimens, can be compared with the acceptance criteria given in Table 2. According to the approach adopted by *S.A.* specifications, by meeting these requirements it is ensured that the bituminous binder is suitable for heavy duty applications. In fact, since the binder is included in a porous aggregate structure in which interlocking effects are negligible, it is subjected to very severe tensile and shear stresses; moreover, in such conditions the global response of the mixture is strongly affected by binder-aggregate interactions.

Table 2: Acceptance criteria defined for indirect tensile strength test results

Temperature [°C]	R_t [N/mm^2]	CTI [N/mm^2]
25	0.38-0.56	≥ 20
40	≥ 0.10	≥ 8

EXPERIMENTAL PROGRAM

Materials

Four different modified binders were considered in the experimental investigation. They were prepared in the laboratory with a controlled-temperature mechanical mixer by using four different polymers in combination with a single base bitumen. In all cases, coherently with recipes adopted in practice for the production of heavy duty modified binders, a 6% polymer content was employed.

The base binder used in the investigation, a 70/100 pen grade bitumen, was preliminarily subjected to chemical analysis by means of the IATROSCAN equipment which leads to an estimate of the relative quantities of saturates, aromatics, resins and asphaltenes (SARA analysis). The results are reported in Table 3, which also contains, for each of the bitumen fractions, the optimal ranges indicated in *S.A.* specifications for binders to be modified with SBS polymers.

Table 3: Results obtained from SARA analysis

Fraction or parameter	Test results	Optimal
Saturates	8,2%	< 15%
Aromatics	47,5%	> 50%
Resins	30,3%	> 20%
Asphaltenes	14,1%	8-13%
Colloidal Instability Index	0,286	-
Coefficient of dispersion	3,492	-
Asphaltenes/Resins Ratio	0,464	-
Saturates/Aromatics Ratio	0,173	-

The polymers employed for the preparation of the modified binders are listed in Table 4. They were selected in order to assess the effects caused by variations of polymer type (SBS vs. EVA), composition (variable styrene content in radial SBS) and structure (radial vs. linear SBS).

Table 4: Polymers considered in the investigation

Code	Type	Description
SBS-R-L	Elastomer	Radial Styrene-Butadiene-Styrene (22% Styrene)
SBS-R-H	Elastomer	Radial Styrene-Butadiene-Styrene (31% Styrene)
SBS-L-H	Elastomer	Linear Styrene-Butadiene-Styrene (30% Styrene)
EVA	Plastomer	Ethylene-Vinyl-Acetate

Reference mixtures were prepared by employing basaltic aggregates which, as illustrated in the previous section, are admitted for use by *S.A.* specifications. Each aggregate fraction was preliminarly subjected to sieve analysis the results of which are given in Table 5. The recipe of the mixture, given in Table 6, was thereafter determined by means of an appropriate optimization process the goals of which were to respect *S.A.* limits and to minimize the difference between the design and target curves.

Table 5: Results obtained from the sieve analyses

Sieve size [mm]	10/16 "Biagioli"	10/16 "Orvieto"	5/10 "Biagioli"	0/3 "Orvieto"	Limestone filler
20	100	100			
15	97.3	76.4	100		
10	13.7	4.4	96.0		
5	0.3	0.6	7.1	100	
2	0.2	0.5	0.5	96.5	
0.4	0.1	0.5	0.1	47.6	100
0.18	0.1	0.5	0.1	30.6	97.8
0.075	0.1	0.4	0.1	18.3	81.1

Table 6: Recipe adopted for the reference mixture

	10/16 "Biagioli"	10/16 "Orvieto"	5/10 "Biagioli"	0/3 "Orvieto"	Limestone filler
Percent by weight in the mixture [%]	31	51	9	4	5

Methods

The experimental program included all the laboratory tests indicated in the *S.A.* acceptance system with the only exception of the LaBer method. Additional investigations were carried out in order to gather further information related to the expected performance of the binders: thus, supplementary rheological tests were carried out on the binders and the reference mixtures prepared with each of them were also subjected to adequate characterization for a direct evaluation of workability and stiffness.

Depending upon test temperature, rheological measurements were performed by making use of a Dynamic Shear Rheometer (DSR) or of a Bending Beam Rheometer (BBR). The employed DSR is a high-resolution controlled-stress rheometer equipped with electrical resistances which in combination with a circulating cooling fluid allow temperature to be controlled with an accuracy of at least 0.1°C. The BBR equipment is of the thermoelectric type, perfectly complying to SUPERPAVE requirements.

DSR testing was carried out adopting the cone-plate geometry (35 mm diameter, 4° angle) which induces in the test samples a uniform level of strain. In the intermediate to high temperature range (40-80°C) tests were carried out in the oscillatory mode by means of frequency sweeps (in the 0.1 to 10 Hz range, corresponding to 0.0628-62,8 rad/s) with a constant amplitude of the imposed shear stress (equal to 100 Pa). The corresponding estimate of the complex modulus and of the phase angle allowed the Authors to give a description of the viscoelastic properties of the binders and to check, at two different temperatures, their compliance to *S.A.* specifications.

In the same temperature range, the DSR equipment was also used to carry out creep-recovery tests. According to the Authors' experience, this type of test more clearly highlights the effects related to variations of the structure of the polymer matrix which is progressively stretched during loading. Moreover, viscous and elastic components of strain can be directly evaluated by analyzing the data obtained both in the loading and recovery (zero-load) phases. Tests on the four modified binders were performed in the cone-plate configuration, with an applied shear stress of 100 Pa and a total duration of 1800 seconds (900 seconds loading; 900 seconds recovery).

In the low temperature range, between -10 and -30°C, BBR tests were carried out by following the SHRP protocol [1]. Before testing, the binder beams were conditioned for 60 minutes in a constant-temperature bath, set at the test temperature; a constant load of 1 N was then applied and deflections of the center point of the specimen were measured. The resulting data were processed for the evaluation, at different loading times (ranging from 0.5 to 240 s), of visco-elastic parameters S(t) (creep stiffness) and m(t) (slope of the time-stiffness curve represented in the log-log scale).

As proven by laboratory experience and field observations, binder rheology strongly affects the workability of bituminous mixtures [5]. In the case of modified binders the presence of a polymer structure leads to an increase of the viscosity which requires an increase of handling and compaction temperatures in order to obtain a satisfying workability. This specific topic was addressed by subjecting the reference mixtures to compaction tests carried out, by means of a gyratory shear compactor, both at the temperature indicated in *S.A.* specifications (150°C for all mixtures) and at optimal compaction temperatures. For each mixture optimal compaction temperature was determined by considering the temperature-viscosity curve of the corresponding binder obtained from measurements carried out by means of a Brookfield device operated according to the ASTM 4402 protocol. Based on the information obtained from suppliers, for some of the considered binders the optimal temperature values calculated in equi-viscosity conditions had to be conveniently reduced in order to reduce the risk of damaging polymer structure.

The *S.A.* acceptance system requires reference mixtures to be tested in the indirect tensile mode in order to obtain information regarding the failure behaviour of the binder films subjected to severe tensile and shear stresses. However, indirect tensile tests carried out in the repeated load mode for the measurement of elastic stiffness (E) were also included in the experimental program in order to obtain information on the pre-failure behaviour of the binders. Tests were performed by making use of a Nottingham Asphalt Tester which was operated according to the prEN 12697-26 protocol at a temperature of 20°C (5μm target horizontal deformation, 124 ms rise time). Since these tests are non-destructive, the same specimens were used for the determination of indirect tensile strength in quasi-static loading conditions.

RESULTS AND DISCUSSION

Empirical tests

Empirical tests are required in the *S.A.* preliminary qualification system [4] which refers to the acceptance limits set within technical specifications [3]. A summary of the experimental data obtained from empirical characterization tests carried out on the modified binders is reported in Table 7, in which the *S.A.* specification requirements are also shown.

None of the considered binders yields a penetration value which falls within the indicated acceptance range. This is not surprising since previous experience and published literature prove that the use of 6% polymer for the modification of a 70/100 pen grade bitumen can lead to a substantial increase of consistency [6]. The extent to which the binders violate the acceptance range depends upon polymer type (maximum in the case of the EVA polymer, minimum for the radial SBS with high styrene content) but does not give any indication of the relative expected performance of the binders. Due to the empirical character of the test and given the peculiar properties of modified binders, the violation of criteria expressed in terms of penetration cannot be considered sufficient for rejecting the considered materials.

In the case of ring and ball tests, the above described consistency increase was reflected by softening point values which always exceed the lower threshold value indicated in specifications. However, a clear distinction can be made between high styrene content polymers (both radial and linear), which exhibit values above 100°C, and the other two polymers (SBS-R-L and EVA), which give results of the order of 80.

All binders met the requirements defined for elastic recovery and Fraass tests: as expected, while the breaking point values do not differentiate clearly the contribution of the various polymers to low-temperature resistance to cracking, it was observed that the recovery measured on specimens subjected to ductility tests is quite sensitive to polymer type. The use of elastomers leads to an almost full recovery, whereas plastomer modification, which leads to the formation of a stiffer but less elastic polymer matrix, yields lower values (80%).

Table 7: Results obtained from empirical tests

	SBS-R-L	SBS-R-H	SBS-L-H	EVA	Specification
Penetration @ 25°C [dmm]	45	48	46	30	**50-70**
R&B softening point [°C]	86	109	103	77	**≥ 70**
Fraass breaking point [°C]	-16	-13	-15	-14	**≤ -12**
Elastic recovery @ 25°C [%]	98	100	98	80	**≥ 80**

Storage stability test results are given in Table 8, which contains the softening point values measured on the upper and lower portion of the samples subjected to the Tube test procedure (3 days storage at 180°C). The other parameters listed in Table 8, $\Delta R\&B_{upper-lower}$ and $\Delta R\&B_{before-after}$, respectively indicate the difference in softening point which exists between the upper and lower portion, and the softening point increase caused by high temperature storage. The former parameter gives an idea of the degree of non-homogeneity of the binder after treatment, whereas the latter is an indicator of the degree of degradation of the polymer matrix.

Widely different results were obtained for the considered set of binders. Those containing radial SBS proved to be sufficiently homogeneous after the treatment, as shown by the very small $\Delta R\&B_{upper-lower}$ values, with sufficiently contained degradation effects. By considering the use of a linear SBS, very similar results were obtained with respect to homogeneity; however, as a result of its different structure the polymer matrix was severely damaged by the high temperature treatment, with a value of $\Delta R\&B_{before-after}$ greater than 20. Finally, in the case of the binder containing EVA it was observed that storage causes the separation of the two phases, with a value of $\Delta R\&B_{upper-lower}$ equal to 15. For this type of modification it is thus recommended to adopt adequate countermeasures such as the inclusion in the system of stabilizing agents and mechanical mixing during tank storage.

S.A. specifications address the issue of phase separation only, by setting a maximum acceptance limit to the value of $\Delta R\&B_{upper\text{-}lower}$, equal to 3. As shown in Table 8, only the SBS-modified binders met this requirement.

Table 8: Results obtained from Tube tests

	SBS-R-L	SBS-R-H	SBS-L-H	EVA	Specification
$R\&B_{upp}$ [°C]	78,8	100,1	82,9	79	-
$R\&B_{low}$ [°C]	78,7	99	82,5	64	-
$\Delta R\&B_{upper\text{-}lower}$ [°C]	0,1	1,1	0,4	15	≤ 3
$\Delta R\&B_{before\text{-}after}$ [°C]	7,3	9,5	20,3	5,5	-

Viscosity tests

The results obtained from viscosity tests carried out with the Brookfield device in the 150-210°C temperature range are plotted in Figure 4. Flow behaviour, which in all cases is non-Newtonian as a result of the presence of a polymer matrix, is strongly affected by changes in polymer type. In the case of SBS polymers, the factor which plays a dominant role in the enhancement of the viscosity level is the styrene content which is an indicator of the volume of the rigid domains which form within the system. Thus, the highest temperature-viscosity curves are those associated to the use of polymers SBS-R-H and SBS-L-H, the former being more viscous as a result of a more interconnected butadiene rubbery network . As shown in Figure 4, the other elastomer, SBS-R-L, characterized by radial structure but lower styrene content, yields much lower viscosity values, which are close to those which correspond to the use of the only plastomer considered in the investigation (EVA).

As in the case of empirical tests, the *S.A.* qualification system [4] refers to the viscosity requirements set in technical specifications [3]. In particular, an admissible viscosity range, comprised between 0.15 and 0.40 Pa·s, is defined at 160°C. Based on the test results plotted in Figure 4, the binders modified with high styrene content SBS polymers should be rejected.

The usefulness of setting a maximum acceptance limit to viscosity values should not be undermined: in fact, such a limit is often introduced in specifications in order to ensure that the binder can be easily pumped in the mixing plant. However, the use of high polymer contents, necessary in order to meet other requirements which are more directly linked to field performance, inevitably leads to results as those shown in Figure 4, which indicate that pumping should be carried out at a temperature which depends upon the specific flow behaviour of each binder. As a consequence, it would be more realistic to introduce in the acceptance system an upper viscosity limit which should refer to a binder-dependent temperature.

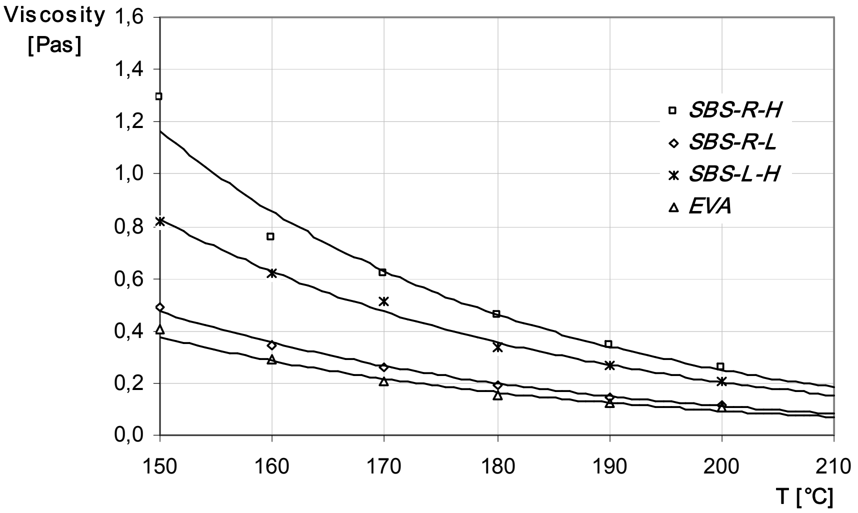

Figure 4: Results obtained from viscosity tests

Rheological tests

As illustrated in the previous section, rheological tests were carried out in various configurations (cone-plate shearing and beam bending) and modes (oscillatory and creep) in order to cover a wide range of temperatures and loading times (or frequencies), thus obtaining the necessary information for a complete description of the visco-elastic behaviour of the binders. This is considered by the Authors as the necessary approach for a performance-related evaluation and comparison of modified binders.

The results obtained in frequency sweep oscillatory tests carried out at 40 and 80°C by using the DSR are shown in Figure 5 and Figure 6, where, for each binder, the average values of complex modulus G* and phase angle δ (derived from two independent tests) have been plotted as a function of frequency.

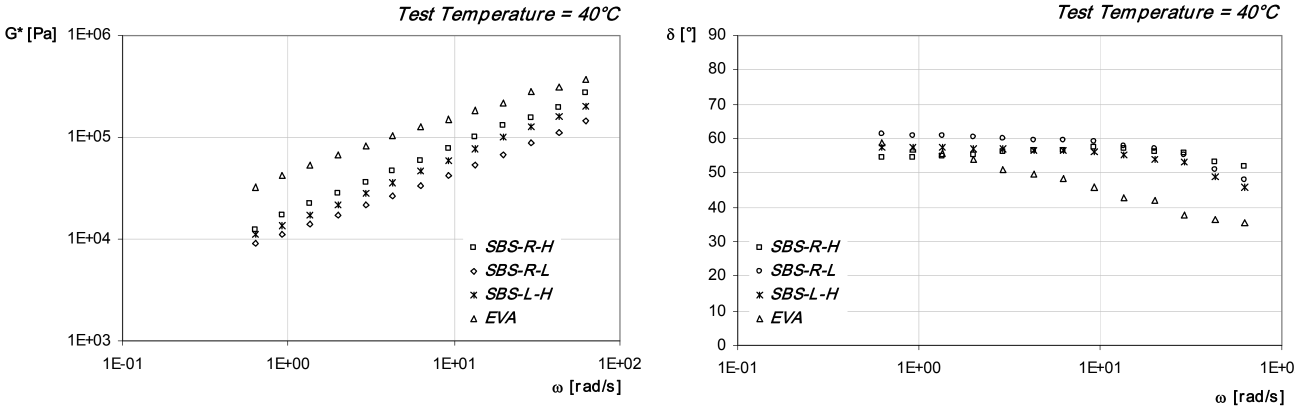

Figure 5: Results obtained from frequency sweep tests at 40°C

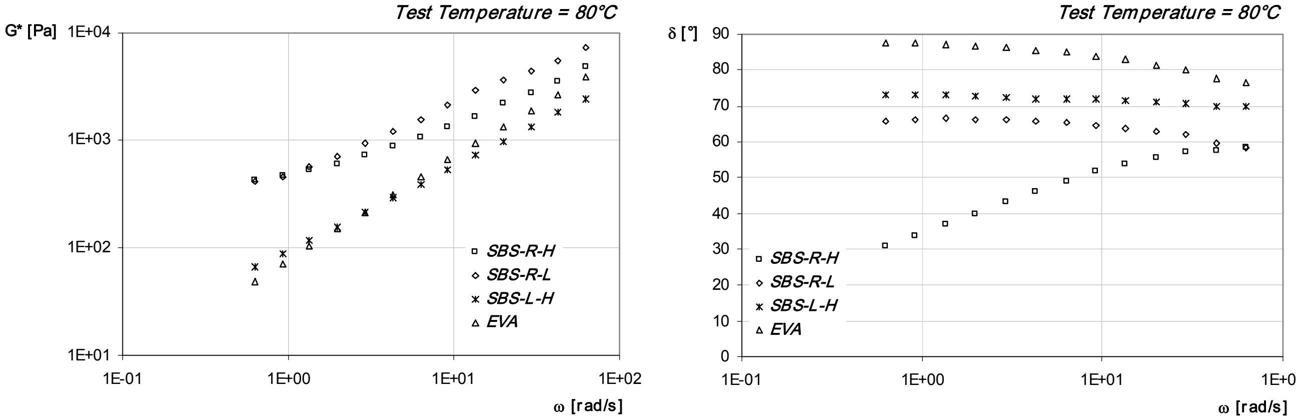

Figure 6: Results obtained from frequency sweep tests at 80°C

It can be observed that the relative effects produced by elastomers and plastomers on the stiffness and elastic response of the base binder depend upon temperature. At 40°C the bitumen modified with EVA exhibits, due to its tough and rigid polymer matrix, the highest G* values and the lowest δ values over the considered range of frequency. On the contrary, when test temperature is raised to 80°C, SBS polymers, especially those with a radial structure (SBS-R-L and SBS-R-H), provide a higher increase both in stiffness and in elastic response. At such a temperature the difference in the rheological effects caused by the two polymer types (elastomer and plastomer) becomes more evident as the frequency is decreased.

As shown in Table 9, if the data measured at 10 rad/s are compared with the requirements set by the *S.A.* specification, only the binder containing polymer SBS-L-H can be considered acceptable. The three remaining binders violate different acceptance ranges: in the case of plastomer-modification violations occur at 40°C, where the binder is considered too stiff (G* exceeding maximum admitted value) and too elastic (δ below the lower admitted value); binders modified with radial SBS have to be rejected due to their rheological response at 80°C where they either exhibit an excessive stiffness (SBS-R-L) or an excessive elasticity (SBS-R-H).

Table 9: Results obtained from frequency sweep tests at 10 rad/s

	G* [kPa] @ 40°C		δ [°] @ 40°C		G* [kPa] @ 80°C		δ [°] @ 80°C	
	Test	*S.A.*	Test	*S.A.*	Test	*S.A.*	Test	*S.A.*
SBS-R-L	43.6	24-150	58.2	45-69	2.27	0.16-1.80	63.9	58-87
SBS-R-H	80.9		57.1		1.40		53.3	
SBS-L-H	62.2		55.4		0.57		71.5	
EVA	153.9		43.3		0.70		83.1	

The conclusions drawn from the direct application of the *S.A.* qualification system require a critical analysis. In fact, the rejection of the radial SBS-modified binders is based on violations which appear to be beneficial with respect to required field performance. Moreover, as shown in Figure 6, in conditions (higher temperatures, shorter loading frequencies or longer loading times) which are typically associated with the possible occurrence of rutting, the use of elastomers both of the linear and radial type, leads to improvements which are greater than those produced by plastomers.

Also the rejection of the EVA-modified binder needs to be thoroughly discussed since the specification criteria may not be fully coherent with performance evaluation principles. In fact, at intermediate temperatures, where the main performance concern is fatigue cracking, the analysis of the visco-elastic properties of paving binders cannot be based on the observation of the $G^*(\omega)$ and $\delta(\omega)$ plots. In this case, derived visco-elastic parameters, such as $G^* \cdot \sin\delta(\omega)$ which gives an idea of energy dissipation and is suggested by the SUPERPAVE system, need to be considered [1]. The corresponding values calculated at 40°C and 10 rad/s for the four binders subjected to investigation are given in Table 10 together with the range derived from the G^* and δ specification limits (see Figure 2). These data highlight the fact that elastomers provide the base binder with a rubbery-like structure in which energy dissipation is lower than that associated with the stiffer EVA matrix. According to this approach, a better fatigue performance is expected for the SBS modified binders; however, derived specification limits lead to an acceptance of the EVA-modified binder.

Table 10: Fatigue parameter $G^* \cdot \sin\delta$ evaluated at 40°C

	$G^* \cdot \sin\delta$ [kPa] @ 10 rad/s	
	Measured	*S.A.* range (derived)
SBS-R-L	37	17-140
SBS-R-H	68	
SBS-L-H	56	
EVA	116	

In the context of a detailed assessment of the visco-elastic behaviour of the modified binders, other interesting information can be drawn from the analysis of the shape of the $\delta(\omega)$ plots given in Figures 5 and 6. In the case of the radial SBS polymers, $\delta(\omega)$ curves do not always decrease monotonically as a function of increasing frequency, and in specific conditions (as in the case of SBS-R-H tested at 80°C) they may exhibit a clear increase. Such an evidence, which is well documented in literature for this type polymer modification [7], is due to the networking effect produced by the rubberized polymeric structure and can be highlighted more clearly by making use of Black diagrams.

For comparison purposes, Black diagrams of the binders containing the high styrene content radial SBS polymer and the EVA plastomer are reported in Figure 7. The shape of the δ-G^* curve obtained for the EVA-modified binder is similar to what is expected for unmodified binders. Since data points corresponding to both test temperatures can be easily fitted with a single, smooth monotonic curve it can be implied that the measured visco-elastic behaviour respects the time-temperature equivalency principle.

In the case of the SBS-modified binder, it is apparent that this principle does not hold: the data obtained at the two temperatures cannot be described by a continuous, smooth curve and the unique correspondence between G^* and δ is lost. The presence of humps in the δ-G^* trends observed at each temperature has already been reported in literature and can be explained by taking into account the actual contribution of the polymer matrix to the overall response of the binder. At each temperature, above a certain G^* value the visco-elastic behaviour of the material is controlled by the base bitumen for which, as expected, stiffness increases are accompanied by a decrease of the phase angle. Below this G^* threshold value, which depends upon a number of factors (temperature, polymer content and type, bitumen), the rubbery polymer network starts to dominate, with a completely opposite rheological behaviour: the phase angle decreases as the complex modulus decreases.

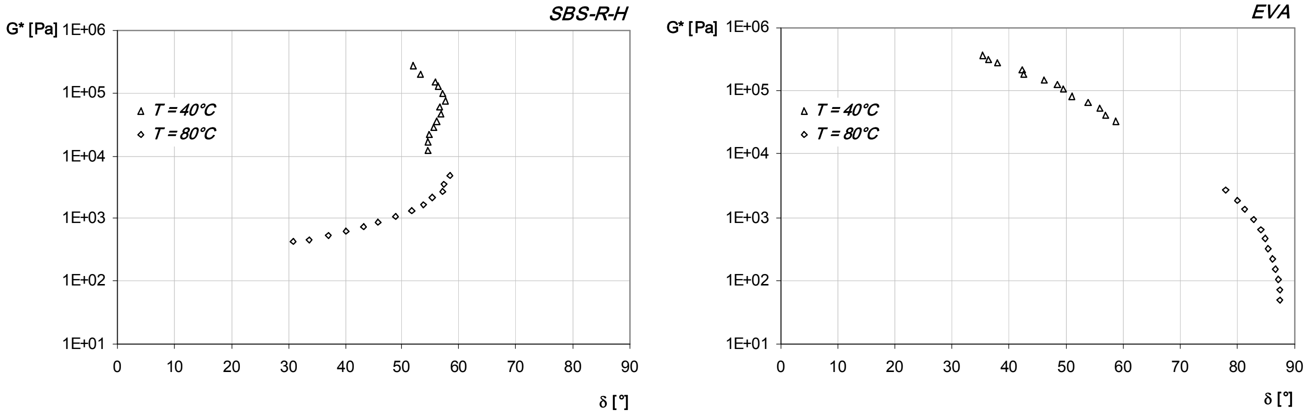

Figure 7: Black diagrams of the binders containing SBS-R-H and EVA

Further information useful for a comparison between the four modified binders at intermediate to high service temperatures can be drawn from the results obtained by means of creep-recovery tests, which can be represented in the time-strain domain as shown in Figure 8. From the first portion of the curve, which corresponds to the (loading) creep phase, the shear stiffness G(t), equal to $\tau_0/\gamma(t)$, can be determined. The data recorded in the (zero-loading) recovery phase, which define the second part of the curve, can be used for the evaluation of the recoverable and permanent compliance (J_R and J_P) which are respectively defined as a function of the recovered and permanent strain (γ_R and γ_P).

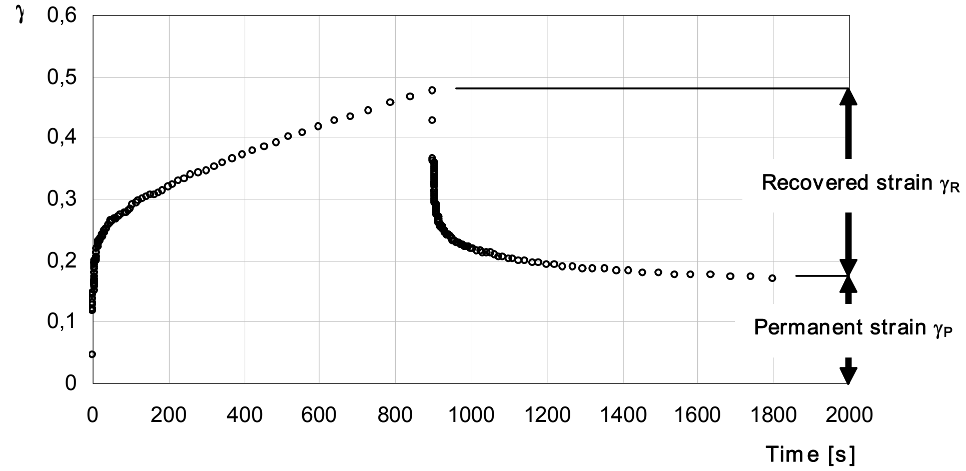

Figure 8: Typical results of a creep-recovery test

In Figure 9 through 11, the G(t) curves obtained at each test temperature are reported for all the binders considered in the investigation. Coherently with the results derived from oscillatory tests, at 40°C and short loading times the plastomer-modified binder is stiffer than all the other elastomer-modified materials; this stiffness ranking is reversed at higher temperatures (60 and 80°C) for all the loading times taken into consideration. In the case of G(t) curves, however, binders containing different polymers can be differentiated more clearly, especially at higher temperatures and longer loading times, as a result of non-negligible slope variations which are observed for some materials.

SBS-modified binders exhibit, in a loading time range which depends upon temperature, an almost horizontal plateau in which rheological behaviour is governed by the polymer; such an effect, which in the oscillatory mode can be detected only by means of Black diagrams, is much more evident for binders containing polymers SBS-R-H and SBS-L-H due to the higher presence of styrene within their molecular structure. When the polymer network is completely stretched under the constant applied stress, the stiff polystyrene domains show their effects and contribute to the response of the system by allowing very smalls strain increments to take place.

The EVA-modified binder considered in the study did not display the same kind of behaviour. The observed trend of the G(t) curve is almost linear in most of the loading time ranges investigated at the various temperatures. Non-negligible slope variations were recorded only at 60°C for short loading durations, when the contribution of the though structure of the plastomeric skeleton was highlighted.

Previous research performed by the Authors proves that the use of creep-recovery test data can be extremely useful in determining the contribution of binders to permanent deformation resistance [8]: in particular, by analysing the recovery portion of the time-strain curve, a number of parameters can be determined by means of adequate data processing and their relationships with mixture stress-strain behaviour can be used with the purpose of selecting and comparing binders on a performance-related basis. However, in this study, which is focused on the critical analysis of the *S.A.* preliminary qualification system and on the comparison of different heavy duty modified binders, recovery data were processed more simply by calculating the percent recovered strain $\%\gamma_R$. Such a parameter is defined as the percent ratio between the strain recovered at the end of the zero-loading phase (γ_R) and the total strain (γ_T) measured at the end of the loading phase (see Figure 8). In principle, $\%\gamma_R$ gives information of the same type of the elastic recovery; however, it is evaluated in controlled stress-strain conditions and is therefore a full rheological parameter. Moreover, the test is carried out at higher temperatures (40-80°C), where viscous components of strain are certainly non-negligible: in such conditions the assessment of the degree of elasticity of the binders has a greater significance since it is more directly related to performance (i.e. to rutting resistance).

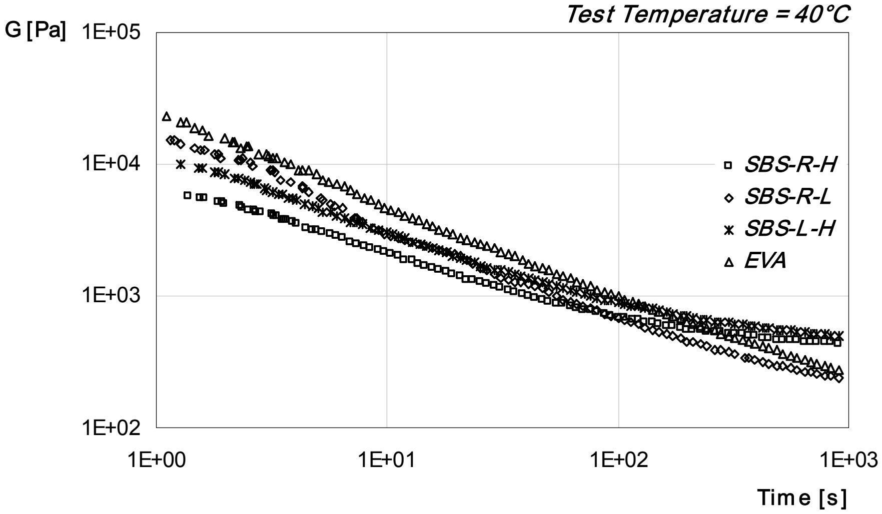

Figure 9: Results obtained from creep tests at 40°C

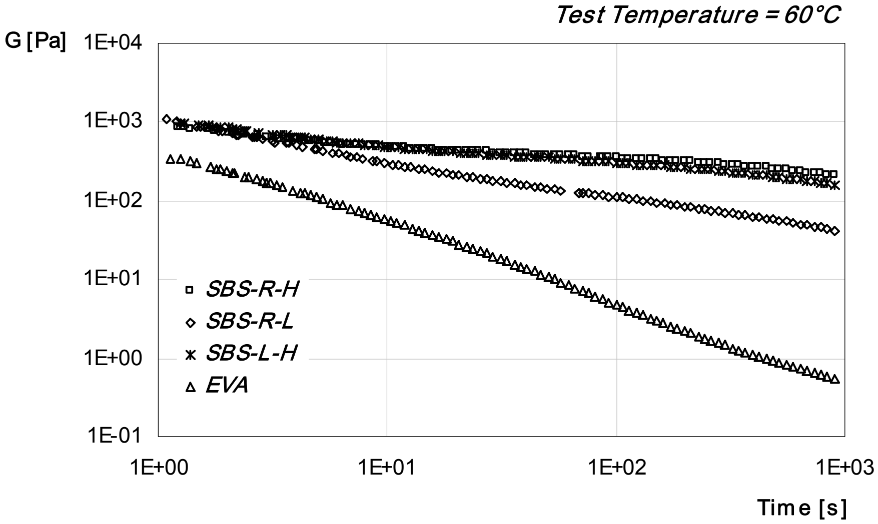

Figure 10: Results obtained from creep tests at 60°C

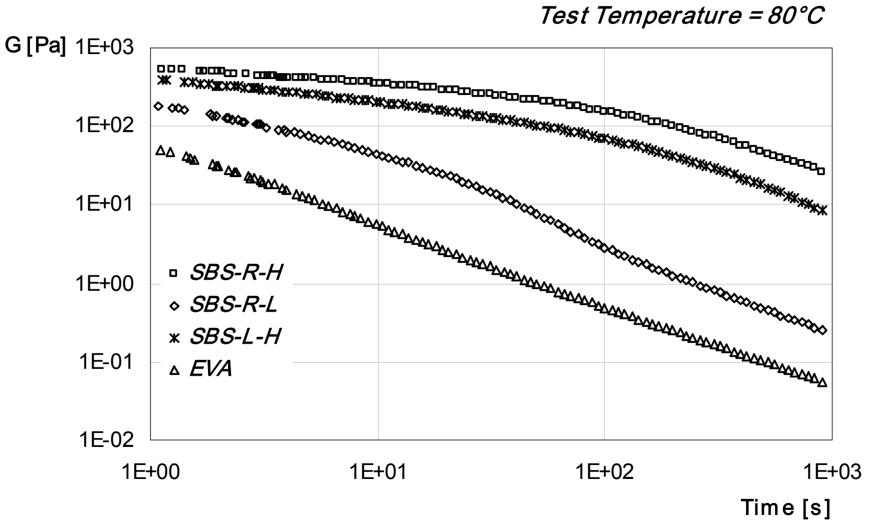

Figure 11: Results obtained from creep tests at 80°C

The average $\%\gamma_R$ values, calculated by considering two independent tests for each binder, are given in Table 11. Results are more sensitive to polymer type (elastomer vs. plastomer) than those derived from ductility tests (see Table 7). Quite obviously, elastomer-modified binders are confirmed to be the most resilient, but it is also noted that a distinction can be made within this group. As proven by the similar behaviour recorded for polymers SBS-R-H and SBS-L-H, which exhibited the greatest values of $\%\gamma_R$, the attitude of SBS-binders to recover after loading primarily depends upon styrene content, the increase of which causes an increase of $\%\gamma_R$. Results also suggest that the type of structure (radial or linear) has a smaller effect, even though a slight recovery increase is observed when passing (for a given level of styrene content) from a linear to a radial structure (i.e. from SBS-L-H to SBS-R-H).

Table 11: Percent recovered strain $\%\gamma_R$

Temperature [°C]	SBS-R-H	SBS-R-L	SBS-L-H	EVA
40	87	71	83	49
60	64	34	56	0
80	14	0	6	0

As illustrated in the previous sections, in *S.A.* specifications the low temperature behaviour of heavy duty modified binders is addressed by means of acceptance limits which are set on the results obtained from empirical Fraass tests. The significance of this type of test in the case of modified binders is questionable since the presence of a polymer matrix may cause problems in the identification of the onset of cracking on the surface of the test samples. This was indirectly confirmed by the results obtained for the four binders considered in this study, which yielded very similar results (Table 7). As a consequence, the binders were subjected to BBR tests which in the low temperature range should highlight the peculiar visco-elastic response associated to each material [9,10].

BBR test results, plotted in Figures 11 through 13 in the loading time - stiffness plane, were coherent with those derived from DSR tests at intermediate to high temperatures. The SBS-modified binders displayed, at all testing temperatures (-10, -20 and -30°C) and loading times, a lower creep stiffness than the EVA-modified binder, characterized by a completely different polymer matrix.

A performance-related comparison between the four binders can be carried out by considering, at each temperature, the parameters which are used in the SUPERPAVE system to prevent the occurrence of low temperature cracking: creep stiffness and m-value at 60 seconds loading time. With respect to the resistance to thermal cracking, lower stiffness values are beneficial since they limit the stresses caused by restrained contraction; moreover, due to the visco-elastic nature of bituminous binders, benefits also derive from greater m-values, which allow a quicker relaxation of thermal stresses to take place, thus reducing the risk of failure. The average values of these parameters are reported in Table 12. Unfortunately, these cannot be compared with SUPERPAVE limiting values which are referred to measurements carried out on binders previously subjected to simulative long-term ageing.

By looking at the S(60s) values, a clear distinction can be made between the resistance to thermal cracking of binders containing elastomers or plastomers; moreover, the three elastomers can be differentiated one from another and can be subjected, if required, to a performance-related stiffness ranking. Further information stems from the analysis of the m(60s) values: it can be observed that the binder containing EVA is not only the stiffest, but also the one with the lowest tendency to relax thermal stresses (i.e. lower m-values), while the distinction between elastomers from this viewpoint is progressively more difficult as temperature is reduced since the behaviour of the binder is increasingly dependent from the response of the bitumen phase.

Table 12: Creep stiffness and m-value at 60 s

	S(60s) [MPa]			m(60s)		
	T = -10°C	T = -20°C	T = -30°C	T = -10°C	T = -20°C	T = -30°C
SBS-R-H	61.4	263.5	758.3	0.44	0.30	0.18
SBS-R-L	73.5	301.0	741.8	0.43	0.28	0.18
SBS-L-H	66.0	287.3	718.3	0.42	0.28	0.18
EVA	102.4	417.4	1063.8	0.41	0.29	0.16

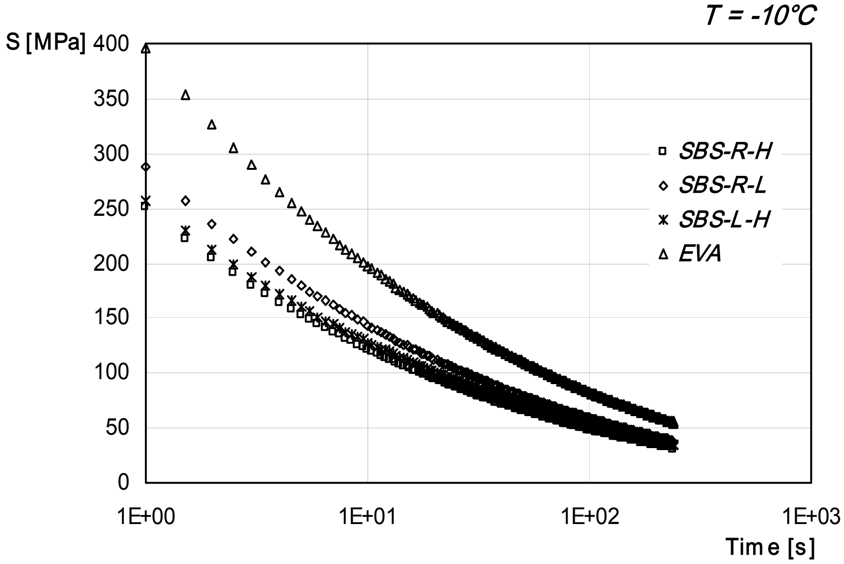

Figure 12: Results obtained from BBR tests at -10°C

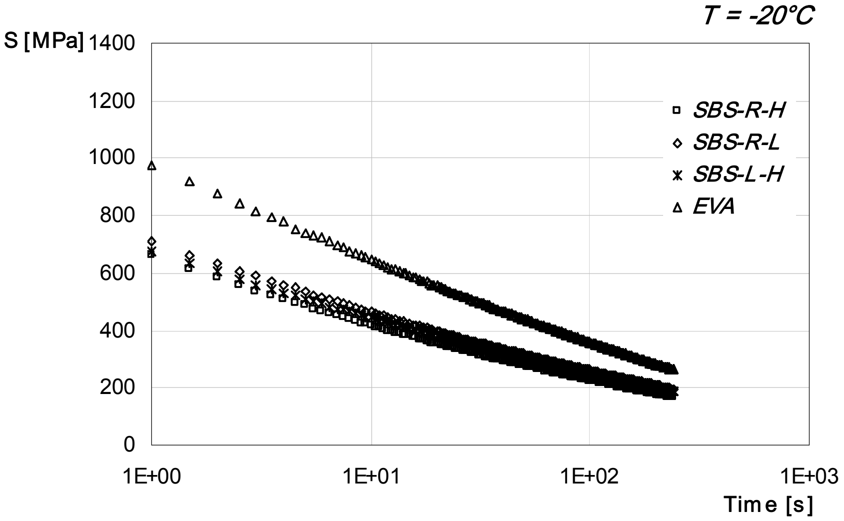

Figure 13: Results obtained from BBR tests at -20°C

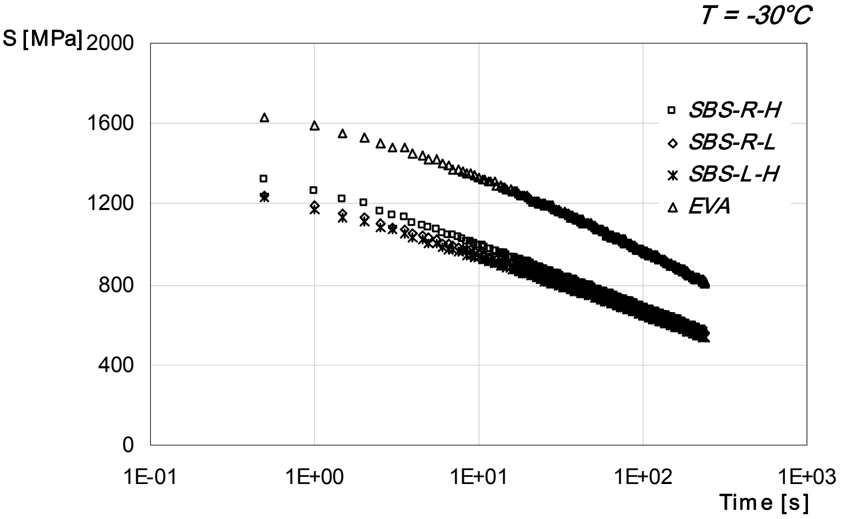

Figure 14: Results obtained from BBR tests at -30°C

Mixture tests

The four reference mixtures were compacted by means of the gyratory shear compactor at a common standard temperature (T_{std}) indicated by *S.A.* specifications and at an optimal compaction temperature (T_{opt}) which was selected according to the previously illustrated viscosity and handling criteria. Subsequent activities included volumetric characterization of the specimens, which required the determination of the theoretical maximum density (TMD) of each mixture, modelling of the compaction curves and mechanical characterization by means of indirect tensile tests carried out at various temperatures in the repeated loading and quasi-static mode. A synthesis of the experimental results obtained in this phase of the study is given in Table 13.

Table 13: Results obtained from mixture tests

	SBS-R-H		SBS-R-L		SBS-L-H		EVA	
Compaction temperature [°C]	150 (T_{std})	195 (T_{opt})	150 (T_{std})	180 (T_{opt})	150 (T_{std})	190 (T_{opt})	150 (T_{std})	180 (T_{opt})
TMD [g/cm^3]	2.459		2.527		2.522		2.507	
Density [g/cm^3] @ 130 gyrations	1.908	1.913	1.917	1.902	1.922	1.927	1.917	1.926
Voids [%] @ 130 gyrations	22.4	22.2	24.2	24.7	23.8	23.6	23.5	23.2
C_1 [%]	61.6	61.8	60.2	61.2	60.9	62.8	61.3	62.9
k	7.39	7.35	7.63	6.91	7.08	6.88	7.25	6.71
E @ 20°C [MPa]	1449	1877	1348	1716	1416	2031	2299	2493
R_t @ 25°C [N/mm^2]	0.358	0.380	0.327	0.335	0.314	0.456	0.259	0.302
CTI @ 25°C [N/mm^2]	91.0	109.5	71.0	106.5	84.2	111.9	86.4	83.0
R_t @ 40°C [N/mm^2]	0.100	0.127	0.121	0.113	0.208	0.136	0.131	0.102
CTI @ 40°C [N/mm^2]	29.9	33.3	45.2	24.1	44.1	25.0	30.8	34.9

As a result of the very high number of gyrations employed for compaction, the average void content values obtained for each mixture were very close to each other, thus proving to be associated to the mixture recipe, and almost independent from binder type and compaction temperature. For three of the considered mixtures, an increase of compaction temperature of 30-45°C caused a reduction of the void content of the order of 0.2%, which is absolutely negligible in the case of open-graded mixtures. For one of the mixtures, containing polymer SBS-R-L, an increase of the void content was observed, which can be attributed to the dispersion of experimental results.

In order to assess whether the binder affects compaction mechanics, compaction curves plotted in the C-N (percent of theoretical maximum density vs. number of gyrations) semi-logarithmic plane were fitted with linear equations. The slope of the regression line, indicated with the symbol k, gives a measure of the workability of the mixture, while parameter C_1, which is the degree of compaction calculated for N = 1, represents the self-compaction of the mixture. As expected, by increasing compaction temperature C_1 tended to increase while k tended to decrease; both parameters are only marginally affected by changes of the polymer type and general conclusions could not be drawn from the recorded data since characteristic trends were not identified.

The results obtained from repeated load indirect tensile tests carried out at 20°C with short loading times (associated to load pulses with a rise time of 124 ms) are consistent with the rheological measurements performed on the binders. The EVA-modified binder exhibits its stiff behaviour even when distributed in thin films within the mixture specimens subjected to pulse loading, thus yielding the maximum E values. Lower elastic stiffness values are associated to the mixtures containing SBS polymers, which yielded approximately equivalent results. It is interesting to note that in the case of SBS-binders the results obtained on the specimens prepared at an optimal compaction temperature (T_{opt}) were in all cases approximately 30% greater than those corresponding to the standard temperature indicated in *S.A.* specifications (T_{std}). This is probably due to the fact that at the optimal compaction temperature a more homogeneous dispersion of the polymer structure within the base bitumen is maintained, thus resulting in a structural strengthening of the porous aggregate skeleton subjected to testing. The same effects were not observed in the case of the EVA-modified binder, for which the percent increase of stiffness was less than 10%, comprised within the scatter of experimental results.

The failure behaviour of the binders subjected to quasi-static loading during the indirect tensile strength test was also coherent with their rheological behaviour. The response measured at 25°C confirmed the fact that the stiffer EVA structure is also more brittle, thus leading to the smallest R_t and CTI values. SBS-modified binders exhibited a greater resistance and ductility as proven by the corresponding higher values of R_t and CTI. Variations of the SBS polymer produced small effects which however suggest that the use of high styrene content polymers leads to an enhancement of both strength and stiffness at failure. As in the case of elastic stiffness, in most cases both R_t and CTI were positively affected by an increase of the compaction temperature.

At 40°C the failure behaviour of the binders was partially shadowed by the response of the aggregate structure of the mixtures which is highlighted as a result of the greater deformations which take place during quasi-static loading. This explains why the four mixtures, regardless of compaction temperature, polymer type, composition or structure, yielded very similar results in terms of both R_t and CTI.

Part of the experimental results obtained on the mixtures can be used to check compliance with *S.A.* specifications. This is shown in Table 14, which contains the R_t and CTI data recorded for specimens compacted at the standard temperature (T_{std}).

Table 14: Results obtained from indirect tensile strength tests

	SBS-R-H	SBS-R-L	SBS-L-H	EVA	Specification
R_t @ 25°C [N/mm^2]	0.358	0.327	0.314	0.259	≥ 0.38
CTI @ 25°C [N/mm^2]	91	71	84.2	86.4	≥ 20
R_t @ 40°C [N/mm^2]	0.100	0.121	0.208	0.131	≥ 0.10
CTI @ 40°C [N/mm^2]	29.9	45.2	44.1	30.8	≥ 8

All the considered binders satisfy most of the *S.A.* acceptance criteria but nevertheless should be rejected since none of them meets the requirement set at 25°C on the indirect tensile strength R_t. The Authors cannot provide a discussion on the performance-related significance neither of the threshold value in correspondence of which violations have been identified, nor of those which have been widely satisfied. It is also difficult to evaluate the inner coherency of the limits set by the specification since the parameters, although expressed in fundamental units, derive from the combination of empirical and rational concepts and cannot be directly used in the modelling of pavement performance. Nevertheless, it was observed that if the binders are considered for use at a compaction temperature which takes into account their specific viscosity characteristics (see Table 13), for two of them (SBS-R-H and SBS-L-H) all the requirements are met.

CONCLUSIONS

The experimental investigation described in this paper was focused on the analysis of the properties of four different modified bituminous binders which were obtained from the use of three different SBS elastomers and one EVA plastomer. These were added to a single base bitumen in the same percentage (6%), typical of heavy duty paving applications. The starting point of the project was the critical analysis of the preliminary qualification system which has been recently developed for the selection of this type of binders by *Autostrade per l'Italia S.p.A.*, the Italian Road Agency which manages and maintains most of the motorway network. Additional rheological tests, which according to the Authors provide more performance-related information, were included in the testing program and proved to be extremely useful both for the interpretation of the results obtained within the specification system and for the identification of its limitations.

Moreover, peculiar aspects of the rheological behaviour of the modified binders were highlighted and discussed with respect to their molecular structure.

Experimental results indicate that the rheological behaviour of modified binders is strongly dependent upon polymer type. This was observed by analysing the results obtained in all testing configurations (cone-plate shear and beam bending) and modes (oscillatory and creep) in a wide range of temperatures and loading times (or frequencies).

The effects associated to the tough and rigid EVA matrix were highlighted at intermediate to low temperatures especially when loads were applied for short time durations and/or high frequencies: in such conditions these binders exhibited a stiffer and more elastic response than SBS-modified materials. This was observed both in rheological tests and in tests carried out on reference mixtures. However, in the case of mixture testing, by means of which the binder is subjected to severe tensile and shear stresses within a porous aggregate structure, stiffening effects were associated to a reduction of tensile strength and ductility.

As expected, plastomer stiffening effects were observed to be greatly reduced at higher temperatures and/or when approaching flow conditions, where binders containing SBS elastomers proved to be stiffer and more elastic, with a greater potential in contributing to permanent deformation resistance.

These rheologically-based conclusions are fully coherent with the results obtained by means of empirical test methods. The binder containing the EVA polymer yielded penetration, elastic recovery and softening point values which are smaller than those obtained on the elastomer-modified binders. By means of an empirical approach it was also observed that during high temperature storage the separation phenomena which may occur within the binder are more evident in the case of the plastomer-modified material.

Further differences in the response of the plastomer and elastomer modified binders were detected by analysing Black diagrams and creep-recovery curves. It was shown that different shapes are associated to different mechanisms with which the polymer matrix contributes to the overall response of the binder. It was thus proven that the use of these graphical representations can be useful in the interpretation of visco-elastic data.

Test results were also analysed in order to evaluate the effects caused by variations of the composition (styrene content) and structure (radial or linear) of the SBS polymer. In most testing conditions increases of the stiffness and elasticity of the binder were associated, especially in the intermediate to high temperature range, to increases of the styrene content, which affects the volume of the corresponding rigid domains created within the rubbery polymer network. These effects were more evident for long loading times and/or in flow conditions, when the polymer network is completely stretched. In the oscillatory mode, at an extremely high temperature (80°C), these same effects were associated to variations of the polymer structure: in these specific conditions radial SBS polymers provide a stiffer response due to the presence of great number of interconnections within the rubbery network which is not totally stretched during testing.

With some exceptions, empirical tests did not allow a clear distinction to be made between the various SBS modifiers. Coherently with rheological measurements, it was observed that the greater softening point values, which are linked to the rheological properties of the binders while approaching flow conditions, were associated with the use of high styrene content polymers. The effects caused by variations of the polymer structure were reflected by storage stability test results: in this case, the linear SBS proved to severely damaged by high temperature conditioning.

The results obtained in the experimental investigation, not only yielded the above illustrated information, but also allowed the Authors to critically analyse the contents of the *S.A.* preliminary qualification system.

The use of high polymer contents is necessary in order to obtain the desired enhancements of ductility and high temperature stiffness and elasticity; however, this inevitably leads to stiffness increases at intermediate temperatures which are reflected by low penetration values recorded at 25°C. The minimum penetration value admitted by the acceptance system thus appears to be exceedingly high and the corresponding requirement cannot be easily satisfied.

Acceptance criteria defined in terms of complex modulus and phase angle also need to be discussed. The use of control charts seems to be more appropriate in the context of quality-control investigations carried out on a single binder, the target behaviour of which is already known. When considering different products, characterized by rheological peculiarities which can be detected by looking at Black diagrams or creep-recovery curves, such an approach may yield misleading results. It was also observed that the G^* and δ acceptance ranges may not be totally compatible with performance-related principles, with which rheological data are linked to specific pavement distresses.

Finally, some comments can be made on the use of mechanical tests performed on reference mixtures as an indirect means of evaluating binders. In principle, this type of approach is extremely interesting, but according to the Authors it needs to be appropriately implemented in the specification system. As suggested by experimental results, indirect tensile tests carried out on open-graded mixtures containing modified binders are extremely sensitive to variations of the compaction temperature, which is certainly binder-specific. Thus, this should be adequately taken into account in the specification system by allowing specimen compaction at either an equi-viscosity temperature or at a temperature defined by the supplier of each binder.

In extremely general terms it may be concluded that the analysed qualification system constitutes an interesting improvement over the previously employed systems. Conventional empirical tests are combined with rheological measurements and reference bituminous mixtures are subjected to mechanical testing: given the complexity of a system obtained by assembling information which stems from such different testing approaches, it appears that it requires to be carefully fine-tuned in order to make set requirements fully compatible with each other or, as an alternative, in order to establish a hierarchy among them.

In the Authors' opinion, improvements to the system may stem, in the future, from the introduction of additional rheological tests such as those described in this paper, from the analysis of a wide database of test results, from the harmonization of the specification approach with other validated systems (such as SUPERPAVE) and from the observation of actual field performance.

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