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## Performance-Related Characterization of Bituminous Binders and Mixtures Containing Natural Asphalt

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### Abstract

Natural asphalt as an additive in bituminous mixtures was subjected to evaluation by means of performance-related laboratory tests carried out on materials sampled from a production plant and a test section. Compaction issues were discussed by referring to binder viscosity and by considering the volumetrics of laboratory-compacted specimens. Mixture stiffness was assessed by means of repeated load indirect tensile tests carried out on field cores and by constructing pseudo master curves. Models were employed for the quantification of effects associated to enhanced compaction and binder ageing. Fatigue was also investigated by means of indirect tensile tests.

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### 1. Introduction

Bituminous mixtures for paving applications are composed of aggregates, filler and bitumen which create a stable composite material, resistant to traffic- and temperature-induced stresses and durable with respect to the aggressive actions of environment. In several cases, standard composition can be complicated by the presence of additional components, generally introduced in mixtures as additives at the production plant, which have the function of enhancing specific performance-related properties or of compensating for the reduced quality of the base materials. Additives which have been employed with success in full-scale applications include polymers (either natural or synthetic), fibres (of different types and lengths), oxidants, antioxidants and anti-stripping agents [1].

During the construction of the Fier-Tepelene highway in Albania, natural asphalt available from the Selenice mine, located in the south-western area of the Country, was suggested as an additive for bituminous mixtures of

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the flexible pavement [2]. Even though this option was not envisioned in design documents [3], it was carefully considered by Road Administration, Project Engineers and Contractor due to problems encountered in locally finding bitumen meeting requirements of technical specifications. Moreover, since it is well known that natural asphalts may significantly harden bitumen as a result of their composition and content of fine minerals, the use of such an additive was seen as an opportunity for reducing total thickness of the bitumen-bound portion of the pavement.

Based on the discussion synthesized above, the pavement cross section which was proposed for construction was constituted, above the embankment or natural subgrade, by an unbound granular sub-base (15 cm thick), an unbound granular foundation (20 cm thick) and three layers of continuously-graded bituminous mixtures complying to ASTM standard D3515 (9, 6 and 4 cm of base, binder and wearing course respectively). The use of natural asphalt was suggested for all bituminous mixtures with a dosage of 8% by weight of the base bitumen.

Due to the fact that limited information was available on the field performance of bituminous mixtures containing the above mentioned additive, detailed experimental investigations were recommended. This paper gives an overview of the tests carried out on the bituminous binder and on the corresponding base and binder course mixtures. Conclusions were drawn with respect to the optimization of construction operations and to expected field performance.

## 2. Experimental investigation

### 2.1. Bituminous binder

Base bitumen sampled from the field production plant was combined in the laboratory with 8% by weight of natural asphalt (locally available in granular form). Mechanical mixing was carried out at 170°C for approximately 15 minutes, until a homogeneous mass was obtained. The resulting binder was subjected to tests for the determination of density at 25°C and viscosity in a temperature range comprised between 120 and 165°C. Moreover, by considering three different degrees of ageing (unaged, short-term aged with the Rolling Thin-Film Oven, RTFO, and long-term aged with the Pressure Aging Vessel, PAV), the binder was fully characterized by a rheological viewpoint with the corresponding evaluation of master curves, temperature-dependency functions and SUPERPAVE PG-grade.

Average density of the binder at 25°C (determined as prescribed by CNR standard 67/78) was found to be equal to 1.039 g/cm<sup>3</sup>. Such a value is higher than those typically obtained for standard bitumen (1.018-1.020 g/cm<sup>3</sup>) due to the presence of natural asphalt, for which a density of 1.16 g/cm<sup>3</sup> was reported by producers [2].

Viscosity tests were performed by means of a Brookfield DVIII-Ultra apparatus by making use of a SC4-21 spindle and by adopting two different rotating speeds, equal to 20 and 100 rpm, which respectively correspond, for the given geometry, to a shear rate of 18.6 and 93 s<sup>-1</sup>. Average results are listed in Table 1. The negligible shear rate effects show that the binder exhibits a pure Newtonian behaviour in the considered temperature range.

Table 1. Viscosity of the binder

Temperature [°C]	Viscosity at 18.6 s <sup>-1</sup> [Pa·s]	Viscosity at 93 s <sup>-1</sup> [Pa·s]
120	1.23	-
135	0.53	0.52
150	0.26	0.26
165	0.14	0.14

Temperature-viscosity data were fitted to an exponential function for the evaluation of reference plant mixing and field compaction temperatures, which respectively correspond to viscosity values equal to 0.17 ( $\pm 0.02$ ) and 0.28 ( $\pm 0.03$ ) Pa·s [4]. Calculated values of such temperatures, respectively equal to 160 and 149°C, are totally compatible with those recorded on site by the Contractor.

Samples of the binder in its three states of ageing were subjected to rheological characterization by making use of an Anton Paar MCR-301 Dynamic Shear Rheometer (DSR). Tests were carried out with 8 mm parallel plates (2 mm gap) in the 4–34°C temperature range, and with 25 mm plates (1 mm gap) between 34 and 70°C. At each temperature, frequency sweeps were performed (from 1 to 100 rad/s) with five acquisition points per decade. Imposed strain levels were defined by following the testing protocol developed by Clyne and Marasteanu [5]. Master curves of the complex modulus ( $G^*$ ) and phase angle ( $\delta$ ) were found by making use of the CAM and WLF models [6, 7] which can be written as follows:

$$\text{CAM} \quad |G^*(\omega)| = G_g \cdot \left[ 1 + \left( \frac{\omega_c}{\omega} \right)^k \right]^{\frac{m}{k}} \quad (1)$$

$$\text{WLF} \quad \log(a_{(T, T_{ref})}) = \frac{-C_1 \cdot (T - T_{ref})}{C_2 + T - T_{ref}} \quad (2)$$

where  $|G^*(\omega)|$  is the norm of the complex modulus (in Pa) at loading frequency  $\omega$  (in rad/s);  $G_g$  is the glassy modulus ( $\log G_g = 9.1$ );  $T_{ref}$  (in °C) is the reference temperature;  $a_{(T, T_{ref})}$  is the shift factor at temperature  $T$ ;  $\omega_c$ ,  $k$ ,  $m$ ,  $C_1$  and  $C_2$  are model parameters.

Values of the abovementioned model parameters are given in Table 2, which refers to master curves built at a reference temperature of 34°C (Fig. 1). As expected, ageing phenomena produce an overall stiffening of the binder accompanied by an enhancement of elastic response (i.e.  $G^*$  increase and  $\delta$  decrease), which are especially relevant in the low frequency (high temperature) range.

Master curves and temperature-dependency functions were employed for the assessment of the performance grade (PG-grade) of the binder according to the approach proposed within the SUPERPAVE-SHRP system [8]. Thus, limiting temperatures for rutting, fatigue cracking and thermal cracking were calculated by referring to critical values of the rheological parameters listed in Table 3. When required, complex modulus values derived from oscillatory shear data were converted into normal creep stiffness values ( $S$ ) by considering loading time (in seconds) to be equal to the inverse of frequency (in Hz) and by assuming a ratio of 3 between normal and shear moduli [9]. Obtained results, synthesized in Table 3, indicate that the binder belongs to the 70-10 PG-grade.

Table 2. Rheological model parameters of the binder

	Unaged	RTFO-aged	PAV-aged
$\log(G_g)$	9.1	9.1	9.1
$\log(\omega_c)$	3.1	2.2	-0.2
$k$	0.162	0.144	0.115
$m$	1.15	1.17	1.35
$C_1$	13.2	15.0	18.0
$C_2$	133.2	140.9	159.3

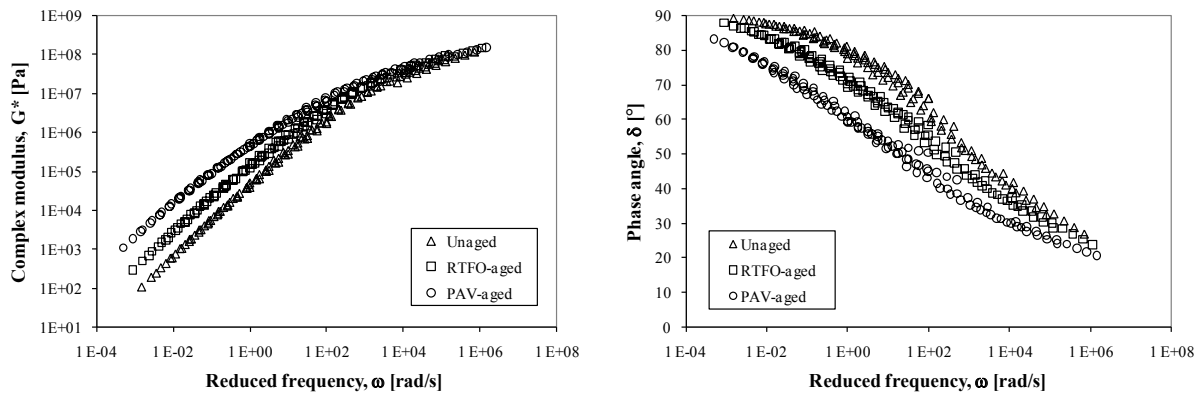


Fig. 1. Complex modulus and phase angle master curves of the binder

Table 3. Identification of PG-grade of the binder

Distress	Ageing	Conditions	Critical values	Limiting temperature [°C]	Specification temperature [°C]	PG-grade
Rutting	Original	$\omega = 10 \text{ rad/s}$	$\min G^*/\sin\delta = 1.00 \text{ kPa}$	70.2	70	70-10
	RTFO	$\omega = 10 \text{ rad/s}$	$\min G^*/\sin\delta = 2.20 \text{ kPa}$	71.3		
Fatigue cracking	PAV	$\omega = 10 \text{ rad/s}$	$\max G^* \cdot \sin\delta = 5000 \text{ kPa}$	26.0	34	
Thermal cracking	PAV	$t = 60 \text{ s}$	$\max S = 300 \text{ MPa}$	-6.8	0	
			$\min m = 0.300$	-3.5		

### 3. Bituminous mixtures

A full-scale test section of the pavement was built by employing base and binder course mixtures produced by the Contractor. Both contained 8% natural asphalt (by weight of the base bitumen), added during the mixing process with an adequate dosage system. Mixtures were laid in the prescribed thicknesses (respectively equal to 9 and 6 cm) on the foundation layer and were thereafter subjected to compaction by employing a double-drum steel roller. During construction operations and after the completion of compaction, samples of loose mixtures and pavement cores (150 mm diameter) were taken for subsequent testing.

Loose mixtures were subjected to tests for the evaluation of composition and theoretical maximum density. Moreover, they were employed for the preparation of gyratory and Marshall specimens which were characterized volumetrically. Further gyratory specimens were prepared to assess the sensitivity of the mechanical properties to compaction level. Cores were employed for the determination of volumetrics, elastic stiffness and resistance to fatigue.

#### 3.1. Composition

Binder content (%B) of the bituminous mixtures was evaluated with the extraction procedure based on the use of solvent (EN 12697-1), while aggregate size distribution was determined by means of wet sieving (EN 933-1). Corresponding results are synthesized in Table 4 and in Fig. 2. It can be observed that for both mixtures, binder content, expressed with respect to total aggregate mass, is close to the target value chosen for production. Moreover, size distributions, of the continuously-graded type, are contained within specification limits provided by ASTM D3515.

Table 4. Binder content (in %) of base and binder course mixtures

	Extraction	Target
Base	4.13	4.1
Binder	5.45	5.1

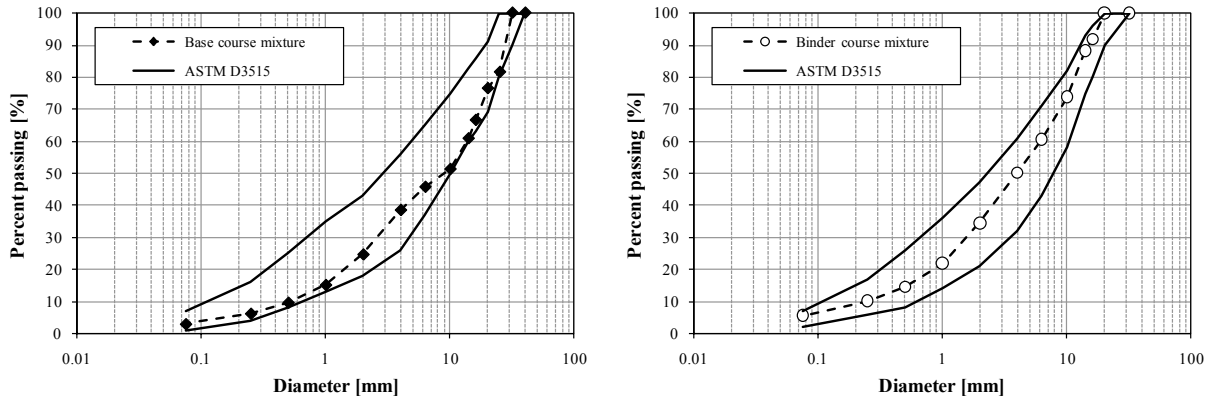


Fig. 2. Aggregate size distribution of base and binder course mixtures

### 3.2. Volumetrics

Compaction of laboratory specimens was carried out by making use of both the Marshall apparatus (CNR 30/73, 75 blows per side) and the gyratory compactor (EN 12697-31, 100 gyrations). Based on information provided by the Contractor, in both cases compaction was performed at a temperature of 165°C, which is higher than the reference value derived from analysis of viscosity data (equal to 149°C).

Volumetric characterization was carried out for both types of specimen and for cores taken from the test section according to EN 12697-6 and EN 12697-8. Average results of measured and calculated parameters are listed in Table 5. Laboratory-compacted specimens were prepared in four replicates per mixture. Thirty and fifteen cores were available for the base and binder course respectively.

It is interesting to observe that for both mixtures, gyratory compaction is much more efficient than the Marshall one. This is shown by the higher density and VFB values, and by the lower void content and VMA values. Such a remarkable difference between the two compaction techniques is quite unusual and may be related to the peculiar properties of the mixtures (and of their component materials) which translate into a significant shear sensitivity. Thus, aggregate particles can be easily packed together when applying shear stresses of variable directions (i.e. with the gyratory equipment), whereas they tend to occupy a significant volume without reorienting adequately when they are mainly subjected to a constant stress regime (i.e. with the Marshall compactor).

Volumetric properties of the cores clearly indicate that density reached in the field by both mixtures is very close to that of Marshall specimens. Such a result is coherent with the type of compaction adopted during construction, based on the use of a steel roller. Given the shear sensitivity of the mixtures, higher compaction levels, closer to reference gyratory compaction conditions, would have probably been achieved if rubber-wheeled rollers had also been employed.

Table 5. Volumetric properties of laboratory-compacted specimens and cores of base and binder course mixtures

	Base mixture			Binder mixture		
	Marshall	Gyratory	Field	Marshall	Gyratory	Field
Density [g/cm <sup>3</sup> ]	2.268	2.361	2.260	2.242	2.348	2.252
Theoretical maximum density [g/cm <sup>3</sup> ]	2.499	2.499	2.499	2.481	2.481	2.481
Void content, %v [%]	9.3	5.5	9.6	10.0	5.7	9.6
Voids in the mineral aggregate, VMA [%]	17.9	14.5	18.2	21.1	17.4	20.8
Voids filled with bitumen, VFB [%]	48.5	62.2	47.6	52.9	67.1	54.1
Percent Marshall compaction [%]	-	-	99.7	-	-	100.4
Percent gyratory compaction [%]	-	-	95.7	-	-	95.9

Table 6. Elastic stiffness of cores of base and binder course mixtures

		5°C		20°C		35°C	
		60 ms	124 ms	60 ms	124 ms	60 ms	124 ms
Base	E [MPa]	11,387	10,113	3,414	2,706	1,097	798
	COV [%]	14.6	17.5	25.0	25.0	44.1	45.7
Binder	E [MPa]	10,098	9,118	4,297	3,423	341	292
	COV [%]	8.7	8.5	9.6	8.7	33.3	25.6

### 3.3. Elastic stiffness

After thickness reduction of the cores (down to 60-80 mm), evaluation of elastic stiffness was carried out by means of repeated load indirect tensile tests as prescribed by EN 12697-26. Tests were performed on 5 cores for each pavement layer by adopting two rise-time values (60 and 124 ms) and three test temperatures (5, 20 and 35°C). Mean stiffness values (E) and corresponding coefficients of variation (COV) are listed in Table 6.

Data given in Table 6 clearly indicate that as a result of insufficient field compaction (Table 5) both mixtures exhibit a low stiffness. With respect to data dispersion, it is interesting to note that COVs are in all cases quite high, with peak values associated to the highest test temperature at which limiting measurement conditions of the equipment are approached. This can be explained by referring to the internal non-homogeneity of the mixtures since it was visually observed that on the surface of the cores in some cases coarser aggregate particles formed clusters. Another contribution to variability may derive from the effective dispersion of the natural asphalt additive, which is added at the production plant in the mixer and not preliminarily blended with bitumen.

In order to give a more complete description of the mechanical properties of the mixtures, elastic stiffness data were subjected to modeling for the construction of pseudo master curves. This was done by referring to the following analytical formulation originally proposed by Zeng et al. [10] for complex modulus data:

$$E^* = E_e^* + \frac{E_g^* - E_e^*}{\left[1 + \left(f_c / f'\right)^k\right]^{m_e/k}} \quad (3)$$

where  $E_e^*$  is the equilibrium complex modulus (for frequency which tends to 0),  $E_g^*$  is the glassy complex modulus (for frequency which tends to infinite),  $f_c$  is a location parameter (with dimensions of frequency),  $f'$  is reduced frequency (function of both temperature and strain),  $k$  and  $m_e$  are dimensionless shape parameters.

The model was employed by considering the formal equivalency between the norm of the complex modulus  $E^*$  and elastic stiffness (E). Moreover, rise-time values ( $t_r$ , in ms) were converted into loading frequencies ( $f$ , in

Hz) by making use of the following simplified formula which assumes a pulse load to be one quarter of a full sinusoidal signal:

$$f = \frac{1,000}{4 \cdot r_t} \quad (4)$$

Results derived from data modeling by assuming a reference temperature of 20°C are listed in Table 7. Due to the limited number of experimental data points,  $E_c$  was chosen equal to 9 MPa [10], while the glassy modulus  $E_g$  was calculated by applying the following relationship [11]:

$$E_g = C \cdot \left( \frac{V_a}{V_l} \right)^{0.55} \cdot e^{-5.84 \cdot 10^{-2} \%v} \quad (5)$$

where C is a constant (equal to  $1.436 \cdot 10^{-4}$ ),  $V_a$  is the percent volume occupied by aggregates,  $V_l$  is the percent volume occupied by the binder, %v is void content.

### 3.4. Effects of ageing and improved compaction

Elastic stiffness values given in Table 6 and calculated by means of pseudo master curve functions (equation (3) and Table 7) are referred to cores which were taken from the pavement immediately after laying. To take into account stiffening effects due to the optimization of construction operations and of the time-dependant ageing of the bituminous binder, the investigation included additional specific tests and modeling activities.

With respect to binder ageing, it was assumed that the bituminous binder contained in the pavement cores has a rheological behavior which can be mimicked by the material subjected to RTFO ageing. The effect of long-term ageing, quantified on the binder only by means of rheological tests carried out on the PAV residue, was transferred to the mixtures by making use of the following models [11] which were fitted to the experimental data:

$$E^* = E_g \cdot R^* \quad (6)$$

$$\log(R^*) = \log(B^*) - C \cdot \left( 1 - e^{-0.13 \cdot \frac{V_a}{V_l}} \right) \cdot \log(B^*) \cdot [1 + 0.11 \cdot \log(B^*)] \quad (7)$$

Table 7. Elastic stiffness model parameters of base and binder course mixtures

	Base	Binder
$E_g$ [MPa]	27,705	23,785
$E_c$ [MPa]	9	9
$\log(f_c)$	-0.56	0.34
k	0.19	0.22
$m_e$	0.89	0.61

where  $E_g$  is the glassy modulus (calculated by means of equation (5)),  $R^*$  is the so-called reduced complex modulus,  $V_a$  is the percent volume occupied by aggregates,  $V_l$  is the percent volume occupied by the binder,  $B^*$  is the reduced complex modulus of the binder,  $C$  is a model parameter.

By assuming once again the correspondence between complex modulus and elastic stiffness, values at 20°C and 10 Hz of the two mixtures (respectively equal to 4,110 and 5,327 MPa for the base and binder course) were fitted to the models by considering the rheological properties of the RTFO-aged binder and the composition and volumetrics of the cores. By using the value of constant  $C$  resulting from fitting, long-term ageing values of the elastic modulus were then calculated by introducing in equation (7) the reduced complex modulus of the PAV-aged binder.

To take into account the effects of improved compaction, additional gyratory specimens of the two mixtures were prepared with a variable void content. Specimens were subjected to tests for volumetric characterization and for the evaluation of elastic stiffness at 20°C and 124 ms rise-time. For each mixture, stiffness values were then expressed as a function of void content with the subsequent calculation of normalized stiffness, expressed as the ratio between actual stiffness and reference values obtained at the same average void content exhibited by the cores (equal to 9.6%).

Corresponding results (Fig. 3) show that the same regression equation can be used for both mixtures for the calculation of the stiffness ratio at any given void content value. In order to estimate the stiffening effects due to the adoption of optimized compaction procedures in the field, it was assumed that normalized stiffness equations given in Fig. 3 are applicable even to data obtained on cores or calculated from their pseudo master curves.

Results deriving from the application of the above described models are synthesized in Table 8, where example values of void content (equal to 8 and 6%) have been considered together with short- and long-term ageing conditions. Combined effects of enhanced compaction and ageing were computed by sequentially applying the two models. It can be observed that by taking into account both effects, stiffness values may increase, with respect to reference values computed in the short-term at the void level obtained in the test section, by approximately 40%.

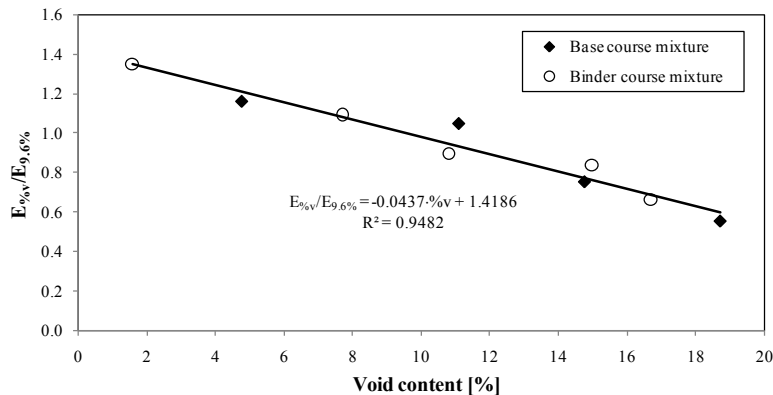


Fig. 3. Normalized stiffness of gyratory specimens as a function of void content

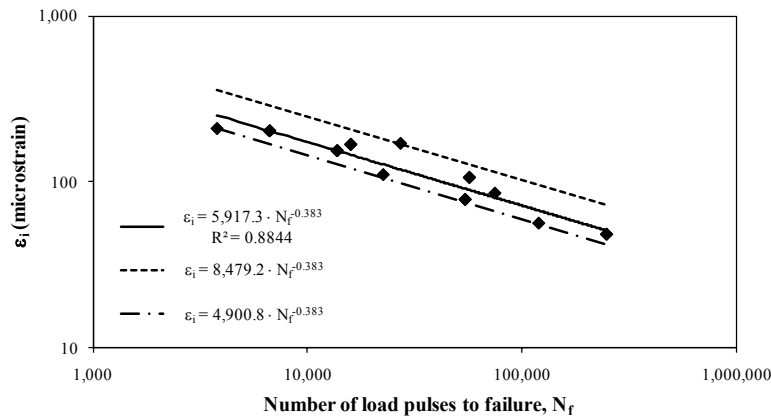


Fig. 4. Indirect tensile fatigue lines of the base course mixture at 20°C

Table 8. Elastic stiffness of cores predicted as a function of void content and ageing (at 20°C and 10 Hz)

	9.6% voids		8.0% voids		6.0% voids	
	Base	Binder	Base	Binder	Base	Binder
E after short-term ageing [MPa]	4,110	5,327	4,394	5,695	4,753	6,160
E after long-term ageing [MPa]	5,108	6,281	5,472	6,729	5,936	7,298

$$\varepsilon_i = a \cdot N_f^{-b} \quad (8)$$

where  $\varepsilon_i$  is expressed in microstrain,  $a$  and  $b$  are regression constants.

Even though cores were characterized by variable volumetrics and stiffness (Tables 5 and 6), a very good fitting was obtained as proven by the high value of the coefficient of determination ( $R^2$ ), equal to 0.8844. Values of the regression constants  $a$  and  $b$  are given in Fig. 4, which also displays values associated to the parallel limiting fatigue lines which include the entire set of experimental data.

### 3.5. Fatigue

A selected number of cores of the base layer was subjected to repeated load indirect tensile tests carried out until failure for the assessment of fatigue properties at 20°C (EN 12697-24). Imposed values of the maximum horizontal stress ( $\sigma_h$ ) were conveniently varied in order to obtain values of the maximum horizontal strain ( $\varepsilon_h$ ) distributed within an adequately wide range. Fig. 4 contains the classical representation of fatigue data in the logarithmic plane by plotting initial horizontal strain ( $\varepsilon_i$ ) as a function of the number of load pulses to failure ( $N_f$ ). Experimental data were fitted to a single fatigue line according to the following power-law equation:

## 4. Pavement performance evaluation

Performance evaluation of the proposed pavement was carried out by employing the above illustrated experimental results as input data in appropriate procedures and methods. In particular, binder data were analyzed within the framework of the SUPERPAVE selection system which is conceived to prevent any binder-related contribution to the occurrence of rutting, fatigue cracking and thermal cracking. Mixture data, processed as indicated in the previous sections, were considered for the calculation of cumulative fatigue damage by making use of a mechanistic procedure.

Table 9. SUPERPAVE design temperatures

	Air	Wearing course	Binder course	Base course
$T_{\max}$ [°C]	34.7	58.8	54.6	51.1
$T_{\min}$ [°C]	-6.0	-9.1	-6.5	-4.7

Pavement maximum and minimum design temperatures ( $T_{\max}$  and  $T_{\min}$ ) were computed by means of the updated SUPERPAVE models [8, 12] and by using data on the latitude of the construction site and on its temperature history retrieved from available on-line archives. As proven by the results shown in Table 9, since design temperatures are comprised within the range associated to PG70-10, it can be concluded that from a rheological viewpoint and with respect to the distress phenomena addressed by SUPERPAVE, the binder subjected to testing can be considered acceptable for use in all pavement layers of the considered infrastructure.

For the assessment of its structural behaviour under loading, the proposed pavement was modelled as a multi-layer elastic system. Elastic moduli of the subgrade and unbound granular layers (respectively equal to 110 and 200 MPa) were derived from technical specifications and by considering the results of field tests performed during construction. A single design temperature, equal to 20°C, was chosen for the pavement. Moduli of the base and binder course were therefore calculated at such a temperature (and 10 Hz frequency) by employing the pseudo master curves derived from experimental data. To take into account the effects of ageing, it was hypothesized that starting from short-term ageing values, elastic moduli increase linearly with time reaching the long-term limiting value after 10 years of service. In the absence of experimental data, in terms of mixture stiffness the wearing course was assumed to be equivalent to the binder course. Traffic prediction data were derived from the presentation document of the construction project [13] and were then converted into 80 kN equivalent single axle loads (ESALs). Cumulative damage was computed in annual incremental steps by considering the time-dependent values of elastic moduli and ESALs, and by referring only to fatigue of the base layer. The corresponding design criterion was obtained from the equations presented in Fig. 4 by introducing a corrective factor which takes into account the specific test configuration and laboratory-to-field shifting [14].

Results obtained in the structural evaluation of the pavement indicate that allowable ESALs, equal to 19.7 million, correspond to a service life of 16.5 years which is greater than the required minimum design life set by the Road Administration at 15 years. Even though the model is affected by several limitations and approximations, it can be concluded that the proposed pavement, which includes natural asphalt as an additive in bituminous mixtures, is technically acceptable.

## 5. Conclusions

The experimental investigation described in this paper focused on the analysis of the performance-related properties of bituminous mixtures containing natural asphalt which were proposed for use in the construction of a highway pavement in Albania. The testing program considered materials sampled at the production plant and taken from a test section. Investigations were supplemented by a pavement performance.

Viscosity tests carried out on the bituminous binder containing 8% by weight of natural asphalt showed that mixing and compaction operations require temperatures which are totally compatible with those normally adopted during construction. Nevertheless, it was observed that cores of the base and binder layer taken from the pavement were characterized by a low level of compaction. This was explained by referring to the significant shear sensitivity of both mixtures which was highlighted by comparing results obtained in the laboratory with Marshall and gyratory compactors. Consequently, recommendations were made to combine the use of steel rollers with that of pneumatic-tyre rollers, capable of transferring higher shear stresses to pavement layers during compaction.

Stiffness of the base and binder course mixtures was assessed on field cores by means of repeated load indirect tensile tests, the results of which were employed for the construction of pseudo master curves. Analysis of experimental data showed that calculated elastic moduli can be adjusted to take into account the enhancement of compaction and progressive ageing of the binder. The effects which derive from variations of the level of compaction can be quantified by referring to a stiffness ratio function which was found to be common for the two mixtures, while ageing effects can be considered by including binder rheological data in a stiffness prediction model based on volumetrics.

Pavement performance evaluation was based on the results of the experimental investigation and required the use of SUPERPAVE distress-related specifications for binders and of a mechanistic structural analysis procedure. It was concluded that the considered bituminous mixtures containing natural asphalt are acceptable for the construction of the highway pavement.

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