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Fatigue Characterization of Bituminous Binders Containing Crumb Rubber from End-of-Life Tires

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***Abstract*—This paper presents the outcomes of an experimental investigation which focused on the fatigue behavior of bituminous binder for paving applications prepared by combining a single reference neat bitumen with different types of crumb rubber products derived from the processing of end-of-life tires. In the laboratory preparation of the blends, crumb rubber dosage was conveniently limited in order to obtain binders characterized by viscosity values compatible with standard operating conditions normally adopted in the production and compaction of bituminous mixtures. Results obtained from time sweep tests carried out in equi-stiffness conditions showed that fatigue performance is enhanced by the use of the considered crumb rubber modifiers with a dosage-dependent improvement of both fatigue life and crack propagation amplitude. Given that the considered crumb rubber products were quite similar, no significant effects were associated to changes of crumb rubber type.**

***Keywords*—road pavements, bituminous binders, fatigue, viscosity, crumb rubber**

I. Introduction

Damage accumulation caused by traffic-repeated loading is one of the most complex phenomena occurring in the surface layers of flexible road pavements that are composed of bituminous mixtures [1]. As a result of fatigue, cracks initiate and propagate in the bituminous binder, leading to a diffused state of microcrack coalescence which can be observed on the pavement surface in the form of randomly-shaped “alligator” cracks [2]. As widely documented in literature, the viscoelastic properties of the binder play a significant role in controlling the development of the abovementioned microcracks [3]. Thus, the selection of an appropriate binder for each paving application is a key issue in pavement design [4].

In order to enhance the engineering properties of bituminous binders, several materials may be added to neat bitumen as modifiers [5]. Styrene-butadiene-styrene (SBS) block copolymer is the one which is more frequently employed in the paving industry, but a range of alternative elastomers and plastomers are also becoming widely utilized, including those derived from the recovery and processing of waste materials [6]. In particular, one of the most attractive solutions is represented by crumb rubber derived from end-of-life tires (ELTs), the use of which can lead to remarkable environmental benefits as a consequence of avoided stockpiling and landfilling [7,8].

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When crumb rubber is blended with bitumen at high temperatures, an interaction between the two components occurs, with the absorption of the bitumen aromatic fraction by the rubber that produces a time-dependent swelling and degradation of the modifying agent [9]. The extent of such an interaction is affected by the mixing conditions and by the chemical nature of employed materials. At the industrial scale, these phenomena occur as part of the so-called “wet” modifying process, which typically requires the use of a crumb rubber dosage of at least 15% (on the weight of the total binder) and yields a binder known as “asphalt rubber” [10].

Previous studies have clearly highlighted the performance-related advantages of the use of asphalt rubber, which include enhanced durability and the possibility of preparing bituminous mixtures with improved functional characteristics [11,12]. However, it has been also pointed out that as a result of the very high viscosity care should be taken in guaranteeing full efficiency of plant mixing and field compaction by means of the adoption of processing temperatures significantly higher than those of standard paving technologies [8].

The aim of the experimental investigation described in this paper was to explore the fatigue properties of bituminous binders containing reduced quantities of crumb rubber. Such a goal stemmed from the desire of exploiting the properties of this peculiar type of modifier with only minimal changes in the operating conditions of production and construction equipment commonly adopted in practice. Other potential advantages are related to the containment of production costs of such binders, although in this perspective the instability of the market worldwide defines an ever-changing scenario to which the paving industry needs to adapt.

The investigation focused on bitumen – crumb rubber blends prepared in the laboratory by employing three different crumb rubber types and two percentages of modification (5 and 10% by weight on the neat bitumen). Preliminary characterization tests focused on the determination of standard empirical properties (penetration and softening point) and of viscosity at temperatures representative of production and compaction conditions of bituminous mixtures. Fatigue properties were assessed by making use of torsional tests carried out in equi-stiffness conditions and by analyzing resulting data according to phenomenological and energy approaches.

Obtained results showed that low-dosage modification by means of crumb rubber yields a limited viscosity increase and non-negligible changes of the fatigue behavior of neat bitumen which however do not seem to be affected by the specific type of employed modifier.

II. Materials and Methods

A. Preparation of Blends and Preliminary Characterization

The experimental investigation involved the analysis of a reference neat bitumen X (50/70 penetration grade) and of six bituminous binders obtained from it by employing three crumb rubber products (identified as A, B and C) added in two dosages (5 and 10% by weight on total binder).

Samples of the three types of crumb rubber were taken from three ELT processing plants which operate the so-called "ambient size reduction" by means of shredding, iron magnetic separation, milling and sieving phases differently combined depending upon available technologies, inflow of material and desired quality of end products [13]. According to owners, managers and operators, plants A and B collected and processed both car and truck tires, while plant C exclusively treated ELTs coming from heavy vehicles [14].

The three crumb rubber products were extensively characterized in previous studies [13-15]. Table 1 shows the particle size distribution data obtained from dry sieving [16], from which it can be observed that the considered modifiers belong to the so-called "standard" category (with a characteristic particle diameter D_{90} corresponding to 90% passing comprised between 0.5 and 1.0 mm), with relevant differences only in the finer fraction (particles smaller than 0.42 mm). In particular, crumb rubber B is definitely finer that products A and C, which in turn are very similar to each other. However, the observed differences in size distribution are not reflected by the surface area per unit mass (SA) which may be calculated according to a previously developed experimental technique by taking also into account the specific morphological characteristics of crumb rubber particles [15]. This is proven by the data listed in Table 2, which indicates that the three crumb rubber products are associated to similar SA values, the highest being attributed, counterintuitively, to the coarser material (crumb rubber C).

TABLE I. CRUMB RUBBER PARTICLE SIZE DISTRIBUTIONS

Sieve size (mm)	Passing (%)		
	Crumb rubber A	*Crumb rubber B*	*Crumb rubber C*
1.000	100	100	100
0.833	99.8	99.4	99.6
0.710	94.9	94.8	95.4
0.589	69.5	73.4	66.5
0.500	32.9	45.5	34.0
0.417	9.39	24.3	12.3
0.250	0.35	2.44	0.86
0.125	0.14	1.04	0.42
0.063	0.07	0.58	0.33

TABLE II. CRUMB RUBBER SURFACE AREA PER UNIT MASS

Crumb rubber	SA (m^2/g)
A	0.0112
B	0.0111
C	0.0118

The six bitumen - crumb rubber blends were prepared in the laboratory operating on 500 g batches by means of a mechanical mixer, equipped with an anchor-shaped stirrer, by imposing a rotation speed of 600-700 rpm for 90 minutes. During production operations temperature was maintained at 180 ± 5 °C by means of a thermostatic oil bath. The mixing protocol was adopted for all blends regardless of specific reaction rates of employed crumb rubber samples [13].

Preliminary characterization of the reference neat bitumen and of the six bitumen – crumb rubber blends was carried out by performing standard empirical tests for the determination of penetration at 25 °C (pen_{25}) [17] and of ring and ball softening point temperature ($T_{R\&B}$) [18]. Moreover, viscosity (η) was measured in in a wide temperature range (135-190 °C) by means of a Brookfield viscometer (DVIII-Ultra) with a SC4-27 spindle at an imposed shear rate equal to 6.8 s^{-1} (corresponding to 20 rpm) [19].

Results obtained from standard empirical tests are listed in Table 3, where blends are associated to codes given by the combination of crumb rubber type and dosage (e.g. A-5 refers to the binder with 5% of crumb rubber A). It was observed that, as expected, the presence of crumb rubber causes a dosage-dependent reduction of penetration and an increase of softening point. However, experimental results did not reveal any effect related to changes of the type of crumb rubber.

Viscosity of the various blends was also found to be dosage-dependent. For each binder, measured viscosity values were fitted to a simple power-law model as indicated by the following equation:

$$\eta_T = \alpha_T \cdot T^{-\beta_T} \tag{1}$$

where η_T is the viscosity at temperature T (expressed in mPa·s), while α_T and β_T are material-dependent model parameters.

TABLE III. RESULTS OF EMPIRICAL STANDARD TESTS

Binder	pen_{25} (dmm)	$T_{R\&B}$ (°C)
X	53	47.6
A-5	37	55.4
A-10	31	62.1
B-5	40	54.0
B-10	32	62.3
C-5	39	55.2
C-10	30	61.9

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Values of model parameters are reported in Table 4 together with viscosity values calculated from power-law equations at 175 °C (η_{175}). Such values were compared to the prescriptions of ASTM standard D 6114, which for asphalt rubber products requires the viscosity at 175 °C to be comprised between 1,500 and 5,000 mPa·s [10]. It was thus observed that as a result of their low crumb rubber dosage the prepared blends exhibit a flow behavior that is significantly different from that of standard asphalt rubber binders, with a characteristic viscosity that is well below the minimum threshold value of the abovementioned range. This observation is encouraging, since it suggests that the use in practice of such blends may lead to only marginal adjustments of operating temperatures during the production and compaction of corresponding bituminous mixtures.

Results provided in Table 4 indicate that at each crumb rubber dosage, viscosity of the considered blends in the investigated temperature range was only slightly affected by variations of crumb rubber type. However, as already highlighted in a previous study [13], crumb rubber C, which was obtained from the processing of truck tires and exhibited the highest surface area per unit mass (Table 2), was characterized by the highest low-temperature stiffness (as revealed by the value of α_T) and temperature sensitivity (β_T), followed by crumb rubber B (the finest product) and crumb rubber A. Such effects suggest that as a result of variations of chemical composition and particle morphology, the different crumb rubber products activated local interactions with bitumen of variable intensity.

TABLE IV. RESULTS OF VISCOSITY TESTS

Binder	α_T	β_T	η_{175}
X	6.24E+15	6.184	84
A-5	1.63E+13	4.801	277
A-10	4.07E+14	5.234	739
B-5	2.56E+14	5.380	219
B-10	1.47E+16	5.918	778
C-5	3.93E+14	5.452	232
C-10	5.45E+16	6.193	701

B. *Fatigue Characterization*

The resistance of bituminous binders to fatigue was evaluated by carrying out oscillatory shear tests by means of a Dynamic Shear Rheometer (DSC). The instrument used was a Physica MCR 301 DSR from Anton Paar Inc., an air bearing stress-controlled device which can also operate in the strain-controlled mode through a feedback controlled loop. The DSR is equipped with a permanent magnet synchronous drive (minimum torque = 0.1 μNm, torque resolution = 0.001 μNm) and an optical incremental encoder for measurement of angular rotation (resolution < 1 μrad). An 8-mm parallel plate sensor system was used with a 2-mm gap between the plates.

Time sweeps were performed in a stress-controlled mode to evaluate the evolution of rheological parameters under sinusoidal loading. In order to avoid the effect of stiffness dependence on the damaging process, tests were carried out at equi-stiffness temperatures [20]. An initial complex modulus value of 15 MPa was selected as appropriate in order to limit machine compliance effects and to guarantee the occurrence of true fatigue associated with internal microdamage rather than instability flow. Equi-stiffness temperatures were preliminarily estimated by the interpolation of temperature ramps performed at a constant low strain levels. Adopted values were equal to 16 °C for the neat binder and the blends with 5% crumb rubber dosage, and 14.5 °C for the binders containing 10% crumb rubber. Oscillating shear stress, applied at a frequency of 10 Hz up to the point of 100% strain in the specimens, was set equal to 300 kPa for all tested binders.

Before running fatigue tests, each specimen was heated for three minutes at 50 °C and then kept at the test temperature for 45 minutes. The first conditioning phase was introduced to prevent slippage effects between the test specimen and steel plates that could lead to adhesion rupture and to limit steric hardening phenomena that might occur in premolded specimens. The second phase allowed a more uniform temperature distribution to be reached within each specimen.

A minimum of three repetitions were performed for each test and average values were considered in the subsequent analyses. All binders were tested in their original state, with no preliminary ageing treatment.

III. Results and Discussion

Examples of the results obtained from time sweep tests carried out on the reference binder and on two blends containing the same type of crumb rubber are displayed in Fig. 1 and Fig. 2, where measured values of the norm (G*) and phase angle (δ) of the complex modulus are plotted as a function of the number of loading cycles (N).

Coherently with the data reported in literature, three stages of response were identified [20-22]. In the first stage, which follows the onset of loading, sudden G* drops and δ increases were recorded, thus suggesting the occurrence of thixotropic effects and of temperature increases due to energy release [23-25]. In the following stage, the binders were progressively damaged by oscillatory loading with the formation of microcracks, with G* and δ decreasing and increasing, respectively, at a constant rate. Finally, in the third stage of testing an abrupt change of G* and δ was observed until failure as a result of formation of macrocracks [26,27].

It is interesting to note that the use of increasing dosages of crumb rubber gradually extended the duration of the second stage of response, thus indicating that the interactions taking place between the two components slowed down the evolution and propagation of microcracks. Moreover, it should be pointed out that the initial δ value was significantly affected by crumb rubber dosage, with a progressive enhancement of elastic response (i.e. lower δ values).

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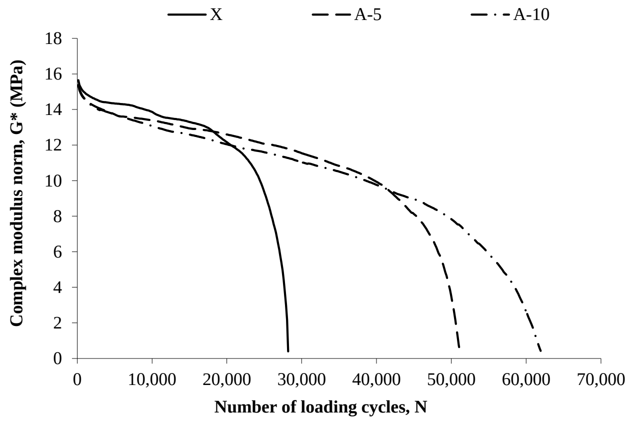

Figure 1. Evolution of G* as a function of loading cycles for blends with increasing crumb rubber dosage (example)

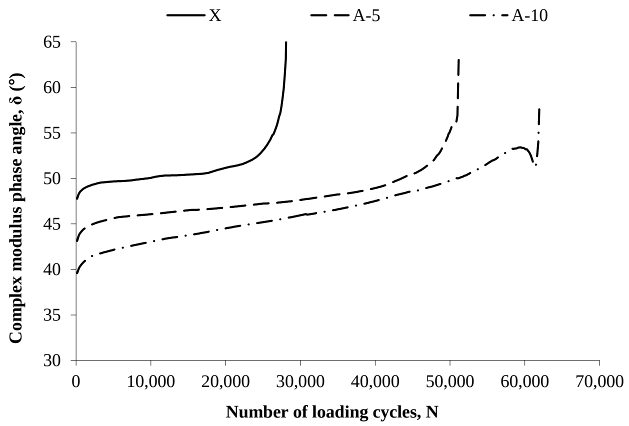

Figure 2. Evolution of δ as a function of loading cycles for blends with increasing crumb rubber dosage (example)

A synthetic description of the fatigue response of the considered binders requires the choice of an appropriate fatigue indicator. According to a phenomenological approach, this can be expressed in terms of the number of applied loading cycles which corresponds, depending upon the case, to a specific level of cumulated strain (N_f, for example relative to 100% strain) or to a predefined percent reduction of the initial modulus (for example $N_{50\%}$, relative to a 50% reduction) [28]. Alternatively, experimental data can be analyzed by referring to the energy dissipated during the evolution of the damage process, expressed in terms of the so-called Dissipated Energy Ratio (DER) [29,30]. Such a parameter is computed according to the formula presented in (2), where w_i and w_n are dissipated energy at cycle i and n, respectively.

$$DER = \frac{\sum_{i=1}^{n} w_i}{w_n} \tag{2}$$

The dissipated energy per cycle per unit volume is calculated as follows:

$$w_i = \pi \cdot \tau_i \cdot \gamma_i \cdot \sin \delta_i \tag{3}$$

where τ_i, γ_i and δ_i are shear stress amplitude, shear strain amplitude and phase angle at cycle i, respectively.

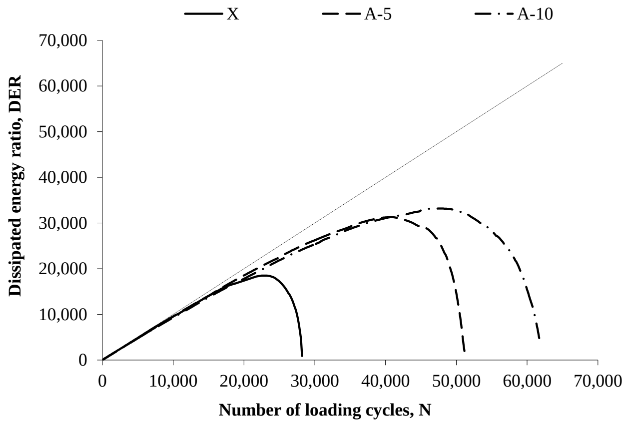

Figure 3. Evolution of DER as a function of loading cycles for blends with increasing crumb rubber dosage (example)

Examples of experimental results are given in Fig. 3, where, according to the energy approach described above, the DER parameter is plotted as a function of the number of loading cycles (N).

According to the interpretation proposed in literature [31], in stress-controlled tests once again three stages of response can be identified. The first one corresponds to initial phase of testing, in which experimental points overlap with the equality line (with 45° inclination), thus revealing that no significant damage occurs since the energy dissipated in each loading cycle remains constant. The following stage occurs as the experimental data points deviate from the equality line, with the corresponding initiation of microcracks which affect to an increasing extent the energy dissipated in each cycle. Such a stage progresses until a peak value of DER is reached, after which the specimen subjected to testing is completely failed. The transition from the crack initiation to the crack propagation phase is usually identified by number of loading cycles (N_p) which corresponds to the intercept between the equality line and the horizontal line passing through the peak value of DER. Moreover, a synthetic parameter which conventionally identifies failure can be obtained by simply considering the number of cycles (N_{DERmax}) corresponding to the peak value itself [32].

As shown in Table 4, all the fatigue life indicators described above, derived either from a phenomenological approach or from an energy-related analysis, were found to increase with crumb rubber dosage.

TABLE V. FATIGUE LIFE INDICATORS

Binder	**N_p**	**N_{DERmax}**	**$N_{50\%}$**	**N_f**
X	19,527	24,100	27,550	29,650
A-5	30,822	40,275	45,600	50,925
A-10	33,592	47,625	51,300	62,925
B-5	25,068	32,550	37,050	41,800
B-10	29,170	40,950	44,450	55,250
C-5	24,505	31,250	35,850	39,850
C-10	30,183	43,550	46,500	58,200

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This clearly confirms the effectiveness of the considered crumb rubber modifiers in enhancing the fatigue resistance of reference neat bitumen X. It should also be pointed out that the observed effects were very similar for the three crumb rubber types considered in the investigation, with recorded differences of the fatigue indicators that can be attributed to the inherent variability of test methods.

It should be underlined that the two energy-related indicators listed in Table 4 (N_p and N_{DERmax}) focus on characteristic points of the response under repeated loading, respectively associated to the onset of crack propagation and to failure. However, previous studies have shown that in the case of modified bituminous binders it may be of interest to refer to their relative difference expressed by means of the so-called Relative Crack Propagation Amplitude (RCPA), defined as [32]:

$$\mathrm{RCPA} = \frac{N_{DER\max} - N_p}{N_{DER\max}} \tag{4}$$

RCPA can be considered as a "fatigue ductility indicator" since it quantifies in relative terms the extension of the crack propagation phase. Low values of RCPA occur for materials which exhibit an abrupt fatigue rupture with a very short crack propagation phase, while high values of RCPA can be found for binders characterized by a greater ability to dissipate energy without collapsing which can be also beneficial in the perspective of damage healing.

As shown in Fig. 4, in the case of the blends considered in the investigation, RCPA values were found to increase almost linearly with crumb rubber dosage. This can be explained by referring to the internal structure of the blends, which as a result of interactions of increasing intensity causes a reduction of the speed of propagation of internal cracks. It is also interesting to observe that the three crumb rubber products provided equivalent effects, with very similar values of the RCPA parameter.

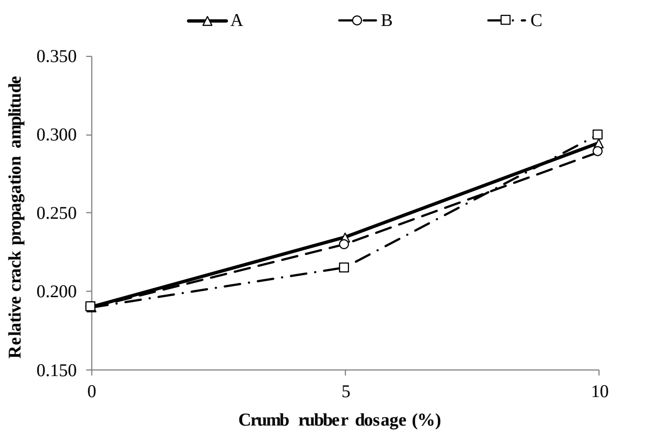

Figure 4. Variation of the RCPA parameter as a function of crumb rubber dosage for the considered blends

IV. Conclusions

The experimental results obtained in the investigation described in this paper clearly suggest that crumb rubber modifiers of the standard type can be employed in reduced quantities (up to a maximum of 10% by weight of the total binder) in order to yield blends with enhanced performance-related characteristics.

Such a conclusion is supported by the results obtained from viscosity tests and from controlled-stress time sweep tests carried out in equi-stiffness conditions. In particular, it was found that viscosity of the abovementioned blends is maintained within a range which is compatible with the adoption of the standard practices which apply to the mixing and compaction of bituminous mixtures. Moreover, it was observed that the use of increasing quantities of crumb rubber improves fatigue performance in terms of both fatigue life and crack propagation amplitude. Although crumb rubber products of different origin were considered in the investigation, experimental results suggest that their fatigue-related effectiveness was approximately equivalent with only marginal differences identified in terms of high-temperature flow behavior.

The results synthesized above are extremely promising from a practical point of view and will need to be validated by supplementary tests carried out on bituminous mixtures containing the same types of modified binders.

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