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Influence of rejuvenators on bitumen ageing in hot recycled asphalt mixtures

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HIGHLIGHTS

- Evaluation of bitumen service life including its reuse in hot recycling.
- Effect of different rejuvenators on the bituminous blends was analysed.
- Rejuvenated bitumens guarantee lower ageing than virgin bitumen alone.
- At the end of service life rejuvenated bitumen can be less stiff than virgin one.

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ABSTRACT

The use of reclaimed asphalt pavement (RAP) in new hot mix asphalt (HMA) by means of hot recycling techniques generates the advantage linked to the exploitation of both lytic and bituminous component, consequently leading to the decrease of both virgin aggregates and bitumen supplying. However, many agencies and public administration authorise RAP percentages ranges from 10% to 30% in hot recycling. The main reason for such a low amount of allowable RAP content is related to the aged bitumen contained in the RAP materials, which is more brittle than a virgin bitumen leading to a final mixture more susceptible to fatigue, thermal and reflection cracking. The use of rejuvenators has the potential to restore rheology and chemical components of aged RAP bitumen, thus allowing a significant increase in the amount of RAP to be properly implemented in HMA.

The experimental investigation is described in this paper and carried out through a dynamic shear rheometer (DSR) which provides the rheological characterisation of a paving grade bitumen during its overall service life including its reuse in hot recycling by adopting different rejuvenators.

Results show that rejuvenators modify bitumen chemistry and consequently rheology by enhancing the viscous response. Moreover, it was observed that oxidation is less harmful, in terms of stiffness increase, on the 50/50 aged bitumen - virgin bitumen blends (rejuvenated or not) than on the virgin bitumen. Moreover, the addition of a rejuvenator in a bituminous blend containing 50% of bitumen reactivated from RAP could lead to a corresponding composite bituminous phase less subjected to ageing phenomena and even less stiff at the end of service life than the associated virgin bitumen alone.

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1. Introduction

Nowadays, the implementation of high reclaimed asphalt pavement (RAP) contents in the production of new hot mix asphalt (HMA) represents a challenging issue for highways agencies and paving companies. The main factors that encourage the use of RAP in HMA are economic and environmental benefits, such as reducing production costs and disposal in landfills as well as preserving natural resources.

Although RAP has its origin from the demolition of damaged asphalt pavements, this material should not be considered as a waste product (Karlsson and Isacsson, 2006), since the important residual properties of bitumen and mineral aggregates contained in RAP could be profitably exploited through hot and cold recycling techniques.

Cold recycling allows reaching the highest amount of RAP to be reused. However, since in cold mix asphalt (CMA) RAP acts as a “black aggregate”, the low production temperature does not allow the reactivation of RAP bitumen that hardly interacts in an efficient manner with new bitumen (Grilli et al., 2012).

As regards hot recycling technique, the use of RAP allows a double advantage through the exploitation of both lytic and bituminous component (Al-Qadi et al., 2007), leading to a reduction of virgin aggregates and bitumen supplying in the production of HMA. However, many agencies and public administrations authorise low RAP percentages ranging from 10% to 30% in hot recycling, due to different concerns.

First, the phenomena of RAP bitumen mobilisation and its blending and interaction with the new virgin bitumen during mix production represent uncertainties which are still under investigation worldwide. Despite the considerable efforts in characterising the interaction between virgin and RAP bitumens (Booshehrian et al., 2013; Navaro et al., 2012; Nguyen, 2009) and their degree of blending (Bressi et al., 2016; Frigio et al., 2015; Shirodkar et al., 2011; Stimilli et al., 2015; Yousefi Rad, 2013), only little fundamental information is available in literature about the physicochemical phenomena and mechanisms during the mixing of a new HMA with RAP. Inaccurate assumptions on the effects of interaction could create problems in both mix design and pavement performance, leading to a final mixture more susceptible to cracking, ravelling, moisture damage and rutting (Dondi et al., 2016; Noferini et al., 2017; Zaumanis and Mallick, 2015).

Moreover, a critical issue is related to the ageing process, which affects physical and chemical characteristics of RAP bitumen entailing a general hardening of the final bituminous blend. As well known, bitumen undergoes two different ageing phases: short- and long-term ageing. The former represents the ageing during plant mixing, transportation and paving and is related to oxidation and lighter components evaporation. Whereas the latter occurs during pavement service life and is mainly linked to oxidation and physical hardening (Lu et al., 2017). Presence of water, local climate, thickness of the bitumen film and, mostly, mix porosity represent the main factors that influence the degree of long-term ageing.

Generally, ageing process causes a progressive change in bitumen rheological and chemical properties, leading to a reduction of the aromatic content and a consequent increase in the amount of resins (which in turn generate asphaltenes), whereas saturates remain essentially unchanged due to their poor reactivity (Lesueur, 2009). Since asphaltenes play a major role in determining bitumen viscosity, it is evident that oxidation causes a stiff behaviour (bitumen hardening) in addition to poor adhesion and reduction of coating properties.

In addition, the maximum amount of RAP to be reused in HMA production depends not only on the ability to correct the physicochemical characteristics of the aged bitumen, but also on the production technology (Mogawer et al., 2012). Considering hot in plant recycling, most conventional drum plants can accommodate 50% RAP, whereas the percentage of reusable RAP in batch plant ranges from 10% to 30% (Kandhal and Mallick, 1997).

When high amounts of RAP (30% or more) are introduced in the production of new HMA, the use of specific additives is strongly recommended to achieve adequate workability and final mechanical performance (Chen et al., 2007; Hajj et al., 2013; Shen et al., 2007; Tran et al., 2017; Xie et al., 2017; Yu et al., 2014). The additives should be non-hazardous and stable over a wide range of temperatures, from production to application. In addition, they must not experience any exudation or evaporation, in order to guarantee a good performance over asphalt pavement lifetime (Bocci et al., 2017; Grilli et al., 2015).

Among recycling additives, a distinction can be made between softening agents and rejuvenators. The softening agents aim at reducing aged bitumen viscosity, whereas rejuvenators attempt to restore the chemical and rheological properties of aged bitumen, thus ensuring long lasting HMA (Grilli et al., 2017, Tabakovi et al., 2017). Rejuvenators can have different nature, which reflects in the molecular structure and polarity (Zaumanis et al., 2014). In the last years, many products including tall oils, organic oils or recycled waste oils have been used worldwide to mobilise the aged bitumen in the RAP with the double benefit of possibly increasing RAP content in the mix and achieving good HMA performance. In particular, it has been observed (Booshehrian et al., 2013; Oldham et al., 2018) that the same target rheological properties, comparable to those of a virgin bitumen, can be obtained by mixing the aged RAP bitumen with a rejuvenator. However, the chemical composition (e.g. the ratio of asphaltenes to maltenes) of the rejuvenated binder is significantly different from that of the virgin bitumen, affecting the long-term performance of the RAP mix (Asli et al., 2012; Shen et al., 2007; Zargar et al., 2012).

Numerous studies have been undertaken on the effect of recycling additives within aged bitumen, but the ageing process of rejuvenated bitumen is still a challenge.

2. Objectives

The main objective of this research is the evaluation of the rheological properties of a neat paving grade bitumen considered during its overall service life, which includes its reuse in hot recycling by adopting different rejuvenators. To this purpose, bitumen properties have been investigated simulating different conditions of its service life: before and after HMA production, at the end of pavement service life, during hot recycling process, after laying of hot recycled HMA and, finally, at the end of the hot recycled HMA service life. In detail, the following aspects have been studied:

- assessment of the ageing effects on rheological properties of the virgin bitumen selected.
- determination of the interaction among virgin and RAP bitumens with/without rejuvenator and the related effects on the rheological properties of the corresponding composite bituminous blends.
- investigation of the ageing effects on the rejuvenated bitumens.

3. Materials

3.1. *Bitumen*

The binder selected was a 50/70 pen bitumen obtained as the residue from a visbreaking process. Visbreaking (i.e. viscosity breaking) is a relatively mild thermal cracking process operated at 455–510 °C and 0.3–2 MPa (3*20 bars) for a short residence time (1*3 min), which allows to reduce the viscosity of residua without attempting coke formation and significant conversion to distillates. The main limitation of the visbreaking process is that the products can be unstable. In fact, thermal cracking at low pressure gives olefins that, in turn, produces a very unstable material tending to undergo secondary reactions (oxidation) to form intractable residua (Speight, 1999).

The basic properties of the reference bitumen used in this study are summarised in Table 1.

3.2. *Rejuvenators*

In the present research, three commercial rejuvenators, commonly used for hot recycling, have been chosen. The first (rejuvenator type A) is a miscible crude tall oil derived from the processing of pine wood in paper industry, and contains fatty acids, resin acids and unsaponifiables. The second (rejuvenator type B) is a mix of different chemicals and consists of modified polyamines and vegetal oils. The third (rejuvenator type C) is an organic refined additive consisting of alkylates and fatty acids. The physical characteristics of the three rejuvenators are shown in Table 2.

4. Bituminous sample preparation

Starting from the virgin bitumen and the three rejuvenators above-mentioned, fifteen bituminous blends were artificially manufactured in laboratory to simulate the overall service life of a reference bitumen (i.e. ageing of a bitumen within a new HMA, rejuvenation of bituminous blends within new HMA containing high RAP content by means of three different additives and ageing of rejuvenated bitumens).

The first service life of the bitumen selected was simulated by subjecting it to a short- and long-term ageing, by means of rolling thin film oven test (RTFOT) and pressure aging vessel (PAV) testing procedures, in accordance with European Standards EN 12607-1 and EN 14769. In details, the RTFOT procedure requires an electrical heated convection oven at 163 °C that contains a vertical circular carriage to accommodate eight sample bottles covered by a film of bitumen for 85 min. The oven is also equipped with an air jet that blows air in each bottle while the carriage circulates. With respect to PAV, the equipment consists of a vessel including a cylindrical chamber with a top lid secured by means of a shear ring assembly composed of an aluminum lock ring and bronze shear ring segments. The chamber can accommodate ten sample pans placed in a sample rack. The combined action of temperature, which is set equal to 100 °C, and pressure of 2.1 MPa should be able to reproduce in only 20 h for the same effects caused by the climate agents on the pavements over a service period from 5 to 10 years.

Table 2 – Basic properties of the rejuvenators.

Rejuvenator	Density at $T = 20$ °C (g/cm^3)	Kinematic viscosity at $T = 20$ °C (mm^2/s)
A	0.93	98
B	0.80	45
C	0.88	47

Table 1 – Basic properties of the reference bitumen used.

	Code	Penetration at $T = 25$ °C (dmm)	Softening point (°C)	Penetration index	Retained penetration after RTFOT (%)
Virgin bitumen	VB	62	46–54	−1.5–0.7	>50

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It should be highlighted that the same aged bitumen obtained with the above-mentioned procedure was also used to reproduce the RAP bitumen. Hot recycling was simulated by blending 50% of the virgin bitumen, 50% of RAP bitumen and, in case, one of the three additives selected. The dosage of each additive complied with manufacturer instructions and previous experimental results as well (6% by RAP bitumen weight for both additives A and B and 3% by total bitumen weight for additive C).

All the bitumens investigated in this study are coded and summarised in Table 3.

5. Experimental program and test procedure

As bitumen is a viscoelastic material, it exhibits both elastic and viscous components of response and its stress–strain relationship is both temperature- and time-dependent. Bitumen rheology is consequently defined by its strain-stress-time-temperature response. However, within the linear viscoelastic (LVE) region, the interrelation between strain and stress is influenced by temperature and time alone and not by the strain magnitude (Van der Poel, 1954).

Based on the above-mentioned bitumen properties, a rheological characterisation of each bitumen selected was carried out by running tests, under different conditions, which allowed two main parameters to be determined: the LVE strain limit (γ_{lim}) and the complex modulus (G^*). Strain sweep tests were performed in order to determine the LVE region of the bitumens, i.e., where the norm of the complex modulus $|G^*|$ is not influenced by the strain magnitude. In particular, bitumen response was studied at a constant frequency ($f = 1.59$ Hz) by applying a range of strains ($\gamma = 0.3\%$–3%) for each temperature selected ($T = 4$, 16 and 28 °C), in order to monitor $|G^*|$ and thus determine the threshold of LVE region, γ_{lim} (defined as the strain corresponding to a $|G^*|$ deviation equal to 5% referred to its initial value $|G^*_{in}|$). Frequency sweep tests conducted under different temperatures and loading conditions represent a useful tool for a complete characterisation of bitumen viscoelastic properties. In addition, these tests implemented for thermo-rheologically simple bitumens at a strain within LVE region (i.e., $\gamma < \gamma_{lim}$) enable experimental data to be shifted between different frequencies and temperatures by applying the time–temperature superposition principle (TTSP). In particular, frequency sweep tests were carried out at a constant strain ($\gamma = 0.5\% < \gamma_{lim}$) over a range of frequencies (from 0.159 to 15.9 Hz) and temperatures (from 4 to 82 °C, with 6 °C intervals).

A dynamic shear rheometer (DSR) was used for both strain and frequency sweep tests. The tests were set up in plate–plate configuration (diameter of 8 mm and gap of 2 mm for low and intermediate temperatures; diameter of 25 mm and gap of 1 mm for high temperatures) and the sinusoidal load was applied in control-strain mode. In accordance with EN 14770, the test protocol provided 2 repetitions for each test. In case of result dispersion ($|G^*|$ out of the range of 10%), a third repetition was carried out.

Table 3 – Investigated bitumens.

Bitumen type		Bitumen preparation
Code	Name	
VB	Virgin bitumen	The reference bitumen 50/70
STAG	Short-term aged bitumen	Bitumen 50/70 subjected to a short-term ageing by means of RTFOT
LTAG	Long-term aged bitumen/RAP bitumen	Bitumen 50/70 subjected to a long-term ageing by means of RTFOT + PAV
LTAG + VB	Unrejuvenated bitumen	RAP bitumen blended with virgin bitumen 50/70 in proportion 50/50
LTAG + VB + A	Rejuvenated bitumen type A	RAP bitumen blended with 6% of rejuvenator A and virgin bitumen 50/70 in proportion 50/50
LTAG + VB + B	Rejuvenated bitumen type B	RAP bitumen blended with 6% of rejuvenator B and virgin bitumen 50/70 in proportion 50/50
LTAG + VB + C	Rejuvenated bitumen type C	RAP bitumen blended with virgin bitumen 50/70 in proportion 50/50 and 3% of rejuvenator C
LTAG + VB_STAG	Short-term aged unrejuvenated bitumen	Unrejuvenated bitumen subjected to a short-term ageing by means of RTFOT
LTAG + VB + A_STAG	Short-term aged rejuvenated bitumen type A	Rejuvenated bitumen type A subjected to a short-term ageing by means of RTFOT
LTAG + VB + B_STAG	Short-term aged rejuvenated bitumen type B	Rejuvenated bitumen type B subjected to a short-term ageing by means of RTFOT
LTAG + VB + C_STAG	Short-term aged rejuvenated bitumen type C	Rejuvenated bitumen type C subjected to a short-term ageing by means of RTFOT
LTAG + VB_LTAG	Long-term aged unrejuvenated bitumen	Unrejuvenated bitumen subjected to a long-term ageing by means of RTFOT + PAV
LTAG + VB + A_LTAG	Long-term aged rejuvenated bitumen type A	Rejuvenated bitumen type A subjected to a long-term ageing by means of RTFOT + PAV
LTAG + VB + B_LTAG	Long-term aged rejuvenated bitumen type B	Rejuvenated bitumen type B subjected to a long-term ageing by means of RTFOT + PAV
LTAG + VB + C_LTAG	Long-term aged rejuvenated bitumen type C	Rejuvenated bitumen type C subjected to a long-term ageing by means of RTFOT + PAV

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6. Analysis and discussion of the results

6.1. Identification of LVE region

Before running the frequency sweep tests, strain sweep tests were carried out on the long-term aged unrejuvenated bitumen (LTAG + VB_LTAG), which exhibits the worst condition in terms of brittleness, as a consequence of hardening due to ageing effects. In fact, the more severe the oxidation subjected by the bitumen considered, the higher the $|G^*|$ values measured and the lower γ_{lim} (Grilli et al., 2017). Results summarised in Table 4 and depicted in Fig. 1 allow the threshold of LVE region, γ_{lim}, to be identified and consequently a proper strain value within LVE region to be selected for frequency sweep test implementation.

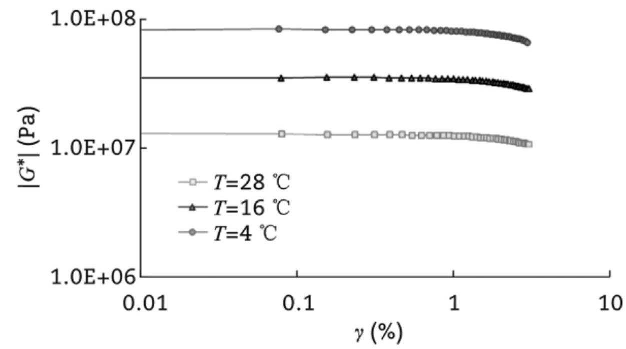

Fig. 1 – Strain sweep test isotherms for the long-term aged unrejuvenated bitumen.

6.2. Data analysis and modelling

Before the construction of the master curves, Black and Cole–Cole diagrams were plotted as useful representations of raw data obtained from frequency sweep tests for all the bitumens investigated. The Black diagram is the graph of the norm of the complex modulus $|G^*|$ versus the phase angle δ. A smooth curve in Black diagram is a useful indicator of time–temperature equivalency and thermo-rheologically simplicity for the bitumens tested (Lesueur et al., 1996) as well as no discrepancies in experimental results (Airey, 2002); moreover, through this plot the magnitude of the glassy asymptote can be reasonably estimated and used in the master curve construction. The Cole–Cole diagram is the graph of the loss modulus, G_2 as a function of the storage modulus G_1; it allows immediately appreciating the ratio between the two complex modulus components and thus estimating the preponderance of the viscous or elastic behaviour at the different temperatures investigated.

As resulted in the Black Diagrams depicted in the following sections of the current paragraph, all the bitumens studied can be considered as linear viscoelastic under the conditions considered and characterised as thermo-rheologically simple, thus TTSP was applied to generate complex modulus master curves and shift factor relationship. A reference temperature equal to 34 °C was selected and the rheological data at all the other temperatures were shifted with respect to time until the curves merge into a single smooth function. The modified Christensen-Anderson-Marasteanu (CAM) Model was adopted to relate the complex modulus norm to the reduced frequency, following a shift factor variation based on the Williams-Landel-Ferry (WLF) law. In detail, according to the modified CAM model, the equation for complex modulus norm is given by Eq. (1)

$$G^* = G_e^* + \frac{G_g^* - G_e^*}{\left[1 + \left(\frac{f_c}{f'}\right)^k\right]^{\frac{m_e}{k}}} \tag{1}$$

where $G_e^* = G^*(f \to 0)$ is the equilibrium complex modulus ($G_e^* = 0$ for bitumens while $G_e^* > 0$ for mixtures in shear), $G_g^* = G^*(f \to \infty)$ is the glass complex modulus, f_c is the location parameter with dimension of frequency (i.e., crossover frequency), f' is the reduced frequency (function of both temperature and strain), k and m_e are dimensionless shape parameters.

Fig. 2 illustrates the complex modulus master curve in Eq. (1). It can be seen that G_g^* is the horizontal asymptote at $f \to \infty$, and G_e^* is the horizontal asymptote at $f \to 0$. The G_e^* asymptote is zero for bitumens. The third asymptote is the one with a slope of m_e and represents the viscous asymptote. The G_g^* and m_e asymptotes intercept at f_c, whereas the intercept between G_e^* and m_e asymptotes is defined by Eq. (2)

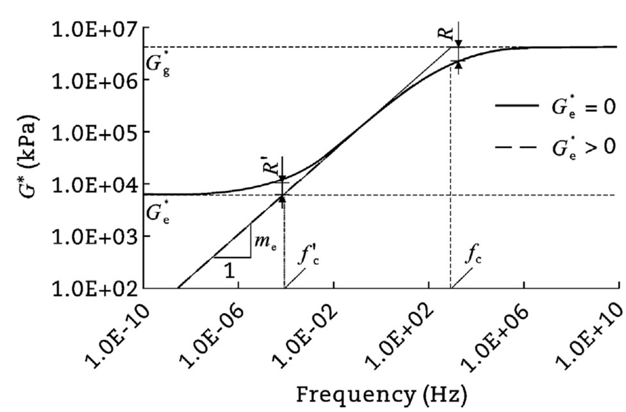

Fig. 2 – Model for complex modulus master curve (Bahia et al., 2001).

Table 4 – Strain sweep test data for the long-term aged unrejuvenated bitumen.

Bitumen	Repetition	T = 28 °C		T = 16 °C		T = 4 °C	
		$\|G^*\|$ (Pa)	γ_{lim} (%)	$\|G^*\|$ (Pa)	γ_{lim} (%)	$\|G^*\|$ (Pa)	γ_{lim} (%)
LTAG + VB_LTAG	1	1.21E + 07	1.53	3.33E + 07	1.44	7.85E + 07	1.40
	2	1.21E + 07	1.68	3.35E + 07	1.51	7.84E + 07	1.47

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$$f_c' = f_c\left(\frac{G_e^*}{G_g^*}\right)^{\frac{1}{m_e}} \tag{2}$$

where $f_c' = 0$ for bitumens.

The distance between $G^*(f_c')$ and G_g^* for bitumens is given by Eq. (3)

$$R = \log\frac{2^{\frac{m_e}{k}}}{1+\left(2^{\frac{m_e}{k}}-1\right)\frac{G_e^*}{G_g^*}} \tag{3}$$

where $G_g^* = 0$, thus $R = m_e/k \log 2$ for bitumens.

The distance between $G^*(f_c')$ and G_e^* for bitumens is given by

$$R' = \log\left\{1+\left(\frac{G_g^*}{G_e^*}-1\right)\left[1+\left(\frac{G_g^*}{G_e^*}\right)^{\frac{k}{m_e}}\right]^{\frac{-m_e}{k}}\right\} \tag{4}$$

where $G_g^* = 0$, thus $R' = \log 2$ for bitumens.

The WLF formulation is then used in the modified CAM model to express the temperature-shift factor

$$\log\frac{a_T(T)}{a_T(T_0)} = -\frac{C_1(T-T_0)}{C_2+(T-T_0)} \tag{5}$$

where $a_T(T)$ is the shift factor at temperature T, T_0 is the reference temperature, C_1 and C_2 are constants.

6.3. *Effects of ageing on virgin bitumen*

All the curves plotted in Fig. 3 and data summarised in Table 5 clearly show an increase in $|G^*|$ values, indicating a marked bitumen hardening, and a reduction in phase angles δ. Hence, the results indicate a prevalence of the elastic component G_1 on the viscous component G_2, as a consequence of the ageing subjected by the reference virgin bitumen. Moreover, considering the CAM model parameters summarised in Table 6, oxidation phenomena determine the following effects on the reference bitumen:

- lower shape index S, as an evidence of a higher sensitivity to frequency changes;
- lower crossover frequency f_c, suggesting a greater overall elastic component in the behaviour;
- higher C_1 and C_2 values, according to the reduction in fractional free volume (i.e., reduction of molecular mobility) (Mazzoni et al., 2016). Discussion part should explore the significance of the results of the work, not repeat them. A combined results and discussion section is often appropriate. Avoid extensive citations and discussion of published literature.

6.4. *Effects of bitumen rejuvenation*

Rejuvenators modify bitumen chemistry and, consequently, rheology by enhancing the viscous response of the bitumen (Nahar et al., 2014; Yu et al., 2014). This behaviour results in a decrease in $|G^*|$ values, indicating a bitumen softening, and a rise of phase angles δ, corresponding to a lower G_1/G_2 ratio, as shown in Fig. 4 and Table 7, comparing the rejuvenated bitumens to the associated composite bitumen without additive (LTAG + VB + A/B/C versus LTAG + VB). In addition, with respect to the CAM model parameters summarised in Table 8, the presence of each rejuvenator leads to the following effects on the corresponding composite bituminous blend (LTAG + VB + A/B/C versus LTAG + VB):

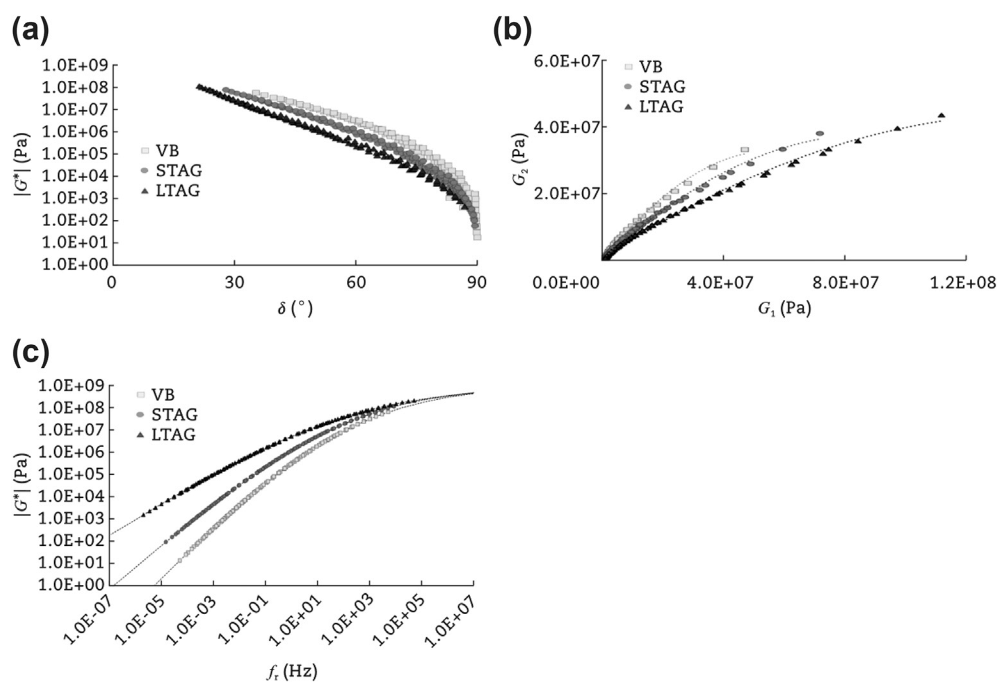

Fig. 3 – Rheological characterisation of the reference bitumen investigated at different stages of ageing (virgin, short- and long-term aged bitumen). (a) Black diagram. (b) Cole–Cole diagram. (c) Master curves of $|G^*|$ at 34 °C.

Table 5 – Rheological data obtained for the reference bitumen investigated at different stages of ageing (virgin, short- and long-term aged bitumen) for $f = 1.59$ Hz.

Bitumen	T = 4 °C				T = 58 °C			
	$\|G^*\|$ (Pa)	G_1 (Pa)	G_2 (Pa)	δ (°)	$\|G^*\|$ (Pa)	G_1 (Pa)	G_2 (Pa)	δ (°)
VB	2.09E + 07	1.49E + 07	1.46E + 07	44.3	3.60E + 03	2.58E + 02	3.59E + 03	85.9
STAG	3.74E + 07	3.12E + 07	2.06E + 07	33.5	1.63E + 04	3.03E + 03	1.61E + 04	79.3
LTAG	6.79E + 07	6.18E + 07	2.81E + 07	24.4	1.19E + 05	4.97E + 04	1.08E + 05	65.1

Table 6 – Calibrated parameters of the modified CAM model and WLF law for the reference bitumen investigated at different stages of ageing (virgin, short- and long-term aged bitumen).

CAM modified model + WLF law	$G^* = G^*_e + \frac{G^*_g - G^*_e}{\left[1+\left(\frac{f_c}{f'}\right)^k\right]^{\frac{m_e}{k}}}$							$\log(a_T) = \frac{C_1(T-T_0)}{C_2+(T-T_0)}$		
	G^*_e (Pa)	G^*_g (Pa)	f_c (Hz)	k	m_e	$S = \log\frac{2^{\frac{m_e}{k}}}{1+\left(2^{\frac{m_e}{k}}-1\right)\frac{G^*_e}{G^*_g}}$	R^2	C_1	C_2	T_0 (°C)
VB	0	1E + 09	360	0.16	1.12	2.107	0.996	15	168	34
STAG	0	1E + 09	290	0.16	0.96	1.797	0.995	21	210	34
LTAG	0	1E + 09	280	0.14	0.70	1.479	0.987	26	210	34

- higher shape index S, as an indication of a more gradual transition from the elastic to the viscous behaviour;
- higher crossover frequency f_c, and thus a remarkable retrieval of viscous component in the behaviour;
- lower C_1 and C_2 values, as a consequence of higher molecule mobility.

On the overall, the additives used are even approaching to restore the rheological properties of the virgin bitumen contained in each composite bituminous blend where they are implemented (LTAG + VB + A/B/C versus VB). This result is confirmed by the comparable values of the calibrated parameters summarised in Table 8 and the nearing of the rheological data and curves shown in Table 7 and Fig. 4 (LTAG + VB + A/B/C versus VB).

6.5. *Effects of ageing on hot recycled bituminous blends*

The results from the bituminous blends including 50% of RAP bitumen and 50% of virgin bitumen, with or without

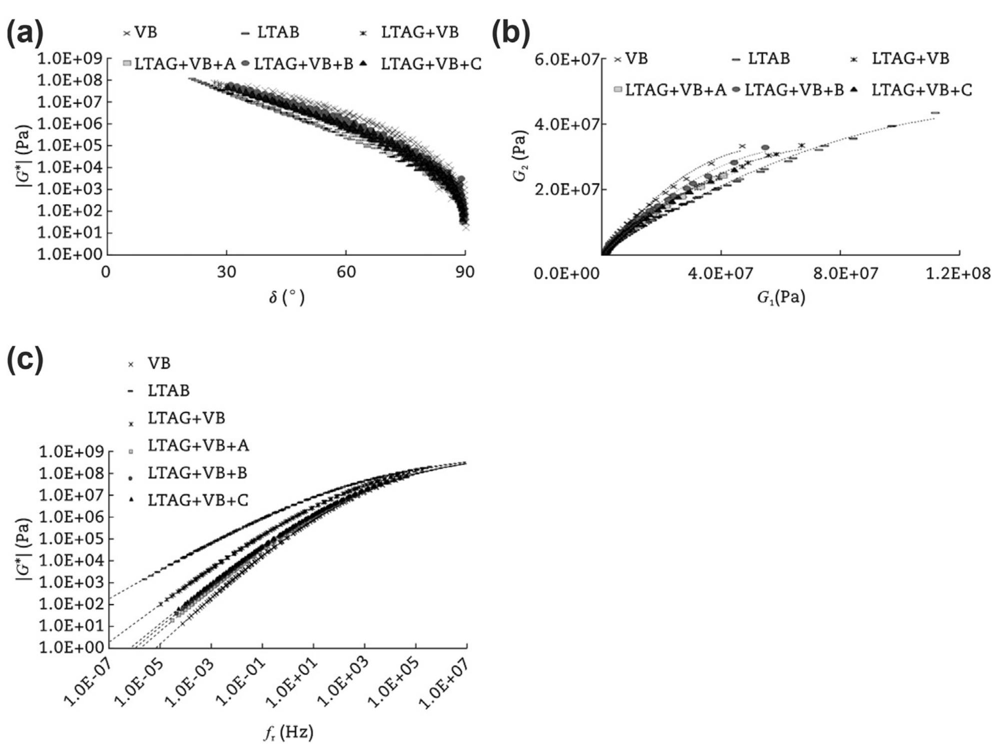

Fig. 4 – Comparison among the unrejuvenated bitumen and the rejuvenated bitumens with the associated virgin and long-term aged bitumens. (a) Black diagram. (b) Cole–cole diagram. (c) Master curves of $|G^*|$ at 34 °C.

Table 7 – Rheological data obtained for the unrejuvenated bitumen and the rejuvenated bitumens with associated virgin and long-term aged bitumens at $f = 1.59$ Hz.

Bitumen	$T = 4$ °C				$T = 58$ °C			
	$\|G^*\|$ (Pa)	G_1 (Pa)	G_2 (Pa)	δ (°)	$\|G^*\|$ (Pa)	G_1 (Pa)	G_2 (Pa)	δ (°)
VB	2.09E + 07	1.49E + 07	1.46E + 07	44.3	3.60E + 03	2.58E + 02	3.59E + 03	85.9
LTAG + VB + A	1.98E + 07	1.59E + 07	1.19E + 07	36.8	7.45E + 03	1.04E + 03	7.39E + 03	82.0
LTAG + VB + B	2.66E + 07	2.11E + 07	1.62E + 07	37.6	7.62E + 03	9.48E + 02	7.56E + 03	82.9
LTAG + VB + C	2.20E + 07	1.77E + 07	1.30E + 07	36.4	1.08E + 04	1.71E + 03	1.07E + 04	80.9
LTAG + VB	4.47E + 07	3.84E + 07	2.30E + 07	31.0	1.64E + 04	3.09E + 03	1.61E + 04	79.1
LTAG	6.79E + 07	6.18E + 07	2.81E + 07	24.4	1.19E + 05	4.97E + 04	1.08E + 05	65.1

Table 8 – Calibrated parameters of the modified CAM model and WLF law for the unrejuvenated bitumen and the rejuvenated bitumens with associated virgin and long-term aged bitumens.

CAM modified model + WLF law	$G^* = G_e^* + \frac{G_g^* - G_e^*}{\left[1+\left(\frac{f_c}{f'}\right)^k\right]^{\frac{m_e}{k}}}$							$\log(a_T) = \frac{C_1(T-T_0)}{C_2+(T-T_0)}$		
	G_e^* (Pa)	G_g^* (Pa)	f_c (Hz)	k	m_e	$S = \log\frac{2^{\frac{m_e}{k}}}{1+\left(2^{\frac{m_e}{k}}-1\right)\frac{G_e^*}{G_g^*}}$	R^2	C_1	C_2	T_0 (°C)
VB	0	1E + 09	360	0.16	1.12	2.107	0.996	15.0	168	34
LTAG + VB + A	0	1E + 09	360	0.16	1.06	1.994	0.993	18.5	190	34
LTAG + VB + B	0	1E + 09	400	0.16	1.03	1.938	0.994	17.0	180	34
LTAG + VB + C	0	1E + 09	400	0.16	1.01	1.900	0.991	15.5	165	34
LTAG + VB	0	1E + 09	380	0.16	0.92	1.731	0.969	22.5	200	34
LTAG	0	1E + 09	280	0.14	0.70	1.479	0.987	26.0	210	34

rejuvenator (LTAG + VB and LTAG + VB + A/B/C versus LTAG + VB_LTAG and LTAG + VB + A/B/C_LTAG), are shown in Tables 9 and 10 and the corresponding master curves, at different ageing levels, are plotted in Fig. 5. For these binders, the oxidation causes the same above-mentioned effects as for the reference virgin bitumen (Fig. 3 and Tables 4 and 5). However, since the hot recycled bitumen blends (rejuvenated or not) are composed of 50% RAP bitumen, oxidation is less detrimental on them than on the virgin bitumen, as confirmed by a lower variation of parameter values with ageing. Moreover, it should be highlighted that the addition of a rejuvenator in hot recycling leads to a composite bituminous blend which can be even less stiff (lower $|G^*|$ values) than the associated virgin bitumen alone at the end of service life, suggesting significant benefits when dealing with high RAP contents (Fig. 6) (LTAG + VB + A/B/C_LTAG versus LTAG). In detail, with respect to the associated reference bitumen alone, the rejuvenated bitumens type B or A guarantee lower ageing effects for $T < 58$ °C and a comparable or slightly higher hardening for $T \geq 58$ °C, respectively (Fig. 7) (LTAG + VB + B/A_LTAG versus LTAG). Whereas the long-term aged rejuvenated bitumen type C exhibits similar stiffness values at temperatures lower than 58 °C and a doubling in stiffness values at higher temperatures ($T \geq 58$ °C), if compared to the associated reference bitumen alone (Fig. 7) (LTAG + VB + C_LTAG versus LTAG).

In particular, with respect to the associated reference bitumen alone, the additive A provides a lower consistency to the corresponding rejuvenated bitumen also when it is unaged

Table 9 – Rheological data obtained for the hot recycled bitumens (rejuvenated or not) at $f = 1.59$ Hz investigated at different ageing stages.

Bitumen	$T = 4$ °C				$T = 58$ °C			
	$\|G^*\|$ (Pa)	G_1 (Pa)	G_2 (Pa)	δ (°)	$\|G^*\|$ (Pa)	G_1 (Pa)	G_2 (Pa)	δ (°)
LTAG + VB	4.47E + 07	3.84E + 07	2.30E + 07	31.0	1.64E + 04	3.09E + 03	1.61E + 04	79.1
LTAG + VB_STAG	5.88E + 07	5.28E + 07	2.59E + 07	26.2	8.75E + 04	3.45E + 04	8.04E + 04	66.8
LTAG + VB_LTAG	8.36E + 07	7.78E + 07	3.05E + 07	21.4	3.47E + 05	2.01E + 05	2.83E + 05	54.6
LTAG + VB + A	1.98E + 07	1.59E + 07	1.19E + 07	36.8	7.45E + 03	1.04E + 03	7.39E + 03	82.0
LTAG + VB + A_STAG	3.25E + 07	2.77E + 07	1.70E + 07	31.5	3.38E + 04	1.05E + 04	3.21E + 04	71.9
LTAG + VB + A_LTAG	5.39E + 07	4.91E + 07	2.23E + 07	24.5	1.42E + 05	7.10E + 04	1.23E + 05	60.0
LTAG + VB + B	2.66E + 07	2.11E + 07	1.62E + 07	37.6	7.62E + 03	9.48E + 02	7.56E + 03	82.9
LTAG + VB + B_STAG	4.01E + 07	3.46E + 07	2.02E + 07	30.2	3.13E + 04	8.84E + 03	3.00E + 04	73.6
LTAG + VB + B_LTAG	5.90E + 07	5.36E + 07	2.45E + 07	24.6	1.14E + 05	5.04E + 04	1.02E + 05	63.7
LTAG + VB + C	2.20E + 07	1.77E + 07	1.30E + 07	36.4	1.08E + 04	1.71E + 03	1.07E + 04	80.9
LTAG + VB + C_STAG	3.87E + 07	3.32E + 07	1.99E + 07	30.9	3.13E + 04	8.78E + 03	3.00E + 04	73.7
LTAG + VB + C_LTAG	6.57E + 07	6.02E + 07	2.64E + 07	23.7	1.99E + 05	1.02E + 05	1.71E + 05	59.2

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Table 10 – Calibrated parameters of the modified CAM model and WLF law for the hot recycled bitumens (rejuvenated or not) investigated at different ageing stages.

CAM modified model + WLF law	G^*_e (Pa)	G^*_g (Pa)	f_c (Hz)	k	m_e	$S=\log\frac{2^{\frac{m_e}{k}}}{1+\left(2^{\frac{m_e}{k}}-1\right)\frac{G^*_e}{G^*_g}}$	R^2	C_1	C_2	T_0 (°C)
	$G^*=G^*_e+\frac{G^*_g-G^*_e}{\left[1+\left(\frac{f_c}{f'}\right)^k\right]^{\frac{m_e}{k}}}$							$\log(a_T)=\frac{C_1(T-T_0)}{C_2+(T-T_0)}$		
LTAG + VB	0	1E + 09	380	0.16	0.90	1.693	0.970	19.5	175	34
LTAG + VB_STAG	0	1E + 09	260	0.15	0.78	1.565	0.982	22.0	185	34
LTAG + VB_LTAG	0	1E + 09	225	0.15	0.65	1.339	0.983	27.0	215	34
LTAG + VB + A	0	1E + 09	360	0.16	1.06	1.994	0.993	18.5	190	34
LTAG + VB + A_STAG	0	1E + 09	350	0.16	0.89	1.728	0.990	22.0	220	34
LTAG + VB + A_LTAG	0	1E + 09	240	0.16	0.75	1.457	0.988	27.5	240	34
LTAG + VB + B	0	1E + 09	400	0.16	1.03	1.938	0.994	17.0	180	34
LTAG + VB + B_STAG	0	1E + 09	340	0.15	0.86	1.726	0.990	22.0	200	34
LTAG + VB + B_LTAG	0	1E + 09	260	0.15	0.75	1.495	0.988	27.5	230	34
LTAG + VB + C	0	1E + 09	400	0.16	1.01	1.900	0.991	15.5	165	34
LTAG + VB + C_STAG	0	1E + 09	340	0.15	0.86	1.726	0.992	21.5	200	34
LTAG + VB + C_LTAG	0	1E + 09	250	0.17	0.73	1.332	0.990	24.0	220	34

or short-term aged, especially at $T < 58$ °C, as shown by the rheological data in Tables 9 and 5 (LTAG + VB + A and LTAG + VB + A_STAG versus VB and STAG). Whereas the presence of additives B in the bituminous blends allows obtaining optimum performance during all the service life, as confirmed by the positive rheological values in Table 9 compared to those from the associated reference bitumen in Table 5 (LTAG + VB + B pre- and post-ageing versus VB/LTAG). This positive behaviour results in a slightly higher stiffness for this rejuvenated bitumen when it is unaged or short-term aged (LTAG + VB + B, LTAG + VB + B_STAG), and lower stiffness after long-term ageing process (LTAG + VB + B_LTAG), suggesting a likely higher resistance to rutting, fatigue and thermal cracking, respectively. As concerns the rejuvenated bitumen type C, its mechanical response is quite comparable to that of the associated reference bitumen alone at the different stages of ageing, except for $T \geq 58$ °C when doubled stiffness values occur, as suggested by the rheological data in Tables 9 and 5 (LTAG + VB + C pre and post ageing versus VB/LTAG).

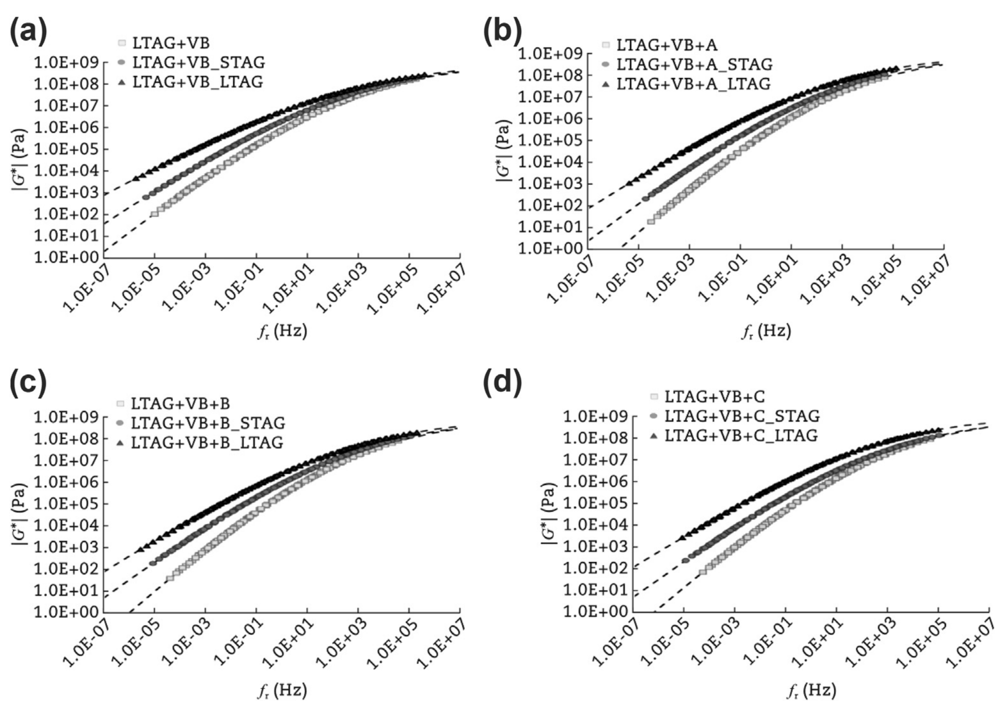

Fig. 5 – Master curves of |G*| at 34 °C for the hot recycled bitumens (rejuvenated or not) investigated at different ageing stages.

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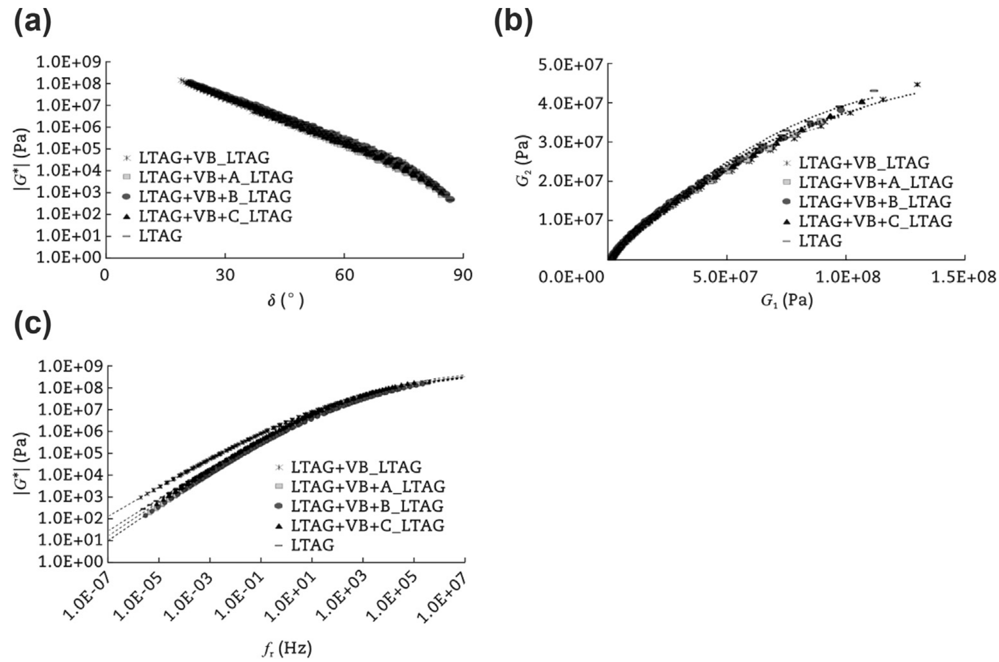

Fig. 6 – Comparison among all the long-term aged bitumens. (a) Black diagram. (b) Cole–cole diagram. (c) Master curves of |G*| at 34 °C.

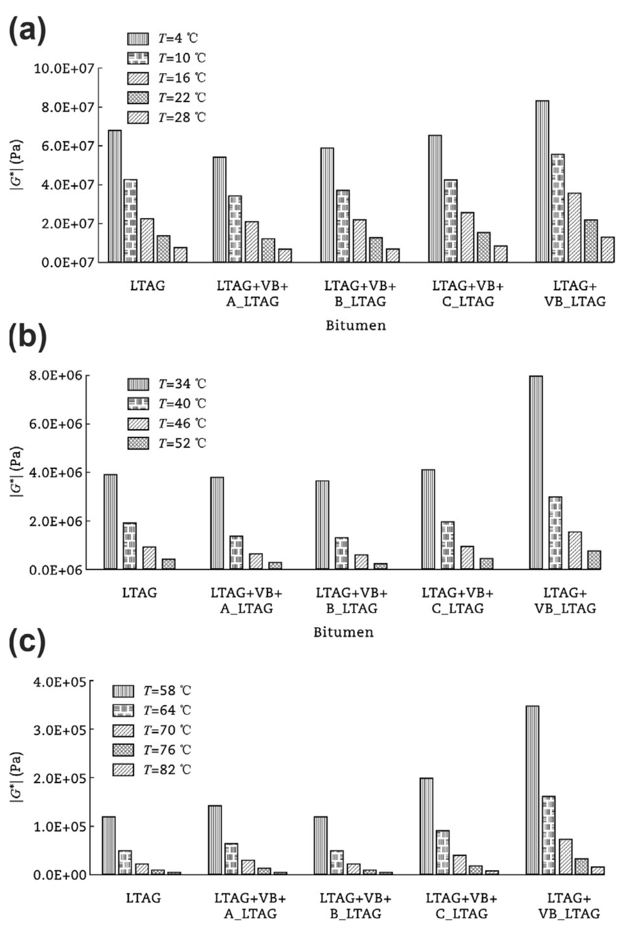

Fig. 7 – Comparison among the long-term aged bitumens investigated in terms of |G*| at f = 1.59 Hz.

7. Conclusions

The experimental investigation described in this paper focused on the assessment of the rheological properties of a reference bitumen considered during its overall service life that includes its reuse in hot recycling by adopting different rejuvenators.

Based on the results obtained, the following conclusions can be drawn:

- Ageing entails an increase in |G*| values, indicating a bitumen hardening, and a reduction in phase angles δ, corresponding to a prevalence of the elastic component G_1 over the viscous component G_2. In addition, the analysis of the master curve parameters shows a decrease in shape index S, as an evidence of a higher sensitivity to frequency changes, and in crossover frequency f_c, suggesting a greater overall elastic behaviour. Moreover, higher C_1 and C_2 values are a further consequence of oxidation according to the reduction in fractional free volume (i.e., reduction of molecular mobility).
- Rejuvenators modify bitumen chemistry and consequent rheology by enhancing the viscous response of the bitumen, as confirmed by a decrease in |G*| values and a rise of phase angles δ, corresponding to a lower G_1/G_2 ratio. In terms of master curve parameters, compared to the corresponding unrejuvenated bitumen, bitumens including any rejuvenator exhibit higher shape index S, as an indication of a more gradual transition from the elastic to the viscous behaviour, and crossover frequency f_c, and thus a remarkable retrieval of viscous component in the behaviour. Moreover, all the additives used are even

approaching to restore the rheological properties of the virgin bitumen, because of the comparable values obtained for both model and rheological parameters.

- Ageing has a similar effect on virgin bitumen and bituminous blends including RAP bitumen. However, as confirmed by a lower variation of parameter values pre and post ageing, oxidation is less detrimental on the hot recycled bitumens (rejuvenated or not) than on the virgin bitumen, since the former are composed of 50% of aged RAP bitumen.
- The addition of a rejuvenator in hot recycling leads to a corresponding composite bituminous blend which can be even less stiff than the associated virgin bitumen alone at the end of service life, suggesting significant benefits when dealing with high RAP contents. In particular, if compared to the associated virgin bitumen alone, the rejuvenated bitumens type B and A guarantee lower ageing effects in a wide range of temperatures (up to 58 °C), whereas in the same range of temperatures the rejuvenated bitumen type C exhibits similar stiffness values pre and post ageing. At higher temperatures ($T \geq 58$ °C) a comparable, slightly higher or even doubled hardening occur, when the additive B, A or C is selected as rejuvenator, respectively.

In conclusion, this experimental study provides an important contribution to understand the effect of ageing on the rheological properties of the bituminous blends containing virgin and RAP bitumens.

Based on the above-described findings, it is reasonable to expect that recycled asphalt mixtures prepared with virgin bitumens, rejuvenators and high amounts of RAP (which releases a percentage of the same aged bitumen) suffer less ageing phenomena and could also be less stiff than virgin mixtures. However, great importance should be given to the choice of the most suitable components of the mixture due to compatibility. In particular, type and dosage of rejuvenator should be properly selected, since their interaction strongly affects the final mixture performance. In addition, it is likely expected that using the same rejuvenators with a different base bitumen could change the corresponding bitumen response. Therefore, further analyses on different types of base bitumens are in progress for a better understanding of their influence on rejuvenation.

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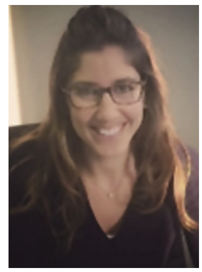

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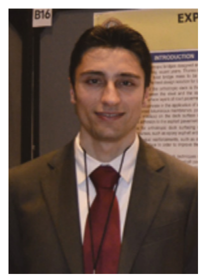

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