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Laboratory and In-Plant Validation of Hot Mix Recycling Using a Rejuvenator

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Abstract: This paper shows an experimental investigation developed in laboratory and in mix-plant for the validation of an asphalt concrete (AC) produced through the hot mix recycling (HMR) technique. A specific rejuvenator was used to improve the mechanical characteristics of the AC containing reclaimed asphalt (RA). The optimum dosage of the rejuvenator was selected on the basis of the viscosity of the bitumen recovered from RA. The AC produced with the optimum dosage of rejuvenator and 30% of RA was characterized in laboratory by means of indirect tensile strength (ITS) and indirect tensile stiffness modulus (ITSM). The results obtained in laboratory were validated by testing the AC produced in a mix-plant. The Fourier transform infrared spectrometry (FT-IR) was used to evaluate the interaction between rejuvenator and bitumen and to check the presence of the rejuvenator to produce the AC in the mix-plant.

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Key words*: Hot mix recycling; In-plant validation; Rejuvenator.*

Introduction

The new awareness of accounting sustainable constructions has led to standardize the characterization of reclaimed materials and to improve the recycling technologies [1, 2].

The most common technique for pavement maintenance provides the milling of the old asphalt layers and the overlay with new asphalt concrete (AC). As a consequence, a large amount of reclaimed asphalt (RA) is produced. The RA contains asphalt binder and mineral aggregates that have important residual properties. For this reason, RA should not be demoted to a waste material but it should be adequately reused.

The hot recycling of RA allows a typical AC to be obtained as final product. Indeed, no modification of the performance-related technical specification is expected when using RA during the AC production process. The main advantages of hot recycling are basically related to the use of RA as constituent materials, entailing the reduction in virgin aggregate and bitumen supply [3].

The hot recycling of RA can be carried out in-place or in-plant. The hot in-place recycling (HIPR) requires heating the old pavement to facilitate the milling and the mixing of the distressed AC with additional virgin aggregate, new bitumen and recycling agents [4]. Even though HIPR allows up to 100% of RA to be recycled, in some countries it is no longer used due to the substantial emission of fumes during production.

The hot in-plant mix recycling (HMR) considers limited amounts of RA, generally no more than 40%, for the production of AC. Particularly, the maximum amount of RA depends on the mix-plant facility and on the characteristics of RA [5, 6].

Usually the traditional production facility is modified to allow the RA to be heated before the mixing phase [3]. As a rule of thumb, higher the RA temperature before mixing, higher the amount of RA to be recycled. Indeed, if cold and wet RA is added directly in the mixing chamber, the virgin aggregates have to be superheated in order to ensure an appropriate mixing temperature. The excessive temperature of aggregates can cause the oxidation, or even the burning, of the bitumen that can compromise the durability of the final AC. On the contrary, a high RA temperature during the mixing phase improves the blending of binders and the mixture workability.

The characteristics of RA such as gradation, type and properties of the residual bitumen play a fundamental role in the mix design phase. Other characteristics such as homogeneity, foreign matters and water content significantly influence the quality of the production [7].

In order to increase and optimize the use of RA, the EN 13108-8 specifies requirements for the classification and description of RA as a constituent material for AC. Moreover, EN 13108-1 up to EN 13108-7 consider the use of RA in asphalt products (i.e. asphalt concrete, asphalt concrete for very thin layers, soft asphalt, hot-rolled asphalt, stone mastic asphalt, mastic asphalt, porous asphalt).

As it is well known, the bitumen during the service life of the pavement is subjected to an aging process which causes a progressive change in its physicochemical properties. As a consequence, bitumen in the RA often shows a stiff behavior, poor adhesion and reduced coating properties.

Therefore, when using a high content of RA, adequate workability and mechanical performance can be ensured by specific additives [8, 9]. Among recycling additives, a distinction can be made between softening agents and rejuvenators. The softening

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agents (e.g. asphalt flux oils) allow the reduction of viscosity of the aged bitumen, whereas rejuvenators (e.g. lube extracts or extender oils) restore the physical and chemical properties of the aged bitumen [10-14]. From a practical point of view, additives are generally easier to be managed than softer paving grade bitumens.

Objectives and Method

This paper summaries a technical experience aimed to improve the hot recycling of RA in a mix-plant by using a specific rejuvenator. The use of a soft grade bitumen was not propose for technical management problems at the specific mix plant. Indeed there was no possibility to store an additional bitumen. In this case the use a small amount of a rejuvenator facilitated the treatment.

The mix-plant was integrated with a crushing and screening system for the RA and a proportioner for liquid additives. The procedure was validated for recycling of 30% RA in the production of an AC for a base course. The optimization process consisted of four main phases:

- identification of the rejuvenator by means of Fourier transform infrared spectroscopy (FT-IR);
- determination of the optimum dosage of rejuvenator through viscosity tests;
- comparison between AC using the optimum dosage of rejuvenator and the reference AC with no rejuvenator in terms of mechanical characteristics;
- validation of the in-plant production.

Experimental Program

FT-IR can be used as a tool to identify the lab-produced and in-plant-produced binders [11, 15]. The spectroscopy uses of electromagnetic waves within a wide, continuous range of frequencies to study the interactions of matter with electromagnetic radiation. Fundamental vibrations, mainly stretching and bending of chemical bonds as well as some rotational motions in molecules, can be detected in the middle infrared region within the interval of wavelengths from 4000 to 400 cm^{-1}. Signal interferences are generated at each wavelength within a selected interval of Infrared (IR) frequencies by the interferometer. The intensity of signal, passing through the probing sample, is measured at each specific wavelength (or frequency, or wavenumber) by a detector resulting in an interferogram which is immediately transformed into an IR spectrum by the mathematical function Fourier Transform. In this case spectral data were obtained with a Perkin-Elmer Spectrum GX1 FT-IR, in transmission on NaCl plates. The spectral resolution was 4 cm^{-1}. Baseline (two-points linear fit) was performed in all cases, while second derivative, Fourier Self Deconvolution and LGC Curve Fitting (Low Gaussian character) procedures were used. For data handling, Spectrum v.6.3.1 (Perkin-Elmer) and Grams AI (Galactic Corp.) software packages were used.

The rejuvenator was preliminary identified by means of FT-IR. Then, FT-IR analysis on pure bitumen, lab-aged bitumen and lab-aged bitumen with different dosage of rejuvenator. was carried out to evaluate the interaction between bitumen and rejuvenator.

Viscosity tests were carried out on a plain bitumen before (virgin bitumen) and after long-term aging by means of PAV process (PAV-aged bitumen) at 60, 100, 135 and 160°C. Particularly, a dynamic shear rheometer (EN 13702-1) was used for testing at 60°C, whereas a rotational viscometer (EN 13702-2) was used for all the others temperatures. Moreover, viscosity tests were repeated on PAV-aged bitumen by adding different dosages of the rejuvenator (4, 4.5 and 5% by bitumen weight).

The optimum dosage of the rejuvenator to be added to the bitumen recovered from RA (i.e. the rejuvenator dosage that allows the correspondence between viscosity curve of aged bitumen and virgin bitumen to be obtained) was determined on the basis of the relationship between viscosity and dosage of rejuvenator.

Supported by practical experiences and some research studies [10, 13, 16], the amount of rejuvenator to be employed when producing AC was determined considering total melting of the old bitumen and total blending of binders.

Since viscosity tests cannot precisely predict the performance-related properties of recycled mixtures, two ACs were produced in laboratory with 30% of RA: the first one with the optimum dosage of rejuvenator (labACrej), the second one with no rejuvenator (labAC).

In laboratory, the selected mineral aggregates were heated at 170°C for 3 hours, while the RA was added to the aggregates and kept at 170°C for just 1 minute (simulating the specific in-plant procedure, in terms of aggregate/RA blending before mixing). The granular blend was then mixed with the virgin bitumen at 170°C, filler and, when needed, rejuvenator. After mixing the AC was stored. Afterwards, the AC was heated in oven at 170°C for 2 hours and compacted by means of a shear gyratory compactor (SGC).

Both ACs were compared in terms of compactability, indirect tensile strength (ITS) following the EN 12697-23 and indirect tensile stiffness modulus (ITSM) following the EN 12697-26.

The same ACs were also produced in a mix-plant adopting the same lab mixing conditions and evaluated in terms of ITS and ITSM for the validation of the procedure.

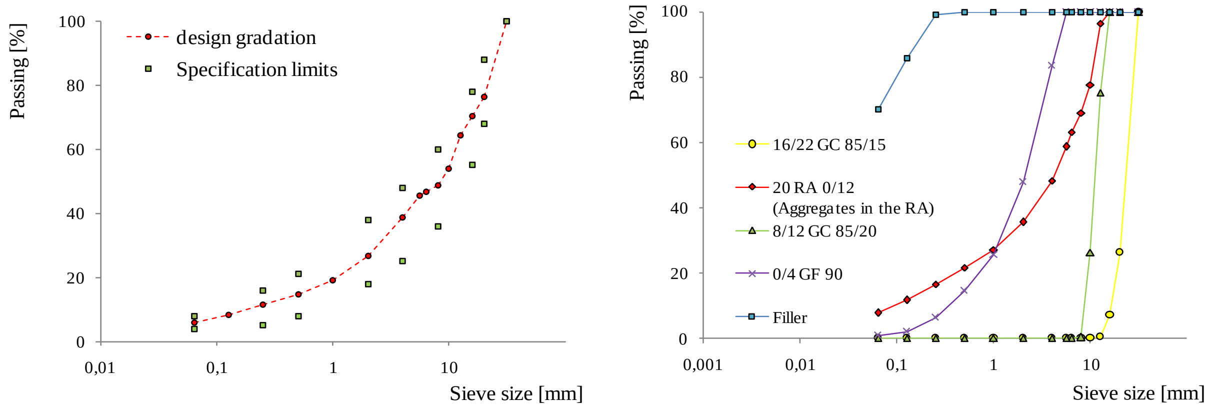
The FT-IR analysis on the bitumens extracted from in-plant produced AC was employed to verify the presence of rejuvenator.

Materials

Fig. 1 a) shows the adopted design granular gradation in comparison with the specification limits for a base course. The design granular mixture consisted of 30% of RA (20 RA 0/12), 32% of coarse crushed aggregate (16/22 G_C 85/15), 10% of medium coarse aggregate (8/12 G_C 85/20), 23% of crushed sand (0/4 G_F 90) and 5% of natural filler. The designation of the mineral aggregates and RA were established according to EN 13043 and EN 13108-8 respectively and Fig. 1 b) depicts the gradation of the single fractions.

Considering complete melting of RA, the gradation of aggregates in the RA (after extraction by solvent) was used to determine the design gradation. The bitumen content in the RA was 4.6% by aggregate weight. The recovered bitumen showed a penetration of 38 dmm and a softening temperature of 54°C.

A 70/100 paving grade bitumen (EN 1259) was selected for both laboratory investigation and mix-plant production. Considering the



(a) (b)

Fig. 1. (a) Design Gradation Curve and Specification Limits; (b) Gradation of Aggregates Used.

residual bitumen contained in RA and the ordinary procedure for base courses adopted by the specific mix-plant, the virgin bitumen content was 3.5% by aggregate weight.

The rejuvenator used has a density of 0.91 g/cm^3 and a viscosity of 60 mPa·s at 25°C, other details are not been disclosed. The amount of rejuvenator to be added was calculated on the basis of the weight of aged-bitumen. The specific tests showed hereafter confirmed its conformity with HMR.

Analysis of Results

Identification of the Rejuvenator by Means of FT-IR

As shown in Fig. 2, the rejuvenator spectrum has bands of a typical aromatic hydrocarbon skeleton with ester carbonyl moietes (free and bonded $\nu_{C=O}$ 1738 and 1726 cm^{-1} and $\nu_{C\text{-}O}$ around 1100 cm^{-1}).

As it is well known, aging induces the oxidation processes of the bitumen that can be revealed by additional absorptions around 1700 cm^{-1}.

For aged bitumen with rejuvenator, the band at 1700 cm-1 due to bitumen oxidation is masked by the doublet of the rejuvenator. The doublet of the latter (1726 and 1738 cm^{-1}) results modified in shape where intermolecular bonding between molecules of the system bitumen-rejuvenator (1726 cm^{-1}) are prevalent on intramolecular bonding (Fig. 3). Indeed the intermolecular bonding (1726 cm^{-1}) prevailed on intramolecular bonding (1732 cm^{-1}).

In addition, the intermolecular mode at 1738 cm^{-1}, is red shifted to 1732 cm^{-1}, that can be considered an evidence of interactions between the rejuvenator and the bitumen. This effect became more evident when increasing the percentage of rejuvenator (Fig. 4). However, it is difficult to hypothesize which groups of the bitumen can be involved in such bonding with the rejuvenator due to the number and complexity of the involved macromolecules [17, 18].

Determination of the Optimum Dosage of Rejuvenator Through Viscosity Tests.

Fig. 5 shows the results from viscosity test at different temperatures on the virgin bitumen (70/100), PAV-aged bitumen (70/100PAV), PAV-aged bitumens integrated with 4% (70/100PAV_4rej), 4.5% (70/100PAV_4.5rej) and 5% (70/100PAV_5rej) of rejuvenator.

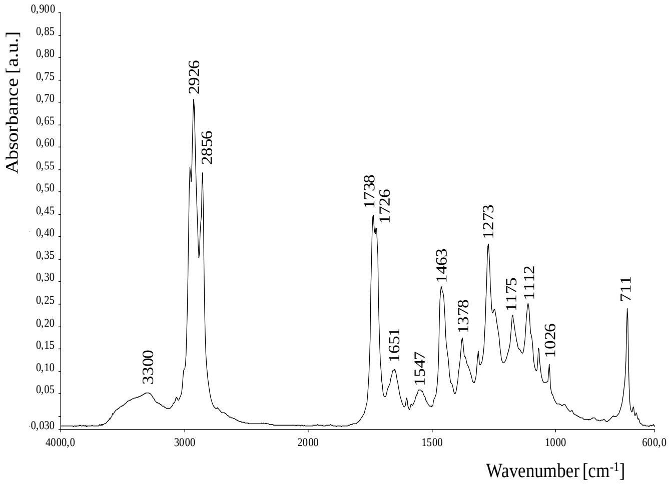


Fig. 2. FTIR Spectrum of the Rejuvenator.

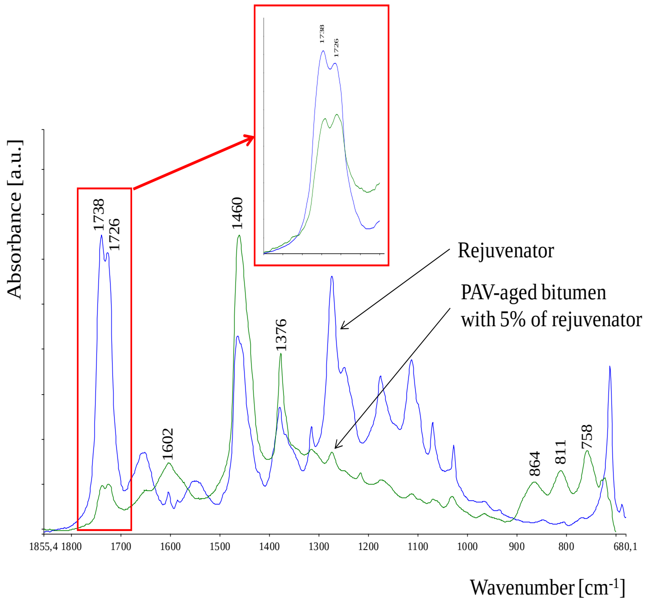


Fig. 3. Detail of the Intermolecular and Intramolecular Bonding.

As expected, the 70/100PAV viscosity curve is significantly higher than the 70/100 viscosity curve over the investigated temperature range, confirming the hardening of bitumen due to the

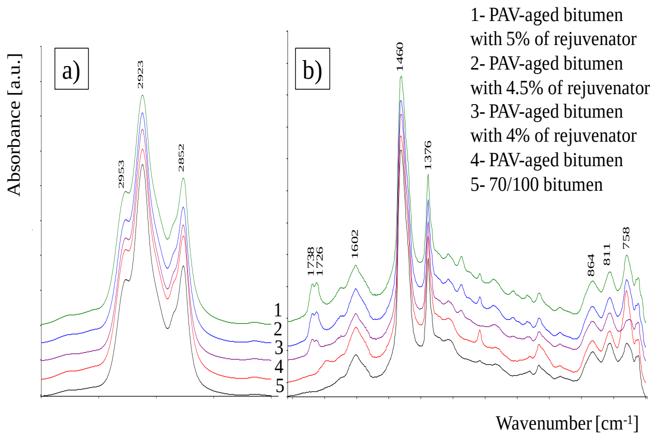


Fig. 4. FT-IR Spectra of Analyzed Bitumen in the Ranges: a) 3100-2700 cm^{-1} and b) 1800-700 cm^{-1}.

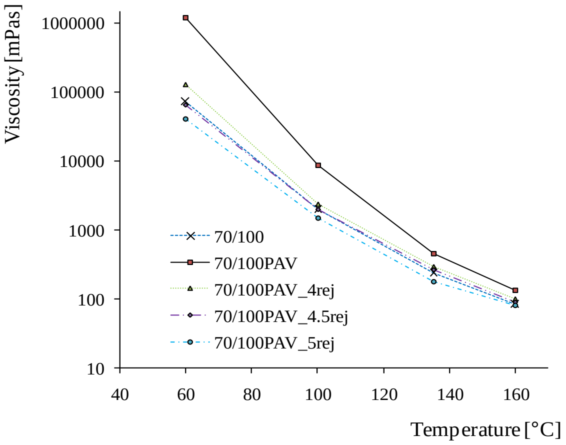


Fig. 5. Results of Viscosity Tests on Virgin Bitumen, PAV-aged Bitumen and PAV-aged Bitumen with Different dDosages of Rejuvenator.

aging process. In regard to viscosity curves related to PAV-aged bitumen integrated with rejuvenator, it can be noticed that the rejuvenator allows a reduction in viscosity at all temperatures to be achieved. Particularly, by adding 4.5% of rejuvenator to the PAV-aged bitumen, the viscosity curve of the aged-bitumen superimposed the viscosity curve of the virgin bitumen.

The specific rejuvenator reduced the viscosity of aged-bitumen at any temperatures and does not yield excessive fluidity at 60°C. This preliminary finding confirmed the selection of the rejuvenator for HMR.

The relationships between viscosity and dosage of rejuvenator for the PAV-aged bitumen are showed in Fig. 6. The exponential regression appeared suitable to well fit the experimental data at all testing temperatures.

For this specific case (i.e. considering the available RA and the selected bitumen), the optimum dosage of rejuvenator at all temperatures was calculated by operating a vertical shifting of the exponential curves before defined to meet the viscosity values of the bitumen recovered from the RA and fixing as target viscosity value the viscosity of the virgin bitumen at the corresponding temperature (Fig. 7).

Table 1 shows the calculated optimum dosage of rejuvenator at all testing temperature according to above mentioned procedure. As

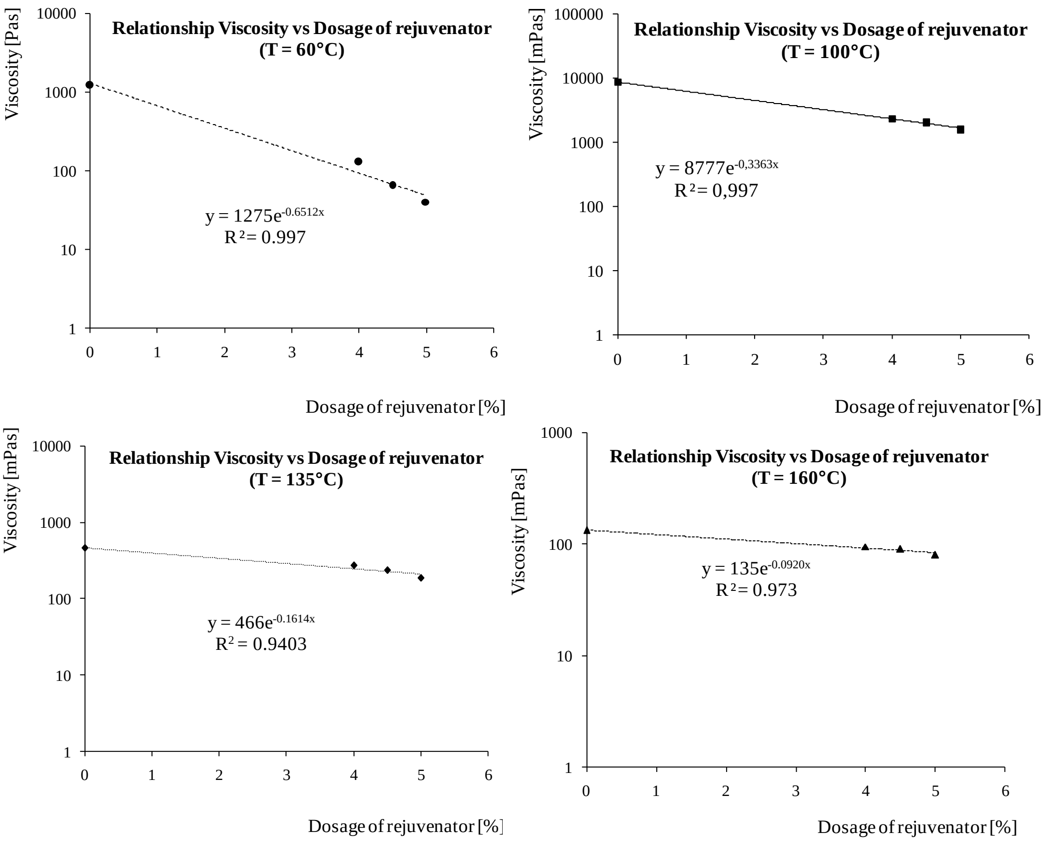


Fig. 6. Effect of the Rejuvenator Dosage on PAV-aged Bitumen

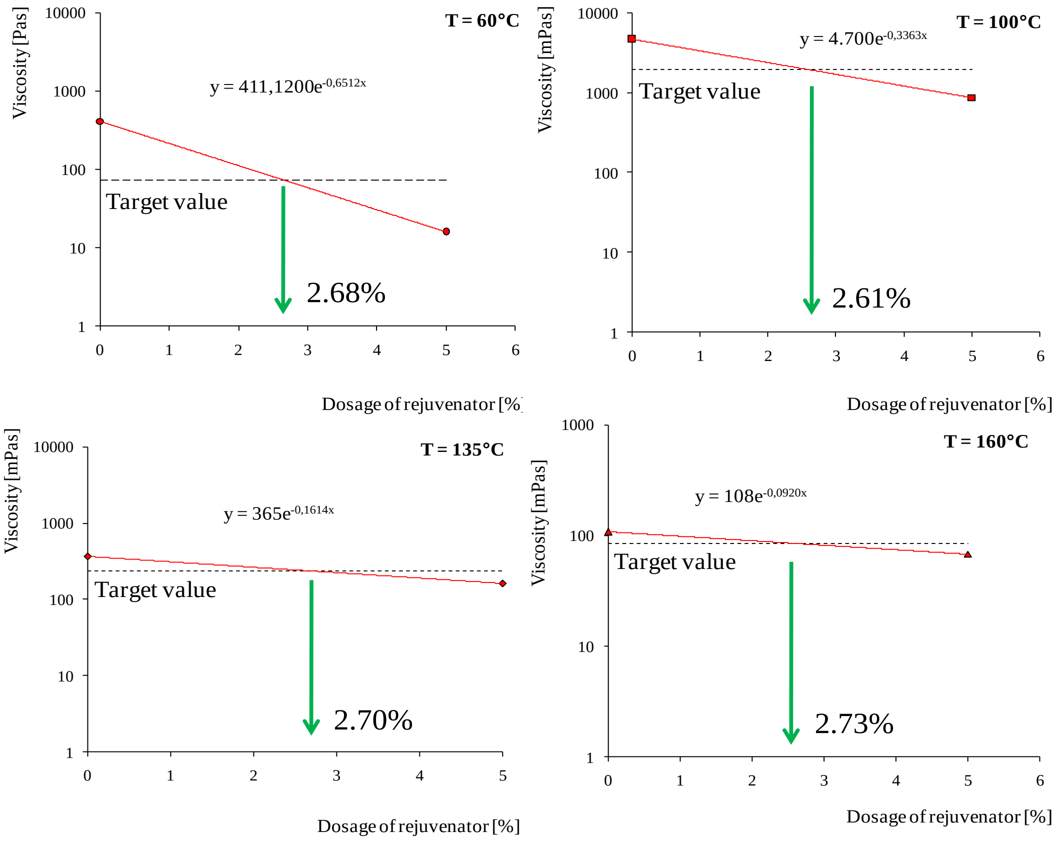


Fig. 7. Determination of the Optimum Dosage of Rejuvenator.

Table 1. Parameters Used for the Determination of the Optimum Dosage of Rejuvenator.

Determination of the Optimum Dosage of Rejuvenator				
Temperature [°C]	60	100	135	160
Regression slope	-0.6512	-0.3363	-0.1614	-0.0920
Viscosity of Recovered Bitumen [mPa.s]	411120	4700	365	108
Target Viscosity (Bit. 70/100) [mPa.s]	71880	1955	236	84
Optimum Dosage [% by Bitumen Weight]	2.68	2.61	2.70	2.73

it can be noticed, similar values of the optimum dosage of rejuvenator, ranging between 2.61 to 2.73% by bitumen weight, were obtained at different temperature. The average value (i.e. 2.68%) was selected as the amount of rejuvenator to obtain viscosity curve correspondence between recovered bitumen and virgin bitumen.

Further viscosity tests were carried out on recovered bitumen from RA with 2.68% of rejuvenator. Fig. 8 compares the viscosity curve of recovered bitumen from RA (bitumen characteristics to be corrected), virgin 70/100 (target viscosity curve) and recovered bitumen from RA with 2.68% of rejuvenator (results of the modification). Results show a similar viscosity trend between the virgin bitumen and the recovered bitumen with rejuvenator, thus validating the estimated optimum dosage of rejuvenator.

Comparison between AC Using the Optimum Dosage of Rejuvenator and the Reference AC.

The ACs produced in laboratory were compacted by means of a SGC under a compaction energy of 100 gyrations. Compactability

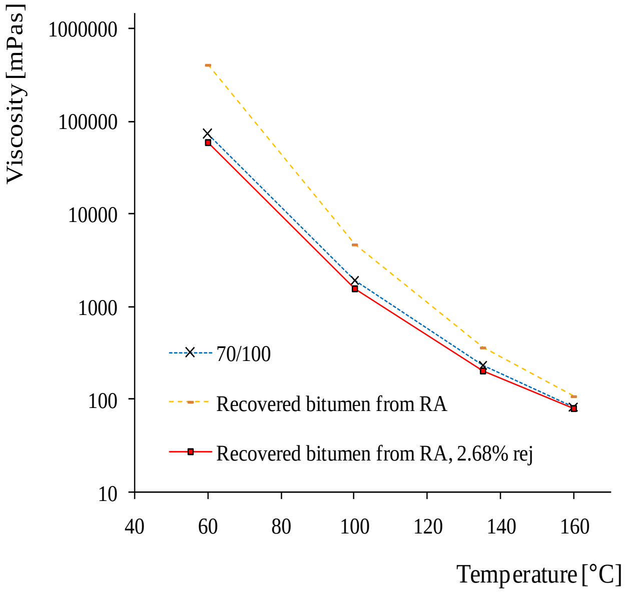


Fig. 8. Modification of Recovered Bitumen form RA with the Optimum Dosage of Rejuvenator.

Table 2. Compactability of ACs.

Air Void Content at 100 Gyrations [%]		
Specimen	LabAC	LabACrej
1	4.0	5.9
2	3.7	4.1
3	5.8	4.4
4	4.2	3.8
5	3.6	6.0
6	5.2	4.7
Average [%]	4.4	4.8
Specifications [%]	3 ÷ 5	

of ACs was evaluated in terms of air void content (EN 12697-8).As shown in table 2, both recycled ACs respected the specifications for a base course. Moreover, the compactability of ACs showed not significant differences. This probably depends on the high compaction energy transferred by SGC that seems to hide the lubricating effect of the rejuvenator.

Table 3 reports the ITS and ITSM values for both ACs. From the analysis of results, it is evident that the AC with no rejuvenator shows failure strength and elastic modulus significantly higher than AC with rejuvenator. In particular the use of the rejuvenator in the AC allowed a reduction of strength and stiffness of about 30 % to be achieved. The excessive stiffness of recycled mixture is due to the presence of the old and hard bitumen from RA that produce stiffer bituminous bonds [19]. Whereas the rejuvenator makes the binder blend less stiff reducing strength and modulus of AC. This finding allows to state that the rejuvenator improves the ductile properties of recycled AC ensuring the respect of technical specifications and preserving the mixture from fatigue-related issues [20].

Validation of the In-plant Production

This section deals with the in-plant production of the two recycled ACs on the basis of materials and proportions designed in laboratory. The main object was to evaluate the reliability of the in-plant production process in terms of effective dosage of rejuvenator added and dispersed in the recycled mixture. To this end, FT-IR analysis on the bitumen extracted from in-plant produced AC was used as control method. In addition, the mechanical characterization of produced ACs allowed the effectiveness of rejuvenator to be evaluated and thus the laboratory procedure aimed to design rejuvenator content to be validated.

Fig. 9 depicts the infrared spectra in the region 1850-600 cm^{-1} of recovered bitumen from AC productions with and without rejuvenator (bands frequencies are shown in Fig. 2). As expected, the spectroscopic determinations showed that the bitumen with no rejuvenator has the shoulder at 1692 cm^{-1}, due to oxidation processes (blue spectrum). On the contrary, the spectrum of the bitumen integrated with 2,68% of rejuvenator did not clearly show this shoulder which is hidden by the tails of the two near bands. The band at 1726 cm^{-1} (intermolecular bonding of the rejuvenator) confirms the presence of the rejuvenator. Considering that the spectrum of the rejuvenator shows two carbonyl bands of approximately the same intensity at 1738 and 1726 cm^{-1} (intra- and intermolecular bonding), the lack of the former mode means that the rejuvenator undergoes bonding not only with itself but also with the bitumen.

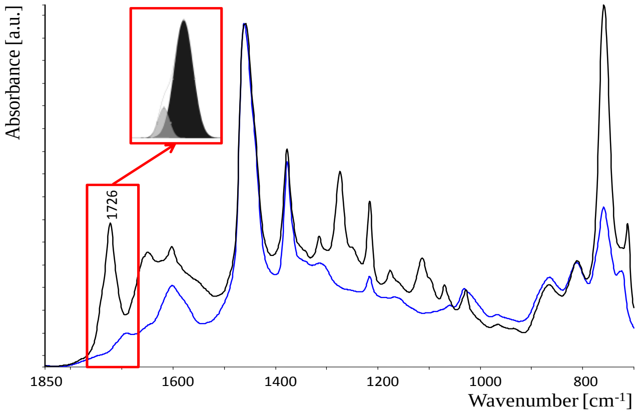


Fig. 9. Infrared Spectra in the Region 1850-600 cm^{-1}of Recovered Bitumen from AC Productions.

Table 4. Compactability of in-plant Produced ACs.

Air Void Content at 100 Gyrations [%]		
Specimen	Mix-plantAC	Mix-plantACrej
1	2.9	3.3
2	3.3	3.2
3	3.3	3.7
4	3.9	3.8
5	3.4	3.3
6	3.0	3.4
Average [%]	3.3	3.4
Specifications [%]	3 ÷ 5	

The ACs produced in the mix-plant were taken and stored in laboratory. In a second phase the material was heated in oven at 170°C for 2 hours and compacted by means of a SGC following the same protocol adopted for lab-produced specimens. As shown in table 4, both ACs meet the technical specifications for a base course as they achieved an acceptable residual air void content. Moreover

Table 3. ITS and ITSM Values of Investigated ACs.

Indirect Tensile Strength [N/mm^2]			Indirect Tensile Stiffness Modulus [MPa]		
Specimen	LabAC	LabACrej	Specimen	LabAC	LabACrej
1	1.42	1.00	4	8674	5791
2	1.39	1.02	5	7282	5616
3	1.55	1.01	6	8255	5850
Average [N/mm^2]	1.45	1.01	Average [MPa]	8070	5752
Specifications [N/mm^2]	0.75 ÷ 1.35		Specifications [MPa]	5500 ÷ 1200	

Table 5. Values of ITS and ITSM of In-plant Produced ACs

Indirect Tensile Strength [N/mm^2]			Indirect Tensile Stiffness Modulus [MPa]		
Specimen	labAC	labACrej	Specimen	Mix-plantAC	Mix-plantACrej
1	1.55	1.30	4	13620	10199
2	1.56	0.99	5	13689	9825
3	1.38	1.33	6	13333	11010
Average [N/mm^2]	1.50	1.21	Average [MPa]	13547	10345
Specifications [N/mm^2]	0.75 ÷ 1.35		Specifications [MPa]	5500 ÷ 1200	

ACs showed not significant differences in terms of volumetric properties confirming the results found for laboratory specimens.

Table 5 shows the result of mechanical characterization of the in-plant produced ACs in terms of ITS and ITSM. Mixture with the rejuvenator showed strength and stiffness properties which are considerably lower than that of mixture with no rejuvenator. Such results are consistent with the laboratory evidences confirming the effectiveness of the rejuvenator to soften the old asphalt binder. The performance of AC with rejuvenator satisfied the technical specifications and gave further confirmation of the reliability of the mix-plant production process. Moreover, it can be seen that in-plant produced ACs showed higher performance values as compared with the lab-produced ACs. This is probably related to the higher compaction level and the different effects of the heating temperature during production.

Conclusions

This paper deals with an experimental investigation intended to optimize the HMR using 30% RA and a specific rejuvenator for the production of an AC for base courses. Particularly, the rejuvenator content was determined on the basis of viscosity criterion of recovered binder. FT-IR analysis was proposed to control the chemical modification of binder due to the rejuvenator. Moreover, the influence of such a rejuvenator on the performance-based properties of AC was investigated by means of ITS and ITSM tests.

The overall result analysis allowed the laboratory procedure for the determination of the design rejuvenator content and the in-plant production process of recycled mixture to be validated. In addition, the FT-IR resulted an useful tool analysis to evaluate the presence of specific additives in the AC and thus to improve the quality control of a mix-plant production process.

In detail, the following conclusions can be drawn:

- the rejuvenator was identified by FT-IR and showed evidence of interactions with the bitumen;
- the use of rejuvenator influenced the viscosity of the aged bitumen, which allows better AC mechanical performance to be expected;
- the analysis of ITS and ITSM for laboratory and mix-plant produced specimens highlighted lower stiffness properties of ACs using rejuvenator denoting the effectiveness of the additive to give higher ductile properties to the recycled mixture. However, a proper characterization for evaluating the rutting potential of recycled mixtures containing the rejuvenator is needed;
- the FT-IR analysis on recovered bitumen from ACs produced in the mix-plant confirmed the presence of the rejuvenator.

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