Case Studies in Construction Materials 15 (2021) e00556

Contents lists available at ScienceDirect

Case Studies in Construction Materials

journal homepage: www.elsevier.com/locate/cscm

Laboratory investigation on the use of recycled materials in bituminous mixtures for dense-graded wearing course

Claudia Nodari*, Maurizio Crispino, Emanuele Toraldo

Department of Civil and Environmental Engineering, Politecnico di Milano, 20133, Milan, Italy

A R T I C L E I N F O

Article history:
Received 25 November 2020
Received in revised form 16 April 2021
Accepted 17 April 2021

Keywords:
Reclaimed asphalt
Stabilized bottom ash
Waste engine oil
Mechanical performance
Recycled bituminous mixtures

A B S T R A C T

Reduce, recycle and reuse of materials are the three crucial procedures that affect the economy of the current century, which are mandatory in preserving and protecting the planet for the next generations. In this field, the construction of road pavements plays fundamental role since huge amount of materials are involved, particularly the bituminous mixtures. This is the reason why the efforts of the scientific community were, and still are, addressed to investigate alternative pavement solutions in which recycled and reused materials, which mostly come from end-of-life pavement removal, can be included with the goal of reducing landfill operations. Moreover, a lot of these efforts were directed to reuse of recycled materials in producing bituminous mixtures for structural pavement layers, and not to the wearing course. Therefore, in order to be a step ahead of the current technical and scientific approaches, this research focused on the study of the effects of the concurrent use of reclaimed asphalt (RA), stabilized bottom ashes (SBA) from urban waste incinerators, and waste engine oil (WEO) in substitution of commercial rejuvenators. Moreover, the benefits deriving from the use of polymer additives were assessed. In this regard, a set of compaction, volumetric and mechanical tests were performed at laboratory scale, demonstrating that it is possible to use high amounts of RA (20 % and 60 %) in bituminous mixtures for wearing courses, also combined with SBAs and WEO. More in detail, the obtained results demonstrate that: both RA and SBAs generally increase mixtures' stiffness (as confirmed by stiffness Master Curves and Indirect Tensile Strength results); SBA can be used as artificial aggregates; and WEO is able to act as fluxing agent to obtain mixture's viscosity reduction.

1. Introduction

Socio-economic development in the past few decades caused nature uncontrolled exploitation. Considering the rapid reduction of natural resources, in order to preserve and protect the planet for the next generations, society is looking for new sustainable alternatives [1]. Within the framework of a sustainable development, to reduce the global environmental footprint, recycling the road pavement materials is of high importance [2].

Among these road pavement materials, bituminous mixtures play an important role. Bitumen is a by-product of crude oil refinery, and aggregates are obtained from quarries; thus, both the main components of the standard bituminous mixtures have undeniable negative environmental footprints. Furthermore, bituminous layers will deteriorate during the pavement

* Corresponding author.
E-mail addresses: claudia.nodari@polimi.it (C. Nodari), maurizio.crispino@polimi.it (M. Crispino), emanuele.toraldo@polimi.it (E. Toraldo).

https://doi.org/10.1016/j.cscm.2021.e00556

service life and are needed to be removed [3]. The bituminous layers removal can be carried out by milling and crushing processes, which produce the so-called reclaimed asphalt (RA) and can be re-used as key material of new bituminous mixtures. Other recycled and artificial key materials are emerging as an environmental-friendly option, such as stabilized bottom ashes (SBAs) from urban waste incinerators and waste engine oils (WEOs) as RA aged fluxing agent. Such recycled and alternative components are currently studied as key materials for pavement structural layers, a lack of scientific literature can be observed about their use for surface wearing courses.

2. Background and literature review

The available literature demonstrated that high amounts of RA content into new bituminous mixtures can affect some performances in terms of workability [4], fatigue life [3,5,6], stiffness [5–7], tensile strength [8] and rutting resistance [5,6,9,10]. It mainly because aged bitumen presents rheological characteristics variation (physical hardening), resulting in an unbalancing of the asphaltenes maltenes ratio. It is the reason why to reactivate the aged binder, rejuvenator additives can be used [1,3,8,9,11–13]. Rejuvenator type must be selected carefully to achieve both short-term and long-term criteria [3]. In fact in the short-term, a rejuvenator must propagate quickly into the RA binder, ensuring a convenient workability during production and laying operations, in the case in which it is the sole benefit deriving from the additive it is better to name it as fluxing agents, and no effects on the aged bitumen composition can be expected. On the contrary, as a long-term criteria, a rejuvenator must modify also the aged binder chemical composition and, thus, its rheological behaviour [3].

In the context of sustainable development, it is possible to replace commercial rejuvenators with recycled oils, such as waste engine oil (WEO). In fact, from a theoretically point of view, WEO, as petroleum-based product, has similar molecular structure as bitumen. Therefore, it can be used as a binder modifier or rejuvenator for RA bitumen [14]. WEO is contaminated engine oil, a product used for engine operation; in which heavy metals (such as lead, zinc, calcium and magnesium) can be expected [14]. The available literature demonstrated that the use of WEO softens bitumen, resulting in decrease of optimum binder content [14], decrease the softening point [11,14], increase the penetration, reduce mixing temperature and improve of bituminous mixtures workability [14].

Stabilized bottom ashes (SBAs) derived from municipal solid waste (MWS) incineration are another promising recycled material that theoretically can be used as aggregate into bituminous mixtures. SBA is one of the products, that is strategic in the waste management process due to advantages of energy production and volume reduction [15,16]. However, SBA contains heavy metals and has variable mechanical properties [16,17]. In general, SBA has composition similar to natural aggregates [17,18]. The use of SBA (as artificial aggregate) into bituminous mixtures does not affect workability and increases optimum binder content [19], voids [17], rutting resistance [19,20] and stiffness of bituminous mixtures [20].

Within the framework of circular economy in which sustainability is the key element, the production of high-quality bituminous mixtures must not be neglected, also considering increased traffic loads and climate changes [21]. In view of this, it is possible to use polymer-modified bitumen or additives (added directly during the bituminous mixtures production) to improve binder and mixture performances [22]. In detail, the addition of polymer increases rutting resistance [21] and affects the dynamic modulus, as shown in mixtures' Master Curves with reduction in stiffness at high frequencies and low temperatures [22].

3. Objectives

Considering the above-mentioned scientific literature, the main objective of this research study is to evaluate the effects of the concurrent use of polymer additives, SBA (in substitution of natural aggregates) and WEO (in substitution of commercial rejuvenator) on compaction properties, volumetric characteristics, and mechanical performance of surface course bituminous mixtures with high RA contents. Therefore, the effort is focused on the development of recycled bituminous mixtures, that meet sustainability and mechanical performance in wearing course.

4. Materials and methods

4.1. Key materials and mixtures

The key materials involved in the investigation were:

- natural lithic aggregates, provided by a local contractor, selected in the range of 0–3 mm, 4–8 mm and 6–12 mm (Fig. 1);
- reclaimed asphalt (RA) sourced by milling a highway porous wearing course, sieved in the range of 0–8 mm (Fig. 1) with an aged polymer modified binder content of 4.2 % by the weight of the RA (EN 12697-1);
- stabilized bottom ashes (SBA) deriving from the incinerator products stabilization, with particle size up to 10 mm (Fig. 1);
- Natural calcareous filler, obtained by local calcareous rocks quarry operations, in the range of 0–0.063 mm;
- 50/70 neat bitumen (EN 1426) obtained from a refinery located in Busalla (Genoa);
- commercial polymers additive (a mix of elastomer and plastomer polymers), in grains and grey coloured, bulk density equal to 350 kg/m^3, softening point at 145 °C and melting point at 170 °C.

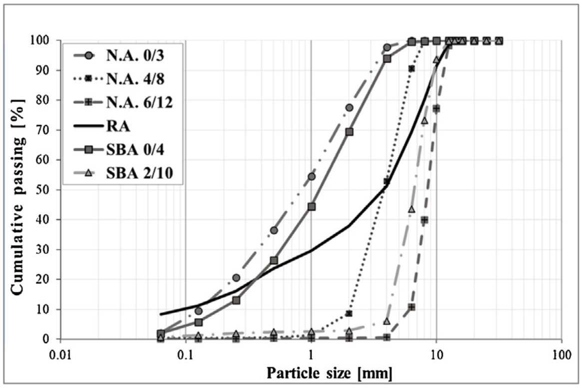

Fig. 1. Particle size distribution of natural aggregates (N.A.), reclaimed asphalt (RA) and stabilized bottom ashes (SBA).

- amine-base rejuvenator available on the Italian market (commercial rejuvenator), amber coloured and liquid at ambient temperature, bulk density equal to 920 kg/m^3, Brookfield viscosity at 25 °C less than 0.05 N/m^{2*}s and Flash Point higher than 170 °C;
- waste engine oil (WEO), black coloured and liquid at ambient temperature, with Brookfield viscosity at 25 °C equal to 0.65 N/ m^{2*}s.

The main characteristics of the key materials are reported in Table 1.

The key materials above described were used to prepare in the laboratory a total of twelve mixtures divided in two main sets containing 20 % and 60 % of RA by weight. In both cases, three reference mixtures were prepared to evaluate the effect of polymer (%RA_P), commercial rejuvenator (%RA_R), and both of them (%RA_P_R).

Then, for each RA percentage, natural aggregates were partially replaced by SBA and the commercial rejuvenator was substituted with WEO (%RA_P_R_SBA, %RA_P_WEO, %RA_P_WEO_SBA).

The key materials were proportioned according to the Italian Specifications [23] (Table 2 and Fig. 2). Thus, no tests for optimum bitumen dosage were performed; the total binder content was selected in the recommended range (between 4.7 and 6.3 %) of the target layer [23]. Because of the high surface porosity of SBAs, as a consequence of the low bulk density (Table 1), the content of bitumen was adjusted according to the following Eq. (1):

$$\%bit_{mix\ WITH\ SBA} = \%bit_{mix\ NO\ SBA} \cdot \frac{Bulk\ Density_{Aggregates\ NO\ SBA}}{Bulk\ Density_{Aggregates\ WITH\ SBA}} \tag{1}$$

Where: $\%bit_{mix\ NO\ SBA}$ is the total bitumen content of mixtures not including SBA (5.3 % by the total mass of mixture); $Bulk\ Density_{Aggregates\ NO\ SBA}$ is the bulk density of the aggregates gradation in mixtures not including SBA, calculated as $\sum_{i=1}(\%aggregate_i \cdot Bulk\ Density_i)$; $Bulk\ Density_{Aggregates\ WITH\ SBA}$ is the bulk density of the aggregates gradation in mixtures including SBA.

4.2. Experimental plan and method

The experimental program is summarized in the flow diagram of Fig. 3. The first step was the key materials characterisation, as described in Section 4.1. Then, the investigated mixtures were prepared using a laboratory mixer at 180 °C ± 5 °C, according to the following phases [24]: (i) heating of the key materials; (ii) first mixing of RA and rejuvenator (or WEO); (iii) addition of natural aggregates and SBA; (iv) addition of polymer and, then, the bitumen; (v) mixing of all materials until the aggregates are homogeneously covers by the bitumen; (vi) addition of filler; (vii) final mixing.

Both sieve size distribution and bitumen content tests were performed at the end of the mixing phase, in order to verify the mixtures' homogeneity. The compaction was performed by using the Gyratory Shear Compactor GSC (EN 12697-31), 210 GSC revolutions (or 230 if polymer is used) were imposed. The compaction temperature was 170 °C ± 5 °C. Four or eight nominally alike cylindrical specimens (100 mm in diameter) were prepared for the mixtures containing 20 % and 60 % of RA, respectively. In addition, two slabs (0.3 × 0.4 × 0.04 m) obtained by rolling compactor (EN 12697-33) were prepared for the mixtures containing 60 % RA, according to the Italian UNI TS 11688.

GSC data were used for a preliminary investigation on the mixtures' compaction behaviour; thus, self-compaction c_1 and workability k were calculated: the average results, with maximum and minimum values as error bars, are shown in Fig. 4a. Results in the figure reveal that the self-compaction parameter appears not to be influenced by the key materials into the mixtures, ranging between 78.45 % and 82.18 %. The workability ranges between 6.67 and 7.75 with a no clear effect of the mixtures' components. This is the reason why the compaction characteristics were studied in a different way, using the

Table 1
Key materials main characteristics.

		Bulk density [kg/m^3] EN 1097-6	LA [%]* EN 1097-2	FI [%]* EN 933-3	SI [%]* EN 933-4	SE [-]* EN 933-8
Natural lithic aggregates	0/3	2734	–	–	–	90.3
	4/8	2747	19.3	7.3	9.9	–
	6/12	2766	16.1	8.8	12.9	–
SBA	0/4	2394	–	–	–	76.7
	2/10	2414	36.2	22.3	–	–
RA aggregates		2682	18.8	8.3	–	–
		Penetration [0.1 mm] EN 1426			Softening Point [°C] EN 1427	
Neat bitumen		51			48.4	
RA bitumen		14			78.5	

* LA: Los Angeles Index; FI: Flakiness Index; SI: Shape Index; SE: Sand Equivalent.

Table 2
Mixtures composition.

	Natural aggregates [%]	RA [%]	SBA [%]	Total bitumen content [%]	Neat bitumen content [%]	Polymer content [% total bitumen content]	Rejuvenator [% RA bitumen]	WEO [% RA bitumen]
20_P	74.70	20	–	5.30	4.50	4	–	–
20_R	74.70	20	–	5.30	4.46	–	2	–
20_P_R	74.70	20	–	5.30	4.46	4	2	–
20_P_WEO	74.70	20	–	5.30	4.46	4	–	5
20_P_R_SBA	54.55	20	20	5.45	4.61	4	2	–
20_P_WEO_SBA	54.55	20	20	5.45	4.61	4	–	5
60_P	34.70	60	–	5.30	2.78	4	–	–
60_R	34.70	60	–	5.30	2.85	–	2	–
60_P_R	34.70	60	–	5.30	2.85	4	2	–
60_P_WEO	34.70	60	–	5.30	2.78	4	–	5
60_P_R_SBA	14.55	60	20	5.45	2.93	4	2	–
60_P_WEO_SBA	14.55	60	20	5.45	2.93	4	–	5

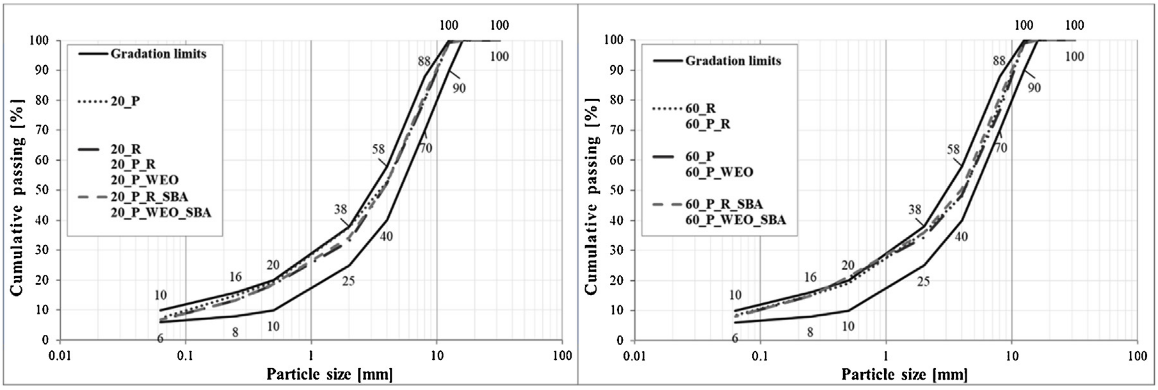

Fig. 2. Aggregates sieve size distribution of the investigated mixtures.

Compaction Energy Index (*CEI*) in comparison with the final volumetric characteristics of the specimens, as proposed by Mahmoud and Bahia [25]. This index represents the energy applied by the roller to achieve a pre-fixed density during construction. It is calculated as the area under the GSC compaction curve, from the 8th gyration (to simulate paver action) to the gyration corresponding to the 92 % of the maximum density. The results of this second stage of investigation are discussed in the following section.

Air voids content (*%v*), voids in the mineral aggregates (*VMA*), and voids filled with bitumen (*VFB*) are the three volumetric parameters considered in the study for evaluating the final structure of the specimens in the compacted state [EN 12697-8]. Average results, with maximum and minimum values as error bars, are shown in Fig. 4b. Volumetric results give a first suggestion about the effects of the key materials on the final volumetric configuration of the mixtures in the compacted state. As a first observation, it is possible to note that the volumetric characteristics are improved because of the RA increase. It might be unexpected, unless one considers that could be due to the friction reduction during compaction because of the film of binder covering the RA aggregates. This sort of pre-coating film plays a positive role during compaction, as also confirmed by the available literature [26].

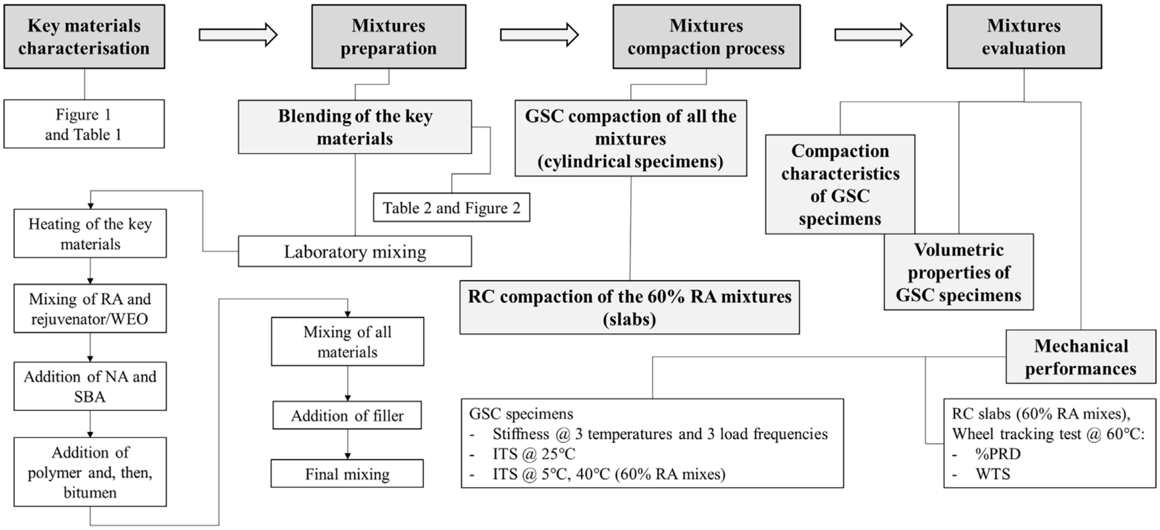

Fig. 3. Flow diagram of the experimental plan.

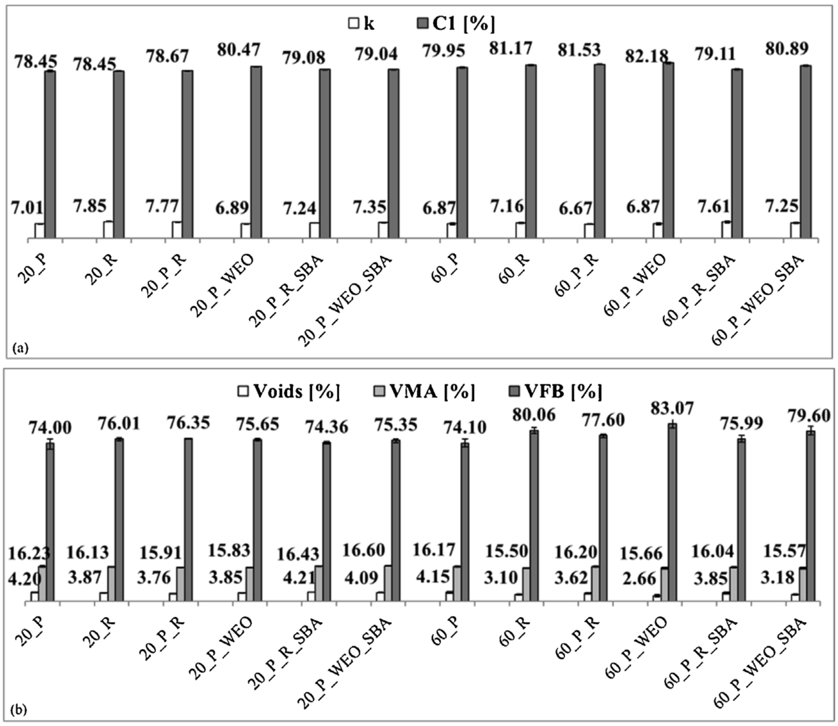

Fig. 4. average GSC (a) compaction results and (b) volumetric characteristics.

Regarding the other key materials, both SBAs and polymer additives appear not to be able to significantly affect final results. Even if two observations must be done regarding the final air voids contents: polymer additives reveal a slight negative effect because it has a hardening role during compaction; the same is for SBAs and it is due to their specific shape that is detrimental for the mixtures' compaction; this is also confirmed by both the high flakiness index results reported in Table 1 and previous studies [17].

Finally, comparing the effects of the commercial rejuvenator and WEO, it is possible to observe that the recycled oil promotes in a better way the mixtures' compaction proneness. It is probably because WEO reduces the bitumen viscosity and softening the binder [27,28], acting as a fluxing agent. Obviously, these preliminary volumetric results must be seen in correlation with the compaction ones (*CEI*), as discussed in the next session.

Table 3
Experimental program and number of replicants of each mixture.

Number of samples tested				
Mixtures	Stiffness at 5-20-40 °C and 1.25 - 2,0 - 4,0Hz EN 12697-26, Annex C	*ITS* at 25 °C EN 12697-23	*ITS* at 5 °C and 40 °C	Rutting at 40 °C EN 12697-22, B-in air
20_P	4	3	–	–
20_R	4	3	–	–
20_P_R	4	3	–	–
20_P_WEO	4	3	–	–
20_P_R_SBA	4	3	–	–
20_P_WEO_SBA	4	3	–	–
60_P	4	3	2	2 slabs
60_R	4	3	2	2 slabs
60_P_R	4	3	2	2 slabs
60_P_WEO	4	3	2	2 slabs
60_P_R_SBA	4	3	2	2 slabs
60_P_WEO_SBA	4	3	2	2 slabs

The mixtures' mechanical performances were assessed by a selected number of tests. As reported in Table 3, stiffness modulus (three test temperatures and three load frequencies), Indirect Tensile Strength (*ITS*, at three test temperatures) and rutting resistance were used as mechanical test parameters to highlight the key materials effects.

Stiffness results were then used to calculate the so-called Master Curves at 20 °C, applying the time-temperature superposition principle to move the test results along the frequency domain. For this reason, three sigmoidal models were compared: Pellinen-Witzack – Eq. (2) -, AASHTO TP-62 and Medani-Huurman. The best data fitting was obtained from the first model.

$$log(S_{mix}) = log\left[a_o \cdot \left(1 - exp^{-((f_r/a_1)^{a_2})}\right)\right] \tag{2}$$

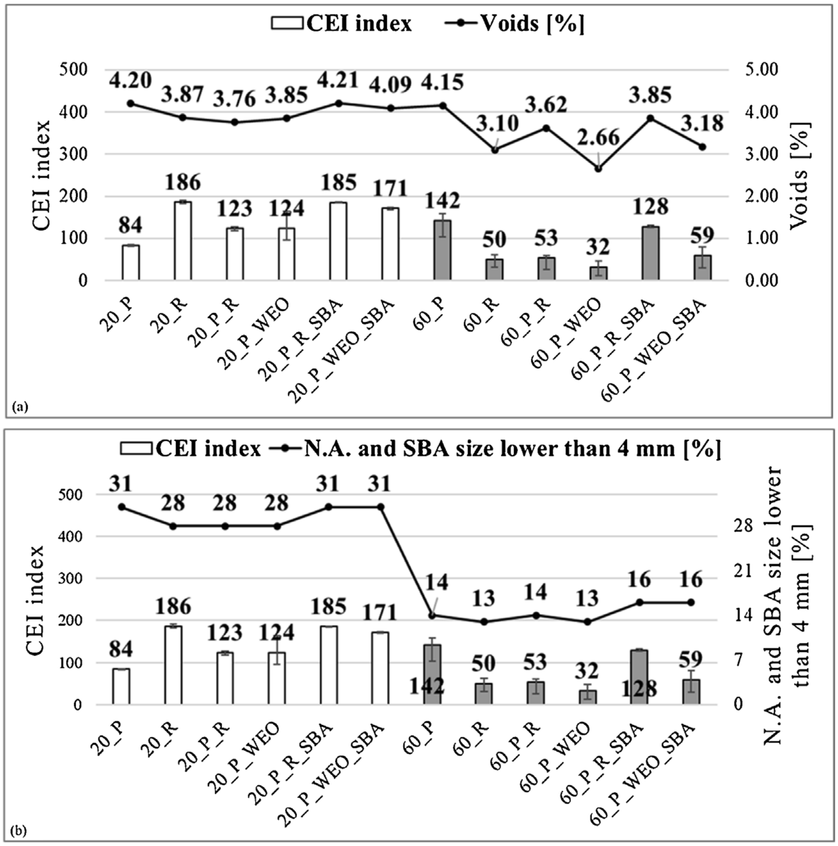

Fig. 5. CEI results versus (a) air voids content, (b) aggregate fine particle content (lower than 4 mm).

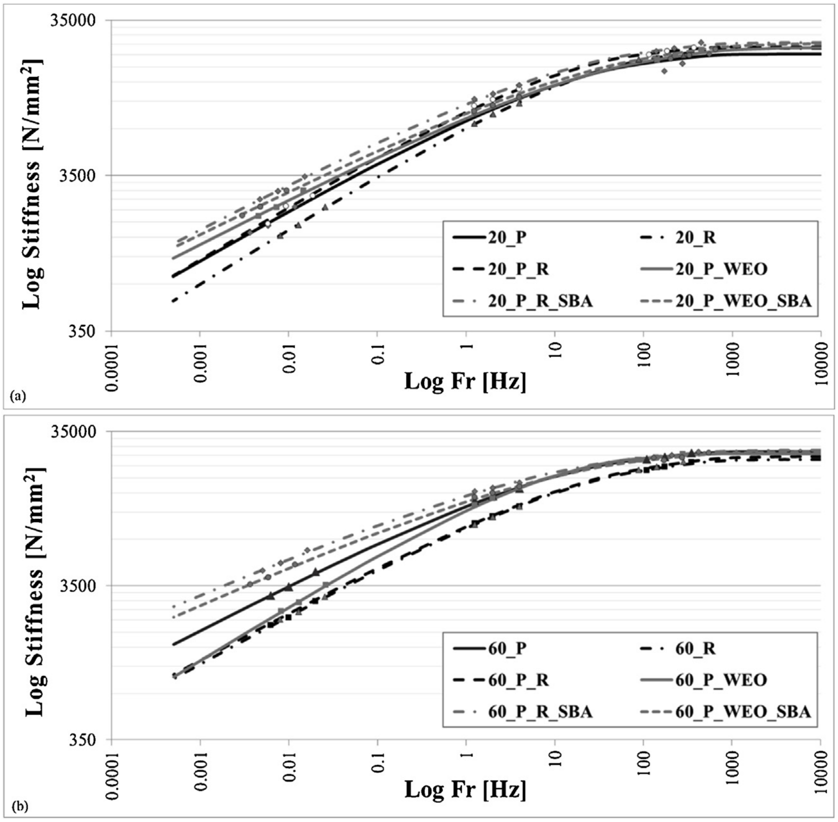

Fig. 6. Master Curves for mixtures containing (a) 20 % and (b) 60 % of RA.

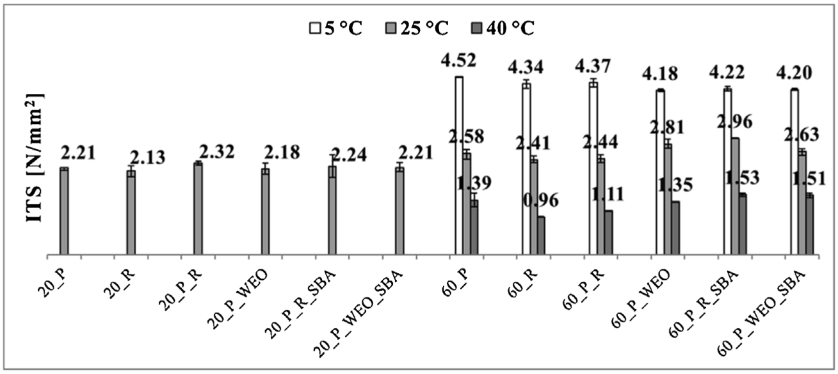

Fig. 7. Indirect Tensile Strength results.

Where: S_{mix} is the stiffness modulus obtained by the model [N/mm^2]; a_o, a_1, a_2 are the shape parameters; f_r is the reduced frequency [Hz].

5. Results and discussion

5.1. Compaction and volumetric results

Fig. 5a reports both the *CEI* compaction parameter and the air voids content in order to assess the way in which the mixtures achieve their final volumetric configuration during the GSC compaction process [26]. As shown in the figure, *CEI* trend is consistent with voids results, except for 20_P; in short, the higher *CEI* values, the higher the voids. In other words, if high compaction energy is required, low volumetric characteristics can be expected. Going in detail to the effects of the key materials and focusing on RA, as previously mentioned, the mixtures in which the higher amount of RA (60 %) is included exhibit the lower air voids content and *CEI*. It because of two main reasons. The first, as previously mentioned, is related to a sort of friction reduction due to pre-coated RA aggregates resulting in lower compaction energy required. The second is due to the mixtures composition, particularly to the mixtures' different aggregate gradation, as stated in [29]: mixtures incorporating small aggregate size require more compaction efforts, due to the higher surface area and therefore higher interaction between particles. The consistency of the obtained results with the literature findings is demonstrated in Fig. 5b, in which is shown that a reduction of natural aggregate and SBA fine aggregate contents implies a decrease of *CEI* (and, thus, of air voids).

Regarding the other key materials, the effects of polymer additives, SBAs, rejuvenators and WEO on the *CEI* parameter are consistent with the results obtained on the volumetric results (Fig. 4b) and previously discussed.

5.2. Mechanical performances

5.2.1. Stiffness master curves

Master Curves obtained by Pellinen-Witzack model at reference temperature of 20 °C (including the shifted experimental values) are shown in Fig. 6a and b, referred to 20 %RA and 60 %RA mixtures, respectively.

Considering mixtures with 20 %RA (Fig. 6a), the Master Curves are quite similar all throughout the frequency domain. Just the Master Curve of the mixture in which the sole polymer is included (20_P) slightly deviates at the frequencies higher than 100 Hz. It is probably due to a bias of the mathematical model, rather than a real different behaviour of the mixture.

Analysing the Master Curves of mixtures with the higher RA content (60 %) reported in Fig. 6b, it can be noted that the curves are split in two different pencil, diverging passing from low to high frequencies. The lower pencil is related to the mixtures containing the sole rejuvenator (60_R) and rejuvenator plus polymer additive (60_P_R). The upper includes the curves of the other mixtures. It appears a very interesting result to highlight the effectiveness of the rejuvenator. In fact, as reported by the available literature [30,31], it is necessary to divide the effects of the rejuvenators, the role of which is the balancing the ratio between asphaltenes and maltenes in the aged bitumen, and the effects of fluxing agents, that mainly acts as a viscosity reducer during the working phases. In this view, just the commercial rejuvenator behaves in the proper way and, thus, reduces the Master Curve level at high frequencies (or at low temperatures, according to the temperature superposition principle), playing a positive role in preventing low temperature cracking phenomena. Of course, when also SBAs are included this behaviour is less evident, because of their shape, as previously discussed. In addition, the positive effect of the commercial rejuvenator is evident comparing mixtures 60_P_R and 20_P, in which similar values of stiffness can be reached.

5.2.2. Indirect tensile strength

Indirect Tensile Strength (*ITS* – EN 12697-23) average results with maximum and minimum values as error bars are reported in Fig. 7.

The first general observation that can be done is the increase of *ITS* at 25 °C according to the RA content, obviously deriving by the high content of aged bitumen, as also confirmed by the available literature [5,32]. Moreover, the positive role played by the rejuvenator is appreciable in comparison to the WEO at high RA contents. In fact, comparing the mixtures named 60_R and 60_P_R to the one named 60_P_WEO, at both intermediate (25 °C) and high (40 °C) temperatures, a *ITS* reduction is appreciable: it is due to the effects that the commercial rejuvenator has on the aged bitumen, as afore discussed.

On the contrary, *ITS* results at the lower test temperature (5 °C) are not able to highlight the contribution of the investigated key components, including rejuvenator and WEO, in fact the results are the same level, ranging between 4.20 and 4.50 N/mm^2.

5.2.3. Rutting resistance

The average results, including maximum and minimum values as error bars, of rutting tests performed according to the EN specification (EN 12697-22, B-in-air) are given in Fig. 8.

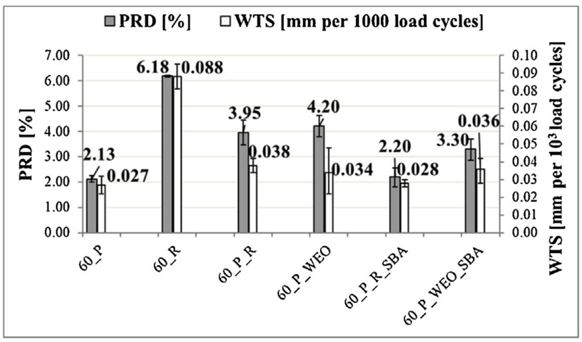

Fig. 8. Rutting Resistance results.

The results in the figure shows that both the commercial rejuvenator and WEO negatively affect the mixtures' rutting proneness. In fact, both the *PRD* (Proportional Rut Depth) and the Wheel Tracking Slope (*WTS*) increase if such additives are used (60_P versus 60_R, 60_P_R, 60_P_WEO). Moreover, it is interesting to note that mixtures 60_P_R and 60_P_WEO show similar *PRD* and *WTS* results, confirming the available literature [1]. However, examining the error bar widths, further studies are worth to investigate the effect of real rejuvenator/fluxing agent on rutting behavior.

Another interesting result is related to the effects of SBAs. In fact, a hardening effect is appreciable, resulting in a decrease of both the rutting parameters (*PRD* and *WTS*). It is in in line with the stiffness results previously discussed.

6. Conclusions

The main scope of this research study was to investigate the effects of the concurrent use of reclaimed asphalt (RA), stabilized bottom ashes (SBA) from urban waste incinerators in replace of the natural aggregates, and waste engine oil (WEO), in substitution of commercial rejuvenators in bituminous mixtures to be used in road wearing courses. Moreover, the benefits deriving from the use of polymer additives in the aforementioned mixtures were also assessed.

Compaction, volumetric and mechanical performances were investigated at laboratory scale, and the obtained results revealed that:

- it is possible to use high contents of RA in bituminous mixtures for wearing courses, even if the stiffening of the mixtures can be expected;
- the RA stiffening effect can be reduced by using rejuvenator additives carefully selected in order to change not only the binder viscosity, but also, the chemical composition and, thus, the rheological characteristics of the aged bitumen;
- WEO is able to act as a fluxing agent, reducing the aged RA bitumen viscosity during compaction, but it is not able to guarantee long-term rheological improvements;
- SBAs can be successfully used as artificial aggregates, even if their shape must be carefully verified case by case since it depends on the incinerator process;
- polymer additives certainly improve the mixtures' performances.

Overall, the obtained results demonstrated that it is possible to use recycled bituminous mixtures for wearing course, ensuring both sustainability and mechanical performances; even if other research activities are needed, especially with regards to cracking susceptibility and durability, through long term aging.

Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Declaration of Competing Interest

The authors report no declarations of interest.

Acknowledgements

The Authors acknowledge Impresa Bacchi s.r.l. for the technical support, especially Mr Mauro Bacchi, Eng. Chiara Panceri and Eng. Livio Trussardi. A great thanks to Eng. A. Anelli, Eng. D. Costa and Eng. G. Volpi for their contribution during the laboratory activities. The Experimental Laboratory of Transportation Infrastructures of Politecnico di Milano (InfraLab@PoliMi) is also gratefully acknowledge.

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