



Sustainable low-temperature asphalt mixtures with marginal porous volcanic aggregates and crumb rubber modified bitumen



Miguel A. Franesqui ^{a,*}, Jorge Yepes ^b, Cándida García-González ^a, Juan Gallego ^c

^a Grupo de Fabricación Integral y Avanzada, Departamento de Ingeniería Civil, Universidad de Las Palmas de Gran Canaria (ULPGC), Campus de Tafira, 35017 Las Palmas de Gran Canaria, Spain

^b Departamento de Ingeniería Civil, IOCAG, Universidad de Las Palmas de Gran Canaria (ULPGC), Campus de Tafira, 35017 Las Palmas de Gran Canaria, Spain

^c Grupo de Investigación en Ingeniería de Carreteras, Departamento de Ingeniería Civil, Transporte y Territorio, Universidad Politécnica de Madrid (UPM), c/ Profesor Aranguren s/n, 28040 Madrid, Spain

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ABSTRACT

This study presents the results of the principal engineering properties of asphalt-rubber warm mixtures (AR-WMA) with waste crumb rubber from used tyres and highly-vesiculated basalt of scoriaceous nature, also considered a residual or marginal aggregate according to standard specifications. The temperature reduction was carried out using a liquid surfactant chemical additive, of easier dosage than granular solid products and in a reduced proportion (0.5% by weight of bitumen). The results were compared both to asphalt-rubber hot mixtures and to hot mixtures with conventional bitumen, all of them with the same aggregates. With the surfactant additive it is possible to lower the production temperatures by up to a maximum of 5–10 °C complying with all the technical specifications for surface courses of pavements, and by up to 25–30 °C for inferior layers or in case of more lenient requirements. Even in the first case, it may compensate for the increase of energy and emissions due to the higher viscosity of the asphalt-rubber binder. With a temperature reduction of 40 °C, certain properties such as the moisture damage strength ratio, rutting resistance and stability were even superior compared to conventional mixtures without rubber (produced at 170 °C). The results obtained may be extrapolated to other volcanic regions both insular and continental areas where this type of aggregates are commonly found and with rigorous environmental requirements.

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1. Introduction

Long-term sustainability of asphalt pavements requires techniques that allow longer on-service life of the materials and so reduce the total costs during their life cycle. It is also fundamental to use local aggregates and materials, reuse and recycle residual products as well as employing low-consumption technologies of raw materials and energy that reduce emissions and waste.

In volcanic territories the most common and abundant aggregates are obtained from eruptive rocks and lava flows with a vesicular and scoriaceous structure of high porosity. However, the higher absorption due to the alveolar structure, particle shape and heterogeneous properties make these aggregates marginal and

inadequate for structural materials according to standard technical specifications (Fransesqui et al., 2010). Hence, this type of aggregates offer inferior performance and limits the use in structural concretes and bituminous mixtures.

In a similar way, the waste from used tyres has also become an important environmental issue in volcanic island territories with natural areas of great value and therefore reuse in situ becomes vital. The recycling of crumb rubber from reclaimed end-of-life tyres (ELT) used as a modifier of the asphalt binder (crumb rubber modifier or CRM) for bituminous mixtures is an interesting alternative. Asphalt-rubber (AR), also called “wet process”, is the blend of an asphalt binder and ground recycled tyre rubber from scrap ELT in which the rubber powder reacts in the hot asphalt bitumen sufficiently to cause swelling of the rubber particles. AR mixtures offer certain environmental advantages regarding natural resources as well as traffic noise reduction. They also provide improved pavement performance properties: moisture sensitivity,

* Corresponding author.

E-mail address: miguel.franesqui@ulpgc.es (M.A. Fransesqui).

rutting resistance and Marshall stability (Liu et al., 2009; Moreno et al., 2013; Shu and Huang, 2014; Kakar et al., 2015; Gibreil and Feng, 2017). This reduces maintenance costs and increases durability. However, the production and compaction temperatures of asphalt-rubber hot mixtures (AR-HMA) are usually at least 10–15 °C higher than with conventional binders as the rubber lends to a greater viscosity. This is due to the digestion process of the rubber in the bitumen (Wang et al., 2017), and thus it reduces workability, decreases the laying and compaction time and increases costs (Lo Presti, 2013). This is an important drawback of this technology as it produces higher energy consumptions (0.5–1.0 L/tonne more) and emissions (1.3–2.5 kg-CO₂/tonne more), which compromises its environmental sustainability. Therefore, in order to guarantee eco-efficiency, AR mixtures should inevitably be associated with warm mix asphalt technologies (AR-WMA) without significantly affecting the mechanical performance.

Of all the methodologies utilized to reduce the manufacturing temperatures of bituminous mixtures that do not use water during the process, the techniques that use microcrystalline waxes to decrease the bitumen viscosity have been thoroughly tried and tested. Most of them are patented products that can improve the binder fluency and reduce the production temperatures by 20–30 °C (Kakar et al., 2015; Rondón-Quintana et al., 2015). Wax additives have also been used in order to reduce the production temperatures of AR mixtures (Rodríguez-Alloza and Gallego, 2017; Saberi et al., 2017) even with reclaimed asphalt pavement (RAP) (Saberi et al., 2017), providing improved performance against moisture susceptibility, fatigue cracking and permanent deformation. In addition, asphalt-wax mixes enable energy saving and reduce emissions (Rodríguez-Alloza et al., 2015) and pollutant fumes (Autelitano et al., 2017).

On the other hand, the tensioactive chemical additives raise the surfactant capacity of the binder with the aggregates. There are also numerous adhesion promoters (anti-stripping surfactant agents or cationic emulsification agents) that allow lower manufacturing temperatures; between 15 and 30 °C (Xiao et al., 2012; Rondón-Quintana et al., 2015). These products contribute to an efficient coating of the asphalt binder on the aggregate surface by reducing the surface tension of the bitumen, increasing the mixture workability and compactability at lower temperatures (Jamshidi et al., 2015; Li et al., 2016; Yang et al., 2017). Consequently, contaminant emissions are reduced and, in certain cases, the bitumen consumption. Although the manufacturers, due to patent policies, do not always offer sufficient information regarding composition or characteristics (Bonaquist, 2011; Rondón-Quintana et al., 2015), in some previous studies with certain additives, it has been verified that resistance to permanent deformations and dynamic resistance of mixtures are maintained or improved (Ouni et al., 2014; Yang et al., 2017). They can also increase resistance to moisture action owing to the presence of polymers within the composition (Rondón-Quintana et al., 2015).

However, the main limitations of the previous studies have been:

- a) Despite the quantitative importance of the marginal volcanic aggregates in many regions, these are usually discarded at the quarries as there are very few studies regarding their applications in bituminous mixtures and these only refer to certain lithotypes such as trachybasalts and volcanic ashes (Faustino et al., 2005; Naji and Asi, 2008; Akbulut et al., 2011). Furthermore, most technical regulations do not usually include specifications adapted to the peculiarities of these mixtures but in volcanic areas the use of these local aggregates is a necessity.
- b) There are no studies regarding the performance of asphalt mixtures with high-porosity vesicular and scoriaceous basaltic

rock as the main mineral aggregate even though these are readily available. In the same way, there are no studies regarding the effect of the AR binders on these porous aggregates or about the possibility of manufacturing these mixtures at low temperatures. Moreover, the use of marginal aggregates does not usually meet the standard specifications for road pavements due to certain performance properties of these mixtures.

- c) Organic additives for WMA of the type microcrystalline waxes are expensive as they are required in high quantities (up to 2–4% by bitumen weight) and some crystallize below certain temperatures (Sánchez et al., 2011; Rodríguez-Alloza et al., 2014). This makes the asphalt mixture stiffer and more difficult to compact and consequently more susceptible to low-temperature cracking (Edwards and Redelius, 2003). They may even worsen fatigue resistance at medium or low temperatures (Rondón-Quintana et al., 2015).

The previous state of the art determined that our study focuses on the use of elastomeric residuals from ELT in order to modify the asphalt bitumen and improve the performance of bituminous mixtures with marginal volcanic aggregates with high degree of vesiculation; the primary aim being to remediate both residuals, reduce the environmental impact and improve pavement durability. Likewise, the use of CRM in the binder composition could compensate for the higher amount of asphalt bitumen required when using high porosity aggregates. In order to meet the environmental challenge implied in the use of AR binders, the temperature reduction is studied by using a tensioactive chemical additive with surfactant properties and with the following characteristics: a) useable in reduced proportions (no higher than 0.5% by weight of the binder, in order to be economically feasible in large pavement production); b) allowing an easier dosage added in a liquid state; c) does not cause crystallization below certain temperatures, as occurs with waxes, achieving higher densities and improved properties for the mixtures.

In order to confirm the practical effectiveness of this solution the main engineering properties of AR mixes with volcanic aggregates from high-porosity vesicular and scoriaceous basalt (6–16% of water absorption) and produced at different low temperatures by using a surfactant liquid additive, were characterized in the laboratory. These are compared both to the AR mixtures manufactured at high temperature and to the hot mixes produced with conventional bitumen and the same volcanic aggregates.

2. Experimental method

2.1. Materials

228 cylindrical specimens, 32 slab specimens and 30 non-compacted samples (for theoretical maximum density tests) were produced in the laboratory, including the reference hot mix asphalt without rubber (Ref. HMA), the reference hot mix asphalt with asphalt-rubber binder (Ref. AR-HMA) and the asphalt-rubber mixtures with warm mix asphalt additive (AR-WMA); these all consisted of semi-dense asphalt concrete (AC16 surf S). This type of paving asphalt concrete was produced following the Spanish technical specifications for roads (PG-3) (Spanish Ministry of Infrastructures, 2014) and is in accordance with European Standard EN 13108-1. This bituminous mixture is widely used for surface courses of different roads, traffic types and climates. It provides better surface macrotexture, lower susceptibility to permanent deformations and is more economical than dense asphalt concrete.

All aggregate fractions used in the mixtures (10–20, 4–10 and 0–4 mm) come from the same type of volcanic rock obtained in the same quarry in Gran Canaria (Canary Islands, Spain). This quarry is

exploited by the Port of Las Palmas to obtain filling material for harbour works. The type of all-in aggregate is a mechanically-crushed vesicular olivinic-piroxenic grey basalt (B-V) of high porosity, one of the most common and abundant lithotypes of rocks in volcanic territories (Fransesqui et al., 2010). The main properties are summarized in Table 1 and a detail may be observed in Fig. 1. This vesiculated aggregate with scoriaceous structure presents a high water absorption (WA_{24}), particularly in the finest fraction (15.5%). The flakiness index (FI), the sand equivalent of fraction 0–4 mm (SE_4) and the polished stone value (PSV) complied with the Spanish specifications for road pavements ($FI \leq 20-30$; $SE_4 > 55$; $LA \leq 20-25$; $PSV \geq 50-56$). These technical specifications are very similar to other international regulations. However, this volcanic aggregate is considered marginal owing to its high percentage of non-prismatic particles, the values of resistance to fragmentation (Los-Angeles coefficient, LA) and resistance to wear (Micro-Deval coefficient, M_{DE}).

A Portland cement with pozzolanic addition, type CEM II/B-P 32.5 R (according to EN 197-1) was used as mineral filler ($\# < 0.063$ mm). This cement is commonly produced in volcanic areas as natural pozzolans are easily found.

The reference mixture without rubber used a commercial bitumen 35/50 pen, whilst the binder of the mixtures with rubber was an asphalt-rubber of the same penetration grade (AR35/50), produced in the laboratory by adding the crumb rubber modifier (CRM). The CRM was manufactured by mechanical grinding at ambient temperature (50% from used truck tyres; 50% used car tyres). To ensure consistency, only one batch of CRM was used. The thermogravimetric analysis revealed the following composition: 57.41% polymer (rubber), 32.22% carbon black, 6.02% ash and 4.67% plasticizer and additives. 100% by weight of CRM passed through sieve 1.0 mm (EN 933-2), 94.1% accumulated of a size smaller than 0.5 mm, 23.7% smaller than 0.25 mm, 3.7% smaller than 0.125 mm and 0.4% smaller than 0.063 mm. The main properties of the binders used for the different types of mixtures are included in Table 2.

The surfactant chemical additive employed to reduce the mixing and compaction temperatures was Cecabase RT[®] (Arkema Innovative Chemistry, France), a commercial liquid water-free tensioactive product (see Fig. 2) composed of at least 50% renewable components. The main physical properties are: viscosity at 25 °C: 600 cP; density: 0.997 Mg/m³; freezing point: -10 °C; flash point: >200 °C; readily soluble in asphalt binder.

2.2. Methodology

In order to compare the effect of the AR binder as well as the temperature reduction additive on the properties of this semi-dense asphalt concrete with highly-vesiculated basalt, three types

of mixtures were formulated in the laboratory:

- 1) In the initial phase, the reference hot mixture (Ref. HMA) specimens of the type AC16 35/50 surf S with 6% (by total weight of mix) of conventional bitumen 35/50 pen were manufactured and tested. Previous Marshall tests, ITSr tests and wheel tracking tests showed this was the optimum binder content according to the resulting properties related to density, air voids, stability, flow value, resistance to water damage and rutting resistance of the mixtures.
- 2) In the next phase, the reference asphalt-rubber hot mixture (Ref. AR-HMA) specimens (AC16 AR35/50 surf S) were produced and tested. These were manufactured with the same type of aggregate, identical particle size distribution (Table 3) and binder content (6%), because laboratory tests shown no significant differences between the optimal binder contents of Ref. HMA and Ref. AR-HMA. However, these last mixtures were made with an AR binder of similar penetration grade (Table 2), previously manufactured in the laboratory by mixing the CRM with a bitumen 50/70 pen. The AR35/50 was obtained with the bitumen 50/70 pen because the elastomer increases the viscosity and consistency of the resulting binder.
- 3) Finally, the asphalt-rubber mixture produced at different lower temperatures with the warm mix asphalt additive (AR-WMA) and identical composition was studied. The mixing temperatures were between 140 and 170 °C, being compacted at 10 °C below (between 130 and 160 °C).

The equipment used to obtain in the laboratory the AR binder, mixing CRM with hot bitumen, and to mix the WMA additive with the AR binder was: a) Mixer unit (IKA Ultra-Turrax T50 digital, with a propeller agitator), maximum velocity 15000 rpm, max. viscosity 5000 mPas (Fig. 2); b) oil bath (max. 225 °C, with temperature probe, stability and accuracy ± 1.0 °C) and 1-L metal container; c) automatically-controlled thermostatic bath (Selecta), range 5–200 °C, stability and accuracy ± 1.0 °C, filled with melted paraffin.

To produce the AR35/50 binder, each 50/70 bitumen sample of 600 g was heated at 180 °C and then 10% (by wt.) of CRM was added in the blending unit with the oil-bath. It was then mixed during 60 min at 4000 rpm at a constant temperature of 180 °C so that the ultraviolet inhibitors, anti-oxidants and other chemicals in the tyre rubber are transferred to the asphalt, including its elastomeric properties. This gives a reacted AR binder of higher consistency. To manufacture the AR-WMA, 0.5% (referred to the bitumen weight) of Cecabase RT[®] liquid additive was carefully added to the bitumen and the blend was subsequently mixed for 10 min at 4000 rpm and 180 °C, ensuring that the additive was properly incorporated into the binder (Fig. 2).

The Ref. HMA was produced in the laboratory by heating the

Table 1
Characterization properties of the aggregate fractions.

Type	# 10–20 mm	# 4–10 mm	# 0–4 mm	Mineral filler ($\# < 0.063$ mm)
	Vesiculated grey basalt (B-V) with scoriaceous nature			100% CEM II/B-P 32.5 R [EN 197-1]
% (by wt. of the total aggregate)	20.93	36.27	38.88	3.92
$\rho_a/\rho_{SSD}/\rho_{rd}$ [EN 1097-6] (Mg/m ³)	2.89/2.63/2.35	2.88/2.56/2.37	2.45/2.36/2.23	–
WA_{24} [EN 1097-6] (%)	5.8	8.3	15.5	–
FI [EN 933-3]	6	6	–	–
SE_4 [EN 933-8]	–	–	73	–
LA [EN 1097-2]	29	28	–	–
M_{DE} [EN 1097-1]	17	23	–	–
PSV [EN 1097-8]	60	60	–	–

(ρ_a) Particle density [apparent]; (ρ_{SSD}) Particle density [saturated surface dry]; (ρ_{rd}) Particle density [dry]; (WA_{24}) Water absorption of particles after 24 h; (FI) Flakiness index; (SE_4) Sand equivalent of fraction 0–4 mm; (LA) Los-Angeles coefficient for resistance to fragmentation; (M_{DE}) Micro-Deval coefficient for resistance to wear; (PSV) Polished stone value for resistance to polishing.

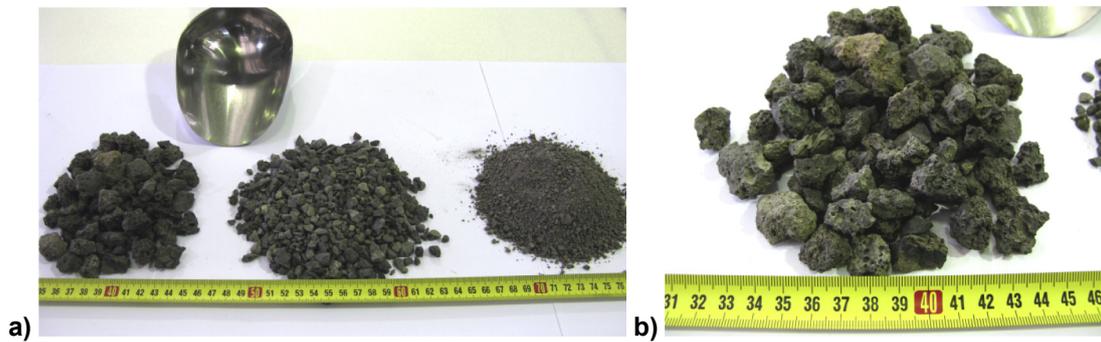


Fig. 1. Aggregate from high-porosity scoriaceous and vesiculated basalt: a) Fractions (10–20, 4–10 and 0–4 mm); b) Detail of the coarsest fraction.

Table 2

Characterization properties of the asphalt binders used for the different types of mixtures.

	Ref. HMA	Ref. AR-HMA	AR-WMA
Binder type	35/50	AR35/50 ^(*)	AR35/50 + 0.5% ^(**) of Cecabase RT [®]
Binder content (% by total weight of mixture)	6.0	6.0	6.0
Density [EN 15326] (Mg/m ³)	1.042	1.028	1.027
Pen [EN 1426] (x10 ⁻¹ mm)	44	38	30
Softening point [EN 1427] (°C)	51.6	64.2	67.4
Viscosity [EN 13302] (cP)	at 60 °C	215,000	211,000
	at 135 °C	600	2000
	at 150 °C	250	850

(Ref. HMA) Reference hot mixture without rubber; (Ref. AR-HMA) Reference asphalt-rubber hot mixture; (AR-WMA) Asphalt-rubber warm mixture; (Pen) Penetration at 25 °C, 100 g, 5s; (Viscosity) Dynamic viscosity by Brookfield rotational viscometer; ^(*) Composition of AR35/50: 10% CRM, 90% bitumen 50/70 pen; (CRM) Crumb rubber modifier; ^(**) % by weight of the bitumen.

Table 3

Aggregate gradation and specified grading envelope for semi-dense asphalt concrete (AC16 S).

Particle size [EN 933-2] (mm)	% passing	Limits of the specified grading envelope (% passing) [PG-3, Spanish Ministry of Infrastructures, 2014]
22	100.00	100–100
16	96.98	90–100
8	68.85	60–75
4	44.71	35–50
2	30.24	24–38
0.5	15.53	11–21
0.25	10.07	7–15
0.063	3.92	3–7

aggregates and the bitumen 35/50 pen to 170 °C (aggregates were heated for 8 h and bitumen 3 h). Subsequently, both were mixed for 1 min coating by hand followed by 2 min in the mixer. The final temperature in the mixer was 170 °C (in no case less than 165 °C) and the compaction temperature 160 °C (no less than 155 °C).

As for the Ref. AR-HMA, due to the higher viscosity of the AR35/50 binder, the heating temperature of aggregates and bitumen was 180 °C and both were in the mixer unit for 3 min. In this case the final temperature in the mixer was 180 °C (no less than 175 °C) and compaction at 170 °C (no less than 165 °C).

Finally, the AR-WMA was mixed between 140 and 170 °C and compacted between 130 and 160 °C on account of the chemical additive. Compaction was always carried out at 10 °C below the corresponding mixture temperature (Fig. 3). Both the aggregates and the AR binder (with the additive formerly incorporated) were previously heated at the same temperature (the required mixing temperature), then coated with the bitumen for 1 min followed by 2 min in the mixer.

The cylindrical specimens (D = 101.6 mm; h = 63.5 mm) were compacted using a Marshall hammer according to EN 12697-30 with 50 or 75 blows per side (depending on the laboratory test). The slab specimens of 300 × 300 × 60 mm were compacted by

rolling according to EN 12697-33 (see Fig. 4).

The compacted specimens and non-compact samples underwent until three series of characterization tests for each type of mixture and production temperature: a) Volumetric properties of bituminous specimens: bulk density (EN 12697-6, Proced. B: saturated surface dry, and Proced. D: geometric), theoretical maximum density (EN 12697-5, Proced. A: volumetric) and void characteristics (EN 12697-8); b) Water sensitivity (EN 12697-12, by indirect tensile test [EN 12697-23]); c) Resistance to permanent deformation: wheel tracking test (EN 12697-22, Proced. B: in air, small device, at 60 °C and 10⁴ cycles) and Marshall test (EN 12697-34). All in all, a total of 290 laboratory specimens and samples were tested, with a total number of 18 tests on the Ref. HMA (without rubber), 18 on the Ref. AR-HMA and 36 on the AR-WMA at different production temperatures. When necessary, the different test samples were previously conditioned in a heater-refrigerator in order to reach the normalized temperature according to standards and maintained during the test if mandatory.

3. Results and discussion

The results of the different characterization properties were



Fig. 2. Mixer unit employed to prepare the AR binder containing WMA additive. In the foreground, liquid WMA additive.

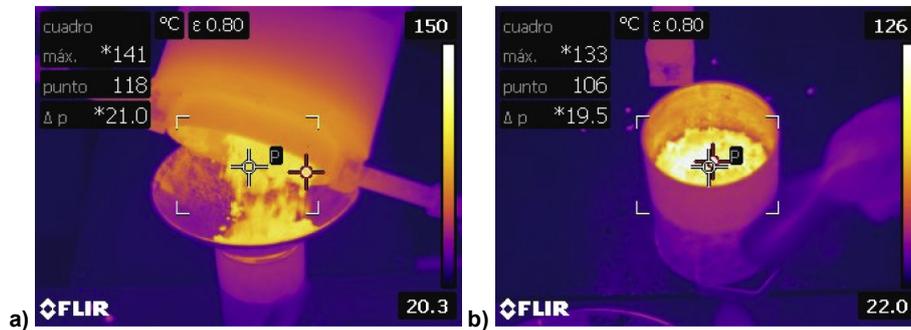


Fig. 3. Infrared thermal images of AR-WMA cylindrical specimen production: a) Mixing temperature: 140 °C; b) Compaction temperature: 130 °C.

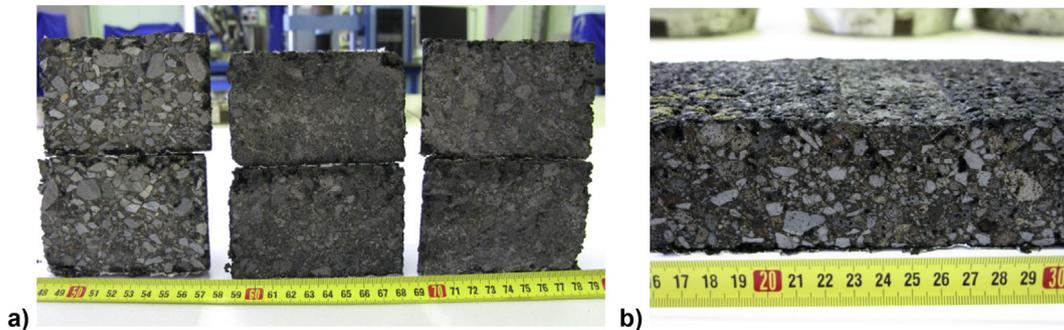


Fig. 4. Cut sections of laboratory samples: a) Cylindrical specimens: left: Ref. HMA without rubber compacted at 160 °C; centre: Ref. AR-HMA compacted at 170 °C; right: AR-WMA compacted at 140 °C; b) Prismatic specimen of AR-WMA compacted at 140 °C.

plotted in relation to the compaction temperature, as these properties not only depend on the mixing temperature (crucial this last in order to achieve an adequate coating of the aggregates with the bitumen) but also on the lowest temperature reached before the end of production. This coincides with the final compaction due to the temperature loss after leaving the mixer and it is critical in order to attain sufficient density. It is necessary to note that the compaction temperature was always 10 °C lower than the corresponding mixing temperature, so results can also be directly read from the graphics as a function of this last temperature if preferred.

3.1. Volumetric properties

The study of volumetric characteristics is normally the first stage in the formulation of a bituminous mixture because these properties are decisive in its design and performance. An excessive air void content may be the main cause of accelerated water damage (Varveri et al., 2014) and lower fatigue life (Ma et al., 2016). In contrast, asphalt mixtures with very low air void content can cause excessive plastic deformations due to traffic loads.

The bulk density of the reference asphalt-rubber hot mixture (Ref. AR-HMA) cylindrical specimens (mixed at 180 °C and compacted at 170 °C by impact with 75 blows/side) is slightly superior to the reference hot mixture with conventional bitumen (Ref. HMA, mixed at 170 °C and compacted at 160 °C). There was a 1.6% difference when the bulk density was measured by saturated surface dry (SSD) procedure and 0.7% if determined by geometric procedure (Fig. 5b and c, respectively). Although all mixtures were made with the same binder content (see Table 2), the theoretical maximum density of the Ref. AR-HMA was superior to the Ref. HMA (2.2% higher) (Fig. 5a).

In Fig. 5b and c it can be observed that the bulk density of the asphalt-rubber warm mixture (AR-WMA) decreases as

manufacturing and compaction temperatures become lower. AR-WMA densities were lower than the Ref. AR-HMA when compacted below 150 °C, whilst they maintained higher densities than conventional Ref. HMA without rubber as long as compaction temperature stayed above 135–140 °C. Densities calculated by SSD procedure were always higher than those obtained by geometric method. The theoretical maximum density of the AR-WMA non-compacted samples did not vary significantly with the different temperatures studied and was higher than both reference mixtures (Fig. 5a).

Table 4 summarizes the averages of the statistical dispersion parameters for the results of the different properties tested. Bulk densities obtained for the three types of mixtures in all tests proved to be homogeneous, especially in the AR mixtures (Coefficient of variation: $C_v \leq 2.1\%$, for AR-WMA; $C_v \leq 1.7\%$, for Ref. AR-HMA; $C_v \leq 3.0$ for Ref. HMA).

Fig. 6 summarizes the void characteristics of the same cylindrical specimens calculated by SSD bulk densities. In Fig. 7 these void characteristic were obtained by geometric bulk densities. The air void content (V_m) and voids in mineral aggregate (VMA) of the Ref. AR-HMA turned out to be slightly superior than the reference hot mixture without rubber (Ref. HMA) for the same compaction energy (by impact with 2×75 blows), although the bulk density of the former also resulted slightly higher. This is due to the superior theoretical maximum density of the AR mixture, as stated before. V_m difference was between 0.6% by SSD procedure (Fig. 6a) and 1.4% by geometric procedure (Fig. 7a). The superior porosity of the AR mixture also suggests a more difficult compaction due to the higher viscosity of the AR binder, even though mixed and compacted at a temperature 10 °C higher. This fact was also observed during laboratory production of AR mixtures. Pérez and Pasandín (2017) obtained similar results in mixtures with the same type of binder but with recycled concrete aggregates. Figs. 6c and 7c show the

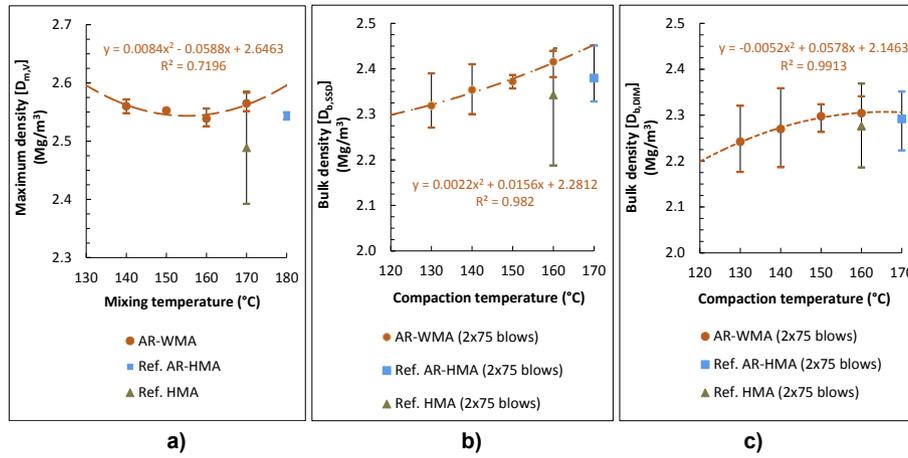


Fig. 5. Densities of the bituminous mixtures: a) Theoretical maximum density (Proced. A: volumetric, EN 12697-5); b) Bulk density (Proced. B: saturated surface dry, SSD, EN 12697-6); c) Bulk density (Proced. D: geometric, EN 12697-6). (Note that 1 Mg/m³ = 1 g/cm³).

Table 4
Statistical dispersion parameters for the different properties tested.

Property of the mixture	Ref. HMA		Ref. AR-HMA		AR-WMA	
	Sd	Cv (%)	Sd	Cv (%)	Sd	Cv (%)
D _{m,v}	0.10 Mg/m ³	3.98	0.03 Mg/m ³	0.99	0.02 Mg/m ³	0.62
D _{b,SSD} (2 × 75 blows)	0.06 Mg/m ³	2.74	0.04 Mg/m ³	1.71	0.05 Mg/m ³	1.94
D _{b,DIM} (2 × 75 blows)	0.07 Mg/m ³	3.03	0.03 Mg/m ³	1.45	0.05 Mg/m ³	2.13
ITSR	6.53%	9.32	11.18%	12.86	5.35%	5.66
ITSw	0.37 MPa	33.19	0.21 MPa	15.78	0.11 MPa	10.31
ITSD	0.44 MPa	27.89	0.11 MPa	7.48	0.24 MPa	19.63
WTS _[air]	0.19 mm/10 ³ cycles	46.47	0.05 mm/10 ³ cycles	58.87	0.08 mm/10 ³ cycles	41.48
RD _[air]	1.66 mm	26.53	0.58 mm	32.08	0.62 mm	19.34
PRD _[air]	1.76%	14.24	1.34%	40.93	1.07%	19.24
Marshall S	0.87 kN	12.75	2.06 kN	12.24	2.05 kN	16.69
Marshall F	0.29 mm	9.99	0.29 mm	10.93	1.11 mm	28.89
Marshall (S/F)	0.31 kN/mm	13.23	1.23 kN/mm	18.36	1.36	38.61

(Ref. HMA) Reference hot mixtures without rubber; (Ref. AR-HMA) Reference asphalt-rubber hot mixtures; (AR-WMA) Asphalt-rubber warm mixtures; (Sd) Standard deviation; (Cv) Coefficient of variation.

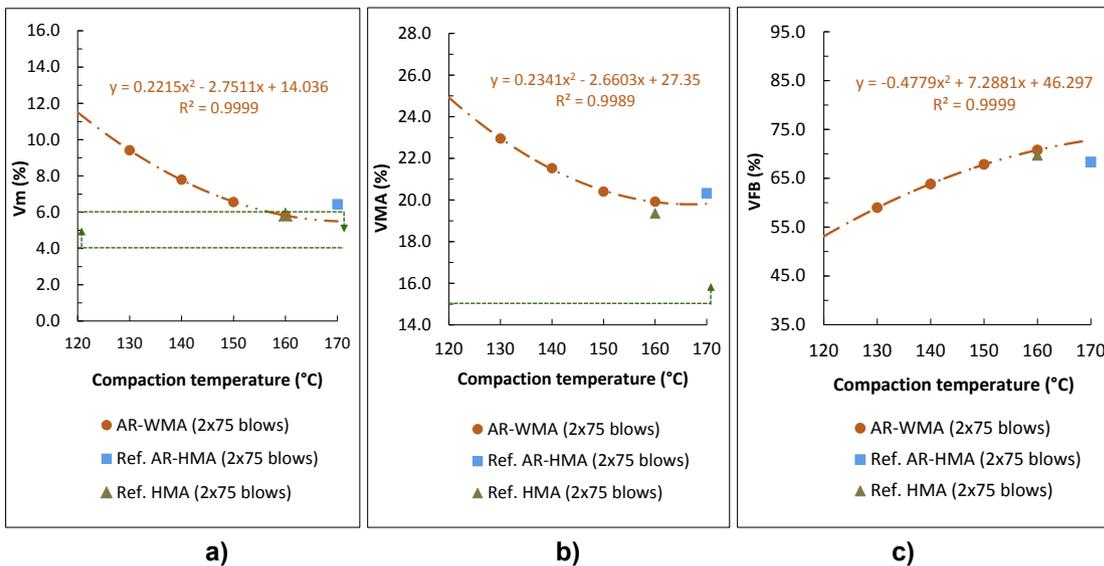


Fig. 6. Void characteristics of the bituminous mixtures by SSD bulk densities according to EN 12697-8 (Limits of specifications are indicated by horizontal dotted lines): a) Air void content in the mixture; b) Voids in mineral aggregate; c) Voids filled with bitumen.

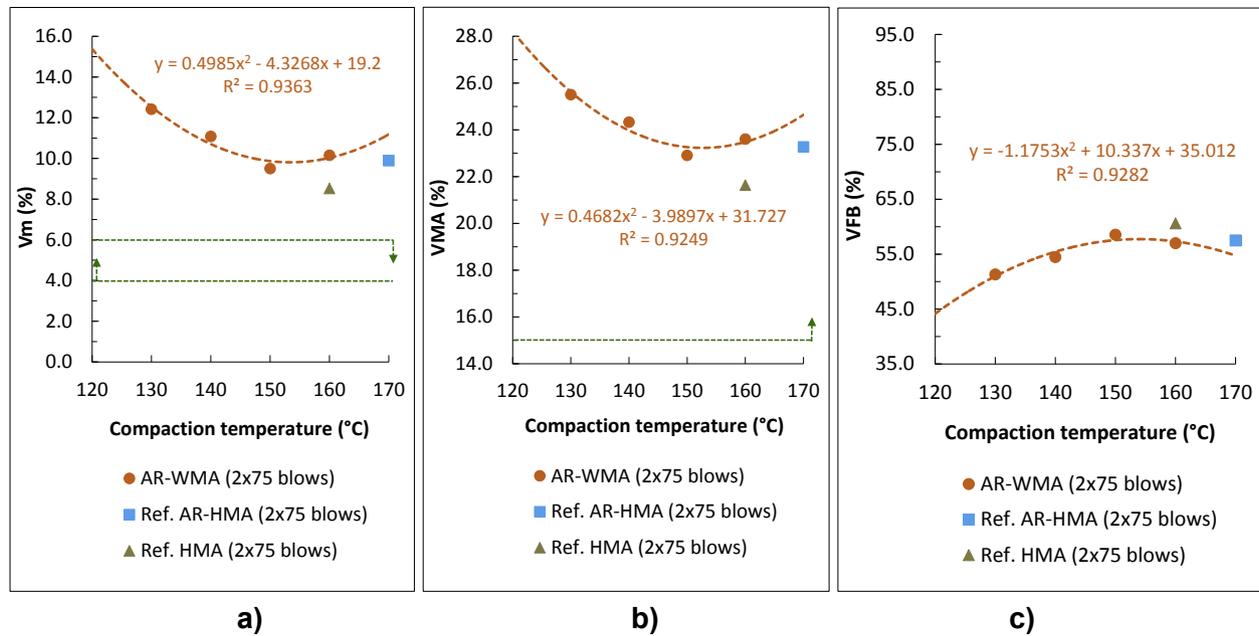


Fig. 7. Void characteristics of the bituminous mixtures by geometric bulk densities according to EN 12697-8 (Limits of specifications are indicated by horizontal dotted lines): a) Air void content in the mixture; b) Voids in mineral aggregate; c) Voids filled with bitumen.

percentage of voids filled with bitumen (VFB), being slightly lower in case of AR mixtures (1.4–3.1%, respectively).

As for the AR-WMA, Fig. 6 also shows that both V_m and VMA resulted higher as the compaction temperature decreases. Similar or inferior air void contents were obtained to both reference mixtures if compaction temperatures are over 150 °C. It is also worth noting that there is an almost perfect experimental value adjustment to the functions obtained by non-linear regression (coefficients of determination R^2 close to 1). However, when these void characteristics were calculated with bulk densities obtained by the geometric procedure, in all cases they resulted superior to those obtained by SSD procedure and with the minimum value for 150 °C (Fig. 7).

Void characteristics of AR-WMA, complied with the specifications of the Spanish standards for roads (PG-3, Spanish Ministry of Infrastructures, 2014) ($4 \leq V_m \leq 6\%$ and $VMA \geq 15\%$, for AC in surface layers, specimens compacted with 2×75 blows and voids determined by SSD procedure) for compaction temperatures equal and superior to 155 °C.

3.2. Water sensitivity

Fig. 8a shows the enhancement of water action resistance on cylindrical specimens compacted by impact with 50 blows/side by means of the indirect tensile strength ratio (ITSR). ITSR represents the quotient between the average indirect tensile resistance at 15 °C of a certain water-saturated (during 72 h at 40 °C) specimen subset (ITS_w) and the corresponding dry specimen subset (ITS_d) at the same temperature, according to European Standard EN 12697-12. The ITSR of the reference asphalt-rubber hot mixture (Ref. AR-HMA, mixed at 180 °C and compacted at 170 °C) resulted 24.1% higher than the reference hot mixture with conventional bitumen (Ref. HMA, mixed at 170 °C and compacted at 160 °C). Indirect tensile strength of water-saturated cylindrical specimens (ITS_w) of the Ref. AR-HMA was (27.0%) superior compared to the Ref. HMA (Fig. 8b). However, this indirect tensile resistance was similar for dry specimens (ITS_d , Fig. 8c). This result proves the higher resistance to moisture damage that the asphalt-rubber (AR) produces in

mixtures with high-porosity marginal volcanic aggregates. This result is noteworthy since previous research with other marginal aggregates (recycled concrete aggregates) reported a lower water damage resistance for AR mixtures (Pérez and Pasandín, 2017).

As for AR-WMA, the ITSR is clearly reduced with the temperature of the mixture although road specifications are met (for AC in surface layers: $ITSR \geq 85\%$) as long as the compaction temperature remains above 140 °C. The improvement of ITSR remains at all temperatures studied compared to the same mixture without rubber thanks to the AR binder. However, this improvement reduces as the temperature drops (Fig. 8a). At 160 °C the ITSR proved to be superior compared to both reference mixtures (30.2% superior to Ref. HMA and even 6.1% superior to Ref. AR-HMA, this last compacted at 170 °C), possibly due to the anti-stripping properties of the surfactant additive.

ITS_w of AR-WMA proved to be similar to the reference mixtures up to temperatures not below 150–160 °C (Fig. 8b). On the contrary, ITS_d of AR-WMA was inferior compared to both reference mixtures at all temperatures (Fig. 8c). Consequently, the chemical additive managed to partially maintain the improvement regarding resistance to water action on these mixtures with high porosity aggregates and manufactured at lower temperatures. This is due to the combined effect of its surfactant properties and the properties of the rubber. This improvement was maintained as long as temperatures do not drop below 150 °C.

According to Table 4, the statistical dispersion of the laboratory test results was lower in the case of the AR mixtures ($Cv \leq 19.6\%$, for AR-WMA; $Cv \leq 15.8\%$, for Ref. AR-HMA) compared to the mixtures without rubber ($Cv \leq 33.2\%$) and Fig. 8 also shows less dispersion for indirect tensile resistance of water-saturated AR-WMA specimens (ITS_w) than for dry specimens (ITS_d).

3.3. Resistance to permanent deformations

Fig. 9 shows the evolution of the rut depth versus the number of load cycles according to wheel tracking laboratory test (proced. B: in air, small device, at 60 °C and 10^4 cycles) on slab specimens compacted with metallic roller and clearly highlights the improved

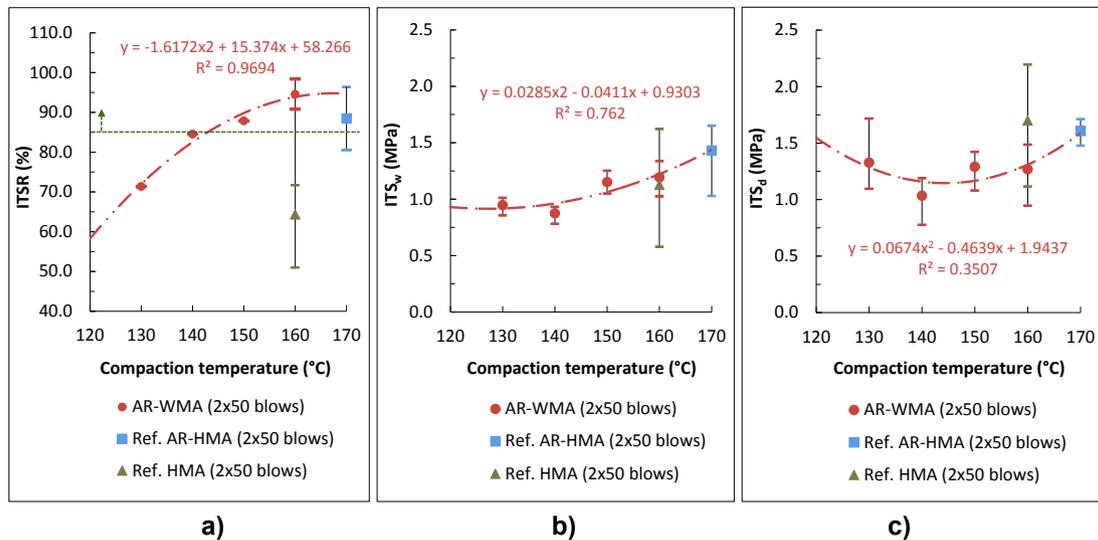


Fig. 8. Water sensitivity of the bituminous mixtures (EN 12697-12) by indirect tensile test on cylindrical specimens (EN 12697-23): a) Indirect tensile strength ratio (Minimum specified ITSR is shown by a horizontal dotted line); b) Indirect tensile strength of saturated specimens; c) Indirect tensile strength of dry specimens.

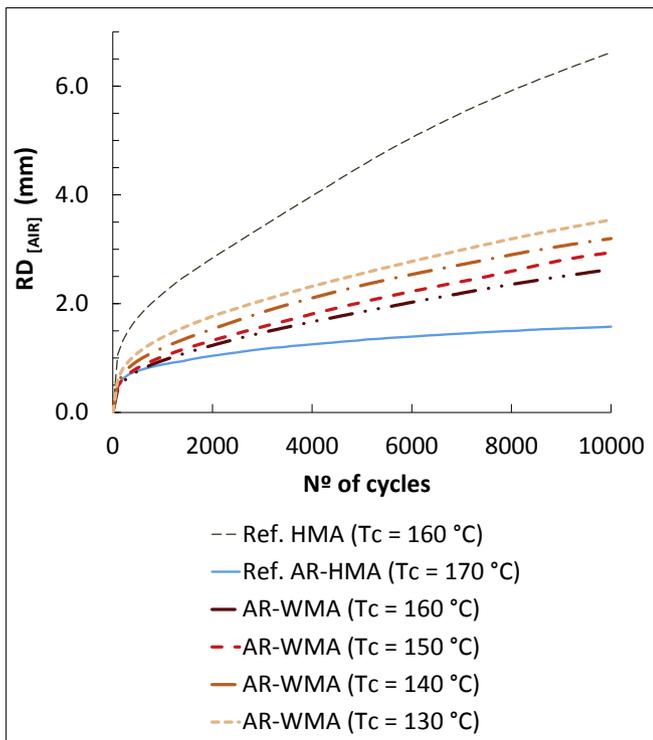


Fig. 9. Rut depth vs. number of load cycles according to wheel tracking test (Proced. B: in air, small device, at 60 °C and 10^4 cycles, EN 12697-22). (Tc: compaction temperature).

rutting resistance of the Ref. AR-HMA compared to the conventional mixture (Ref. HMA). These were both manufactured with the same bitumen content (6.0% by wt. of mixture) but with different production temperatures as aforementioned. This improvement of resistance to permanent deformation of the AR mixture is noteworthy: the wheel-tracking slope of the rut depth ($WTS_{[air]}$ between 5×10^3 and 10^4 load cycles) decreased by 546.0% (Fig. 10a); the rut depth ($RD_{[air]}$ at 10^4 cycles) decreased by 279.6% (Fig. 10b), and the proportional rut depth ($PRD_{[air]}$ at 10^4 cycles) by 8.2%

(Fig. 10c). These results prove that the use of an AR binder in mixtures with this type of volcanic aggregates considerably improves the resistance to plastic deformation owing to the elastic properties of the rubber. This improvement constitutes an important practical application for construction purposes (Fig. 11).

AR-WMA slab specimens results are also shown in Figs. 9 and 10 and compared to the reference mixtures. Both wheel-tracking slope of the rut depth ($WTS_{[air]}$) and rut depth ($RD_{[air]}$) and proportional rut depth ($PRD_{[air]}$) increased as production and compaction temperatures of AR-WMA lowered. The good regression adjustment to the functions for the rut depth and the proportional rut depth at 10^4 cycles is noteworthy (Fig. 10b and c).

Therefore, these low-temperature mixtures only met technical specifications for surfaces ($WTS_{[air]} \leq 0.10$ mm/ 10^3 cycles; $PRD_{[air]} \leq 5\%$) with compaction temperatures above 155 °C (whilst $WTS_{[air]}$ limitation is only achieved above 170 °C). In any event, the improvement of resistance to plastic deformation that the AR have on these mixtures with marginal volcanic aggregates compared to the mixtures without rubber (Ref. HMA) remains at all the temperatures studied (Fig. 9). For instance, at 160 °C the $WTS_{[air]}$ of AR-WMA was 117.8% inferior (more favourable) than the Ref. HMA, the $RD_{[air]}$ 129.4% inferior and the $PRD_{[air]}$ 6.5% inferior (Fig. 10). Nevertheless, the aforementioned improvement is reduced as the compaction temperature decreases.

The standard deviation of the $PRD_{[air]}$ results was lower in the case of the AR mixtures ($Sd \leq 1.1\%$, for AR-WMA; $Sd \leq 1.3\%$, for Ref. AR-HMA; $Sd \leq 1.8\%$, for Ref. HMA), even though the variation coefficients were similar (Table 4). It is also worth highlighting the dispersion reduction of laboratory results as the AR-WMA production temperatures increase (Fig. 10).

Marshall tests provided similar results on cylindrical specimens compacted by impact with 2×75 blows, with a noteworthy increase of stability (152.2%), reduction of Marshall flow value (23.9%) and consequently an increased Marshall quotient (221.5%) for the Ref. AR-HMA compared to the Ref. HMA without rubber. These Marshall tests on the AR-WMA specimens showed a decrease of stability and an increase of deformation as temperatures were lowered (Fig. 12). In a similar manner to the results provided by the wheel tracking tests, the improvement of the Marshall stability that the AR binder has on mixtures with this type of aggregates is maintained at all the temperatures studied. At 160 °C the Marshall

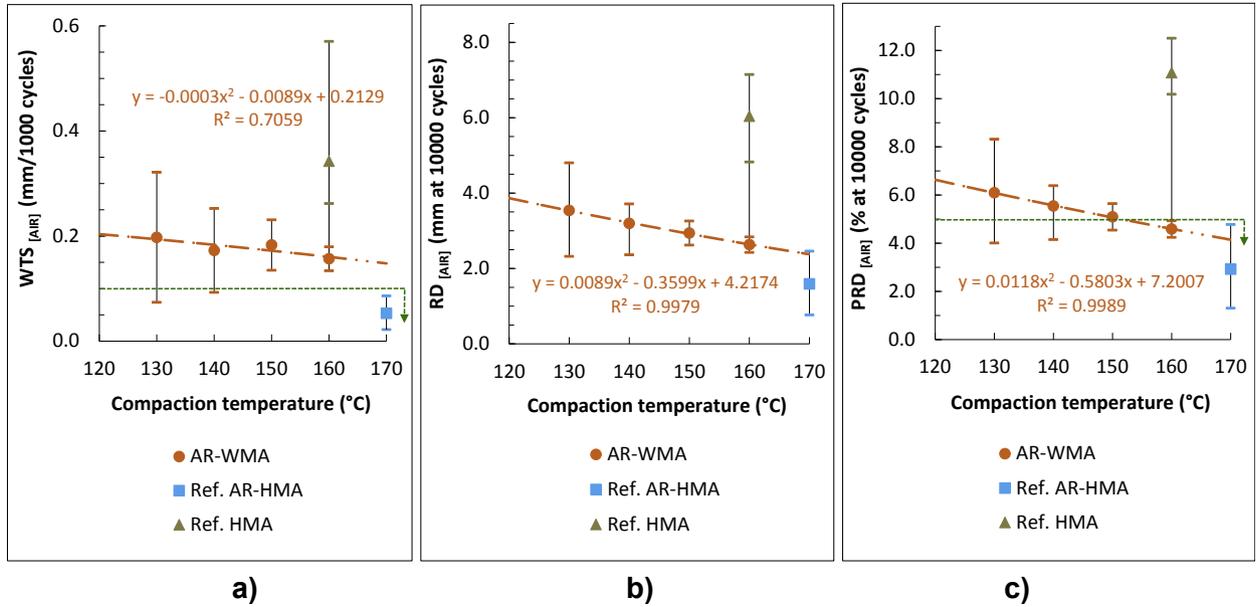


Fig. 10. Wheel tracking test results (Proced. B; in air, small device, at 60 °C and 10⁴ cycles) according to EN 12697-22 (Limits of specifications are indicated by horizontal dotted lines): a) Wheel-tracking slope between 5 × 10³ and 10⁴ load cycles; b) Rut depth at 10⁴ cycles; c) Proportional rut depth at 10⁴ cycles.

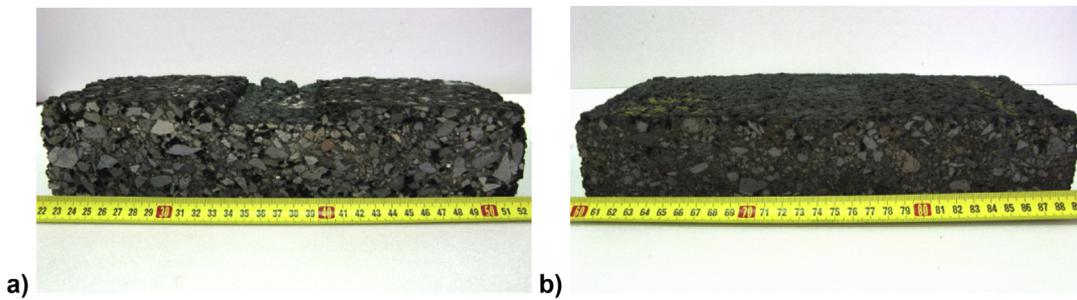


Fig. 11. Improvement of resistance to plastic deformation using the AR binder. Comparison of the rut depth after 10⁴ cycles at 60 °C by wheel tracking test: a) Cut section of a Ref. HMA slab specimen (without rubber); b) Cut section of an AR-WMA slab specimen compacted at 140 °C.

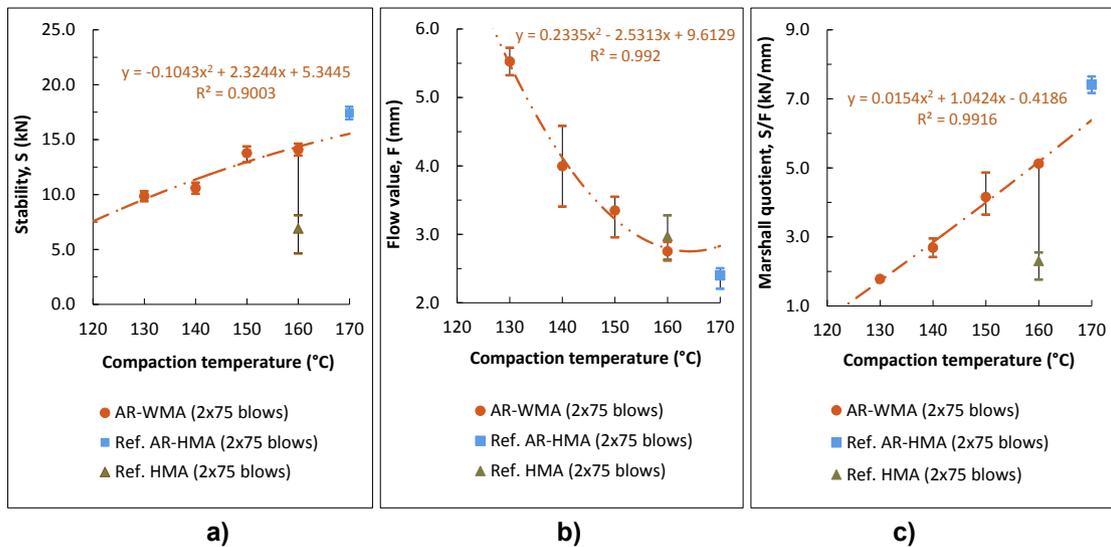


Fig. 12. Marshall test results (EN 12697-34): a) Marshall stability; b) Marshall flow value; c) Marshall quotient.

stability of the AR-WMA was 103.9% higher than the Ref. HMA and the Marshall flow value was 8.0% inferior, while the Marshall quotient continued to be 122.6% superior. However, this improvement of resistance to plastic deformation decreases as the temperature is lowered.

In this test, the statistical dispersions of the results were higher for AR-WMA ($C_v \leq 38.6\%$, for AR-WMA; $C_v \leq 18.4\%$, for Ref. AR-HMA; $C_v \leq 13.2\%$, for Ref. HMA) (Table 4).

The dynamic stiffness modulus by indirect tensile test on cylindrical specimens compacted with 2×75 blows (IT-CY, load surface factor $k=0.6$, at 20°C) was also evaluated, according to Standard EN 12697-26, in order to determine the mechanical performance of these mixtures under traffic loads. The specimens of Ref. AR-HMA provided an average stiffness modulus at 20°C of 6044 MPa and 6830 MPa for Ref. HMA without rubber. Thus, both mixtures (with the same binder content of 6%) showed sufficient stiffness for this type of asphalt concrete, though the mixture with rubber suffered a 13% of modulus reduction due to its slightly higher porosity (as stated in section 3.1), as a result of the higher viscosity of the AR binder.

3.4. Interfacial contact of mixture components

The improved resistance of the AR mixtures, especially to indirect tensile test of water-saturated specimens compared to the mixture without rubber, is closely linked to a superior interfacial adhesion between the binder and the aggregate particles. This can be observed in Fig. 13: cracks fracture the aggregate particles but the adhesion at the aggregate-bitumen interface is maintained (Fig. 13c and d) because of the better adherence of the AR binder. This even occurs on mixtures produced at low temperatures thanks to the use of the surfactant additive; on the contrary, on conventional mixtures cracks mainly go around the interfacial contacts (Fig. 13a and b). Moreover, the superior softening point of the AR binders (see Table 2) contributes to the reduction of the permanent deformation of these mixtures.

According to Mull et al. (2002), it seems that it may have a microstructural origin. CRM binders can increase the interfacial adhesion between the bitumen and the aggregate particles by creating more and finer microscopic ridges and well pronounced dimples with less smooth fracture surfaces. This can be observed on high resolution Scanning Electron Microscopy (SEM) images of the fracture surface. This higher degree of interlock among the mixture components could explain the micromechanical behaviour and thus, the performance enhancement of the rubberized mixtures.

3.5. Compliance with pavement specifications

Fig. 13 summarizes the AR-WMA production temperatures that, according to results, fulfil with standard specifications for pavements, including surface, binder and base courses. As shown on this graph, the worst compliance is with those properties regarding permanent deformations, specially the $WTS_{[air]}$. With this surfactant additive the minimum compaction temperature in order to produce this type of AR-WMA for surface layers is 165°C (mixing at 175°C) if all the requirements must be fulfilled. However, if WTS is not so relevant or if these mixtures are used for binder or base layers, where these requirements are less strict, the compaction temperatures can be reduced to a minimum of $150\text{--}155^\circ\text{C}$. These mixtures even maintain a satisfactory resistance to water damage if compacted over 140°C for surface layers and 135°C for lower pavement layers (see Fig. 14).

4. Conclusions

Based on the results and discussion of this experimental research, the following conclusions can be drawn regarding semi-dense asphalt concrete with highly-vesiculated volcanic aggregates, produced at different temperatures:

The bulk density of the asphalt-rubber warm mixture (AR-WMA) with surfactant additive decreases as production and compaction temperatures are lowered. This low-temperature mixture maintained similar densities to the AR-HMA (mixed at 180°C and compacted at 170°C) as long as compaction is not below 150°C , and similar to the HMA (mixed at 170°C ; compacted at 160°C) if the temperature does not fall below $135\text{--}140^\circ\text{C}$.

Consequently, both the air void content (V_m) and voids in mineral aggregate (VMA) of the AR-WMA increased as manufacture temperature reduced. A void content similar to the AR-HMA and to the HMA was achieved when compaction temperatures were equal or above 155°C . This complies with the road specifications regarding void characteristics for surface layers.

Although a significant enhancement of moisture damage resistance was noted due to the use of the AR binder, the indirect tensile strength ratio (ITSR) of the AR-WMA clearly decreases when temperatures are lowered. ITSR only meets road specifications for surfaces as long as compaction temperature is above 140°C . Nevertheless, the improvement of ITSR owing to the AR binder is maintained at all temperatures studied: at 140°C the improvement still remains around 20%.

Laboratory results clearly highlighted the outstandingly-improved rutting resistance when using the AR binder. However, decreasing temperatures result in a loss of resistance to plastic deformation of the AR-WMA. These low-temperature mixtures only comply with road specifications for surface courses if compacted above 155°C . Nevertheless, the improvement of resistance with respect to the mixture without rubber is maintained at all temperatures studied.

Similar conclusions may be drawn from the Marshall tests: the improvement of resistance to plastic deformation decreases as temperature is lowered, though at the production temperature of conventional mixtures it still remains significantly (at 160°C the AR-WMA Marshall quotient continued to be 122.6% superior than Ref. HMA).

It is possible to produce these AR-WMA and comply with all the standard specifications for road surfaces as long as the compaction temperature remains above $160\text{--}165^\circ\text{C}$ (mixing at $170\text{--}175^\circ\text{C}$). This means a reduction of just $5\text{--}10^\circ\text{C}$, which compensates for the higher energy consumption and emissions owing to the high viscosity of the AR binder. For lower pavement layers or where the requirements regarding plastic deformation are less strict, the compaction temperatures can be reduced to a minimum of $150\text{--}155^\circ\text{C}$.

These AR-WMA can provide adequate performance even with compaction temperatures of $140\text{--}145^\circ\text{C}$, meaning a reduction of the production temperature by up to $25\text{--}30^\circ\text{C}$. This last reduction could imply lower energy use and emissions (approx. 14–18%), safer and healthier conditions for workers, higher transport distances and laying less influenced by climatic conditions. In any case, these mixtures presented certain properties such as the water resistance, rutting resistance and stability superior to the conventional mixtures without rubber at all temperatures studied, even with a reduced proportion of crumb rubber (10% by bitumen weight, in this study).

The proposed methodology could contribute to better development of the environmentally protected volcanic regions by re-using waste from used tyres and marginal aggregates and extending the durability of asphalt pavements.

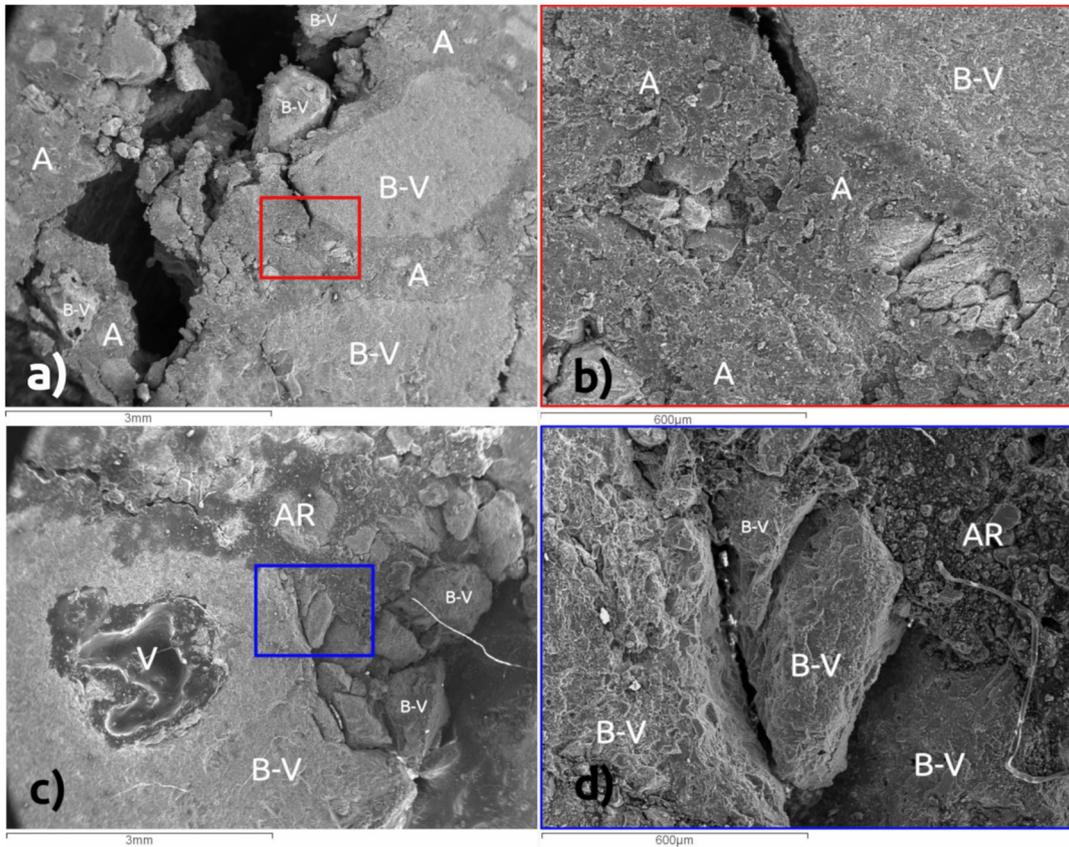


Fig. 13. SEM images of fracture cracks on samples after indirect tensile test: a) & b) Cracks skirt around the aggregate particles in Ref. HMA (without rubber); c) & d) Cracks fracturing the aggregate in AR mixture. (B–V) Aggregate particle of vesicular basalt; (V) Void in aggregate particle; (A) Conventional bitumen; (AR) Asphalt-rubber bitumen.

Properties		Specifications for AC-S		Mixing temperature (°C)													
				Compaction temperature (°C)													
				130	135	140	145	150	155	160	165	170	175	180			
Volumetric	$D_{b,ssd}$ (*)	Maximum value (similar to that obtained with AR-HMA)															
	V_m (*)	Surface courses: 4% - 6%															
		Binder courses: 4% - 7%															
		Base courses: 4% - 8%															
VMA (*)	Surface courses: $\geq 15\%$																
	Binder courses: $\geq 15\%$																
	Base courses: $\geq 14\%$																
Water sensitivity	ITSR (**)	Surface courses: $\geq 85\%$															
		Binder courses: $\geq 80\%$															
		Base courses: $\geq 80\%$															
Permanent deformations	WTS _[air]	Surface and binder courses: $\leq 0.07-0.10 \text{ mm}/10^3 \text{ cycles}$															
		Base courses: $\leq 0.10-0.15$															
	PRD _[air]	All courses: $\leq 5\%$															

Complying specifications for surface courses of pavements
 Complying specifications for binder courses of pavements
 Complying specifications for base courses of pavements

(*) Specimens compacted by impact with 2x75 blows
 (**) Specimens compacted by impact with 2x50 blows

Fig. 14. Production temperatures of AR-WMA complying with technical specifications for road pavements (Specifications for AC mixtures according to Spanish Ministry of Infrastructures, 2014).

The following aspects are considered interesting for future research:

Analyse the fatigue resistance to dynamic repeated loads and durability of this type of AR-WMA, as well as its crack resistance at low temperatures.

Study reclaimed asphalt pavement (RAP) mixtures with this type of aggregates and the efficiency of the AR-WMA additives following subsequent recycling processes.

Implementation of this AR-WMA studied in the laboratory on operational roads.

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