

Valorisation of lignin-rich industrial byproduct into half-warm mix reclaimed asphalt with enhanced performance

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ABSTRACT

To promote a circular economy and sustainable development, the possibility of using a lignin-rich industrial byproduct as a partial substitute for asphalt emulsions in road pavements is analysed. A half-warm mix reclaimed asphalt (HWMRA)-type AC 16 surf S, manufactured with 100% reclaimed asphalt pavement, is selected. Substitution percentages of 0% (control mixture), 5%, 10%, 15%, and 20% are employed.

The microstructure of the blend of the byproduct and asphalt emulsion is observed using scanning electron microscopy. The water resistance of the mixture is investigated using indirect tensile tests, as well as its volumetric properties. The stiffness, thermal susceptibility by indirect tensile tests, and resistance to permanent deformation by confined uniaxial compression tests and Hamburg wheel tracking tests are also studied.

The optimum percentage of byproduct substitution is 5%, resulting in an enhanced HWMRA with a slightly better water resistance, higher resistance to permanent deformation at low and medium environmental temperatures, and higher indirect tensile strength and stiffness than those of the control mix.

1. Introduction

The term lignin encompasses a set of biopolymers with a three-dimensional molecular structure composed of coniferyl alcohol [1]. It is an amorphous and inactive material that constitutes approximately 20/40% of the plant tissue mass of tall plants [1]. Its discovery dates back to 1838 when the French chemist Anselme Payen discovered that, in addition to cellulose, there was another component in the wood [1].

Owing to the nature and availability of this renewable biomass resource, numerous researchers have attempted to find new applications for lignin. In particular, because its chemical compounds and hydrocarbons are similar to those contained in bitumen, lignin has significant potentials for use in road sustainable pavement construction [2].

Several researchers have analysed the effect of using lignin as a bitumen extender or modifier. Pan [3] stated that coniferyl-alcohol lignin can be successfully used as a bitumen antioxidant; however, the temperature should be controlled (e.g. < 130 °C) when blending bitumen and lignin to avoid lignin oxidation. According to Xu et al. [4], when wood lignin powder is blended with neat bitumen in percentages of 5% and 10%, a stiffening effect occurs in the modified asphalt binder. Better high-temperature rutting performance, improved ageing

performance, and worse fatigue resistance of lignin-modified bitumen have been reported. Xie et al. [5] stated that bitumen modified with Kraft lignin improves rutting but worsens low-temperature cracking. In addition, they found that some lignin fractions improved the temperature performance of modified bitumen and the ageing properties. Batista et al. [6] concluded that modified bitumen with 1%, 4%, and 6% commercial Kraft lignin led to binders with higher rutting resistance at high temperatures, higher thermal cracking resistance at low temperatures, and higher photodegradation resistance. Zabelkin et al. [7] demonstrated that when pyrolytic lignin is used as a bitumen modifier, it produces a volume increase (38%) with heating (140 °C), thus decreasing the bitumen consumption. They also discovered that pyrolytic lignin could improve the cohesion of bitumen. Gao et al. [8] analysed the high-temperature rheological performance and fatigue performance of a virgin bitumen PG 58–28 modified with 2%, 4%, 6%, and 8% of commercial lignin from waste wood chips. Improved permanent deformation resistance and retarded oxidation reactions were observed. Nevertheless, a lower fatigue performance was achieved for lignin concentrations over 8%. Norgbey et al. [2] used lignin waste as a bitumen modifier and extender. They found that lignin-modified bitumen had higher viscosity, better aggregate-binder adhesion, and

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better rutting resistance. Peng et al. [9] found that asphalt modified with lignin-based polyurethane improved the adhesion properties of the asphalt binder. Yu et al. [10] stated that asphalt binders modified with soda lignin powder in percentages of 5%, 10%, 15%, and 20% had better thermal storage stability and enhanced fatigue and rutting performance. Wu et al. [11] used lignin from the pulp and paper industry as a bitumen extender with a mass ratio of 1:4 (lignin: bitumen), and observed improved ageing performance, increased stiffness, and lower thermal susceptibility. Nevertheless, they also found that it was necessary to increase the mixing temperature to 10 °C when using lignin as a bitumen extender.

Various researchers have analysed the effect of lignin as a bitumen extender or bitumen modifier. Nevertheless, investigations that analyse how the use of lignin as a bitumen extender or bitumen modifier influences the performance of bituminous mixtures are limited. Hobson [12] analysed two commercial lignins (from softwood and hardwood) as antioxidants for hot-mix asphalt (HMA), concluding that the results of this study do not support the theory that lignin acts as an antioxidant for HMA. Nevertheless, he used lignin as a fine aggregate substitute and not as a bitumen modifier or extender. Yuliestyan et al. [13] used chemically modified Kraft lignin as a cationic emulsifier for bitumen emulsions used for the manufacture of 100% reclaimed asphalt pavement (RAP) half-warm mix asphalt (HWMA). They used a gyratory compactor to manufacture the specimens, obtained the indirect stiffness modulus at 20 °C and the indirect tensile strength at 15 °C. Despite the good mechanical performance of the mixture, they did not compare the performance of this mixture with that of a half-warm mix reclaimed asphalt (HWMRA) made with commercial bitumen emulsion. Subsequently, Yuliestyan et al. [14] studied 100% RAP mixes, manufactured using a bitumen emulsion stabilised by amine-functionalised lignin at temperatures ranging from 80 to 130 °C. These researchers concluded that the mixes met the requirements of French design. They did not use a control mixture with a commercial bitumen emulsion to compare the results. Pérez et al. [15] analysed the possibility of using lignin-rich industrial waste from the hardboard industry as a bitumen extender for HMA. These researchers concluded that the optimum substitution rate was 20% by weight of the bitumen. This percentage improved the water sensitivity of the bituminous mixes because of the introduction of lignin and the foaming of bitumen. They also studied the modified bitumen properties [16] and stated that the use of 20% of industrial waste improved the fatigue and rutting resistance of the modified asphalt. Arafat et al. [17] concluded that the addition of 6% of lignin (obtained from black liquor using softwood Kraft processing) to the neat bitumen led to HMA with improved rutting resistance and similar water sensitivity. These authors also studied lignin-modified bitumen performance and concluded that long-term ageing was enhanced. Zhang et al. [18] analysed the moisture damage resistance, ravelling, rutting, thermal, and fatigue cracking of stone mastic asphalt (SMA) manufactured using 10% organosolv wood lignin powder instead of bitumen. These researchers found that the lignin powder improved the overall mechanical performance of the SMA. Zahedi et al. [19] concluded that using up to 6% lignin improved the Marshall stability of the tested HMA. They used lignin extracted from black liquor, heating it at a maximum temperature of 110 °C and then sieving and milling the residue. Xu et al. [20] used two types of commercial lignin as a partial substitute for bitumen for the manufacture of HMA. They used 5% lignin by weight of bitumen. They found that mixtures made with bitumen modified with lignin exhibited enhanced moisture damage resistance, permanent deformation, and ageing performance.

The use of asphalt mixes with low manufacturing and laying temperatures, that is, cold mixes and warm mixes would be in line with improved health and safety in paving works. In addition, both technologies have lower greenhouse gas (GHG) emissions than those of HMA. The Intergovernmental Panel on Climate Change (IPCC) in its Fifth Assessment Report (AR5) [21] identifies GHGs as the main drivers of climate change. Therefore, a reduction in GHG emissions, is expected

to contribute to the objective of maintaining the average global warming below 2 °C and combating anthropogenic climate change. Moreover, the use of these types of mixtures has other advantages such as lower energy consumption, the availability of a longer construction season, reduced odours, and the possibility of off-peak paving [22]. Therefore, it is of interest to enhance the use of cold and half-warm asphalt mixtures.

HWMA is a mixture that requires lower manufacturing and construction temperatures than conventional HMA. Remarkably, the manufacturing and laying temperatures range from 60 °C to 100 °C [23]. Nevertheless, they perform similarly to the HMA [24]. In particular, HWMA can be made with foamed bitumen or bitumen emulsions. If they are made with bitumen emulsion, additives must be added to improve adhesion, wrapping, and workability [24]. That is, an important conclusion that has been seen throughout the literature review is that although HWMA has a mechanical behaviour similar to that of HMA, it may be more sensitive to the action of water because of its lower manufacturing temperature [24].

2. Aims and scope

According to the Food and Agriculture Organization of the United Nations, 12 million m³ of hardwood panels are produced on our planet every year. In Spain, the National Association of Board Manufacturers [25] indicates that companies producing wood-based panels are distributed throughout the national territory. Solid waste, toxic gases, and wastewater are generated during the production of wood-based panels. For example, in the case of a plant producing hardboard (HB), for every cubic metre of HB produced, 2.49 L of wastewater are generated [26]. Specifically, in the case of HB, a large amount of lignocellulosic waste is generated during the wet manufacturing process and is dissolved in the wastewater [27]. This is the main drawback of this manufacturing process, which prevents the proper development of this industry, as the treatment of this waste generates high production costs.

This study analyses the feasibility of using a lignin-rich industrial byproduct from the manufacture of HB as a partial substitute for bituminous emulsion in the manufacture of reclaimed bituminous mixtures. In this study, the lignin-rich industrial byproduct is directly used, that is, it is used with no physical and/or chemical transformation. Specifically, the performance of an asphalt concrete half-warm mixture manufactured with 100% RAP (HWMRA) and with partial substitution of bitumen emulsion using the industrial lignin-rich byproduct was analysed. The selected mixture is for the surface layer of road pavements and presents an aggregate with a maximum size of 16 mm and a semi-dense grain size (AC 16 surf S).

The three major objectives of this study are:

- First, to reduce the consumption of bituminous emulsion in the manufacture of bituminous mixtures by partially replacing it with an industrial byproduct rich in the biopolymer lignin from the manufacture of HB.
- Second, to promote the use of more environmentally friendly technologies for manufacturing bituminous mixtures and, therefore, contribute to the fight against anthropogenic climate change. Accordingly, the study was performed using a half-warm bituminous mixture.
- Finally, it contributes to the implementation of the circular economy both in the road paving sector and in the wood-based panel industries by reducing the consumption of natural resources, reusing raw materials, and increasing the durability of the end products. Accordingly, not only industrial waste will be used in the manufacture of bituminous mixtures but also RAP from road milling will be used.

This proposal is in line with “*Asphalt is not trashfalt*”, that is, the idea is not to use the road as a landfill but to take advantage of the potential of industrial byproducts to manufacture a product with the same or

higher quality, but with lower consumption of natural resources and, therefore, higher competitiveness. The waste is rich in biopolymer lignin. This makes it particularly suitable for the improvement of the water sensitivity of HWMRA.

Consequently, the performance of an HWMRA manufactured with 5%, 10%, 15%, and 20% of industrial byproducts rich in biopolymer lignin in place of bituminous emulsion is being studied. As shown in the study flowchart in Fig. 1, first, the characterisation of the basic materials is addressed. In particular, the moisture contents of RAP and its particle size were determined. The microstructure of the mix of industrial waste and emulsion was analysed using scanning electron microscopy (SEM). Second, the water resistance of the mixture was investigated using indirect tensile tests, as well as its volumetric properties. Third, for the control mixture (0% industrial waste) and for the mixture manufactured

using the optimum percentage of emulsion replacement by industrial waste, the mechanical properties were analysed and compared: stiffness, thermal susceptibility, resistance to permanent deformation using the Hamburg wheel tracking tests, uniaxial compression tests, and cohesion loss. Finally, penetration tests and ring and ball tests were conducted for the control bitumen emulsion and for the bitumen emulsion mixed with the optimum percentage of industrial waste replacement.

3. Materials and methods

The materials and methods used for the present laboratory investigation are described below.

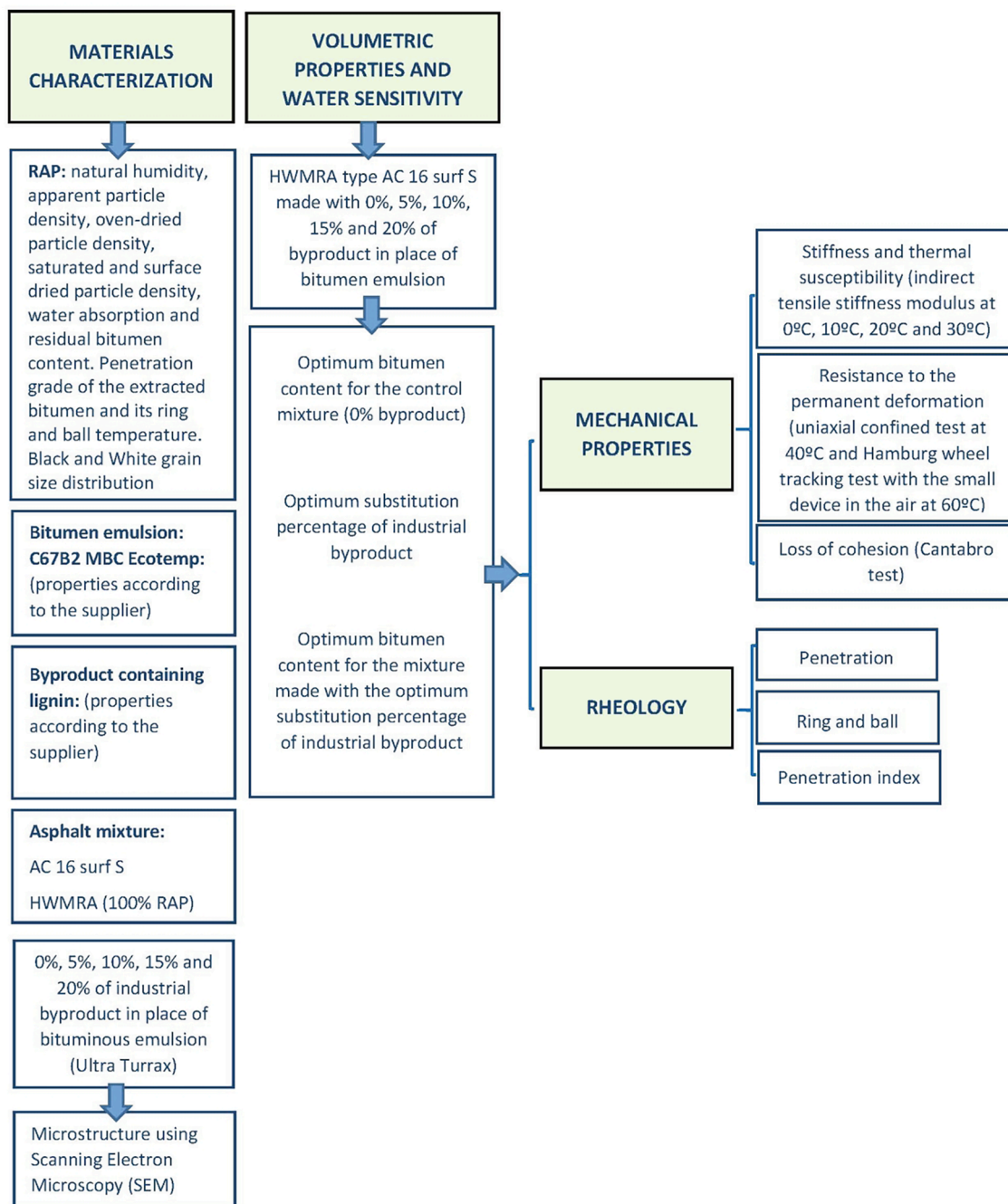


Fig. 1. Study flowchart.

3.1. Materials characterisation

3.1.1. Aggregates

One type of aggregate was used for the manufacture of bituminous mixtures. In particular, RAP from the milling of bituminous surface course layers of existing roads was supplied by a local contractor. According to the ATEB [28], the RAP was divided into two fractions: 0/5.6 mm and 5.6/22.4 mm. As shown in Table 1, the natural humidity, apparent particle density (ρ_a), oven-dried particle density (ρ_{RD}), saturated and surface dried particle density (ρ_{SSD}), water absorption (WA_{24}), and residual bitumen content were obtained to characterise the two RAP fractions. Table 1 also shows the penetration grade of the extracted bitumen and its ring and ball temperatures.

As expected, the natural humidity and water absorption of the fine fraction exceeded those of the coarse fraction owing to the higher specific surface area of the fine fraction. Table 1 shows that the different densities are higher for the coarse fraction than for the fine fraction. This result is followed by the higher residual bitumen content of the fine fraction, and a higher specific surface area.

As expected, the penetration of the extracted bitumen was low, and its ring and ball temperatures were high. These results confirm the ageing of the extracted bitumen.

3.1.2. Bitumen emulsion

Following the supplier's suggestions, a bitumen emulsion C67B2 MBC Ecotemp was used as the binder for the manufacture of bituminous mixtures. According to the supplier and EN-13808 [34], it is a rapid-setting cationic bitumen emulsion that has a bitumen content ranging from 65% to 69% and is specifically designed for the construction and maintenance of close-graded HWMA. The main properties of the bitumen emulsion, according to the supplier, are listed in Table 2.

3.1.3. Byproduct containing lignin

The industrial waste used (Fig. 2) originates from wood extracts that are generated during the manufacturing process of high-density fibreboards. It is a 100% natural co-product containing high value-added compounds, such as lignin, tannins, polyphenols, and sugars. More specifically, according to the supplier, industrial waste comprised 56.31% water and 43.69% dry matter. The main components of this dry matter were 41.46% of sugar (mainly xylose, glucose, galactose, rhamnose, arabinose, and mannose), 23.39% of lignin (16.29% of insoluble Klason lignin and 7.10% of soluble lignin), 13.3% of pectin, 11.8% of polyphenols, 9.05% of mineral matter, and other compounds that appeared in low percentages. It has a pH of 3.2 and a density (at 75 °C) of 1,155 kg/m³.

Table 1
RAP characterisation.

Property	Standard	Fraction 0/ 5.6 mm	Fraction 5.6/ 22.4 mm
Natural humidity (%)	EN 1097-5 [29]	0.37	0.21
ρ_a	EN 1097-6 [30]	2.41	2.51
ρ_{RD}	EN 1097-6 [30]	2.34	2.45
ρ_{SSD}	EN 1097-6 [30]	2.37	2.48
WA_{24}	EN 1097-6 [30]	1.21	0.94
Residual bitumen content (%)	EN 12697-1 [31]	5.49	3.41
Penetration (0.1 mm) at 25 °C of the extracted bitumen	EN 1426 [32]	32	
Ring and ball (°C) of the extracted bitumen	EN 1427 [33]	70°	

Table 2
Main properties of the bitumen emulsion.

Property	Standard	Value
Polarity	EN 1430 [35]	Positive
Recovered binder content (by distillation) (%)	EN 1431 [36]	≥65
Fluidising agent content (by distillation) (%)	EN 1431 [36]	≤2



Fig. 2. Industrial byproduct used in this investigation.

3.1.4. Mix of bitumen emulsion and byproduct containing lignin

In this study, the cationic bitumen emulsion was partially substituted with different percentages of the byproduct containing lignin: 0% (control), 5%, 10%, 15%, and 20% by weight.

To guarantee a correct bitumen modification and a uniform blend of the bitumen emulsion and the byproduct, both products were mixed using an ultra turrax rotor–stator dispersing instrument at 7,000 rpm for 30 min at ambient temperature (Fig. 3).



Fig. 3. Blend of bitumen emulsion with byproduct using ultra turrax.

3.1.5. Asphalt mixture

To determine the type of mixture, both fractions, the fine (0/5.6 mm) and coarse (5.6/22.4 mm), were sieved following EN 933-1 [37]. The distribution of the grain size of RAP (black curve) was determined for the two fractions (Fig. 4). Subsequently, the bitumen was extracted from the RAP according to EN 12697-1 [31] using a mesh cylinder, a continuous flow centrifuge, and perchloroethylene. The distribution of the grain size after bitumen extraction (white curve) was also obtained according to EN 933-1 [37] for the two aforementioned fractions (Fig. 4).

Considering the white curve of the RAP, using 30% of the fine fraction (0/5.6 mm) and 70% of the coarse fraction (5.6/22.4 mm), an HWMRA-type AC 16 surf S, for the surface course of road pavements, was composed according to the limits given by the ATEB [28], as shown in Fig. 5.

To manufacture the AC 16 surf S, the RAP was dried for 24 h at 60 °C. Subsequently, the RAP was heated at 110 °C, the bitumen emulsion was heated at 65 °C and the HWMRA was mixed and compacted at a temperature lower than 100 °C. For sample compaction, the gyratory compactor was used according to EN 12697-31 [38] using 65 or 44 turns, depending on the test.

According to the ATEB [28], all the samples were cured for 3 days at 50 °C in an oven before use.

3.1.6. Microstructure of the asphalt binder using SEM

The microstructure of the blend of bitumen emulsion and lignin-rich byproducts was examined using SEM. Percentages of 0% (control), 5%, 10%, 15%, and 20% of byproduct in place of the bitumen emulsion were analysed.

3.2. Methodology

3.2.1. Volumetric properties

The EN 12697-8 [39] indicates that the air void content (V_a) must be calculated as follows:

$$V_a = \frac{\rho_m - \rho_b}{\rho_m} \times 100 \quad (1)$$

The bulk specific density saturated surface dry (ρ_b) was determined on cylindrical specimens of 60 mm in height and 100 mm in diameter, which was compacted with 65 turns of the gyratory compactor according to EN 12697-6 [40]. The maximum specific density (ρ_m) was obtained for loose asphalt mixtures according to EN 12697-5 [41]. For the bulk specific density and maximum specific density, three samples were used in each case.

According to the ATEB [28], an HWMRA-type AC 16 surf S should have V_a ranging from 3% to 6% to be adequate for the highest heavy traffic categories (annual average daily heavy traffic ranging from 2,000 to 200). For the lowest heavy traffic categories (annual average daily heavy traffic lower than 200), V_a should range from 3% to 5%.

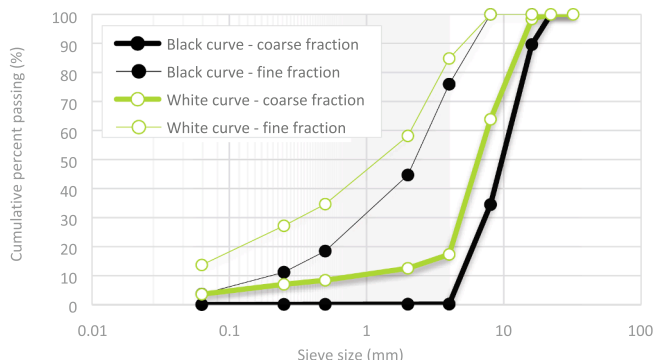


Fig. 4. Black and white curves of the RAP.

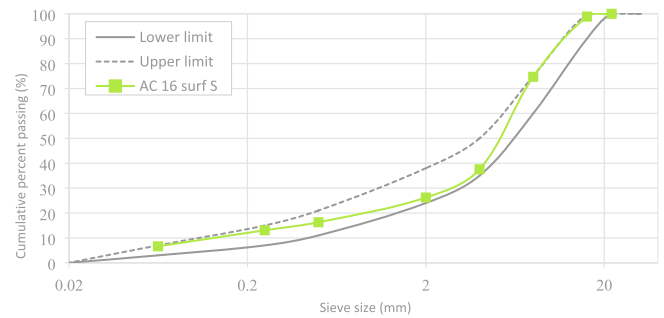


Fig. 5. Distribution of the grain size of the selected AC 16 surf S.

3.2.2. Water resistance

An HWMRA-type AC 16 surf S should have a tensile strength ratio (TSR) of over 85% [28] to exhibit adequate performance against the action of water. According to EN 12697-12 [42], the TSR is calculated as follows:

$$TSR = \frac{ITS_w}{ITS_D} \times 100 \quad (2)$$

where ITS_w is the average tensile strength of four cylindrical specimens that were subjected to a water bath at 40 °C for 3 days and subsequently conditioned for a minimum of 4 h at 15 °C in water. ITS_D is the average tensile strength of four cylindrical specimens that have been at room temperature and subsequently conditioned for a minimum of 4 h at 15 °C in air. For this test, 8 samples were used for each case.

3.2.3. Stiffness and thermal susceptibility

To determine the stiffness of the mixture, the indirect tensile stiffness modulus (resilient modulus) was obtained according to Annex C of EN 12697-26 [43], as shown in Fig. 6.

Thus, the resilient modulus (M_R) was calculated as follows:

$$M_R = \frac{F \times (\hat{\nu}^2 + 0.27)}{z \times h} \quad (3)$$

where F is the maximum applied load (N), z is the horizontal deformation (mm), h is the sample thickness (mm), and ν is Poisson's ratio (assuming a Poisson's ratio of 0.35 for different temperatures [42]).

The test was conducted in a climatic chamber. To analyse the stiffness of the mixture, the resilient modulus was obtained at 20 °C. To analyse the thermal susceptibility of the mixture, the resilient modulus was obtained at 0, 10, 20, and 30 °C. Four samples were used in this test.

There are no requirements in terms of resilient modulus or thermal susceptibility for an HWMRA-type AC 16 surf S.

3.2.4. Resistance to the permanent deformation

In this study, two methods were used to determine the rutting susceptibility of the mixtures: uniaxial confined test based on EN 12697-25 [44] and Hamburg wheel tracking test based on EN 12697-22 [45] with a small device in the air. Two procedures were used to analyse whether both led to similar results.

In the confined uniaxial cyclic compression test (Fig. 7), three specimens of 150 mm in diameter and 60 mm in height were tested for each type of mixture. The samples were subjected to 3,600 vertical, periodic, and rectangular load pulses with a load of 100 ± 2 kPa and a frequency of 0.5 Hz.

The percentage axial cumulative deformation after n load cycles (ϵ_n) was calculated as follows:

$$\epsilon_n = \frac{u_n}{t_i} \times 100 \quad (4)$$

where u_n is the accumulated permanent deformation of the specimen after n load cycles, rounded to the hundredth of a millimetre, and t_i is the

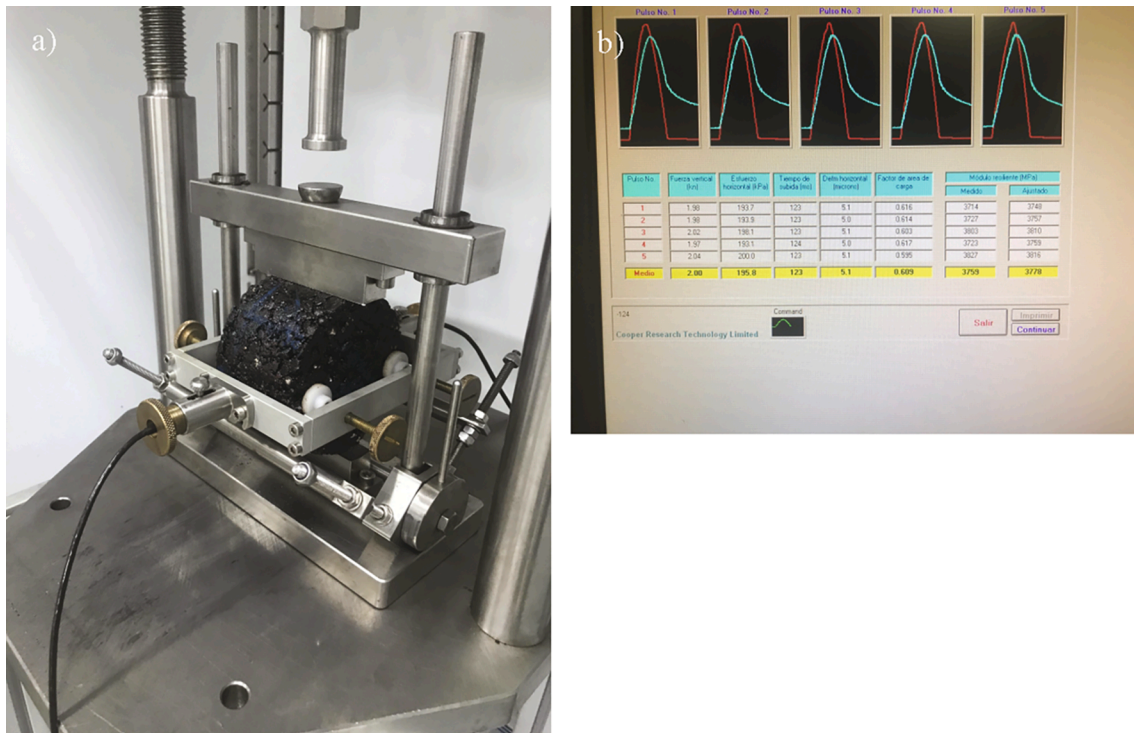


Fig. 6. Resilient modulus test using the Cooper NU-14: a) Cylindrical samples of 100 mm of diameter and 60 mm in height subjected to haversine pulses in two perpendicular vertical diameters and b) after the 10 conditioning pulses, the sample is subjected to 5 pulses.



Fig. 7. Confined uniaxial cyclic compression test.

initial thickness of the specimen (mm).

In the Hamburg wheel tracking test, the susceptibility of the resistance of the mixture to permanent deformation was evaluated by analysing the rut formed as a consequence of the repeated passes of a loaded wheel (700 ± 10 kN) over prismatic specimens at a controlled temperature (60 ± 1 °C). For the AC 16 surf S, the test was conducted in air using a small device followed by procedure B. Two prismatic samples

(400 × 305 × 60 mm) were tested for each type of mixture.

There are no requirements for a uniaxial confined test. However, as seen in Table 3, there are requirements for the HWMRA-type AC 16 surf S for the wheel tracking test [46].

3.2.5. Cohesion loss

The cohesion loss of HWMRA was determined using the mean of the Cántabro test [47]. This test allows us to empirically assess the resistance to the disintegration of the mix in the face of the abrasive and suction effects caused by traffic using the weight loss generated by the abrasion of the bituminous mixture.

Table 3

Hamburg wheel tracking test requirements. Average wheel tracking slope on air (WTS_{AIR}) and rut average depth (%) (PRD_{AIR}) in the interval from 5,000 to 10,000 cycles.

Summer thermal zone	WTS _{AIR} for heavy traffic category (*)				
	T00 and T0	T1	T2	T3 and shoulders	T4
Hot	≤0.07		≤0.07 (**)	≤0.10 (***)	
Medium	≤0.07	≤0.07 (**)	≤0.10 (***)	≤0.15	
Warm	≤0.10	≤0.10 (***)	≤0.10		

(*) Traffic category T00 refers to annual average daily heavy traffic (AADHT) ≥ 4,000.

Traffic category T0 refers to 4,000 > AADHT ≥ 2,000.

Traffic category T1 refers to 2,000 > AADHT ≥ 800.

Traffic category T2 refers to 800 > AADHT ≥ 200.

Traffic category T3 refers to 200 > AADHT ≥ 50.

Traffic category T4 refers to AADHT < 50.

(**) Values higher than the one indicated may be accepted if, simultaneously, it is satisfied that WTS_{AIR} ≤ 0.10 and PRD_{AIR} < 5%.

(***) Values higher than the one indicated may be accepted if, simultaneously, it is satisfied that WTS_{AIR} ≤ 0.15 and PRD_{AIR} < 5%.

In this test, four cylindrical test specimens were fabricated for each case. The samples were compacted at 44 turns on a gyratory compactor. After curing, the specimens were conditioned at the test temperature (22 ± 1 °C) and were introduced consecutively in the Los Angeles machine without the abrasive charge (steel balls). Each sample was subjected to 300 revolutions at a rotation speed of 30–33 rpm. At the end of the process, it is possible to calculate the weight loss (in percentage) owing to the degradation of each sample. This loss, defined as IC (or Cántabro Index), can be determined using the following equation:

$$IC (\%) = \frac{P_{ini} - P_{fin}}{P_{ini}} \times 100 \quad (5)$$

where: P_{ini} : initial weight of the sample (g) P_{fin} : final weight of the sample (g)

There are no requirements for the AC 16 surf S in terms of the Cántabro test; however, for porous asphalt, a maximum value of 20–25% [48] is allowed.

3.2.6. Rheology

To determine the consistency of the modified bitumen and control binder, a penetration test was conducted according to the EN-1426 standard [49]. In the present test, the penetration in 0.1 mm of a needle in the bitumen sample was measured. The needle had a charge of 100 g and was allowed to act for 5 s at a temperature of 25 °C. Six measurements were performed in each case.

Similarly, to determine the thermal susceptibility of the modified bitumen and the control binder, the softening point was determined using ring and ball tests. This test was conducted according to the EN-1427 standard [50]. In this test, two annular bitumen samples were heated in a water bath in a controlled manner (5 °C per minute) with a ball on top, until the samples touched the lower surface, which was at a distance of 25 ± 0.4 mm. The temperature at which each sample touched the lower surface was noted, and the average of both measurements was the ring and ball softening temperature. Two measurements were performed in each case.

After conducting both tests, the penetration index (IP) was calculated using the NLT-181 [47]. This index provides a measure of the thermal susceptibility of bituminous material and is calculated as follows:

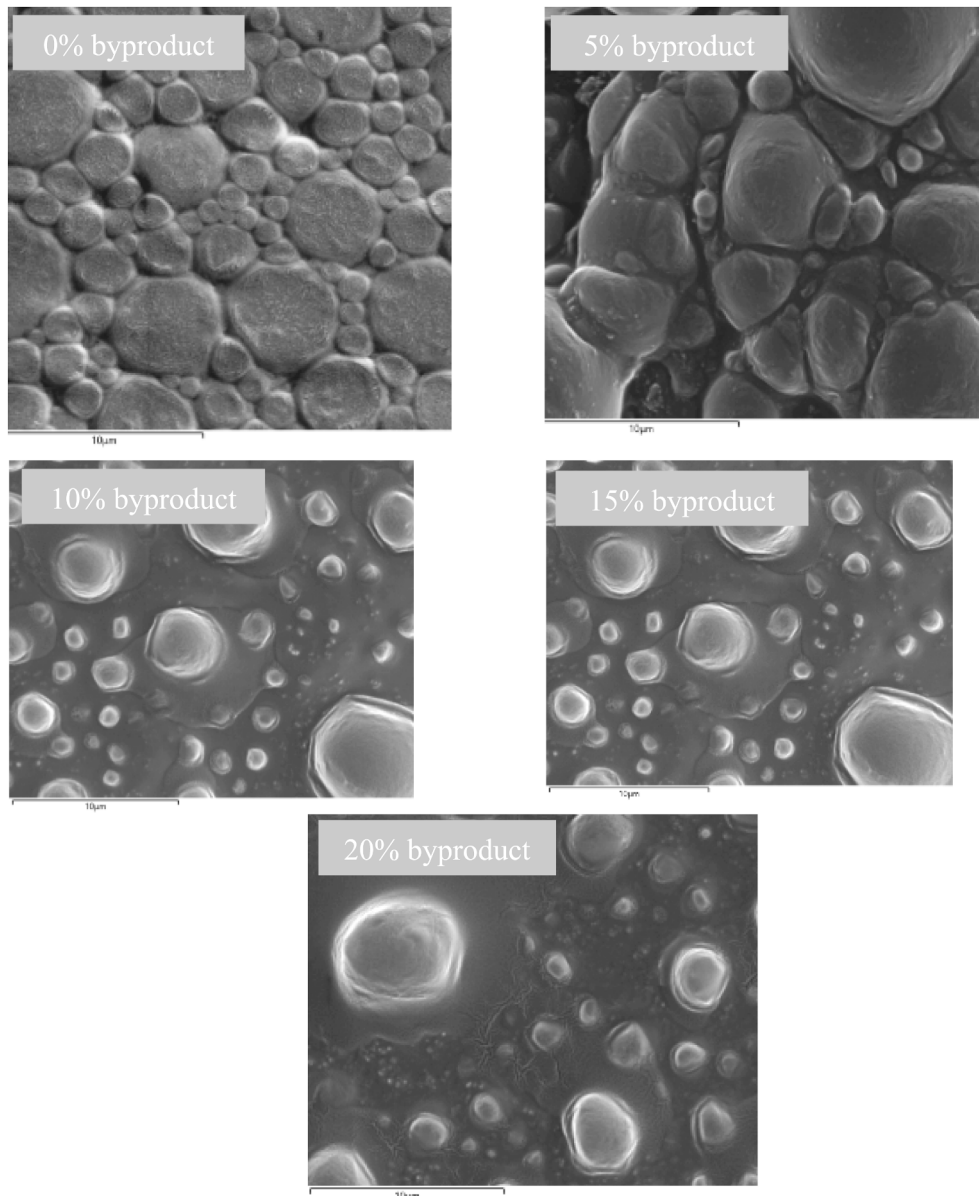


Fig. 8. SEM images of the bitumen emulsion (0% byproduct) and the bitumen emulsion mixed with different byproduct percentages (5%, 10%, 15%, and 20%).

$$IP = \frac{20u - 300v}{u + 30v} \quad (6)$$

where $u = (t_{AB} - t_p) \cdot \log 4v = \log 800 - \log Pt_{t_{AB}}$ = ring and ball temperature (°C) t_p = temperature at which the penetration test is conducted (25 °C) Pt = penetration in 0.1 mm

Bitumen for road asphalt pavement applications usually has an IP ranging from -1 to $+1$ [47]. When bitumen has an IP lower than -1 , its thermal susceptibility is higher. When the IP is higher than $+1$, the thermal susceptibility is lower.

4. Results and discussion

The results obtained in this study are described and analysed below.

4.1. Microstructure of the asphalt binder using SEM

SEM observations of the mixture of bitumen emulsion and byproduct in different percentages (0%, 5%, 10%, 15%, and 20%) were conducted to study the microstructure of the binder. Selected SEM images of these observations are shown in Fig. 8.

Small bitumen micelles can be seen in the microstructure of the bituminous emulsion (0% byproduct), as expected. In addition, Fig. 8 shows that as the byproduct is added to the bitumen emulsion (from 5% to 20%), the small bitumen micelles disappeared, giving rise to a different microstructure from that of the bitumen emulsion. This is likely because the mineral matter included in the byproduct acts as a demulsifier of the bitumen emulsion. The microstructure of the bituminous emulsion is partially preserved only in the mixture of emulsion and 5% byproduct, because of the low quantity of byproduct used. Consequently, the performance of mixtures manufactured with a byproduct content ranging from 10% to 20% is expected to differ significantly from that of HWMRA-type AC 16 surf S manufactured with 0% (control) and 5% byproduct.

4.2. Volumetric properties

Table 4 shows the air void content (V_a) for a total rate HWMRA-type AC 16 surf S, manufactured using 0% (control), 5%, 10%, 15%, and 20% of the lignin-rich byproduct in place of the bitumen emulsion. In Table 4, the values that comply with the air void content required for this type of mixture (3% to 6%) are shaded in grey. As seen, for the control mixture, the first binder content that leads to an adequate air void content was 3.00%.

As shown in Table 4, for some binder contents (3.50% and 4.00%), in general, as the byproduct percentage increases, the air void content decreases. In addition, for all binder contents, the air void content was lower for the HWMRA-type AC 16 surf S manufactured using all byproduct percentages than for the control mixture (0%). Nevertheless, despite these observations, there is no clear trend in terms of the air void

Table 4

Air voids content (%) for HWMRA-type AC 16 surf S manufactured using lignin-rich byproduct.

Residual binder content (%) ^(*)	Byproduct				
	0%	5%	10%	15%	20%
2.50	7.50	6.16	5.00	6.22	6.77
3.00	5.69	5.38	5.54	4.77	5.43
3.50	5.15	4.77	4.78	4.50	3.55
4.00	4.41	3.59	2.94	3.79	2.90
4.50	3.41	–	–	–	–

^(*) The term residual binder refers to the mixture of the residual binder content of the bitumen emulsion C67B2MBD Ecotemp and byproduct in mass over the dry aggregate. For example, for a binder content of 2.5% and 0% of byproduct, 2.5% is bitumen; however, for a binder content of 2.5% and 20% of byproduct, 2.0% is bitumen, and 0.5% is the byproduct.

content evolution as the byproduct content increases.

It is also interesting to analyse the apparent density SSD (Table 5) and the maximum density (Table 6).

In the case of the apparent density (Table 5), for all the binder contents, the mixture made with all the byproduct percentages displayed higher values than the control mixture (0%). For some binder content (3.50% and 4.00%), in general, as the percentage of the byproduct increases, the apparent density also increases. However, similar to the air void content, there is no clear trend for the apparent density values as a function of the percentage of byproduct.

Nevertheless, for the maximum density (Table 6), a clear trend is shown. In general, as the percentage of the byproduct increases, the maximum density increases. The slightly higher density of the byproduct (1.155 Mg/m³) compared with a typical bitumen density (ranging from 1.01 to 1.06 Mg/m³ [51]), could be mainly responsible for this trend.

In general, the volumetric properties of bituminous mixtures made with lignin as a partial substitute for bituminous binders are not well addressed by previous studies. Accordingly, some studies did not determine the volumetric properties [13] or use a target air void content for the design of the mixtures [18,20]. Nevertheless, Zahedi et al. [19] stated that for lignin percentages of 3% and 6%, the air void decreased when compared to the control mixture and increased by 9% and 12%, respectively. These results contrast with the results obtained by Arafat et al. [17], because these researchers, for a 6% lignin in place of bitumen, found that the air voids increase when compared to the control mixture. The different types of lignin used in both investigations could explain the contrasting results.

4.3. Water sensitivity

The first residual binder content resulted in a control mixture (0%) with a suitable air void content of 3.00%. As a result, water sensitivity analysis of the mixtures was initiated with this binder content. The results are listed in Table 7.

As previously mentioned, for the type of mixture under analysis (a total rate HWMRA-type AC 16 surf S), a TSR higher than or equal to 85% is required [28]. The only two byproduct percentages that meet this requirement, that is, the control mixture (0% byproduct) and the mixture manufactured with 5% byproduct in place of bitumen emulsion, are shaded in grey in Table 7. The TSR is very similar in both cases, although slightly higher (1.35% higher) in the case of the mixture made with a 5% byproduct than in the case of the control mixture. As previously mentioned, the low quantity of byproduct used in this case has no negative effects on the microstructure of the bitumen emulsion; however, it provides a certain amount of lignin (0.51%), which helps to improve the water resistance of the mixture.

Furthermore, it is interesting to note that both the unconditioned (ITS_D) and conditioned (ITS_W) strengths are substantially higher for the 5% byproduct mixture than for the control mixture. Specifically, the ITS_D was 13.27% higher, whereas the ITS_W was 14.76% higher. It is also interesting to note that ITS_D increases with an increase in the percentage of the lignin byproduct of up to 10%, whereas ITS_W only increases up to 5% of the lignin byproduct.

For byproduct percentages of 10%, 15%, and 20%, the TSR value is inadequate and is significantly far from the 85% required for this type of mixture. Therefore, the water sensitivity analysis was continued with the control mixture and with the mixture manufactured with the 5% byproduct, analysing two additional binder contents (3.5% and 4.0%). The results are shown in Table 8 and confirm that the use of the 5% byproduct instead of bitumen emulsion leads to mixtures with adequate water sensitivity. The TSR results were similar for the control mixture and the mixture with the 5% byproduct. For the 3.0% and 4.5% binder content, the TSR values are slightly higher for the mixture containing 5% byproduct and slightly lower for the mixture containing 4.0% binder content. For the 3.0% and 4.0% binder content, the ITS_D and ITS_W are higher for the mixture manufactured using 5% of byproduct and lower

Table 5
Apparent density (Mg/m^3) SSD for HWMRA-type AC 16 surf S manufactured using lignin-rich byproduct.

Residual under content (%)	Byproduct									
	0%		5%		10%		15%		20%	
	A (*)	S (**)	A (*)	S (**)	A (*)	S (**)	A (*)	S (**)	A (*)	S (**)
2.50	2.248	0.016	2.261	0.018	2.285	0.020	2.266	0.008	2.252	0.005
3.00	2.252	0.015	2.269	0.010	2.269	0.011	2.295	0.009	2.283	0.022
3.50	2.260	0.014	2.271	0.025	2.272	0.038	2.282	0.000	2.314	0.012
4.00	2.259	0.027	2.288	0.008	2.296	0.004	2.292	0.006	2.312	0.001
4.50	2.266	0.024	–	–	–	–	–	–	–	–

(*) A = Average.

(**) S = Standard deviation.

Table 6
Maximum density (Mg/m^3) for HWMRA-type AC 16 surf S manufactured using lignin-rich byproduct.

Residual binder content (%)	Byproduct									
	0%		5%		10%		15%		20%	
	A (*)	S (**)	A (*)	S (**)	A (*)	S (**)	A (*)	S (**)	A (*)	S (**)
2.50	2.431	0.002	2.410	0.011	2.406	0.006	2.417	0.014	2.416	0.010
3.00	2.388	0.001	2.398	0.005	2.402	0.005	2.410	0.002	2.414	0.010
3.50	2.383	0.007	2.385	0.005	2.386	0.006	2.390	0.003	2.400	0.003
4.00	2.363	0.001	2.373	0.005	2.365	0.006	2.382	0.002	2.381	0.006
4.50	2.346	0.002	–	–	–	–	–	–	–	–

(*) A = Average.

(**) S = Standard deviation.

Table 7
Water resistance results for HWMRA-type AC 16 surf S manufactured using lignin-rich byproduct and a 3.00% of binder.

Byproduct (%)	ITS_D (MPa)		ITS_W (MPa)		TSR (%)
	A (*)	S (**)	A (*)	S (**)	
0	2.027	0.096	1.802	0.076	88.90
5	2.296	0.248	2.068	0.236	90.08
10	2.410	0.125	1.389	0.104	57.62
15	1.964	0.178	1.369	0.165	69.69
20	2.157	0.239	1.387	0.278	64.32

(*) A = Average.

(**) S = Standard deviation.

for that using 4.5% of binder.

Based on the results, a 3.0% residual binder content (3.0/0.67 = 4.48% of bitumen emulsion C67B2 MCB Ecotemp) was determined to be optimum for the control mixture and also for the mixture manufactured with a 5% byproduct instead of bitumen emulsion.

Comparing the results with those obtained by other researchers, it is interesting to note that the water sensitivity of the mixture has not been thoroughly analysed in previous investigations. Consequently, Arafat et al. [17] obtained a bitumen content of 4.6% for the control mixture; however, they directly used the same bitumen content for the mixture made with 6% lignin in bitumen. That is, the lignin mixture was not designed. They found adequate moisture damage resistance for both

Table 8
Water resistance results for HWMRA-type AC 16 surf S manufactured using lignin-rich byproduct.

Byproduct	0%					5%				
	ITS_D (MPa)		ITS_W (MPa)		TSR (%)	ITS_D (MPa)		ITS_W (MPa)		TSR (%)
	A (*)	S (**)	A (*)	S (**)		A (*)	S (**)	A (*)	S (**)	
3.0	2.027	0.096	1.802	0.076	88.90	2.296	0.248	2.068	0.236	90.08
3.5	2.029	0.174	2.001	0.087	98.60	2.611	0.256	2.418	0.203	92.60
4.0	2.668	0.252	2.385	0.229	89.37	2.379	0.235	2.176	0.237	91.50

(*) A = Average.

(**) S = Standard deviation.

mixtures, but slightly lower for the mixture made with lignin. Zhang et al. [18] followed the same procedure. That is, for the control SMA, they obtained a 6.2% of optimum bitumen content and used the same bitumen content for the mixture made with 10% lignin in the bitumen. They found adequate moisture damage resistance for both mixtures, but slightly better for the mixture made with lignin. In addition, the mixture made with lignin had higher conditioned and unconditioned strengths. Moreover, the different types of lignin used as well as the different grain size distributions and manufacturing temperatures of the mixtures could explain these differences in their performance.

4.4. Resilient modulus and thermal susceptibility

Percentages of 10%, 15%, and 20% of byproduct in place of bitumen emulsion were discarded owing to their inadequate performance in terms of moisture damage resistance. Thus, the research continued with the control mixture (0%) and the mixture containing 5% of lignin-rich byproduct in place of bitumen emulsion, at the optimum binder content (3.0% of binder). Fig. 9 shows the resilient modulus for both mixtures with respect to temperature.

As seen in Fig. 9, for all the tested temperatures, the stiffness is higher for the mixture using the byproduct than for the control mixture. This result agrees with the results obtained by other researchers that studied this property [20]. Thus, in this range of environmental temperatures (0 °C to 30 °C), a better rutting performance is expected for the mixture made with the 5% byproduct. Nevertheless, the thermal susceptibility of

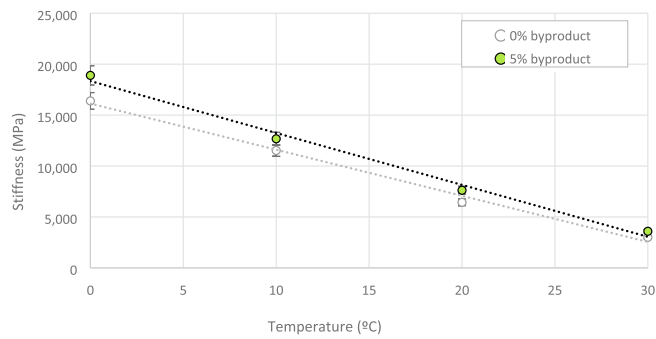


Fig. 9. Resilient modulus versus the temperature for the control mixture (0% byproduct) and mixtures manufactured using 5% of the byproduct.

the total rate of HWMRA-type AC 16 surf S made with the 5% byproduct is similar, although slightly higher (slope of the curve 4.4% higher) than that of the control mix (0% byproduct). Consequently, slightly lower performance in terms of permanent deformation at the highest in-service temperatures and fissuration at low in-service temperatures of the mixture made with 5% byproduct are expected.

4.5. Resistance to the permanent deformation

To analyse the resistance to permanent deformation, two different tests were conducted: the uniaxial confined compression test and the Hamburg wheel tracking test.

As seen in the previous section, the rutting performance of the mixture made with 5% of the byproduct compared to that of the control mixture, at least in the range of 0 °C to 30 °C was better. However, at the highest in-service temperature, an opposite trend is expected to occur. That is, the control mix behaves better than the mixture made with a 5% byproduct at the highest temperatures. To determine the temperature at which the trend reverses, tests were conducted at different temperatures. The confined uniaxial test was conducted at 40 °C and the Hamburg wheel tracking test at 60 °C.

Figure 10 shows the results of the axial strain ($\mu\epsilon$) versus the load cycles for the uniaxial confined compression test for both mixtures. As seen, at 40 °C, the mixture made with 5% byproduct still outperforms the control mixture in terms of rutting resistance. Consequently, the creep rate is 0.16 $\mu\text{m}/\text{m}/\text{load cycle}$ for the control mixture and 0.14 $\mu\text{m}/\text{m}/\text{load cycle}$ for the mixture made with 5% of byproduct in place of bitumen emulsion. The creep modulus is 29.0 MPa for the control mixture and 30.2 MPa for the mixture made with 5% of byproduct.

Figure 11 shows the rut depth (mm) versus the load cycles for the Hamburg wheel tracking test for the control mixture (0%), and the mixture made with the 5% byproduct in place of the bitumen emulsion. Table 9 shows the average slope of the curve between 5,000 and 10,000

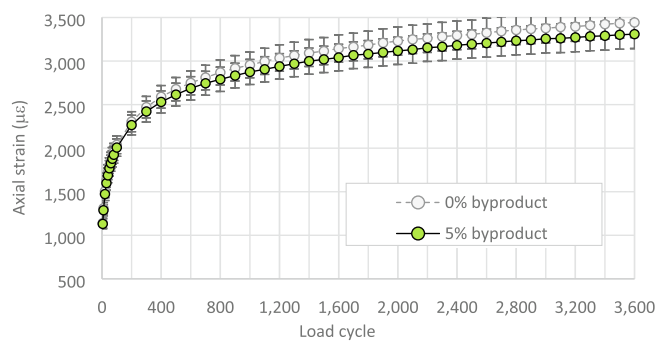


Fig. 10. Uniaxial confined compression test results (axial strain vs load cycle) for the control mixture (0% byproduct) and the mixture made with 5% byproduct.

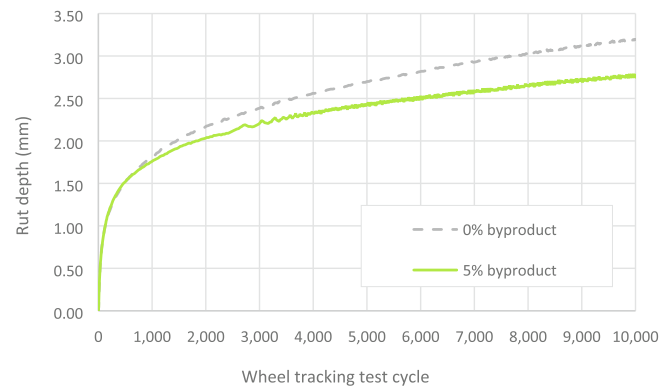


Fig. 11. Hamburg wheel tracking test results (rut depth vs load cycle) for the control mixture (0% byproduct) and the mixture made with 5% byproduct.

cycles (WTS_{AIR}), final average rut depth (RD_{AIR}), and percentage of final average rut depth (PRD_{AIR}) for both mixtures. Because of the results, and similar to the results obtained from other studies [17,18,20], it can be noticed that, at 60 °C, the mixture made with 5% byproduct still outperforms the control mixture, in terms of resistance to permanent deformation. According to the specifications (see Table 3), the control mixture is adequate for T3, T4, and shoulders, whereas the mixture made with 5% byproduct is adequate for all the heavy traffic categories in terms of rutting resistance.

According to Khan et al. [52], the in-service asphalt temperature depends on several parameters, such as wind speed, air temperature, relative humidity, and solar radiation. It has also been observed that surface colour [53] can influence the pavement temperature. For porous asphalt, Higashiyama et al. [54] conducted a series of field temperature measurements, and it can be seen that up to a temperature of approximately 35 °C in the environment, the asphalt did not exceed 60 °C.

Based on these studies and results, regions with environmental temperatures of up to 30–35 °C are expected to exhibit a better permanent deformation performance of the mixture made with a 5% byproduct than that of the control mixture.

4.6. Cohesion loss

For the control mixture (0% lignin) a Cántabro Index of 22.6% was obtained, whereas for the mixture made with 5% lignin a slightly higher index of 23.1% was obtained. Therefore, it can be concluded that the introduction of a byproduct in the mixture slightly impairs cohesion. This property was previously studied by Zhang et al. [18], who obtained the opposite result, that is, the lignin improved the cohesion loss of the mixture. However, the results are not comparable because these researchers use pure lignin and not a byproduct (which contains other components in addition to lignin), and they also investigated an HMA SMA type.

Table 9
 WTS_{AIR} , RD_{AIR} , and PRD_{AIR} for the control mixture (0% byproduct) and the mixture made with 5% byproduct.

Parameter	Byproduct			
	0%		5%	
	A (*)	S (**)	A (*)	S (**)
WTS_{AIR} (mm each 1000 load cycles)	0.10	0.014	0.07	0.035
RD_{AIR} (mm)	3.2	0.304	2.8	0.509
PRD_{AIR} (%)	5.3	0.507	4.6	0.849

(*) A = Average.

(**) S = Standard deviation.

4.7. Rheology

The penetration grade, softening point temperature, and IP for the commercial bitumen emulsion C67B2 MBC Ecotemp (0% byproduct) and the bitumen emulsion mixed with 5% byproduct, are shown in Table 10.

The C67B2 Ecotemp datasheet provided by the supplier indicates that the penetration of the residual binder of the bitumen emulsion should be lower than 100 0.1 mm and the softening point temperature should be higher than 43 °C. As shown in Table 10, both values comply with datasheet indications. Similar to other studies [19,20], for the bitumen emulsion mixed with 5% of the lignin-rich byproduct, the penetration grade decreased and the softening point increased. That is, the residual binder of the mix of bitumen emulsion and 5% of byproduct is stiffer than that of the commercial bitumen emulsion. This could explain the higher ITS values, higher stiffness, and higher cohesion loss for the HWMRA made with the 5% byproduct in place of bitumen emulsion.

In addition, as shown in Table 10, the IP for the residual binder of the commercial bitumen emulsion indicates that this bitumen is a conventional form used for road asphalt applications. The residual bitumen of the bitumen emulsion mixed with 5% byproduct shows a higher thermal susceptibility, which is in agreement with the results obtained for the thermal susceptibility of the HWMRA previously indicated in Section 4.4.

5. Savings

A 3.0% residual binder content (bitumen) was chosen as the optimum binder content for the control mixture, and 3.0% was also selected for the mixture manufactured with a 5% byproduct instead of bitumen emulsion. However, in this last case, not all is bitumen, because 5% is a lignin-rich byproduct. That is, the quantity of byproduct is $3.0 \times 5 / 100 = 0.15\%$ and the quantity of bitumen is $3.0\% - 0.15\% = 2.85\%$. That is, in the case of the mixture containing 5% byproduct, 0.15% bitumen is saved when compared to the control mixture.

For the control mixture, 3.0% of residual binder content equals $3.0 / 0.67 = 4.48\%$ of the bitumen emulsion type C67B2 MBC Ecotemp. For the mixture made with 5% of byproduct instead of bitumen emulsion, 3.0% of residual binder content equals $4.48 \times 0.95 = 4.25\%$ of bitumen emulsion type C67B2 MBC Ecotemp and $4.48 - 4.25 = 0.22\%$ of lignin-rich byproduct. Assuming a bitumen emulsion price of 528.77 €/t and considering that the byproduct is free, these bitumen emulsions lead to savings of approximately 1.18 €/t of bituminous mixtures ($0.22 / 100 \times 528.77 = 1.18$). This bitumen and economic savings lead to mixtures with slightly better water resistance and higher indirect tensile strength.

6. Conclusions

In this study, the feasibility of using a lignin-rich industrial byproduct, as a partial substitute for bitumen emulsion in HWMRA-type AC 16 surf S, manufactured with 100% RAP, was analysed. Substitution percentages of 0% (control mixture), 5%, 10%, 15%, and 20% were employed. The main conclusions drawn from this investigation are as follows:

- The use of up to 5% of industrial byproducts from the hardboard industry in place of bitumen emulsion in full-rate HWMRA-type AC 16 surf S constitutes a perfect way to promote the circular economy, sustainability, and protection of the environment. This is because this mixture is manufactured with 100% recycled aggregate at lower temperatures (<100 °C) than those used for HMA, reduces the consumption of raw materials derived from petroleum, and value waste (industrial lignin-rich byproduct).

Table 10

Penetration, ring and ball temperature, and penetration index for the commercial bitumen emulsion and the bitumen emulsion mixed with 5% byproduct.

Parameter	Byproduct			
	0%		5%	
	A (*)	S (**)	A (*)	S (**)
Penetration (0.1 mm)	91	3.7	39	1.8
Softening point (°C)	46	0.32	49	0.23
Penetration index	-0.8	-	-1.9	-

(*) A = Average.

(**) S = Standard deviation.

- The industrial byproduct can be used directly, with no form of transformation.
- An optimum residual binder content of 3.0% (4.48% of bitumen emulsion content C67B2 MBC Ecotemp) was selected for the control mixture (0% byproduct) and for the mixture made by replacing 5% of bitumen emulsion with the byproduct.
- That is, it is possible to save 0.15% bitumen (0.22% bitumen emulsion) in the manufacture of full-rate HWMRA for the surface layer of road pavements, with the replacement of 5.0% of the bituminous emulsion by an industrial residue rich in lignin at the optimum residual binder content.
- This lower emulsion consumption leads to savings of approximately 1.18 €/t of the bituminous mixture.
- The mixture made with the 5.0% byproduct at the optimum residual binder content has enhanced properties. Accordingly, it displays a slightly higher water resistance (TSR 1.35% higher) and higher indirect tensile strength resistance, both in the unconditioned state (13.27% higher) and in the conditioned state (14.76% higher).
- In addition, for regions with environmental temperatures up to 30 or 35 °C, the permanent deformation performance of the mixture made with a 5% byproduct is better than that of the control mixture. Therefore, it is foreseeable that it will suffer less damage throughout its useful life.
- The use of 5.0% byproduct leads to stiffer mixes in the range of 0 °C to 30 °C. Nevertheless, the thermal susceptibility was slightly higher for this mixture (the slope of the curve was 4.4% higher).
- In addition, the cohesion loss is slightly higher for mixtures made with 5% byproduct.
- The residual binder content of the bitumen emulsion mixed with the 5% byproduct was stiffer than the residual binder content of the commercial C67B2 MBC Ecotemp.

Despite the encouraging results, further investigation is needed, and other properties, such as fatigue performance, should be studied.

CRediT authorship contribution statement

A.R. Pasandín: Funding acquisition, Project administration, Investigation, Methodology, Conceptualization, Supervision, Writing – original draft. **E. Nardi:** Investigation, Conceptualization, Writing – original draft. **N. Pérez-Barge:** . **E. Toraldo:** Supervision, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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