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Mechanical performance of sustainable asphalt mixtures manufactured with copper slag and high percentages of reclaimed asphalt pavement

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Abstract

The use of reclaimed asphalt pavement (RAP) in the construction of hot mix asphalt (HMA) pavements is a widely used alternative in the development of sustainable road infrastructure. For this reason, the use of higher RAP contents continues to be a topic of interest. This study evaluated the performance of semi-dense asphalt mixtures incorporating 50% to 70% RAP and 7.5% and 15% copper slag (CS) as partial replacement of aggregate, to determine the influence of CS on the effective increase of RAP in the design of sustainable asphalt mixtures. The mixtures were evaluated using macrotexture, fatigue strengths, water sensitivity, and the Hamburg wheel tracking test. The results show that the use of RAP improves the permanent deformation resistance (RD) and fatigue life of the mixtures, while the mean texture depth (MTD) and indirect tensile strength ratio (ITSR) decrease. The incorporation of CS in the mixtures with RAP improved MTD and ITSR compared to the mixtures including only RAP and allowed higher RAP contents in fatigue and Hamburg wheel tests without showing a negative influence.

Keywords: Copper slag; Reclaimed Asphalt Pavement; Macrotexture; Moisture sensitivity; Fatigue, Rutting.

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1. Introduction

The progressive social awareness of the need for sustainable industrial development that launched in recent years worldwide has resulted in increasingly demanding environmental agreements and public policies on production [1]. Minimizing the exploitation of natural resources and the emission of pollutants in the manufacturing processes [2–4], extending pavement life [5,6] and the management of generated waste have been topics of interest concerning the development of road infrastructure [7].

The use of reclaimed asphalt pavement (RAP) as a replacement for a fraction of natural aggregates in the construction of hot mix asphalt (HMA) pavements is one of the most suitable alternatives to achieve a sustainable approach [8–10]. Its reuse has made it possible to reduce the

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waste generated in pavement rehabilitation processes, promote the circular economy, and show significant economic, energy, and environmental benefits [11–13]. For this reason, the use of higher RAP contents in asphalt mixtures is being highly demanded by the productive sector. However, its use is currently limited by various technical design manuals and international pavement construction standards [14–16]. In general, the results presented in these documents suggest the use of RAP in quantities lower than 30% (between 10% and 30%) in the manufacture of HMA, since in this way mixtures with similar characteristics to conventional ones without the addition of RAP are obtained. Likewise, a particular design and evaluation are recommended for each case when the mixtures contain values higher than 30% of RAP [14–16].

Among the main limitations in the standardization of high RAP mixtures is the variability in the performance demonstrated by these mixtures, due to the variability of the RAP itself and to a partial knowledge of the interaction mechanisms with virgin materials [14,17]. The effective binder content of RAP depends on the degree of oxidation of the RAP binder and the size of the RAP particles, since RAP is a material composed of aggregate particles coated by an aged binder [18,19]. Their contribution corresponds to an intermediate state between black rock (no interaction and no binder contribution) and a total diffusion in which all the aged RAP binder is mixed with the aggregates and virgin binder [18]. A review of previous research suggests a higher active binder contribution from coarse RAP particles than the shown by fine RAP particles, revealing the importance of controlling RAP gradation in the design of recycled mixtures [20–22]. In this sense, the literature agrees that neither of the two gradations used in the design of recycled mixtures are representative of reality, the black curve corresponding to the disaggregated RAP particles nor the white curve corresponding to the RAP aggregate recovered after binder extraction. This results indicate that variables related to the source of origin of the RAP are more influential, such as the depth at which the mixture was obtained, the original properties and environmental and traffic conditions to which the RAP source was subjected during its service stage [17,21,23].

Researchers have studied the incorporation of different types of materials and design methodologies to avoid an excessive increase in the stiffness modulus, which is a characteristic of mixtures with high RAP content that can affect long-term performance, turning them into a brittle material susceptible to the appearance and propagation of cracks. The use of rejuvenating additives (oils and waxes) [24,25], polymer modified binders [26,27] or lower grade PG (stiffness compensation) [10], the use of warm mixtures (reduction of short-term aging of RAP) [28,29] and the use of stabilizing additives (slag, fibers, and minerals) [30–35] are some of them. On the one hand, these techniques make it possible to counteract the aging of RAP by controlling its viscoelasticity and, on the other hand, to stabilize the behavior of RAP mixtures against the phenomena of loss of adhesiveness due to moisture conditions, fatigue damage, and permanent deformation, the main causes of deterioration of this type of mixtures [36]. In the particular case of slags, they have attracted prominent interest in the area of civil engineering due to its potential use as an aggregate and its limited reuse at present [37,38].

Copper slag (CS) is a by-product obtained from the pyrometallurgical production of copper, and its use could minimize the stiffening effect of RAP in HMAs while helping to reduce the environmental impact associated with its accumulation and lixiviation [38,39]. About 2.5 tons of slag are generated for every ton of copper produced, reaching a production of about 24.8 million tons of CS per year worldwide [38,40]. Although CS was approved as a conventional aggregate in HMA in the United States in the 1990s [41], its use has been limited [42,43]. Likewise, previous research on the use of CS in HMAs has been generally limited to its use as a replacement for fine aggregate and filler without the presence of other non-conventional materials [39,44], dismissing the potential of larger particle sizes.

CS exhibits several desirable characteristics in HMA aggregates, including high hardness, stability and wear resistance, high friction angle and many fracture faces, and a surface with a higher amount and pronunciation of voids, cavities, and pores than the surface of conventional aggregates [44,45]. These properties vary mainly depending on the type of CS used (granulated or cooled) related to the cooling rate (in water or air, respectively). The CS presents an intermediate degree of alkalinity due to its chemical composition, with the presence of ferrous (Fe 20-40%), silicon oxide (SiO_2 35-50%), alumina (Al_2O_3 0-10%), calcium oxide (CaO 0-25%) and copper oxide (CuO 0.5-2.1%) among others, with a slight tendency to acidity due to the marked presence of SiO_2 [38,46]. This characteristic gives it a certain degree of hydrophilicity compared to the binder [39,44]. However, the high porosity and angularity gives it a better adhesion and interaction with the new and aged binder on the RAP surface, increasing the adhesiveness, the optimum binder content (OBC), and consequently, more elastic behavior of the mixtures with RAP [42,44,47,48].

Despite the existing components in CS and the presence of different types of oxides, previous studies have shown low levels of leaching associated with the use of CS being classified as a special non-hazardous material [45,49]. These low levels of leaching shown by CS increase when it is analyzed in acidic environments with increasing concentrations of Copper and Zinc, always under the maximum permitted levels [50,51]. It has also been shown that any possible leaching risk is considerably reduced in bituminous applications due to the binder film that protects them from direct contact with moisture [39]. Finally, tests carried out on CS against alkali-silica reactions, resistance to sulfate attack, and moisture saturation conditions have allowed them to be categorized as "harmless" aggregate and not producing harmful expansions in cement mortars, indicating the low presence of reactive silicates [52,53].

Accordingly, this research evaluated the mechanical and functional performance of HMA incorporating up to 70% RAP and 15% CS to replace the natural aggregate. These mixtures were compared with the performance shown by a conventional mixture (0% RAP and 0% CS) without the addition of these recovery by-products.

2. Objective and scope

The objectives of this study are as follows:

- To evaluate the mechanical and functional performance of semi-dense type hot mix asphalt mixtures incorporating different contents of RAP (0%, 50%, 60% and 70%) and CS (0%, 7.5% and 15%).
- To determine the influence of CS on the effective increase of RAP in recycled asphalt mixtures by statistical analysis of performance results.

The percentages of CS used are due to recommendations made in previous studies on the maximum replacement percentage by volume of the combination of both materials in HMAs [47,48].

The designation used in the study for the mixture corresponds to the percentage of RAP included, followed by the percentage of CS incorporated, thus, the mixture M50/7.5 corresponds to a sample incorporating 50% RAP and 7.5% CS. The total combinations used are detailed in section 3.2.

The tests performed correspond to moisture sensitivity tests using the indirect tensile strength ratio (ITS and ITSR), rutting resistance using the rutting test (HWTT), initial stiffness (IS) and fatigue resistance using the four-point bending test (4PBT), and evaluation of the macrotexture using the sand patch test (SPT). Once the mechanical and functional results were obtained, a statistical analysis was performed to determine the usefulness of CS in the performance of the mixtures with high RAP contents. Fig. 1 shows a scheme of the study performed.

The difference observed in the aged binder content of the coarse and fine RAP in Table 1 is mainly due to the RAP stockpiling conditions (no separation and homogenization of the RAP) and the higher binder content observed in the fine RAP fractions [20,21] due to their higher surface area per unit weight and the formation of coarser RAP milling aggregates predominantly consisting of coalescence of fine RAP particles [22,54].

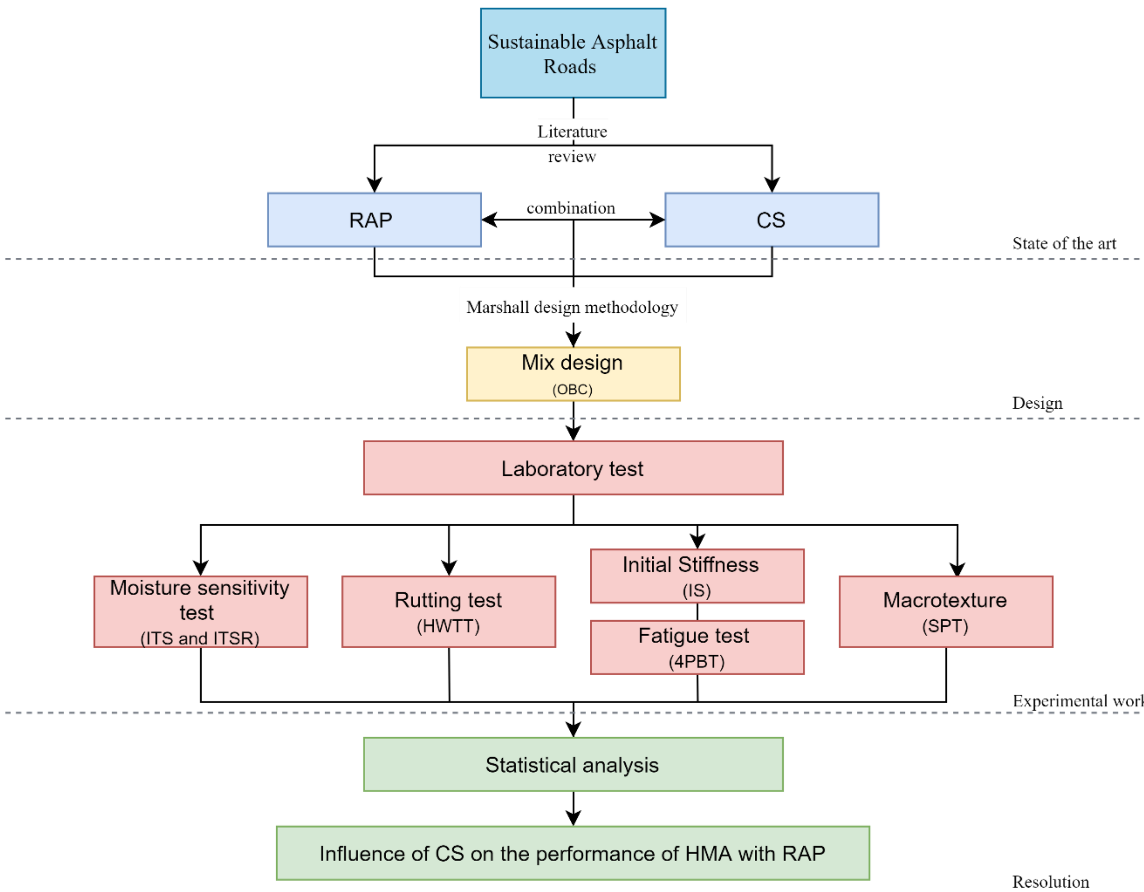

Fig. 1. Scheme of the study

3. Materials and mix design

3.1. Materials

The binder used in this study corresponds to a conventional CA-24 binder classified according to its absolute viscosity at 60°C according to Chilean technical standards. The conventional aggregate (AG) and reclaimed asphalt pavement (RAP, Fig. 2a) came from an HMA production plant, and the pavement removal and rehabilitation processes were carried out by it in southern Chile. The copper slag (CS, Fig. 2b) was obtained from a copper extraction mine in central Chile. Here, CS is deposited in dumps and air-cooled (refrigerated), resulting in slag with glassy characteristics and low water absorption. The physical properties of the binder and aggregates used are shown in Table 1.

Table 1
Properties of materials used.

Properties	Standards	Result
Bitumen CA-24		
Viscosity at 60°C, 40000 Pa (Pa·s)	EN 12596	303.9
Penetration at 25°C, 100 g. 5 s. (0.1 mm)	EN 1424	59
Softening Point R&B (°C)	EN 1427	52.6
Ductility 25°C (cm)	EN 13589	>150
Density (gr/cm^3)	EN 15326	1.035
aggregates course / fine		
Density (g/cm^3)	EN 1097-6	2.812 / 2.750
Water absorption (%)	EN 1097-6	1.22 / 1.01
Los Angeles (%)	EN 1097-2	15.54 / -
Crushed particles (%)	EN 933-5	89.21 / -
RAP course / fine		
Density (g/cm^3)	EN 1097-6	2.782 / 2.767
Water absorption (%)	EN 1097-6	1.15 / 0.99
Bitumen content aged (% by weight of the sample)	EN 12697-39	4.2 / 9.1
CS course / fine		
Density (g/cm^3)	EN 1097-6	2.971 / 2.936
Water absorption (%)	EN 1097-6	0.90 / 0.61
Los Angeles (%)	EN 1097-2	12.64 / -
Crushed particles (%)	EN 933-5	100 / -

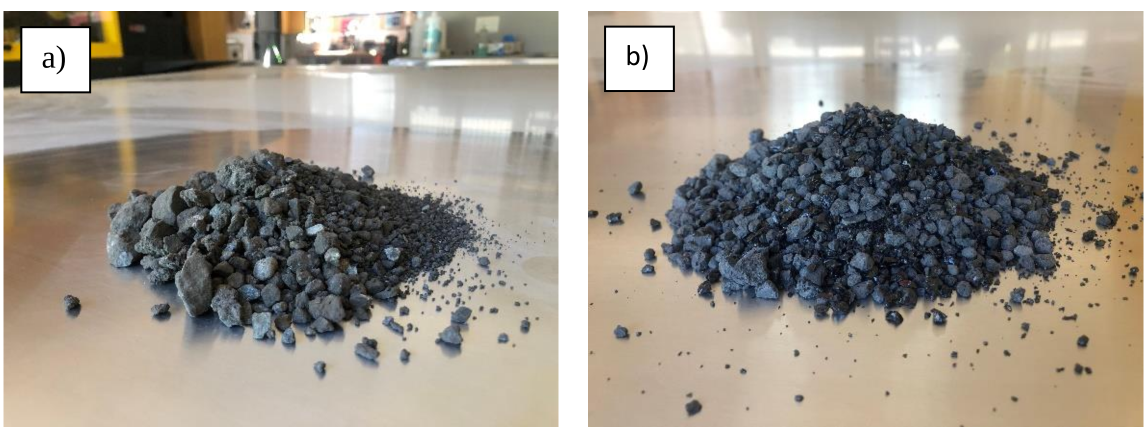

Fig. 2. By-products used in this study: (a) recycled asphalt pavement (RAP); (b) Copper slag (CS).

3.2. Design and samples preparation

In this study, a total of 12 different asphalt mixtures were designed, combining the different RAP and CS contents selected. The gradation of the aggregates (RAP, CS, and AG) was combined to obtain a semi-dense gradation curve type IV-A-12 according to the Chilean Highway Manual used as wearing and binder courses. Fig. 3 shows the gradation of the designed mixtures and the RAP and CS aggregates used. The RAP gradation shown in the figure corresponds to the black curve, due to the low presence of fines in the CS. The design of the IV-A-12 mixtures and the determination of the optimum binder content (OBC) were carried out following the Marshall design methodology and the requirements of Chilean regulations. According to the Marshall design and the OBC, a mean void content of 5.5 ± 0.2% was obtained for all the mixtures designed. Table 2 shows the mixtures designed in this study and their respective combinations of RAP, CS, and design parameters.

A total of 288 test samples were prepared. Before the manufacturing and mixture process, the AG and CS aggregates were heated at 175°C for 6h, while the RAP was heated for 2h at 90°C to avoid over-aging of the binder included in RAP and to achieve softening of the RAP. The virgin binder is heated for 2 hours at 155°C and mixed for a maximum of 5 minutes with previously homogenized aggregates. Once the previous stage is completed, the mixtures are compacted according to the type of sample and test sample. The cylindrical samples for the moisture sensitivity tests (ITS, ITSR) and obtaining the OBC were compacted using an impact compactor applying 50 blows and 75 blows per side, respectively, according to EN 12697-30. Prismatic samples (plates) for the HWTT and SPT tests, and plates for the 4PBT and IS tests were compacted by a plate compactor controlling the compaction energy according to EN 12697-33. The plates for the 4PBT were sawn after curing (storage for 15 days at 20°C) to form beam samples with dimensions 380x50x50 mm, according to EN 12697-24.

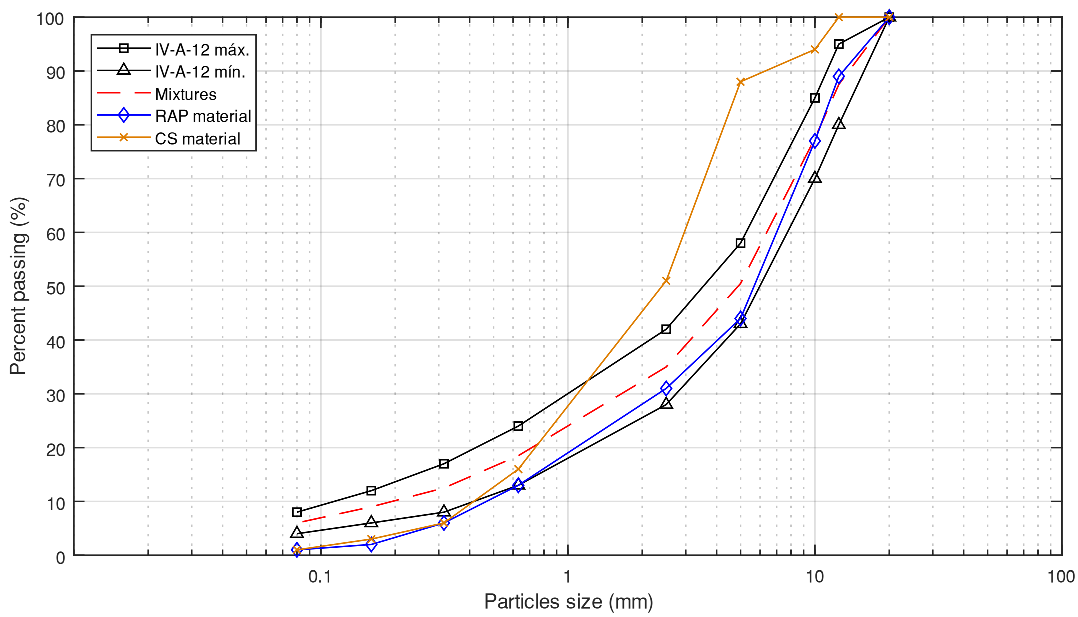

Fig. 3. Design curve for IV-A-12 mixtures and grading curves of the RAP and CS aggregates.

Table 2
Mixtures and design parameters.

Mixture	Content (%)			Bulk Density	
	RAP*	CS*	Bitumen (OBC)**	(g/cm^3)	Standard deviations
M0/0 (ref.)	0	0	5.25	2.288	0.009
M50/0	50	0	5.28	2.342	0.006

M60/0	60	0	5.51	2.373	0.001
M70/0	70	0	5.24	2.372	0.001
M0/7.5	0	7.5	5.09	2.386	0.010
M50/7.5	50	7.5	5.49	2.481	0.001
M60/7.5	60	7.5	5.61	2.470	0.003
M70/7.5	70	7.5	5.60	2.476	0.003
M0/15	0	15	5.82	2.363	0.008
M50/15	50	15	5.18	2.417	0.009
M60/15	60	15	5.16	2.416	0.007
M70/15	70	15	5.22	2.407	0.003

* % by volume of total aggregates

** % by weight of mix

4. Test methods

4.1. Macrotexture

The volumetric or sand patch method (SPT) was used to evaluate the macrotexture of the sample surface according to EN 13036-1. In this study, 25000 mm^3 of sand type 80/200 spread over the sample surface in a circular shape was used. The area of the circle formed in each sample is obtained from the mean value of the measurement of 5 different regularly spaced diametric lines. The mean texture depth (MTD) is obtained from the ratio between the volume of sand used and the area of the circle formed (Eq. (1)).

$$MTD = \frac{4\,V}{\pi\,D^2} \quad (1)$$

where V is the volume of sand used (mm^3) and D is the average diameter of the circle formed (mm).

4.2. Fatigue tests and initial stiffness

The four-point bending test (4PBT) was used to determine the fatigue resistance of the designed samples under repeated application of a sinusoidal load (without rest periods) according to EN 12697-24, Annex D. In this study, deflection control (constant deformation) was adopted for the load application mode at a temperature of 20°C and a frequency of 30 Hz. The applied deflection levels correspond to 300, 400 and 500 μm/m measured at the center of the beam. Before each test, the prismatic samples were kept for 24 h in a ventilated thermostatic chamber at the test temperature (20°C). The phenomenological approach failure criterion ($N_{f/50}$) was used to determine the fatigue life of the samples, based on the number of load cycles (N_f) in which a 50% reduction of the initial stiffness or a complete fracture of the sample takes place [26,55,56]. The fatigue curve is obtained from analytical interpolation of the data obtained using the power function shown in Eq. (2).

$$\varepsilon_i = a\left(N_{i,f/50}\right)^b \quad (2)$$

where ε_i is the value of the initial strain (μm/m), $N_{i,f/50}$ is the number of loading cycles or repetitions of sample up to the failure criterion *(f/50)* and *a, b* interpolation coefficients related to the mixture type [10,57].

The initial stiffness (IS) of the samples calculated in this study corresponds to the dynamic flexural stiffness modulus (complex) in cycle 100 of the fatigue test (4PBT) according to EN 12697-24, Annex D. According to previous studies, at this stage of the test the HMA samples still do not show significant changes in their original properties so they can be considered undamaged and in their initial state [27,58]. The initial dynamic flexural modulus of stiffness $S_{mix,0}$ is obtained from Eq. (3).

$$S_{mix,0} = \frac{\sigma_{100}}{\varepsilon_{100}} \tag{3}$$

where σ_{100} y ε_{100} correspond to the maximum cyclic amplitude of the stress (Pa) and strain (μm/m) function at cycle 100, respectively.

4.3. Moisture sensitivity test

The indirect tensile strength ratio (ITSR) according to EN 12697-12, (Method A) was used to determine the moisture sensitivity of the mixtures designed in this study. This method uses the calculation of the indirect tensile strength (ITS) of dry and wet cylindrical samples according to EN 12697-23.

The procedure consists of selecting 6 cylindrical samples for each type of mixture prepared according to section 3.2 and forming two subgroups of 3 samples each with equivalent physical characteristics. The subgroup used to calculate the ITS under dry conditions is kept in the air at a temperature of 20 ± 5°C, while the subgroup used to calculate the ITS under wet conditions is subject to saturation and vacuum attrition in distilled water for 30 ± 5 minutes and then kept immersed in a water bath at 40°C for 68 h to 72 h. The ITS test is carried out at 15°C according to Spanish regulations for road and bridge works (PG-3), so the samples were previously conditioned at this temperature in their different media (dry and wet). The indirect tensile strength ratio (ITSR) is obtained from Eq. (4) and Eq. (5).

$$ITS = \frac{2P}{\pi\, D\, H} \tag{4}$$

$$ITSR = 100 \times \frac{ITS_w}{ITS_d} \tag{5}$$

where *ITS* is the indirect tensile strength (kPa), *P* is the maximum applied load (kN), *D* is the sample diameter (mm), *H* is the sample height (mm), ITS_w is the indirect tensile strength of the wet sample (kPa) and ITS_D is the indirect tensile strength of the dry sample (kPa).

4.4. Rutting test

Rutting resistance was determined by the Hamburg wheel tracking test (HWTT) according to AASHTO T324. This method helps to determine the susceptibility to viscoplastic deformation of HMA immersed in water at controlled temperature (50 ± 0.5°C) through the measurement of the depth of the tread formed by repeated passes of a steel wheel loaded with 705 N at a speed of 52 passes/min. The samples were kept for 30 minutes in a 50°C water bath before starting the test.

The test ends at 10000 cycles (20,000 passes) or when a permanent vertical deformation of 20 mm has been reached on the sample surface. The mean rut depth (RD) is obtained after testing and recording the vertical deformation of 2 samples for each type of asphalt mixture designed.

This test also helps to determine signs of a loss of adhesiveness (stripping) due to the effects of water [20]. This phenomenon is observed (if available) by an increase in the slope of the deformation curve [59].

The strain rate calculation (WTS_w) was also calculated, corresponding to the slope of the strain curve in its creep stage (between cycles 5000 and 10000 if stripping is not available), in addition to the percentage of average tread depth (PRD_w) calculated as the ratio between the tread depth and the average thickness of the sample.

4.5. Statistical analysis

Statistical analysis was used to determine scientifically the significance between the results obtained and to avoid analytical errors given the dispersion of the experimental data. The null hypotheses of normality and homoscedasticity were tested separately for all the data obtained using the Shapiro-Wilk test and the Levene test, respectively, to determine the types of statistical tests to be used. In this study, a significance level of 95% was considered for all tests in the hypothesis testing.

The parametric test used for data with a normal distribution was the one-way ANOVA and the Fisher-Snedecor F statistic. In the post hoc analysis, Turkey tests and the Games-Howell test for equality of variances were used. The Kruskal-Wallis H test and Mann-Whitney U test were used for data that did not follow a normal distribution.

5. Results and discussion

5.1. Macrotexture

Fig. 4 shows the SPT test results as a measure of the surface macrotexture of the mixtures and the optimum binder content (OBC) of each mixture. All the designed mixtures showed a significant reduction ($p\text{-value} < 0.05$) in mean texture depth (MTD) compared to the reference mixture (M0/0). The addition of high RAP contents as the sole aggregate replacement material shows a greater negative effect on the reduction of macrotexture than the shown by mixtures containing only CS. Reductions of up to 47.7% in the MTD are observed when 70% RAP is used compared to the M0/0 mixture. These results are in agreement with findings presented in previous studies that point to a decrease in skid resistance with increasing RAP content due to increased wear and polishing of RAP compared to natural aggregate [60]. The mixtures containing only CS as an aggregate replacement by-product presented reductions of 28.3% in the MTD when 15% CS was added and 9.36% when 7.5% CS was used, outlining a slight improvement in the results compared to the high RAP mixtures, but always below the reference mixture. This slight improvement in results compared to RAP and decrease compared to the reference mixture may be due to the combined presence in CS of particles with higher angularity and roughness than RAP and the presence of particles with vitreous characteristics, especially in sizes below 4 mm (see Fig. 2), not available in mixtures made with natural aggregates only [14,38].

The addition of CS in the mixtures with RAP and the combined effect of both materials shows mixed results with increases and decreases in macrotexture. The mixture M70/7.5 generates a significant increase ($p\text{-value} = 0.000$) in the MTD value of 27.0% (with respect to M70/0), while the use of 15% CS caused a significant decrease in the macrotexture (p-value = 0.000) in the mixtures with 60% RAP. The mixtures M70/0, M60/7.5, M60/15, and M70/15 would not comply with the minimum values of macrotexture for newly manufactured mixtures corresponding to 0.6 mm according to Chilean regulations (in reference to the mean profile depth or MDP). However, all the mixtures would meet the "acceptable" values proposed by some researchers and the "alert threshold" of some regulations corresponding to an average texture depth of 0.4 to 1.6 mm [61].

According to the results obtained, it was not possible to visualize a direct influence of the OBC (Fig. 4.) nor of the void content on the values of the mean depth of texture, contributing with the results obtained in previous studies that point out that only the assessment of these factors is insufficient to characterize the surface properties of friction and macrotexture in asphalt mixtures [62,63].

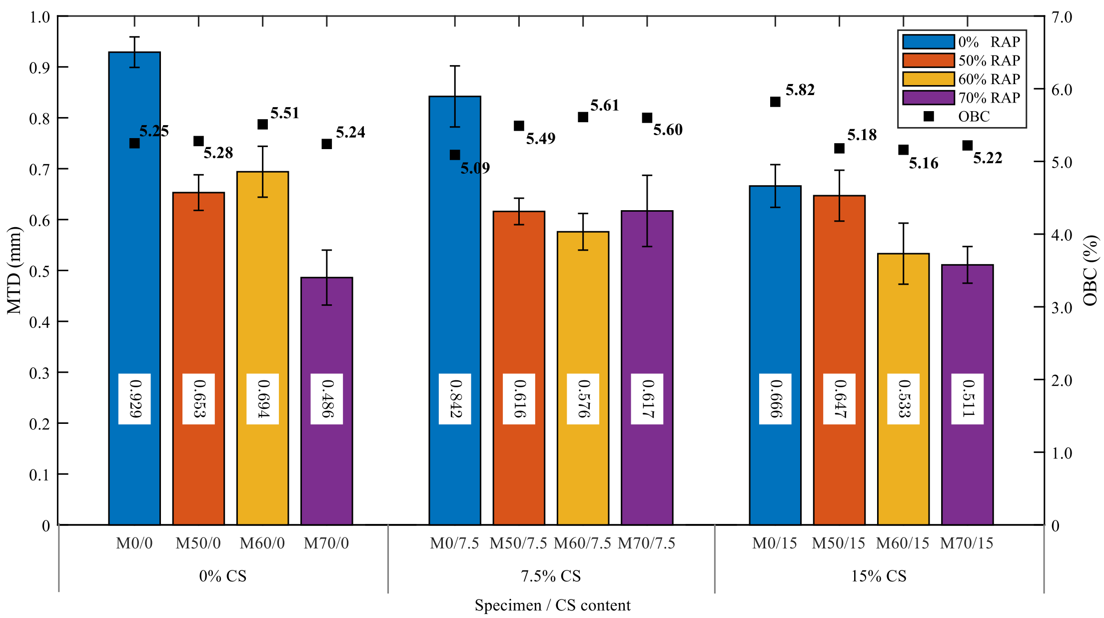

Fig. 4. Mean depth of surface texture and OBC for mixtures with error bar of +/-2 standard deviations (+/-2 σ) in MTD.

5.2. Fatigue tests and initial stiffness

The results of the initial stiffness and initial phase angle test for all the mixtures are shown in Fig. 5. Although there is no clear trend between the initial stiffness (IS) values and applied microstrain level, all the mixtures containing RAP present IS values higher than the M0/0 mixture at all the microstrain levels studied. This is due to the incorporation of an active fraction of aged binder (coming from RAP) in the optimum binder content (OBC) that modifies the viscoelastic properties of the virgin binder, stiffening the mixture [21]. An initial increase in the dynamic stiffness modulus (IS) is observed for all microstrain levels with the addition of 7.5% CS followed by a reduction of the modulus when 15% CS is added when only CS is used to replace the AG in the mixture, reaching similar stiffness values to the reference mixture. This increase and decrease of the stiffness modulus as the CS content increases was already obtained in previous studies by Ziari et al. [44], assigning this behavior to the higher angularity of the CS compared to the natural aggregate and the interlocking that it generates inside the mixture.

Regarding the results of the initial phase angle recorded in this study due to its direct relationship with the dynamic stiffness modulus and its elastic and viscous components, it can be seen in the mixtures with RAP and CS (grouped by CS content) a trend exists similar to that observed in the mixtures without RAP and with increasing addition of CS in the value of the initial stiffness, with a peak of this at the 7.5% CS content analyzed in the previous paragraph. In this case, the initial phase angle in the mixtures with 0% CS and 15% CS tends to decrease with the increase of the RAP content, which agrees with previous studies outlining a greater elastic component [64], while the initial phase angle in the mixtures with 7.5% CS tends to grow increasing its viscous component. This behavior is obtained because the OBC in the mixtures with 7.5% CS increased with the RAP content, adding a greater amount of virgin binder. This trend is reflected in the fit lines drawn between the initial phase angle values in Fig. 5.

The results of the fatigue life of the mixtures under the stiffness reduction and deformation control criteria are presented in Fig. 6. Table 3 shows the regression (*a* and *b*) and determination coefficients (R^2) calculated from the fatigue life shown by each mixture, as well as the deflection level (ε) that ensures a fatigue life $N_{f/50}$ of 10^6 loading cycles. These parameters help to determine the performance of each of the studied mixtures under repeated loading cycles and to compare them with eah other.

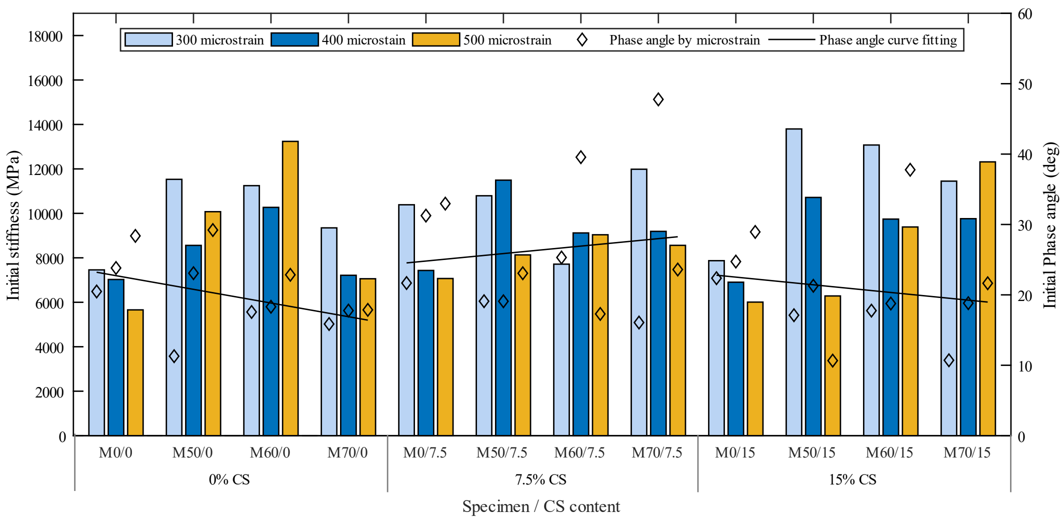

Fig. 5. Results of initial stiffness modulus and phase angle at 20°C in 4PBT test with deflection control by mixture type.

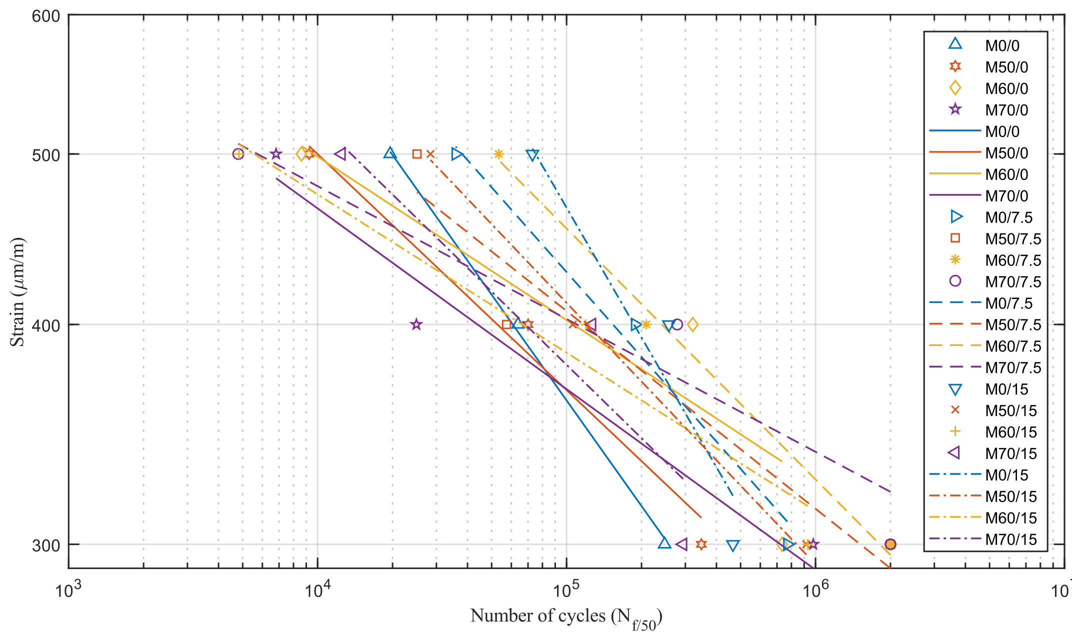

Fig. 6. Fatigue lifeline by 4BPT test at 20°C, controlled deformation and $N_{f/50}$ failure criteria.

Table 3
Fatigue curve regression coefficients and deflection level for 10^6 cycles.

Mixture	*a* (μm/m)	*b* (-)	ε (10^6)	R^2
M0/0 (ref.)	3601.0	-0.1995	229	0.9978
M50/0	1719.0	-0.1341	270	0.9637
M60/0	1172.0	-0.0929	325	0.7274
M70/0	1199.0	-0.1027	290	0.9098
M0/7.5	2703.0	-0.1600	296	0.9730
M50/7.5	1485.0	-0.1124	314	0.8207
M60/7.5	2342.0	-0.1426	327	0.9898
M70/7.5	960.9	-0.0755	339	0.8768
M0/15	7824.0	-0.2450	265	0.8697
M50/15	2277.0	-0.1486	292	0.9905
M60/15	1086.0	-0.0899	314	0.9297
M70/15	1869.0	-0.1385	276	0.8169

As predicted according to the literature review [20,27,65], all the mixtures manufactured with RAP showed the ability to withstand a higher level of deflection ε (for 10^6 load cycles) than the reference mixture M0/0 and a decrease in the slope (-b) of the fatigue curve, independent of the RAP content incorporated (Table 3). Several authors agree that this behavior may be due to a multilayer system formed by the added virgin binder and the aged binder of the RAP, which would serve to reduce the stress concentration inside the mixture and improve the fatigue life [19,20]. As for the mixtures incorporating only CS, the fatigue life curves have a more accentuated shift towards higher values of load cycles than the shown by the mixtures incorporating only RAP. This shift towards higher values of $N_{f/50}$, is accompanied by steeper slopes, decreasing the difference between the fatigue life at the microstrain levels studied, which is in agreement with the results described in previous studies [39]. The incorporation of 7.5% CS in the mixtures with RAP (combined effect of both materials) has a positive influence on the fatigue life performance of the semi-dense mixtures, showing in all cases higher deformation stress values (associated with ε) than the mixtures that only incorporate RAP independently. An increase of up to 16.9% can be observed in the level of deflection of the M70/0 mixture when 7.5% CS (M70/7.5) is incorporated. This effect is no longer noticeable when 15% CS is added to the mixtures with RAP, showing increases and decreases of ε. On the other hand, the mixture that showed the best performance in the fatigue strength test is the mixture M70/7.5 (R^2= 0.8768) with an increase in the deflection level (ε) of 48.03% concerning the reference mixture, followed by the mixture M60/7.5 (R^2= 0.9898) with an increase of 42.8% concerning M0/0. It is important to consider in the comparison of these mixtures with better performance (M60/7.5 and M70/7.5) not only the value of the deflection level ε to reach 10^6 load cycles, but also the value of the slope (-*b*) shown by each of them. In this case, the slope of the mixture with better performance (M70/7.5) is almost half of the slope shown by the mixture M60/7.5 (Table3), so although it shows better values of deflection level for 10^6 loading cycles, its performance for increasing values of initial deformation (*e.g.*,500 μm/m) will be lower, showing the latter (M60/7.5) a more stable behavior at different microdeformations.

5.3. Indirect tensile strength and moisture sensitivity test

Fig. 7 shows the indirect tensile strength ratio (ITSR) of wet and dry samples and the average individual value of each of the mixtures in the ITS tests under dry (ITS_d) and wet (ITS_w) conditions, in addition to the minimum values required by European standards for the ITSR corresponding to 80% for base and intermediate layers and 85% for wearing courses. All mixtures incorporating RAP as the sole aggregate replacement material showed a significant increase (p-value < 0.05) in indirect tensile strength in dry conditions concerning the reference mixture, with a rate of improvement of up to 48.2% for the M70/0 mixture. Although the ITS_d value increases when increasing the RAP content, no significant differences are observed between the mixtures with 50%, 60%, and 70% RAP (M50/0, M60/0, and M70/0) with p-value > 0.07 and 95% significance level. As for the results under moisture conditions, the same trend as in dry conditions is obtained, with increasing values of ITS_w when increasing the RAP content. However, this rate of improvement is lower than the observed in the previous group, obtaining non-significant statistical differences between the M0/0 mixtures and the M50/0 and M60/0 mixtures. This decrease in the rate of improvement of ITS_w concerning the observed in the dry condition for increasing values of RAP means that the mixtures with 50% and 60% RAP (M50/0 and M60/0 respectively) despite their better results, do not meet the minimum value required for the ITSR strength ratio of 80%, revealing the damage caused by water in the mixtures with RAP. This improvement of the mixtures with RAP in the individual ITS tests (dry and wet) and decrease in the ITSR value is due to an increase in the stiffness of the active binder in the mixture, due to the aged binder of the RAP [47] which is accentuated by the presence of water.

The mixtures incorporating only CS presented slightly higher ITS_d values than the reference mixture; however, this increase is not significant (p-value > 0.05). Concerning the test under wet conditions, the mixtures with CS presented a significant reduction in ITS_w compared to the M0/0 mixture with p-value =0.005 and p-value =0.001 for the M0/7.5 and M0/15 mixtures, respectively. This behavior is due to the CS having a higher affinity for water than for the binder given their hydrophilic nature [39,44] and a slight tendency to acidity (reported in the literature). This negative effect of CS is even higher than that shown by the mixtures with RAP obtaining the lowest ITSR values of the study (ITSR=70.4% for M0/7.5 and ITS=68.2% for M0/15).

The combined effect of RAP and CS improved (in almost all cases) the performance of the mixtures in the ITS tests (in particular for ITS_w), compared to those incorporating only these materials individually. The inclusion of 7.5% and 15% CS in the mixtures with 50% RAP shows a significant positive influence on the ITS_w value, increasing its value up to 19.9% for the mixture with 15% CS (M50/15). This positive effect of the interaction of both materials is further evidenced in the ITSR evaluation, where the mixture M50/0 goes from an ITSR of 72.9% (< ITSR 80%) to an ITSR of 85.7% with the inclusion of 7.5% CS (M50/7.5) and to an ITSR of 85.8% when 15% CS is added, mixtures that can be used as wearing course according to European standards. A similar case occurs for the mixtures with 60% RAP (M60/0), where the inclusion of CS improves the ITS_w values and allows an increase in the ITSR value of 7.7% with 7.5% CS reaching an ITSR value of 80%. The incorporation of CS reduced the ITS values in both conditions (dry and wet) in the mixtures with 70% RAP (M70/0). However, despite the significant reduction (p-value < 0.05) shown by the inclusion of CS, the ITSR value in both mixtures (M70/7.5 and M70/15) remained with values above 80%, with a peak of 87.6% ITSR in the M70/7.5 mixture.

Complementarily to the improvement observed above with the inclusion of CS in mixtures with RAP, a stabilizing effect of CS in the ITS_d and ITS_w tests is observed when analyzing independently the groups of mixtures with contents of 7.5% CS and 15% CS and the inclusion of increasing percentages of RAP (Fig. 7). In these groups, there are no significant differences (p-value > 0.117) between the mixtures with 50%, 60%, and 70% RAP. This stabilizing and enhancing effect of CS in the mixtures with RAP may be due to a good mechanical affinity between CS and the aged RAP binder due to the higher presence of pores that CS possesses [44], particularly in the particle sizes larger than 8 mm.

It should be noted that despite the good affinity shown by RAP and CS, all the mixtures designed in this study incorporating RAP and/or CS have lower ITSR values than the reference M0/0 mixture (ITSR=94.8%). However, practically all the mixtures (except for the M0/7.5 and M0/15 mixtures in wet condition) obtained values in the individual ITS_d and ITS_w tests higher than the M0/0 mixture, so it seems important to consider not only the ITSR values but also the performance of ITS in their individual tests.

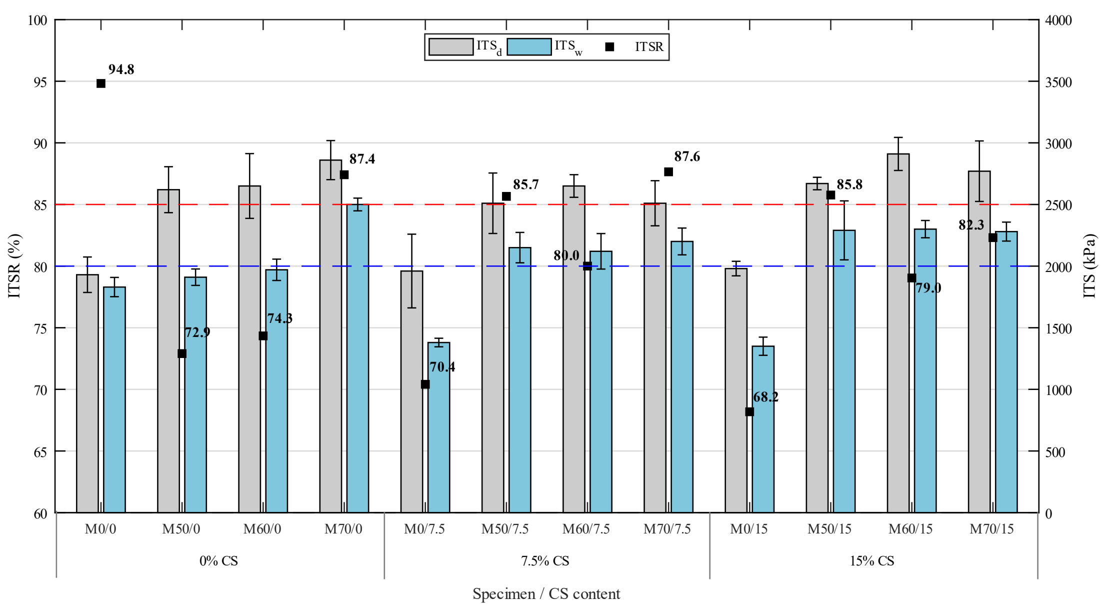

Fig. 7. Indirect tensile strength and moisture sensitivity test results with an error bar of +/- 2 standard deviations (+/-2 σ) about the mean.

5.4. Rutting test and deformation rate

The results on the mean rut depth (RD) of the HWTT test are shown in Fig. 8. Table 4 lists the strain rate (WTS_w), percent mean rut depth (PRD_w) and mean rut depth (RD) parameters obtained in the HWTT test under saturated conditions in the water at 50°C temperature.

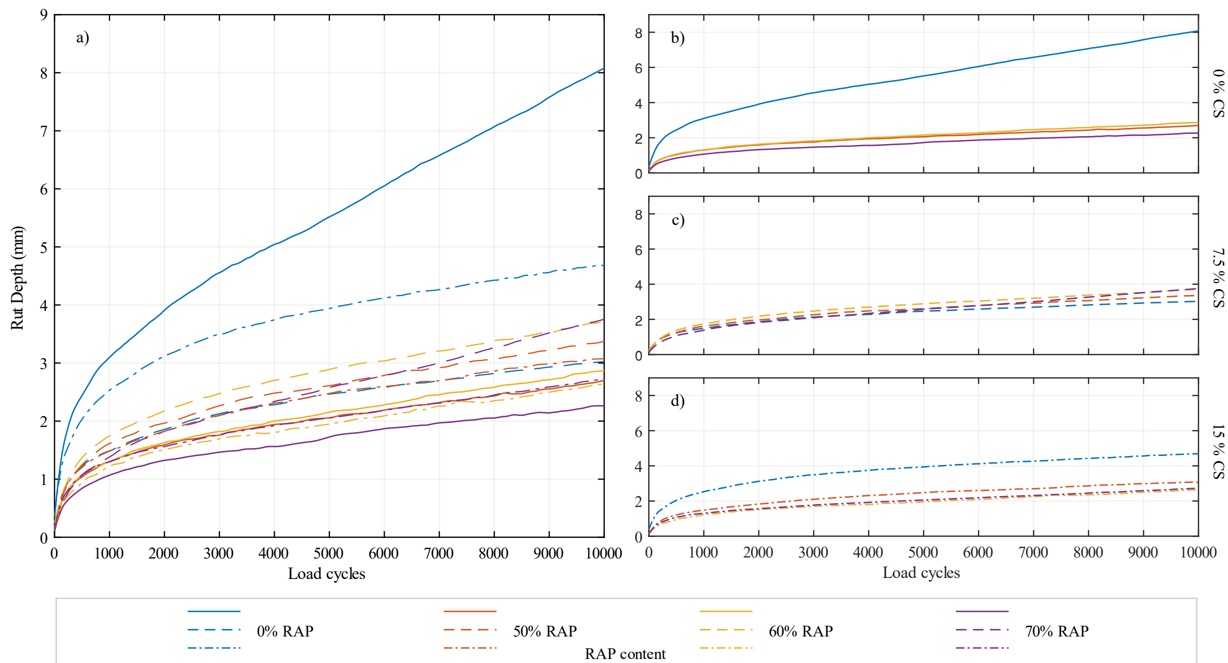

Fig. 8. Rut depth v/s load cycles: (a) All mixtures; (b) Mixtures with 0% CS; (c) Mixtures with 7.5% CS; (d) Mixtures with 15% CS.

Table 4
Parameters of the Hamburg wheel tracking test (HWTT) at 50°C, 705 N and speed of 52 passes/min.

Mixture	WTS_w ($mm/10^3$)	PRD_w (%)	Ruth Depth (RD) (mm)
M0/0 (ref.)	0.504	13.56	8.076
M50/0	0.122	4.410	2.699
M60/0	0.147	4.686	2.901
M70/0	0.102	3.624	2.291
M0/7.5	0.112	4.996	3.014
M50/7.5	0.141	5.480	3.385
M60/7.5	0.162	6.140	3.709
M70/7.5	0.239	5.912	3.729
M0/15	0.135	7.933	4.615
M50/15	0.123	5.058	3.071
M60/15	0.137	4.334	2.659
M70/15	0.132	4.399	2.738

The reference mixture M0/0 is the mixture that presented the highest WTS_w and PRD_w parameters and the highest levels of vertical deformation in the Hamburg test, with a significantly higher RD rut depth (p-value < 0.05) than the shown by the rest of the mixtures manufactured in this study. On the other hand, the mixture M0/0 shows a steeper slope in its curve between the beginning of the test (cycle 0) and cycle 1000 (see Fig. 8a), showing a higher degree of post-compaction. This may be due to the greater initial flexibility shown by the M0/0 mixture (see Fig. 5) given by its virgin binder and a lower angularity of its aggregates.

The inclusion of RAP in the mixture (Fig. 8b) significantly decreases (p-value < 0.05) the value of rut depth and strain rate compared to the M0/0 mixture, independent of the incorporated RAP content, reaching values of up to 71.6% improvement when 70% RAP is added. This improvement with the inclusion of RAP is because the aged binder of RAP increases the stiffness of the virgin binder and tends to decrease its phase angle (see Fig. 5) decreasing its viscous (non-recoverable) component [64]. No significant differences (p-value > 0.179) are observed in the WTS_w and RD parameters with increasing addition of RAP, obtaining final values in rut depth close to 2.3 and 2.9 mm for the mixtures with 50%, 60%, and 70% RAP (M50/0, M60/0, and M70/0 respectively).

The addition of 7.5% CS in the mixtures with RAP (Fig. 8c) increased for all RAP contents the rut depth (RD) compared to the mixtures with only RAP. However, these differences are not statistically significant (p-value > 0.089) so a negative influence is not evident. A similar case occurs in the mixtures with 15% CS and RAP (Fig. 8d) where despite presenting mixed results (with increases and decreases) in the rut depth compared to their counterparts incorporating only RAP, no significant differences are observed in the RD value with p-value > 0.184. It should be noted that despite there being no significant differences between the RD values obtained between the mixtures with RAP and the mixtures with RAP and CS, the mixture that presented the best performance in the HWTT test is the M70/0 mixture with a rut depth of 2.291 mm. Likewise, the mixtures M60/15 and M70/15 presented similar numerical results in the rut depth to the mixture M50/0, which incorporates the lowest percentage of RAP studied, with a difference of 0.04 mm and 0.039 mm, respectively. According to these results, the inclusion of CS would allow adding higher percentages of RAP to the mixture without practically modifying its performance in the HWTT test.

The mixtures incorporating only CS as replacement material showed lower values of deformation slope and rut depth than the M0/0 mixture. However, this rate of improvement is lower than the shown by the mixtures containing only RAP as a replacement material, mostly evidencing the suitability of its joint action with RAP in improving the permanent deformation resistance of HMAs.

According to the graph shown in Fig. 8a, there is no evidence of a loss of adhesiveness (stripping) in the mixtures studied, with the beneficial effects of RAP and CS predominating over the possible negative impacts of these materials in the presence of water. On the other hand, it is worth noting that the mixtures with low ITSR test values (mixtures M50/0, M60/0, M0/7.5, M0/15, and M60/15), in particular the mixture M60/15 which combines both materials, show a superior performance to the reference mixture in the rutting test under wet conditions. This suggests that moisture damage to the asphalt mixtures, in terms of increased modulus of stiffness, is not significant in tests with repetitive loading over time where there are recovery periods.

6. **Conclusions**

Based on the results obtained and their statistical analysis, the following conclusions can be drawn:

- The use of high percentages of RAP in semi-dense HMAs significantly reduces the mean texture depth (MTD) value, showing the lowest performance with the inclusion of 70% RAP. The addition of CS in the mixtures with RAP does not help to reverse the lower performance shown by these in the MTD value.
- In the initial stiffness value, the use of RAP increased the dynamic stiffness modulus of the mixtures and decreased the phase angle, modifying the viscoelastic properties of the virgin binder.
- In the fatigue life analysis, RAP increased the deformation stress values ε associated with the 10^6 loading cycles compared to the reference mixture showing a better performance. On the other hand, the use of 7.5% CS helps to incorporate higher RAP contents in the semi-dense HMAs, maintaining, and even improving in some cases, the fatigue strength.
- Regarding the results of the moisture sensitivity test, practically all the mixtures designed with RAP improved the ITS in both dry and wet conditions. However, the ITSR between both conditions decreases, evidencing greater water damage in the mixtures with RAP.
- The use of CS as the only replacement material significantly increases the moisture sensitivity of semi-dense HMAs. However, when added in conjunction with RAP, it improves the performance of the mixtures with RAP in the ITSR test, also showing a stabilizing effect with increasing RAP contents.
- The inclusion of high percentages of RAP significantly decreases the susceptibility to permanent deformation of HMAs. On the other hand, it was not possible to confirm significant negative effects on the rut depth and strain rate parameters with the inclusion of CS in the mixtures with RAP.
- According to the results obtained, the incorporation of RAP and CS in HMAs would help the design of sustainable asphalt mixtures without compromising the mechanical performance of asphalt pavements. The use of 15% CS in mixtures with 60% RAP (M60/15) and 7.5% CS in mixtures with 70% RAP (M70/7.5) is recommended.

CRediT author statement

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Declaration of interest statement

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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