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The feasibility of using copper slag in asphalt mixtures for base and surface layers based on laboratory results

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ABSTRACT

This study aims to assess the feasibility of using copper slag (CS) in asphalt mixtures in both the surface and base layers of road pavements. For this purpose, two sets of asphalt mixtures with different CS content (15 and 30%) and fraction sizes (0/8 and 0/16 mm) were prepared. The optimal binder content of each mixture was determined using the Marshall mix design method. Their laboratory performances were investigated and compared with the properties of reference asphalt mixtures, i.e. mixtures consisting exclusively of virgin materials. Test results showed that adding CS to the mixture for the surface layer improved stiffness and rutting resistance and had almost no negative impacts on the remaining properties apart from cracking resistance. Further, the addition of CS to the base-layer mixture improved cracking resistance, but negatively affected stiffness, rutting and fatigue resistance. In both cases, adding CS did not influence the strength, water sensitivity or freeze–thaw resistance. From a leaching perspective, the results showed that leachates gathered from the asphalt mixtures designed for the surface layer did not contain heavy metals. Overall, it can be concluded that CS has a potential to be used as an appropriate aggregate substitute for virgin aggregates in asphalt mixtures for both surface and base layers.

1. Introduction

The use of high quality aggregate in the construction of heavily trafficked roads is a mandatory requirement in the (re)construction of road pavement structures in many developed countries. However, the availability of such natural raw materials is finite. Consequently, contractors and road authorities have been exploring alternatives to avoid further depleting natural stocks. In this context, recycling existing paving materials and reusing non-hazardous solid waste materials are often considered as potentially viable options. Although seemingly attractive from economic and environmental standpoints, the latter option can raise concerns due to such materials potentially not complying with the rigorous technical requirements for the production of asphalt mixtures.

Indeed, any material used as an aggregate in asphalt mixtures must have good physicochemical properties [1]. Previous studies have investigated the possibilities of using reclaimed asphalt pavement (RAP) [2], recycled concrete aggregate (RCA) [3], steel slag [4], and other alternatives in asphalt mixtures. These studies have shown that it is possible to produce asphalt mixtures with properties very similar to

those of mixtures made of exclusively virgin materials. Furthermore, testing has highlighted the importance of several properties such as homogeneity, low water absorption, good wear resistance, favourable particle shape and a good affinity with bitumen.

One waste material that has been shown to have a good potential for application in the construction industry is copper slag (CS) [5]. CS is a by-product obtained during the smelting, converting and refining of copper matte [6]. It is usually stockpiled near a copper mining and smelting complex and must be crushed, or graded, before use in new applications. Granulated CS has numerous favourable properties for use as an aggregate, such as the angular or irregular shape of its grains, good abrasion resistance, low water absorption and excellent soundness characteristics [7,8]. Despite its favourable properties, CS is not yet widely recognised as a construction material in technical regulations, but still considered a waste material (including in Serbia). In addition to its use in the production of abrasive and cutting tools, CS has also been used to produce tiles, roofing material granules, concrete and asphalt mixtures [6].

The use of CS in asphalt mixtures dates back to the mid-1980 s. At that time, it was suggested that CS could be a suitable aggregate

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substitution where it was available locally [9]. This possibility has been further investigated in many studies where CS has been used as a partial replacement of filler (<0.075 mm) [10,11], fine (<4.75 mm) [12–17,23] and medium aggregate fractions (0.15/9.5 mm) [18] to produce Hot or Warm Mix Asphalt (HMA or WMA). Different CS proportions have been considered in these studies (up to 100% for filler, and up to 40% in other fractions) to determine its optimal content in asphalt mixtures and its influence on mixtures' performances. Fig. 1 presents an overview of previous studies where different proportions of various size CS granules were used to produce asphalt mixtures for surface, binder and base layers. This shows that most of the research has been performed in Chile, which is the world's largest copper producer, India and the Middle East. Conversely, there are no studies published in indexed scientific journals originating in Europe or North America. From this figure it is also notable that the only one study has considered a medium (coarser than 4 mm) CS fraction.

The optimal CS content in HMA/WMA mixtures is typically found to be between 10 and 20% [13,16,17,19,20]. The statements of Raposeiras et al. [18] and Mohi Ud Din and Mir [21] that suggest the use of a limited content of CS because of potential aggregate segregation are in line with these findings.

In seeking to verify to what extent such statements hold, researchers have investigated the impact of different CS contents on the properties of multiple mixtures. The general trend is that increasing CS content leads to increased Optimal Binder Content (OBC), consequently leading to fewer air voids, less stability and lower stiffness [12–14,17]. However, some studies have also shown that it is possible to produce asphalt mixtures with the same [11,22] or even lower OBC [10], regardless of the CS content. This seems possible because of the low water absorption of CS (usually below 0.3%). Indeed, it has been concluded that the addition of CS can reduce the water sensitivity of asphalt mixtures, irrespective of the actual CS content [10,15–17].

A considerable amount of research has been dedicated to understanding the leaching, permanent deformation, fatigue and cracking resistance of asphalt mixtures incorporating CS. However, the results of these studies have occasionally been inconsistent. Some results have shown that adding CS enhances resistance to permanent deformation [12] and fatigue resistance [11], regardless of the CS content. Others have claimed that such improvements can only be observed if the CS content does not exceed 20% [16,17]. Other studies have gone further in claiming that the benefits of using CS are even more evident if used in mixtures containing RAP [17–19,23]. This was explained by the fact that

RAP provides additional stiffness, while the angularity of the CS particles provides adhesion and increases interlocking [21,24]. Overall, only one study has considered leaching, and this concluded that the use of CS should not harm the environment [11].

Notwithstanding the merits of the studies mentioned above, the authors of this study are unaware of any studies where a 0/16 mm CS fraction has been used, and even a 0/8 mm CS fraction has rarely been used to produce HMA/WMA. Moreover, only one study [15] has been identified where the various CS fractions (ranging from 0.08 to 2.5 mm) were used as aggregate replacements to determine their impact on mixture performance made with the same virgin aggregate. Acquired knowledge might be very useful for asphalt producers with relatively modest plants because they would then be able to determine what CS fraction could be used for the production of multiple mixtures without affecting their quality. Further, the assessment of cracking resistance of mixtures including CS using the newly developed cracking tolerance index (CT_{index}) [25] has not yet been reported.

To overcome this knowledge gap, the research work presented in this paper aims to assess the possibility of using different CS content and fraction sizes in asphalt mixtures that are intended for application in surface and base layers of road pavements. The expectation is that the findings from this study can be used to demonstrate that it is realistic to consider CS as a construction material and not only a waste.

2. Experimental design

In this study, two sets of asphalt mixtures (one for the construction of a surface layer and the other for a base layer) with different CS contents (15 and 30%) and fraction sizes (0/8 mm and 0/16 mm) were prepared and tested. After determining the OBC of all the mixtures according to the Marshall mix design method, their properties (displayed in Fig. 2) were compared to the properties of reference asphalt mixtures, i.e. mixtures composed entirely of virgin materials. Furthermore, because the surface layer is exposed to rainfall, the potential leaching susceptibility of the asphalt mixtures was also assessed. The rutting resistance of both sets of asphalt mixtures were likewise investigated. Although such distress is mostly observed in the pavement surface layer because of its direct exposure to high temperatures and heavy traffic loads, rutting tends to be most severe in the middle asphalt layer, where the shear stresses are greatest. Consequently, the base layer mixture, which is also very often used for the binder layer in some countries, was also investigated. The base layer asphalt mixtures were subjected to a fatigue test

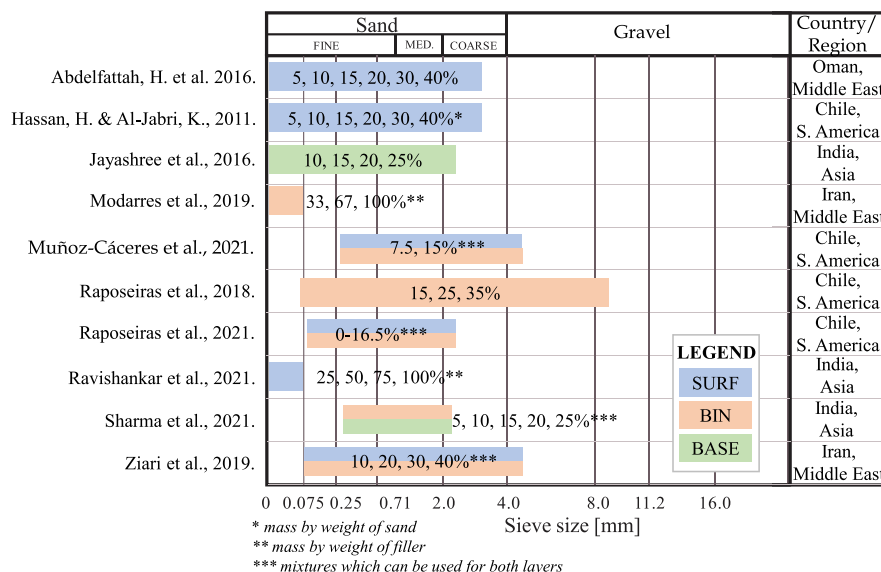


Fig. 1. CS content and sizes used in testing different asphalt mixtures.

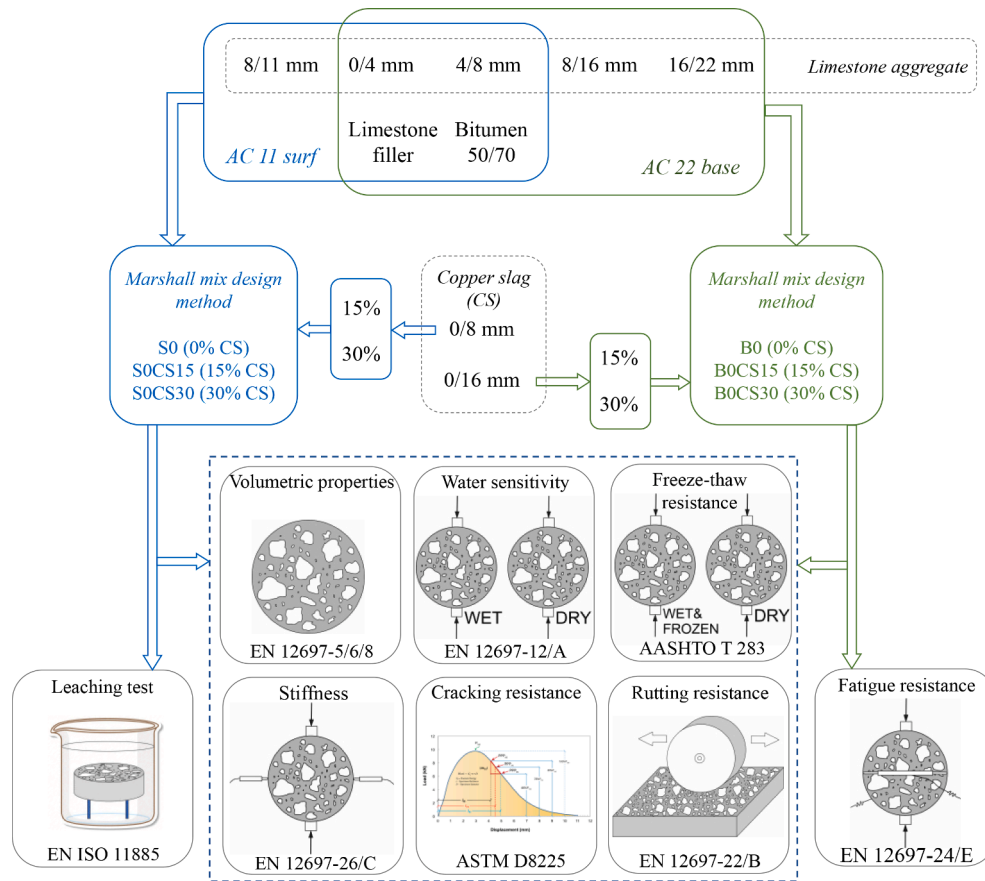


Fig. 2. The plan of the experimental study.

since they are exposed to bending under traffic loading, leading to the fatigue cracking seen on pavement surfaces. The experimental plan of the study is shown in Fig. 2.

3. Materials

3.1. Asphalt binder

Plain bitumen of type 50/70 was used in all the asphalt mixtures and its properties are given in Table 1.

3.2. Filler, aggregate and copper slag

A mineral filler of limestone origin was used in this study. The basic properties of the filler are given in Table 2.

Crushed limestone aggregate from a single source was used in all the asphalt mixtures. In addition to the mineral filler, mixtures for the surface layer (AC11 surf) contained 0/4, 4/8 and 8/11 mm fractions, whereas mixtures for the base layer (AC22 base) contained 0/4, 4/8, 8/16 and 16/22 mm fractions. The basic properties of all the fractions are shown in Table 3.

The CS was gathered from slag dumps near a copper mine located in Serbia. The CS was formed by abrupt air-cooling of hot copper slag. Although a 0/16 mm fraction has not been used as an aggregate

Table 1
Properties of the plain bitumen.

Property	Value	Unit	Standard
Penetration value	50 × 0.1	mm	EN 1426
Softening point	51.5	°C	EN 1427
Penetration index	-0.80	-	EN 12591, Annex A

Table 2
Basic properties of the filler.

Property	Value	Unit	Standard
Particle size distribution (percentage passing)	Sieve size [mm]		
	0.063	84.9	
	0.125	94.7	EN 933-1
Particle density	2.0	100.0	
Void content of dry compacted filler (Rigden voids)	31	Mg/m ³	EN 1097-7
Methylene-blue value	0.45	%	EN 1097-4
		g/kg	EN 933-9

replacement in previous studies because of possible issues concerning homogeneity, it was selected for this study because it can be used without additional crushing, thus increasing the amount of material that can be used without increasing processing costs. The physical properties of the CS, as measured, are shown in Table 3, and the chemical composition (determined using wavelength dispersive spectrometry – X-ray fluorescence (XRF)) are shown in Table 4.

Table 3 shows that, due to the crushing process, both CS fractions have very similar gradation in the range below 8 mm. As such, it can also be concluded that both fractions have at least the same if not better properties than virgin aggregate fractions. The one exception to this was that the 8/16 mm CS fraction had a slightly worse shape index than the equivalent virgin aggregate fraction. Given the chemical composition shown in Table 4, it can be concluded that this CS has the potential to achieve crack-healing through induction heating because of its high metallic oxides content [26] or by UV radiation [27]. Finally, the tests showed that the CS particles had a rough surface and an angular shape as

Table 3
Physical properties of virgin aggregate and CS.

Property	Virgin aggregate [mm]					CS [mm]		Unit	Standard
	0/4	4/8	8/11	8/16	16/22	0/8	0/16		
Sieve size [mm]									
0.063	3.2	0.9	0.7	0.5	0.3	4.0	3.0		
0.09	4.2	1.1	0.8	0.6	0.4	5.6	4.3		
0.25	16.6	1.2	0.9	0.9	0.6	15.2	13.3		
0.71	39.7	1.3	0.9	0.9	0.6	39.2	33.6		
2.0	67.3	1.4	0.9	0.9	0.6	57.3	57.3		
4.0	95.7	2.2	1.3	0.9	0.6	76.8	76.8	mm	EN 933-1
8.0	100.0	96.9	10.7	3.7	0.6	97.3	85.3		
11.2	–	100.0	96.6	45.9	0.7	100.0	92.5		
16.0	–	–	100.0	97.7	12.2	–	96.6		
22.4	–	–	–	100.0	98.3	–	100.0		
31.5	–	–	–	–	100.0	–	–		
Oven-dry particle density (ρ_{nd})	2.725	2.710	2.715	2.708	2.704	3.550	3.560	Mg/m ³	EN 1097-6
Water absorption (WA ₂₄)	–	0.8	0.7	0.6	0.5	0.4*	0.2**	%	EN 1097-6
Shape index (SI)	–	6.9	6.4	6.9	6.2	8.9*	6.1**	–	EN 933-4
Na ₂ SO ₄ soundness	1.4	1.3	1.1	1.0	0.9	1.0	1.0	%	ASTM C88
Resistance to fragmentation (Los Angeles coefficient)	–	–	24	–	–	–	15	–	EN 1097-2/Point 5
Sand equivalent (SE)	71	–	–	–	–	–	–	–	EN 933-8

*fraction 4/8 mm, ** fraction 8/16 mm.

Table 4
Chemical composition of CS.

Compound	Concentration [%]
Si	16.44
Al	2.49
Fe	33.08
Ca	2.57
Mg	0.89
Na	0.25
K	0.83
S	1.19
Cl	0.00
P	0.03
Ti	0.11
Cu	0.73
Zn	1.55
V	0.02
Mn	0.06
Cr	0.06
As	0.07
Pb	0.09

shown in Fig. 3, which displays the 0/8 mm fraction in its actual size and at 10, 20 and 30 times magnification.

3.3. Mix design methodology

Two types of asphalt mixtures were prepared for this study, namely an AC11 surf 50/70 and an AC22 base 50/70. The former is typically used for the surface layer of roads with moderate traffic, whereas the latter mixture is used as a base layer for roads with traffic loadings

ranging from light to very heavy.

In addition to the reference mixtures (denoted as S0 (surface) and B0 (base)), two additional mixtures were prepared for each set of tests. In these mixtures, 15 and 30% of the virgin aggregate was replaced with CS. A 0/8 mm fraction was used in the surface layer and a 0/16 mm fraction in the base layer. These mixtures are denoted as S0CS15 and S0CS30 for the surface layer and as B0CS15 and B0CS30 for the base layer. It was not viable to use a higher CS concentration in the base layer mixture due to the particle distribution of the CS 0/16 mm fraction, and it was decided to also exclude higher rates in mixtures for the surface layer. The density of the virgin aggregate fraction was much lower than that of the CS, so the replacement was based on volume, resulting in a higher proportion of CS by mass in the final mixture. The proportion of the virgin aggregate fractions was adjusted with CS to make the final gradations as close as possible to those of the reference mixtures. The OBC of every mixture was determined at an air void content of 4.5% for the AC11 surf mixture and 5.5% for the AC22 base using the Marshall method (EN 12697-34) and in accordance with Serbian specifications. The particle size distributions of the virgin aggregate and the CS on each sieving of the mixtures analysed, along with their OBCs, are given in Table 5. The gradation curves of both mixture types are displayed in Fig. 4.

Table 5 shows that mixtures with 15% CS had lower OBCs than those of the reference mixture which was attributed to the lower water absorption and improved interlocking properties. However, the OBC increases in both cases when 30% of CS is used. This is possibly because of the higher specific surface area and rougher surface which outweighed the CS's low water absorption.

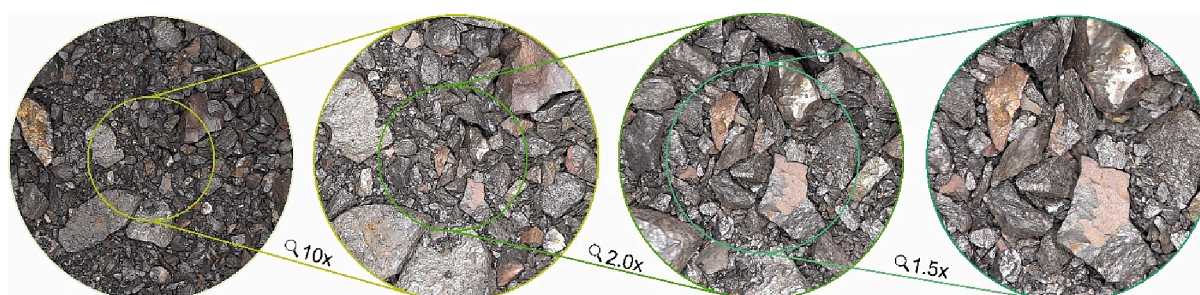


Fig. 3. Fraction 0/8 mm of CS in original size and magnified 10, 20 and 30 times.

Table 5
Grading curves of the asphalt mixtures.

Mixture	OBC [%]	Material	Percentage passing through each sieve size [mm]									
			0.09	0.25	0.71	2.0	4.0	8.0	11.2	16.0	22.4	31.5
S0	4.8*	VA	9.1	16.2	28.5	43.1	58.5	82.4	99.3	100.0	100.0	100.0
SOCS15	4.6* (4.4)**	VA	8.6	14.2	23.7	35.0	46.9	67.5	84.3	85.0	85.0	85.0
		CS	0.8	2.3	5.9	8.6	11.5	14.6	15.0	15.0	15.0	15.0
SOCS30	5.0* (4.6)**	VA	8.0	12.1	18.9	26.9	35.3	52.6	69.3	70.0	70.0	70.0
		CS	1.7	4.6	11.7	17.2	23.0	29.2	30.0	30.0	30.0	30.0
B0	3.7*	VA	7.6	12.8	21.6	32.1	43.0	59.5	70.1	84.5	99.7	100
BOCS15	3.7* (3.5)**	VA	7.0	10.8	16.8	24.0	31.5	46.7	56.3	69.6	84.7	85.0
		CS	0.6	2.0	5.0	8.6	11.5	12.8	13.9	14.5	15.0	15.0
BOCS30	4.0* (3.6)**	VA	6.5	8.7	12.0	15.9	20.0	33.9	42.6	54.6	69.7	70.0
		CS	1.3	4.0	10.1	17.2	23.0	25.6	27.7	29.0	30.0	30.0

*Binder content by weight of a mineral mixture composed of limestone aggregate, ** Binder content by weight of the resulting mineral mixture composed of limestone aggregate and CS.

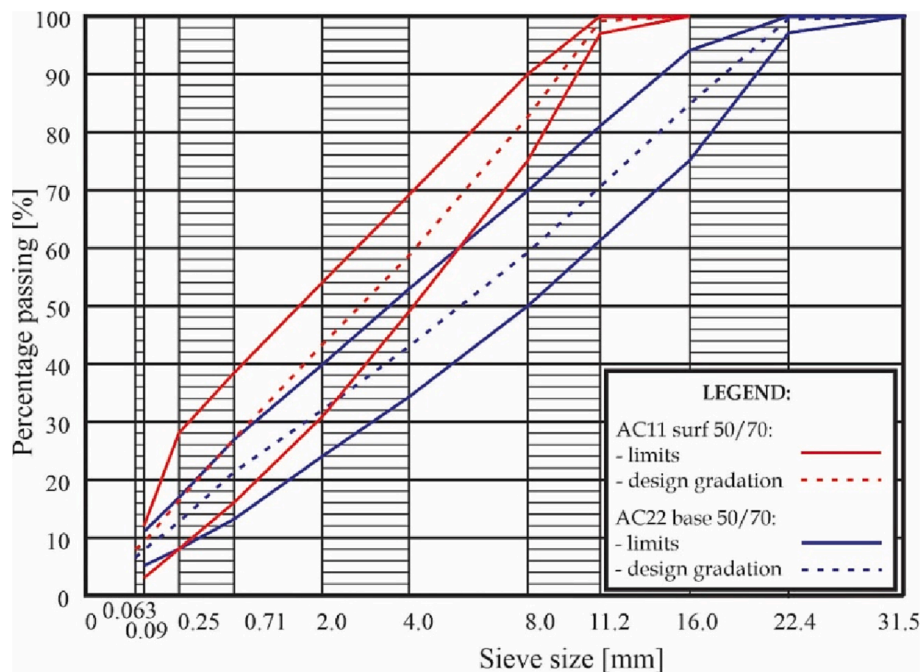


Fig. 4. Gradation curves of asphalt mixtures with limit lines.

3.4. Samples preparation and testing methods

All the asphalt mixtures were prepared at the OBC (Table 5). Virgin aggregate and CS fractions were preheated to 160–170 °C while the virgin bitumen was preheated to 150 °C before mixing. The materials were mixed for five minutes in a laboratory mixer with a 30 l capacity before the addition of a cold filler and a subsequent three-minute mixing. Following this, the loose mixtures were quartered aiming to obtain samples that were as homogenous as possible and then short-term aged in an oven for 4 h at 135 °C to simulate the manufacturing process of an asphalt plant [28,29]. Immediately before compaction, all the mixtures were remixed by hand to ensure representative samples, which were further compacted at 150 °C using a range of methods (Table 6). The volumetric properties (maximum density (EN 12697-5), bulk density (EN 12697-6) and void characteristics (EN 12697-8)) of each mixture/sample were determined before their performances were assessed.

The nominal thicknesses of the slabs for the wheel tracking test were similar to those typically laid on a road (70 mm for the base and 50 mm for a surface layer) as specified in EN 12697-24. Also, 100 mm diameter cylindrical cores were extracted from 70 mm thick slabs for fatigue testing.

Water damage in asphalt mixtures can be defined as the degradation

of mechanical properties due to the presence of moisture. There are two possible damage mechanisms: (i) the deterioration of the adhesive bond between the aggregate surface and the asphalt binder (adhesive damage) and (ii) the cohesive damage of the asphalt mastic [30]. The water susceptibility of the asphalt mixtures is presented in terms of the Indirect Tensile Strength Ratio (ITSR) between Indirect Tensile Strength (ITS) values of wet and dry sets of samples (Equation (1)):

$$ITSR = \frac{ITS_w}{ITS_d} \cdot 100 \tag{1}$$

where ITS_w is the average ITS of the wet group [kPa], and ITS_d is the average ITS of the dry group [kPa].

The impact of water and then freezing on compacted asphalt mixtures (AASHTO T 283) was assessed as the Tensile Strength Ratio (TSR) after n cycles ($n = 1$ or 3):

$$TSR_n = \frac{S_{tm,n}}{S_{td}} \cdot 100 \tag{2}$$

where $S_{tm,n}$ is the average ITS of the freeze–thaw conditioned subset [MPa] after n cycles, and S_{td} is the average indirect tensile strength of the unconditioned (dry) subset [MPa].

Table 6
Sample preparation and testing methods.

Testing method	Standard	Samples		Testing conditions	
		Preparation	Conditioning		
Water sensitivity Freeze-thaw resistance	EN 12697-12, Method A	2 × 35 blows in a Marshall compactor	2 × 3 samples (dry and wet set)	- Dry set: at 25 ± 1 °C - Wet set: 68–72 h in WB* at 40 °C	Determination of ITS (EN 12697-23) at 25 ± 1 °C Determination of ITS at 25 ± 1 °C
	AASHTO T283	Various number of blows in Marshall compactor required to achieve 6–8% air voids	3 × 3 samples (one unconditioned and two conditioned sets)	- Unconditioned set: at 25 ± 1 °C - Conditioned sets: (15 + h at -18 ± 2 °C in F**; 24 h at 60 ± 1 °C in WB*; 4 h in WB* at 25 ± 1 °C) once and twice	
Cracking resistance (IDEAL-CT)	ASTM D8225	Unconditioned samples with an air void content of 7 ± 0.5%	3 samples	-	-
Stiffness	EN 12697-26, Annex C	2 × 50 blows in a Marshall compactor	4 samples	At least 4 h at testing temperature	- Loading time: 124 ± 4 ms - Testing temperatures: 5, 10, 20, 30 and 40 °C
Rutting resistance	EN 12697-22, Procedure B: Small device (in the air)	- Slabs compacted according to EN 12697-33: 1) 260x320x70 mm (for base layer mixtures) 2) 260x320x50 mm (for surface layer mixtures)	2 samples	At least 6 h at testing temperature	- Testing temperature of 60 ± 1 °C - 20,000 wheel-passes
Leaching	EN ISO 11885	Marshall samples used for stiffness determination	3 samples	- In a batch system, without stirring, where the vessels were open - pH value of water: 5.1 and 7.0 - 24 h, 72 h, 120 h and 168 h	ICP-OES*** was used to investigate the presence of heavy metals in water solutions
Fatigue test	EN 12697-24, Annex E (IT-CY)	Cylindrical samples of 100 mm diameter cored from 70 mm thick slabs (for base layer mixture)	At least 10 samples	At least 4 h at testing temperature	- (Ha)versine load with 0.1 s loading time and 0.4 s rest time. - Testing temperature of 10 °C - Stress-controlled mode

*WB – water bath, ** F – freezer, *** ICP-OES – Induced Coupled Plasma with Optical Emission Spectrometry.

The indirect tensile asphalt cracking test (IDEAL-CT) was used to assess the cracking resistance of asphalt mixtures. The test is comparable to other laboratory tests used for cracking resistance, such as the Texas Overlay Test and Illinois Flexibility Index Test [25]. It analyses the data collected during an ITS test (displacement and load) in a different way to the traditional ITS test. As the test is very sensitive to air void content [31], only unconditioned samples that had been prepared for the freeze–thaw resistance test, having an air void content of 7 ± 0.5%, were used. As an output, a performance-related cracking parameter, CT_{index} was used to assess the cracking resistance.

The stiffness was measured at a range of temperatures (between 5 °C and 40 °C) with a single rise time of 124 ± 4 ms (the time taken for the applied load to increase from zero to maximum value).

The resistance to permanent deformation (rutting resistance) was expressed as a mean proportional rut depth (the ratio of the measured rut depth to the thickness of the tested sample) and the wheel-tracking slope.

For the analysis of the experimental leaching test, induced coupled plasma with optical emission spectrometry (ICP-OES) was carried out using a Thermo Scientific iCAP 6500 duo analyser to determine the concentration of heavy metals (Cu^{2+} , Pb^{2+} , Zn^{2+} , $Cr^{3,6+}$) in a sample taken from distilled water solutions.

Given that the highest tensile stresses and strains occur in the lower asphalt layers, the fatigue resistance of base layer asphalt mixtures was investigated. The tests were carried out in a stress-controlled mode at different stress levels with an assumed Poisson's ratio of 0.35. The load was applied as many times as necessary to achieve the established failure criterion. In this study, the dissipated energy concept was used, where failure is defined as the number of loadings ($N_{f,w}$) until cracks are initiated. This occurrence can be visually identified by looking for the peak value of the w_n curve, which was generated using Equation (3):

$$w_n = \frac{n}{\epsilon_{R,n}} \bullet 10^6 \quad (3)$$

where n is the number of loading cycles, and $\epsilon_{R,n}$ is the resilient strain at

load cycle n during cycling loading [$\mu\text{m}/\text{m}$].

The results from the individual tests were later fitted to and presented in the form of a power (fatigue) function, as expressed by Equation (4):

$$\lg(N_{f,w}) = \lg(k_w) + n_w \bullet \lg(\epsilon_0) \quad (4)$$

where $N_{f,w}$ is the number of loading cycles using the resilient approach, k_w and n_w are material constants and ϵ_0 is the initial strain amplitude at load cycle n during the cyclic loading [$\mu\text{m}/\text{m}$].

4. Results and discussion

4.1. Volumetric properties

The average air void content of the different testing samples and the ranges between the minimum and maximum values are displayed in Fig. 5. From this, it can be seen that increasing the CS content of the surface layer asphalt mixtures results in slight increases in air void content. There was a smaller increase in the base layer samples due to the coarser CS used in these mixtures since just a few coarse grains within a sample can considerably increase its mass. Consequently, there was a greater variation in the air void content of samples with a 0/16 mm fraction (Fig. 5). This issue has already been seen as a potential problem, especially when using high CS contents [18,21]. Here, it should be emphasised that not only the quantity but also the size of the grains can cause heterogeneity in a mixture.

4.2. Water sensitivity

Results from the testing, in the form of average values and standard deviations of ITS for the wet and dry subsets, as well as the resulting ITSRs, are displayed in Fig. 6. The figure shows that all the surface layer mixtures with CS, regardless of the binder and CS contents, had similar ITS values under dry conditions, typically 12–13% lower than the values for the reference mixture. A similar pattern was observed with the wet

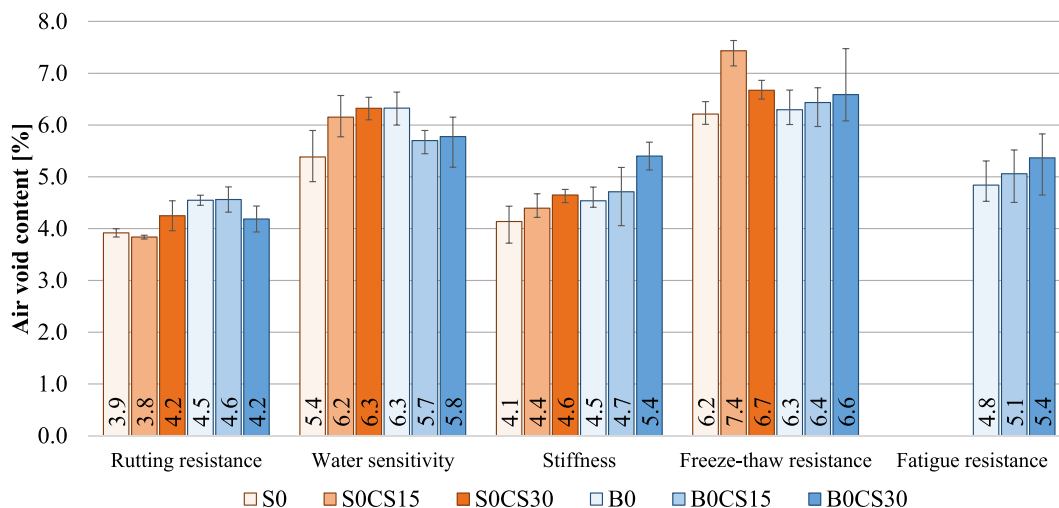


Fig. 5. Air void content of the samples tested.

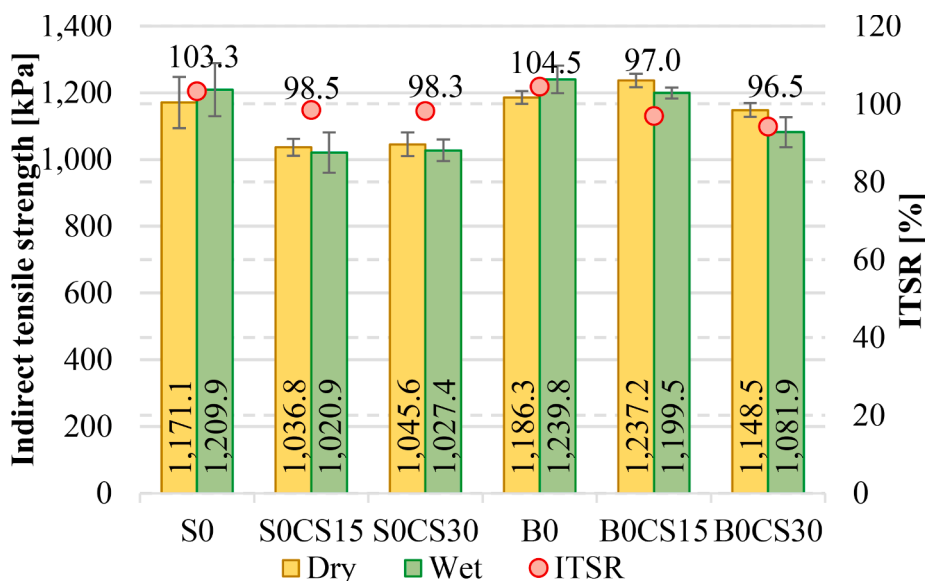


Fig. 6. Results of water sensitivity tests.

subset, where ITS values were approximately 17–18% lower than with the reference mixture. With the base layer mixtures, the mixture with a 15% CS content had a slightly higher ITS value (by around 4%) whereas the mixture with a 30% CS content had a slightly lower ITS value (by around 3%) in dry conditions compared with the reference mixture. This finding is similar to that of a previous study [17], where ITS increased with increases in CS content up to 20%, after which it began to decrease. It can also be seen that after conditioning, the ITS values decreased with an increase in CS content: by 3% in the mixture with a 15% CS content and by 12% in the mixture with a CS content of 30%.

Turning to the ITS values, one can observe that they all decreased with an increase in CS content, although all the mixtures had ITS values of about 100%. ITS values higher than 100% were observed in both reference mixtures, a finding for which many factors have been suggested [32]. However, it should be noted that the variability in the densities between the dry and wet specimens, which is typically stated as a likely cause [30], was negligible, and it is not easy to identify the real cause of this phenomenon. The high ITS values achieved could be due to an increase in internal pressure and the aggregate friction during testing resulting from water replacing air in the voids and the inability of

the compression test to properly measure the effects of moisture damage [33].

Overall, the test results would seem to show that water does not significantly affect the mechanical properties of asphalt mixtures that include CS (all the ITS values were well above the usually recommended 70–80%). Further, with a CS content of 30%, the sensitivity to water is slightly lower than with the 15% CS sample, a finding consistent with Sharma et al. [16]. However, the opposite was found in another study [17], where the OBC increased with an increase in CS content, consequently increasing the water sensitivity.

4.3. Freeze-thaw resistance

The average ITS values of the various mixtures, along with their standard deviations and TSRs, are displayed in Fig. 7.

The results presented in Fig. 7 show that a single freeze–thaw cycle does not cause a decrease in the ITS value of any of the tested mixtures; rather, it increased them resulting in TSR values above 100%, a finding consistent with the results of the water sensitivity test. This led to the conclusion that one cycle may not be sufficient to identify the impact of

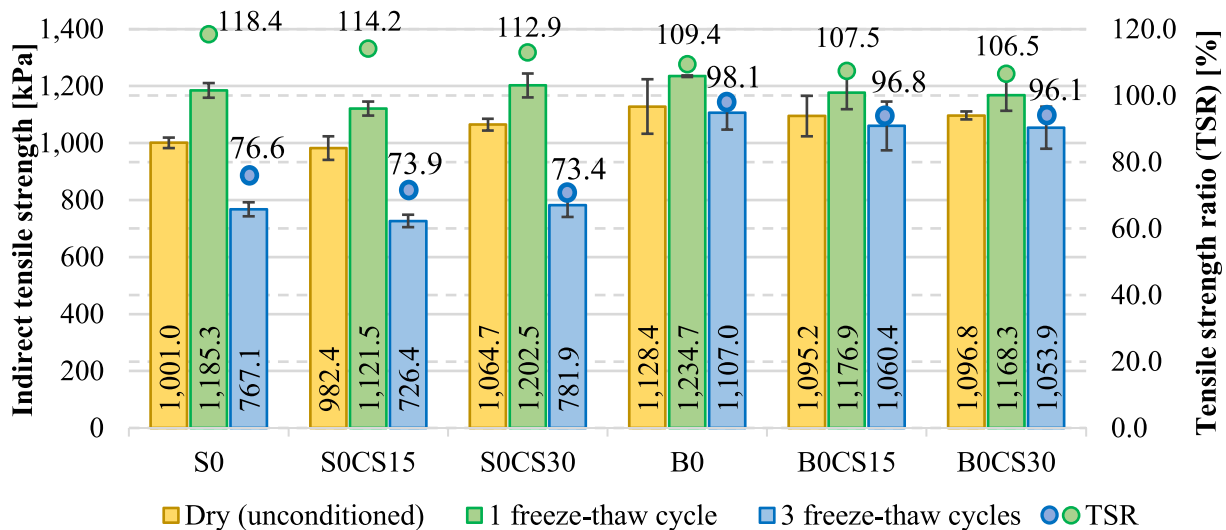


Fig. 7. Results of the freeze–thaw resistance tests.

CS on the freeze–thaw resistance, a finding consistent with previous studies [32,34].

Therefore, it was decided to apply two additional cycles (making three in total) to more deeply investigate freeze–thaw resistance. After the further freeze–thaw cycles, the air void content of the mixture increased due to the change in volume cause by the expansion of water at low temperatures. This led to the degradation of the adhesive bond between aggregate and bitumen, the appearance of micro-cracks and, consequently, a reduction in the mixture’s strength [30,35].

The ITS values of the surface layer asphalt mixtures after the third freeze–thaw cycle were very similar (around 750 kPa) regardless of the CS content (0, 15, 30%), giving a TSR values of around 75%. When considering the performance of mixtures for the base layer, the mixtures with CS always had a slightly lower ITS than the reference mixture, regardless of the number of freeze–thaw cycles applied or the CS content. Nevertheless, the resulting TSR values were close to 100%, with only a very small decrease with the higher CS content. These values are much higher than those for the surface layer mixtures, a finding attributed to aggregate size on the basis that the percentage of stripping in the fine aggregate versus the percentage of stripping in the coarse aggregate may affect the retained TSR [33].

Finally, all the mixtures had a TSR value (after three freeze–thaw cycles) higher than the standard AASHTO T 283 requirement of 70% (after only one cycle) [36]. As such, it can be concluded that the addition of finer CS fraction to the surface layer mixture may reduce its freeze–thaw resistance, whereas including coarser CS fraction in the base layer mixtures should not affect this.

4.4. Cracking resistance

Fig. 8 displays the average values of the CT_{index} for each mixture (“x” mark on the graph) and the range of measured values for each testing sample. The larger the CT_{index} , the slower the cracking growth rate, i.e., the better the asphalt mixture’s cracking resistance.

The test results show that adding CS to the surface layer mixture reduced its cracking resistance to similar levels with both 15% and 30% fractions. Conversely, the addition of CS to the mixture for base layer slightly increased the cracking resistance, although there was a more noticeable scatter in the individual measurements. This was attributed

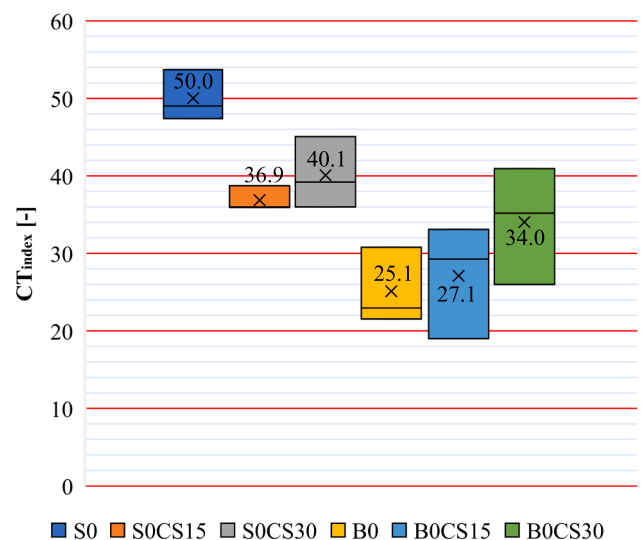


Fig. 8. IDEAL-CT results for the asphalt mixtures.

to the heterogeneity of the testing samples since they contain a 0/16 mm CS fraction. The fact that the 30% CS mixture had a higher CT_{index} than the mixture with 15% CS is linked to the higher OBCs of these mixtures [37].

4.5. Stiffness

Fig. 9 displays the stiffness modulus values of the investigated asphalt mixtures at different temperatures. It can be observed that the mixture with a CS content of 15% has the highest stiffness among the surface layer mixtures over the entire temperature range, which is attributed to the lowest bitumen content. The mixture with a 30% CS content has a slightly higher stiffness than the reference mixture at the lower testing temperatures (5 °C and 10 °C) but a significantly higher stiffness (close to the 15% CS values) at the higher temperatures.

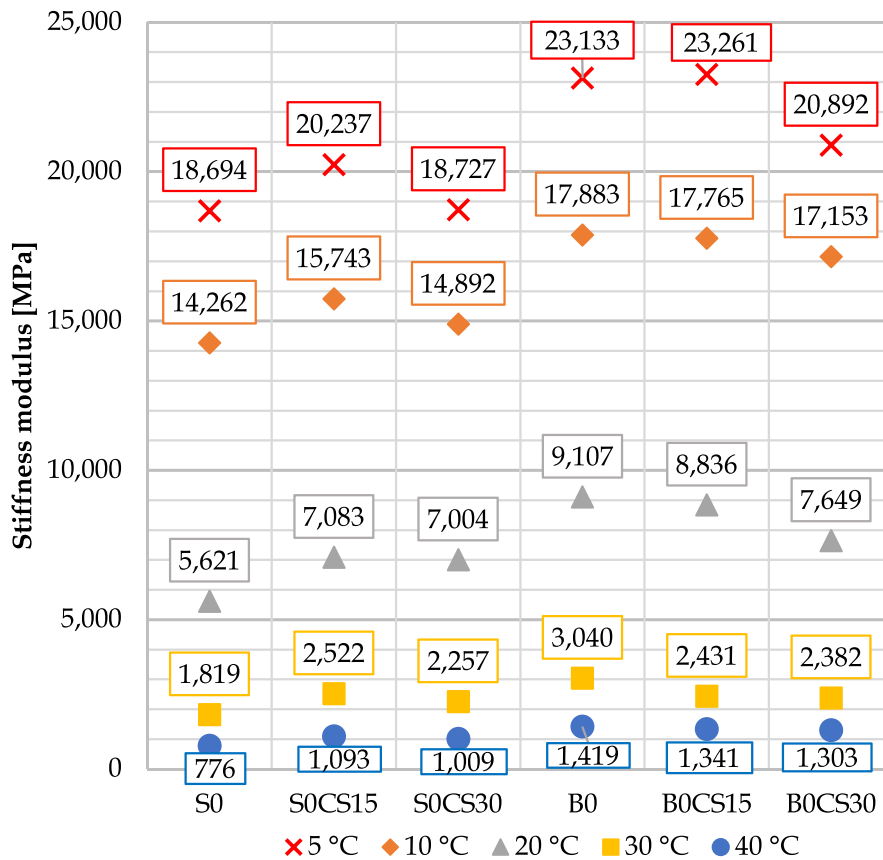


Fig. 9. Stiffness modulus values of asphalt mixtures at different temperatures.

Turning to the mixtures for the base layer, the figure shows that, at the lower and mid testing temperatures (5 to 20 °C), the mixture with a 15% CS content had a very similar stiffness to the reference mixture, whereas the mixture with 30% CS, which contained an additional 0.3% bitumen, had a lower stiffness. However, at higher temperatures (30 and 40 °C), the stiffness decreased with an increase in CS content, despite the increased binder content. Again, this finding can be ascribed to the fact that the aggregate skeleton is largely responsible for the stiffness at higher temperatures.

Very similar behaviours in asphalt mixtures with CS have been observed in previous studies [12,18], where the stiffness increased with increasing CS content up to 20–30%, after which it started to decrease. Although other studies [13,17] have concluded that a mixture’s stiffness decreases with increasing CS content, this was mostly because these mixtures simultaneously had increased levels of OBC, leading to a decrease in stiffness.

It should also be emphasised that the test results at the highest temperature (40 °C) generally agree with the wheel tracking test results (Section 4.6). The addition of CS improves the rutting resistance of

Table 7
Wheel tracking test results.

Mixture	RD _{AIR} [mm]	PRD _{AIR} [%]	WTS _{AIR} [mm/10 ³ load cycles]
S0	7.2	14.3	0.31
SOCS15	5.9	11.8	0.25
SOCS30	5.1	10.6	0.17
B0	5.4	7.7	0.15
B0CS15	6.2	8.7	0.20
B0CS30	7.0	9.9	0.27

asphalt mixtures for the surface layer but lowers the resistance of the base layer asphalt mixtures.

4.6. Rutting resistance

Fig. 10 graphically presents the wheel tracking test results for the investigated mixtures. The measured values of rut depth (RD_{AIR}), the calculated values of proportional rut depth (PRD_{AIR}), and wheel-tracking slopes (WTS_{AIR}) are provided in Table 7. When considering surface layer asphalt mixtures (Fig. 10a), the results show that an increase in CS content increases rutting resistance, which is also clearly visible in the cross-sections of testing samples (Fig. 11). Specifically, the addition of 15% and 30% CS led to corresponding improvements in rutting resistance of roughly 20% and 35% compared to the reference mixture. This addition of CS also slowed rut propagation (Table 7), which is attributed to the angularity of the CS particles increasing the interlocking. This improvement in the rutting resistance of surface layer asphalt mixture is particularly significant given that the reference mixture had a relatively poor rutting resistance (PRD_{AIR} = 14.3%),

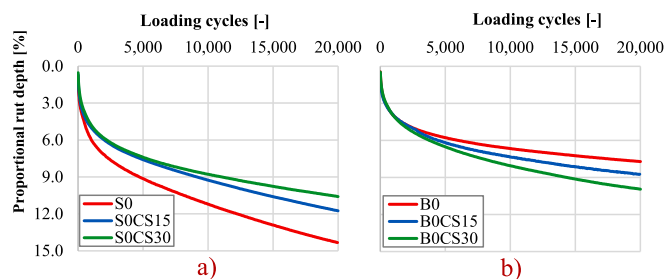


Fig. 10. Proportional rut depth of asphalt mixtures for (a) surface and (b) base layers.

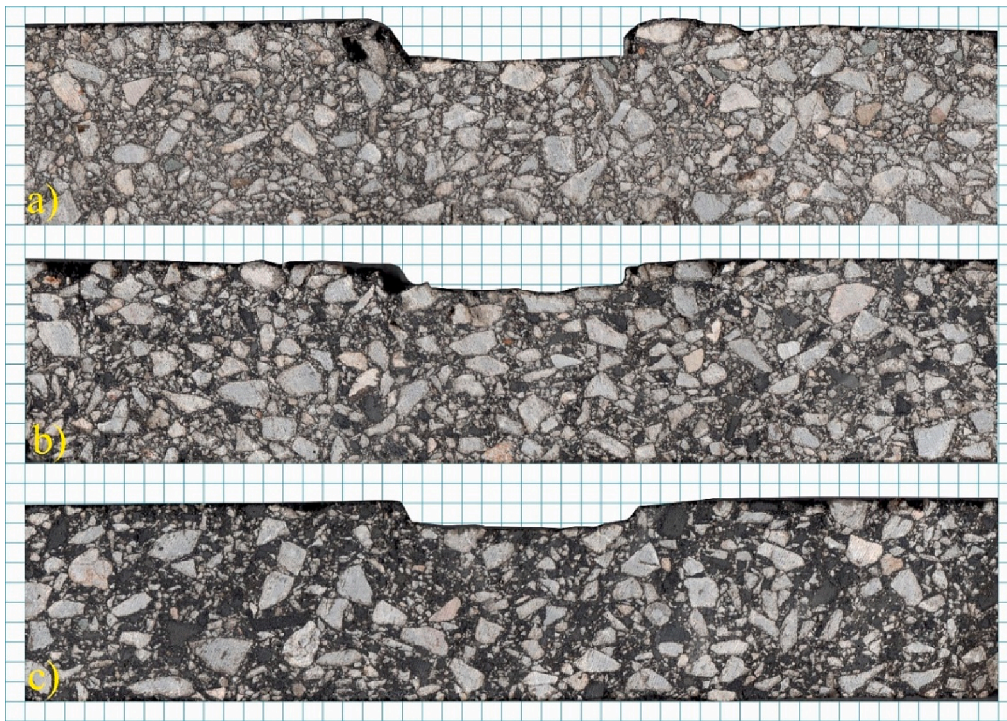


Fig. 11. Cross sections of (a) S0, (b) SOCS15, and (c) SOCS30 asphalt samples after wheel tracking test.

which is why it is typically only used for roads with moderate traffic loading.

In contrast to the surface layer, the effect of including CS on the base layer mixtures resulted in a decrease in the rutting resistance irrespective of the quantity of binder present (Fig. 10b). Replacing 15% and 30% of the virgin aggregate with CS increased PRD_{AIR} by approximately 13% and 29% respectively, resulting in the accelerated development of permanent deformation. Similar results were obtained in another study [12], where rutting resistance decreased with an increase in CS content, attributed largely to the higher OBC in these mixtures.

In this study, all the mixtures contained the same type of binder and all had similar air void volumes. Consequently, the behaviour demonstrated can be attributed to the aggregate skeleton, which is known to have a dominant impact on rutting resistance [38]. Referring back to Table 3, it can be seen that the mixtures for the base layer had higher percentages of 4/8 mm CS fraction than mixtures for the surface layer (for example, more than 50% of the 4/8 mm fraction came from the CS in the case of B0CS30 mixture). This CS fraction had a higher SI than the virgin aggregate fraction (Table 3) and this is probably the main reason for the weaker rutting resistance [39].

4.7. Leaching

The chemical analyses undertaken did not detect the presence of heavy metals within the aqueous solution regardless of the initial pH value, the sampling interval and the CS content. In other words, their concentration was below the detection limits: $Cu^{2+} < 0.01$ mg/L, $Pb^{2+} < 0.02$ mg/L, $Zn^{2+} < 0.05$ mg/L and $Cr^{3,6+} < 0.01$ mg/L. As such, this research supports the results of a previous study [11], namely that CS can be considered an environmentally friendly aggregate from a leaching perspective when used in asphalt mixtures because the asphalt film, which covers the CS particles, neutralises the potential toxicity of any heavy metals.

4.8. Fatigue resistance

The fatigue resistance of the samples was assessed by comparing the critical strain values (ϵ_6) that were deduced for a fatigue life of one million cycles. These were calculated based on fatigue laws, where the results from fatigue tests were fitted and presented as a power function (Equation (4)) as illustrated in Fig. 12. The regression coefficients of the

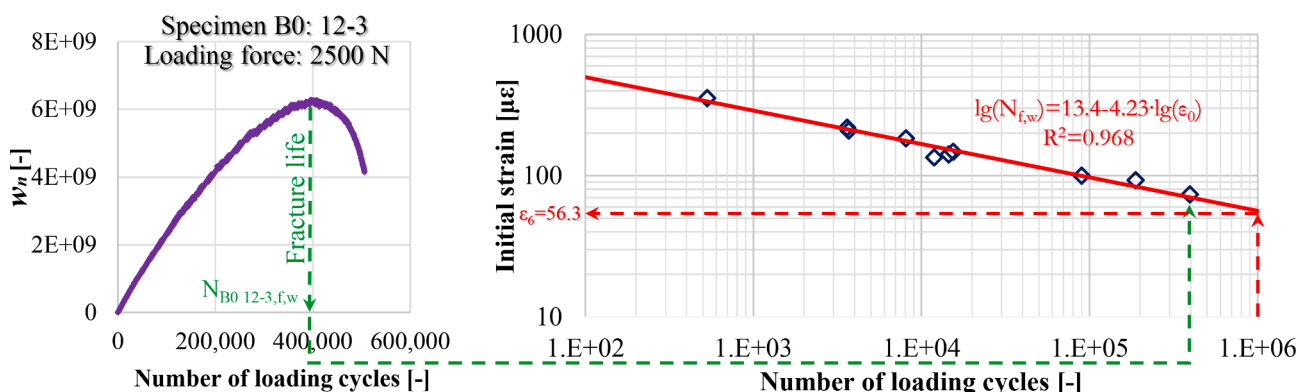


Fig. 12. An example of energy ratio evolution in the IT-CY fatigue test and the corresponding fatigue law.

Table 8
Regression coefficients of the fatigue lines.

Mixture	lg(k_w) [-]	n_w [-]	R^2 [-]	ϵ_6 [$\mu\text{m/m}$]
B0	13.40	-4.23	0.968	56.3
B0CS15	12.95	-4.02	0.967	53.5
B0CS30	12.90	-4.00	0.987	52.7

fatigue lines are presented in Table 8. This shows that the addition of CS slightly decreases the fatigue life in that it lowers the critical strain value (ϵ_6) compared to that of the reference mixture. However, the mixture with a 15% CS content only reduced the critical strain by roughly 5% and, as such, it is reasonable to state that they have comparable fatigue resistance. The critical strain value of the mixture with a 30% CS content was roughly 7% below that of the reference mixture, supporting the conclusions drawn by Sharma et al. [16], but still very close to that for the 15% CS mixture despite the higher binder content. The difference in fatigue resistance is mainly attributed to the differences in the air void content of the tested samples, which varied with changes to the CS and binder contents: a 15% increase in CS content saw an increase in the air void content of approximately 0.3%.

4.9. Overview of the test results

The research performed in this study show that adding a relatively fine CS fraction (0/8 mm) to the surface layer asphalt mixture has almost no negative impacts on mixture performance (the exception being its cracking resistance). However, some base layer asphalt mixtures' properties (stiffness, rutting, and fatigue resistance) did worsen slightly with an increase in CS content, whereas the cracking resistance improved. This is a consequence of the less advantageous composition with a coarser CS fraction (0/16 mm) and the consequent heterogeneity in mixtures containing it. Further, in both cases, the inclusion of CS did not influence the strength, water sensitivity, and freeze–thaw resistance of asphalt mixtures. Table 9 summarises how different CS contents and fraction sizes changed the properties of asphalt mixtures for both surface and base layers relative to the CS-free control mixtures.

Table 9
Comparison of properties of asphalt mixtures with CS with the properties of reference mixtures.

Property	S0CS15	S0CS30	B0CS15	B0CS30
Indirect tensile strength (ITS)	↔	↔	↔	↔
Water sensitivity	↔	↔	↔	↔
Freeze-thaw resistance (after three cycles)	↔	↔	↔	↔
Cracking resistance	↓	↓	↑	↑
Rutting resistance	↑	↑	↓	↓
Stiffness 5, 10, 20 °C	↑	↔	↔	↓
Stiffness 30, 40 °C	↑	↑	↓	↓
Leaching	↔	↔	-	-
Fatigue resistance	-	-	↔	↓

↑ - indicates an improvement in the property relative to the reference mixture.
↔ - indicates a property similar to the reference mixture.
↓ - indicates a worsening of the property relative to the reference mixture.

5. Conclusions

Two types of asphalt mixtures, one for the surface layer for road pavements carrying moderate and low traffic loads (AC11 surf 50/70) and another one for the base layer (AC22 base 50/70) for road pavements carrying heavy traffic loads were prepared using copper slag (CS) with different fraction sizes (0/8 mm and 0/16 mm), and in different proportions (15 and 30%). In a range of laboratory tests, their performances were then compared with those of reference asphalt mixtures made entirely of virgin materials. The main conclusions of the study are as follows:

- Before using CS in asphalt mixtures, it is crucial to investigate the following properties: (i) water absorption, since this affects OBC; (ii) shape index, which has a significant effect on aggregate interlocking and consequently on stiffness; (iii) strength and rutting resistance; and (iv) adhesion between CS and a bitumen, since this may lead to water sensitivity issues.
- The tests results showed that, based on the Marshall mix design method, asphalt mixtures with a CS content of 15% contain the same, or even less, bitumen than the control mixtures, whereas mixtures with a 30% CS content require an additional bitumen content of 0.2% and 0.3% compared to the reference mixtures for the surface and base layers respectively.
- Considering a broad range of both mechanical and chemical properties of asphalt mixtures, it can be concluded that the addition of CS does not compromise these properties, and where it does, not to a significant extent.

Overall, the conclusion can be drawn that CS has a good potential for use as a substitute for a significant proportion of the virgin aggregate in asphalt mixtures, although the use of fractions coarser than 0/8 mm should be avoided because of the possible heterogeneity in such mixtures.

To assess the extent to which this conclusion can be further generalised, the low temperature cracking resistance of mixtures incorporating CS should be determined. Future work on this subject will focus on assessing the environmental and economic performance of pavement structures incorporating asphalt mixtures as tested in the present study.

CRedit authorship contribution statement

Marko Orešković: Conceptualization, Methodology, Formal analysis, Investigation, Visualization, Writing – original draft. **João Santos:** Writing – original draft, Writing – review & editing, Conceptualization, Supervision. **Goran Mladenović:** Resources, Writing – review & editing. **Vladana Rajaković-Ognjanović:** Resources, 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.

Data availability

Data will be made available on request.

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Standards

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- [2] ASTM C88-13 — Standard Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate.
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- [20] EN 1427:2017 — Bitumen and bituminous binders – Determination of the softening point – Ring and Ball method.
- [21] EN 933-1:2013 — Tests for geometrical properties of aggregates – Part 1: Determination of particle size distribution – Sieving method.
- [22] EN 933-8:2016 — Tests for geometrical properties of aggregates – Part 8: Assessment of fines – Sand equivalent test.
- [23] EN 933-9:2014 — Tests for geometrical properties of aggregates – Part 9: Assessment of fines – Methylene blue test.
- [24] EN ISO 11885:2009 — Water quality – Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES).