

## Article

# Laboratory Evaluation of Porous Asphalt Mixtures with Cellulose Ash or Combustion Soot as a Filler Replacement

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**Abstract:** Porous asphalt (PA) mixtures have become a common and very useful pavement technology for preventing dangerous driving conditions in wet weather and as a permeable surface in permeable pavement systems due to their capacity to filter water. The increased social awareness of sustainability matters has expanded the interest in reducing the impact of construction materials on the environment, mainly by using recycled materials in their composition and, hence, reducing the depletion of raw materials. In this research, two innovative recycled filler materials, namely, cellulose ash (CA) and combustion soot (CS), have been used as a total filler replacement in PA mixtures to look for improved mechanical strengths and characteristics. Four different filler dosages were assessed for each filler material, and the produced PA mixtures were tested for their particle loss, water sensitivity, freeze-thaw durability, Marshall stability, resilient modulus and permeability in order to obtain a full picture of their performance. The obtained results showed that both filler materials, adequately dosed, can generate PA mixtures that significantly overcome reference mechanical values for PA mixtures that maintain adequate infiltration capacities to satisfy the main international standards, proving to be suitable replacements for conventional filler materials commonly used in asphalt mixture production.

**Keywords:** porous asphalt; permeable pavements; sustainable pavements; sustainable urban drainage systems; SUDS; recycled filler; cellulose ash; biomass soot



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**Citation:** Andrés-Valeri, V.C.; Muñoz-Cáceres, O.; Raposeiras, A.C.; Castro-Fresno, D.; Lagos-Varas, M.; Movilla-Quesada, D. Laboratory Evaluation of Porous Asphalt Mixtures with Cellulose Ash or Combustion Soot as a Filler Replacement. *Sustainability* **2023**, *15*, 15509. <https://doi.org/10.3390/su152115509>

Academic Editor: Syed Minhaj Saleem Kazmi

Received: 5 October 2023

Revised: 26 October 2023

Accepted: 28 October 2023

Published: 1 November 2023



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## 1. Introduction

In the recent years, flood risks have attracted the attention of academia due to the great impact that flood events have on the society, particularly in urban areas, where they can generate economic losses, amenity disturbance and other environmental impacts. Some authors have quantified the economic losses due to flooding effects to be more than USD 140 billion in the US during the period 1970–2006 [1]. In order to alleviate these problems, some authors look for solutions that can help to reduce the effects of stormwater in the building environment. It is within this framework where sustainable drainage techniques have arisen as a powerful tool to improve stormwater management, providing a set of techniques that can be used all over the building environment in order to promote stormwater retention and infiltration while providing water quality treatment to polluted runoff waters [2]. Permeable pavements (PPs) are a special pavement technology that allows for water infiltration through the whole pavement structure, providing source control for rainwater. Stored water in PPs can later infiltrate into the soil for groundwater recharge or can be retained for delayed drainage purposes [3].

Permeable pavements are multi-layered pavement structures that are basically made up of a permeable surface placed over a reservoir layer, also called sub-base layer, that must provide enough porosity to allow water storage and enough bearing capacity to support traffic loads applied over the pavement structure [3]. There are many permeable surfaces that can be used in PP structures: concrete pavers, porous concrete, grass reinforced surfaces, etc. [3]. However, since most exterior pavements are asphalt, due to its lower price, one of the most commonly used and well-known permeable surfaces in permeable pavements is porous asphalt (PA). PA materials form a special type of pavement surface that is basically composed of a mineral skeleton coated by a bituminous binder, which possesses great potential for application in both conventional pavements and trafficked permeable pavement systems [3]. PA friction courses have been used during the last two decades as a surface layer in conventional pavements where they showed effectiveness in managing rainwater and reducing dangerous driving conditions during wet weather [4,5]. This pavement material is mainly characterized by its open structure with high void ratios, which provide infiltration paths for rainwater and allow water infiltration through the pavement. This open structure helps to drain water from the pavement surface, increasing pavement skid resistance and road safety by reducing hydroplaning, splash and spray and reducing visibility loss in wet weather conditions when used in conventional pavement systems.

In spite of the great benefits of this material as a pavement surface layer, it has a short service life, normally less than 10 years [6], limiting its application to very specific uses. The open structure of PA mixtures and their low fine content limit the bituminous binder content that can be added to the mixture in order to prevent draindown problems that give rise to the reduced hydrological functionality of PA surfaces. The limited binder content together with the open structure of the material has led to its reduced pavement durability. The shear stresses produced by vehicle movement produce raveling damage due to the open structure of the material [7] and the reduced stone-on-stone contact and thin binder films over mineral aggregates [8]. Additionally, the successive application of vehicle loads can cause excessive plastic deformations [9], resulting in rutting and fatigue damage.

On the other hand, aging is considered a functional failure of asphalt pavements, and it is associated with the main failures produced in asphalt-wearing courses, especially in PA friction courses. The mechanisms behind asphalt-mixture aging are related to chemical, mechanical, physical and rheological changes in the bituminous binder [10]. These changes begin during the fabrication and construction of asphalt pavements (short-term aging) and extend along the pavement service life (long-term aging) mainly due to climatic and environmental conditions that produce the progressive oxidation of asphalt mixtures [10–12]. Chemical alterations produced during mixture aging point to modifications in the bituminous-binder molecular structure increasing their stiffness and cracking potential [10]. PA mixtures are affected by severe long-term aging due to their open structure, which allows binder oxidation through the whole pavement structure [13], resulting in premature aging [7]. Additionally, other environmental conditions, such as solar radiation, contribute to the aging of PA mixtures, making them brittle and increasing their raveling potential [14]. For all these reasons, the mechanical capacity and durability of PA surfaces have traditionally been questioned [15].

Much research has been conducted to overcome these limitations, giving way to a new generation of porous asphalt mixtures with improved characteristics. The literature reviewed showed that the aggregate characteristics, binder type and properties and the additions used are key factors in the properties of the resulting PA mixtures. In fact, different authors have reported important benefits by replacing conventional or polymer-modified bituminous binders with high-viscosity bitumen and by including various types of fibers and additives in PA mixes [16]. Other authors have looked for natural fibers for reinforcing PA mixes, reducing draindown, particle losses and moisture sensitivity [17].

It is widely accepted that the overall performance of asphalt pavements is dependent on the intrinsic properties of the bituminous binders and mastics [18,19], the bituminous mastic being the combination of bituminous binder and mineral filler which ultimately

favors the agglomeration of coarse aggregates in hot-mix asphalt [18]. Additionally, as reported in previous works [18], the filler–bitumen interaction is relevant to the properties that most influence asphalt pavement failures, so the filler plays a fundamental role in pavement functionality. The addition of mineral filler to asphalt mixtures has been traditionally used to improve their mechanical strength and durability, increasing the mastic stiffness and the aggregate coat and improving the adhesiveness between the aggregates and the binder. The reinforcing effect of filler materials in asphalt mixtures has been studied recently by some researchers [20,21]. Different studies pointed out that specific surface area as well as composition are key characteristics that influence the filler effectiveness on asphalt performance [22,23]. Some recent works report that the reinforcing effects of filler particles on asphalt binder include volume-filling effects, physicochemical interactions and filler–particle interactions [21,24]. The importance of reinforcement effects by filler particles on asphalt mixtures depends on the filler concentration in the bituminous mastic [21]: for low filler concentrations, the volume-filling effect is the main improvement mechanism, while asphalt–filler interfacial interaction and filler–particle interaction effects play important roles for medium and high filler-volume concentrations, respectively.

Numerous studies have evaluated the potential use of different materials as contribution fillers in asphalt mixtures. These materials include cement, calcium hydroxide, calcium carbonate or vegetable filler [23]. The increased social awareness of sustainability matters has led to an increased interest in reducing residues, reusing them and giving them a second life. For this reason, in recent years, some authors have attempted to use residues and byproducts as replacement raw materials in asphalt pavements. Some authors attempted to use recycled glass fiber from wind turbine blades in asphalt mixtures, showing that this can provide interesting benefits to the bituminous binder and reduce pavement deformation and rutting effects [25]. Other authors looked for recycled filler alternatives in asphalt mixtures, like phosphorus tailing powder, obtaining interesting benefits in terms of resistance to water damage [26]. Bottom ashes from different industries have been also used in asphalt mixtures with interesting results [10,23,27–31]. For instance, bottom ash from coal-fired powerplants was shown to be a suitable replacement for fine aggregates in asphalt concrete due to their lipophilic molecules, which react with asphalt cement and develop effective asphalt films, enhancing the bond strength between aggregates and the asphalt cement [31]. Other studies showed that the use of bottom ash from biomass combustion as a filler replacement can provide excellent characteristics to dense graded asphalt mixtures. This is due to its calcium-rich composition and its absorption by aggregate particles in asphalt mixtures, which prevent the premature separation of the aggregate–bitumen interface [18], leading to stronger mixtures because of the increased bonding between aggregates and the bituminous binders [10,23,32]. Bottom ash from the cellulose industry, named cellulose ash (CA), is a common residue produced during the combustion of cellulose products by incineration processes during the pulp-mill production, mainly from burning bark and chipping wood to generate energy for biomass boilers [23]. Due to its nature as the residue with the second greatest contribution to total industrial solid waste (ISW), it is considered a potential pollutant waste product and is actually disposed of in authorized ISW landfills [10]. Results obtained in previous research [10,23,32], together with the composition and particle size of CA, suggest that it can be a potential filler replacement material in PA mixtures as well as a potential candidate to be used for concrete production or soil stabilization.

Some authors have also attempted to use carbon-based byproducts in bituminous mixtures and showed that their use can be an efficient way to improve bituminous mastic properties, providing increased strength and anti-aging properties to bituminous mixtures with limited cost increments in relation to the polymer modification of asphalt binders [19]. Different studies [19,33] have pointed out that some carbon-based byproducts, like carbon black, have a physical and chemical affinity with the asphaltene and maltene constituents of bituminous binders and fortify the carbonyl functional group bonds of asphalt binders, leading to stronger asphalt mixtures. Biomass combustion soot (CS) is a cluster composed

of a large number of carbon particles and is mainly produced through the incomplete combustion process using fossil fuels and open biomass [34]. Some researchers pointed out that soot is the second largest contributor to the greenhouse effect, only surpassed by carbon dioxide [34,35], due to its high radiative-forcing potential. It is also the cause of significant environmental and health concerns [36], mainly because it is a major source of particulate matter (PM) in the atmosphere [37]. Even if soot is composed of air-borne particles, it commonly settles in the chimneys of biomass combustion systems (wood stoves or wood burners for residential heating), forming a layer of carbon-based particles that progressively reduce the cross-section of the chimney. To solve this problem, several times during the year, maintenance is required to eliminate the soot residues settled in the chimneys by brushing them and collecting the brushed material. Residential biomass combustion soot is a significant component of global anthropogenic combustion emissions and, in fact, once collected during the maintenance operation of wood stoves, it is directly thrown into landfills without a recycling process. Previous works indicate that its carbon-rich composition can provide anti-aging benefits and increased strength to asphalt mixtures, and the very fine particle sizes of CS suggest its potential reuse as a filler replacement in PA mixtures.

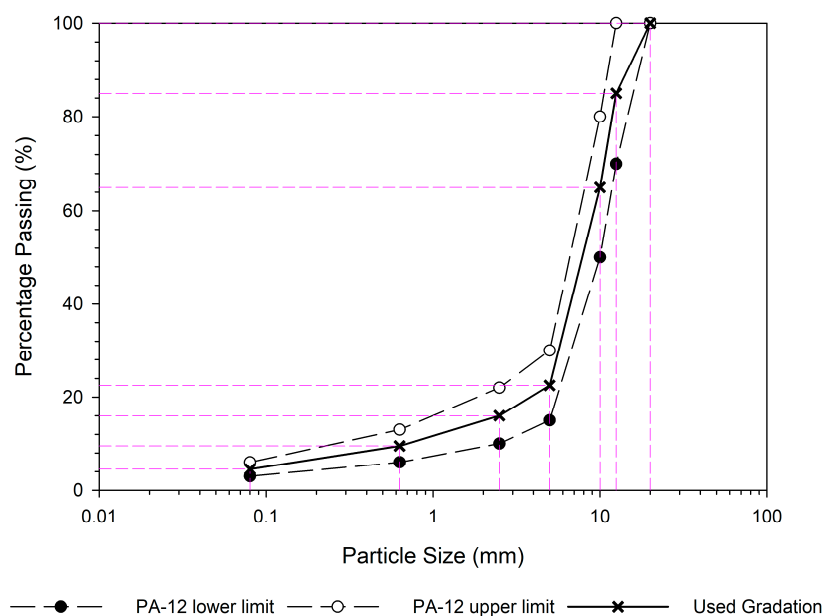
CA and CS are very common residues in a number of countries, where they can cause significant environmental impacts because they are normally thrown into landfills. For this reason, authors worldwide have attempted to reuse them in construction materials like asphalt mixtures as replacement raw materials [10,19,23,32,38]. Previous research showed that both materials can be suitable filler replacements in asphalt mixtures, but the prediction of their performance in PA mixtures has yet to be empirically validated. This study aims to evaluate the influence of conventional mineral filler replacement by CA and CS fillers on the mechanical and functional performance of PA mixtures. Experimental PA mixtures were designed and manufactured with different dosages of these filler materials and the obtained mixtures were tested for draindown and for their particle loss, water sensitivity, freeze–thaw durability, Marshall stability and resilient modulus. Additionally, void content and permeability tests were conducted on all mixtures in order to assess their hydrological functionality.

## 2. Materials and Methods

### 2.1. Aggregates and Bituminous Binder

Natural crushed gravel with a density of 2797 g/cm<sup>3</sup> and locally available at the Los Rios Region (Chile) was used in this research, with a gradation fitted to the center of the gradation band for a PA-12 mixture in accordance with the Roads Manual of Chile [39]. The used gradation can be seen in Figure 1, and it is characterized by a maximum aggregate size of 19 mm (3/4") and a nominal maximum aggregate size of 12.5 mm (1/2").

A conventional CA-24 bituminous binder dosed at 5% by mixture weight was used in this research, and its characteristics are shown in Table 1. The binder dosage was fixed at 5% by mixture weight as this is the suggested amount of bitumen recommended in Chile for PA mixes. It should be noted that the Chilean Roads Manual [39] indicates that polymer-modified bituminous binders (PMBBs) should be used in porous asphalt mixtures. However, in order to assess only the filler influence and to avoid potential synergetic, antagonistic, additive or dependent effects due to the combination of other additives, a conventional bitumen was used. Moreover, as a conventional bituminous binder produces weaker mixtures, the effect of the tested alternative materials in the mixture performance should be more significant than by using a PMBB. Additionally, by using conventional bitumen, it was possible to directly assess if the tested recycled filler materials could produce PA mixtures that meet the regulatory requirements in Chile without using PMBBs and, thus, at a lower construction cost. It is expected that by using PMBBs, the tendencies would be similar to those observed in this research, even if it remains to be evaluated in further research.



**Figure 1.** The PA-12 gradation band and the used gradation fitted to the center of the PA-12 gradation band.

**Table 1.** Properties of the used bituminous binder.

Properties	Standard	Result
Viscosity at 60 °C, 40,000 Pa (Pa·s)	EN 12596 [40]	303.9
Penetration at 25 °C, 100 g, 5 s (0.1 mm)	EN 1426 [41]	59
Softening Point, R & B (°C)	EN 1427 [42]	52.6
Ductility 25 °C (cm)	EN 13589 [43]	>150
Density (g/cm <sup>3</sup> )	EN 15326 [44]	1035

## 2.2. Filler Materials

### 2.2.1. Mineral Filler

Mineral filler was used as a reference in order to assess the potential of the experimental filler materials to be used in PA mixtures. Analysis of the chemical composition of the used mineral filler showed that it is rich in siliceous dioxide (SiO<sub>2</sub>), aluminum oxide (Al<sub>2</sub>O<sub>3</sub>), calcium oxide (CaO) and iron (Fe) and was obtained by sieving natural crushed gravel used as a coarse aggregate. As limestone powder is not locally available, and road companies do not normally use limestone powder in the south of Chile to control construction costs, this mineral filler is normally used in spite of the low expected affinity with the bitumen due to the presence of silicon dioxide in its composition. The used mineral filler showed a density of 2.797 g/cm<sup>3</sup>, and its particle size distribution can be seen in Table 2.

**Table 2.** Particle size distribution of filler materials.

Sieve Size (μm)	Percentage Passing		
	Mineral Filler	Cellulose Ash	Combustion Soot
80	100.0%	100.0%	98.8%
63	99.7%	99.2%	86.6%
53	97.0%	94.5%	72.2%
45	90.6%	86.3%	59.7%
38	82.8%	72.0%	49.1%
32	76.7%	50.1%	37.2%
25	62.6%	27.5%	24.4%
20	52.9%	12.9%	15.8%



### 2.2.2. Combustion Soot

The biomass combustion soot used in this research was shown to have a density of 1310 g/cm<sup>3</sup> and was collected by cleaning the chimneys of residential biomass combustion systems, commonly named as wood burning stoves or wood burners. The obtained material was later homogenized and sieved to meet the particle size necessary to be used as a filler material. Analysis of the chemical composition showed that it is mainly composed of ferric oxide (Fe<sub>2</sub>O<sub>3</sub>), calcite (CaCO<sub>3</sub>), carbon (C) and calcium (Ca). In Table 2, the particle size distribution of the CS filler used can be seen.

### 2.2.3. Cellulose Ash

The cellulose ash used in this research showed a density of 2480 g/cm<sup>3</sup> and were provided by a local biomass and cellulose production company. Analysis of the chemical composition of the filler materials showed that the CA used was rich in calcium oxide (CaO), aluminum oxide (Al<sub>2</sub>O<sub>3</sub>), calcite (CaCO<sub>3</sub>) and sylvite (KCl). In Table 2, the particle size distribution of the CA filler used can be seen.

### 2.3. Filler Dosage

The filler was dosed according to the selected gradation for all mixtures. In PA mixtures with filler replacement, the method summarized in IRAM 1542 [45] was used for the filler dosage. The method is based on determining the critical concentration ( $C_S$ ) and the volumetric concentration ( $C_V$ ) of the filler materials. Critical concentration ( $C_S$ ) is an essential parameter when determining the quantity (volume) of filler to use in a mixture and implicitly considers other parameters like specific surface area and surface texture [32]. The volumetric concentration ( $C_V$ ) has been defined by some authors as “the volume of filler per unit volume in the filler/bitumen system” [46]. The deformation capacity of the filler/bitumen system decreases when a certain ratio between both materials is exceeded, and a certain amount of viscosity between the filler particles is produced that makes the system behave as a rigid solid [32]. Values of  $C_V$  and  $C_S$  were calculated according to what is specified in IRAM 1542 [45] by using Equations (1) and (2).

$$C_S = \frac{M}{V_F \cdot \rho} \quad (1)$$

$$C_V = \frac{V_F}{V_F + V_B} \quad (2)$$

where  $M$  is the filler weight (g),  $\rho$  is the filler density (g/cm<sup>3</sup>),  $V_F$  is the filler volume (cm<sup>3</sup>) and  $V_B$  is the bitumen volume (cm<sup>3</sup>).

Once the  $C_S$  is calculated, the filler dosage is obtained by fixing values for the relationship  $C_V/C_S$ . According to IRAM 1542 [45], the  $C_V/C_S$  relationship should be lower than 1 in order to conserve the viscous deformation capacity of the mixture [23,32]. Furthermore, 4 different  $C_V/C_S$  relationships were selected for this research: 0.50, 0.75, 1.00 and 1.25. In Table 3, the results of applying this dosage method to both filler materials are shown.

**Table 3.** Results of applying IRAM1542 to the CA and CS fillers.

Filler	Density (g/cm <sup>3</sup> )	$C_S$	$C_V/C_S$	$C_V$	Filler Content (% b/w of Mixture)
CA	2.48	0.22	0.50	0.1100	1.391
			0.75	0.1650	2.205
			1.00	0.2200	3.118
			1.25	0.2750	4.148

**Table 3.** emphCont.

Filler	Density (g/cm <sup>3</sup> )	C <sub>S</sub>	C <sub>V</sub> /C <sub>S</sub>	C <sub>V</sub>	Filler Content (% b/w of Mixture)
CS	1.31	0.45	0.50	0.2250	1.636
			0.75	0.3375	2.836
			1.00	0.4500	4.480
			1.25	0.5625	6.866

#### 2.4. Experimental Design

In total, 8 experimental PA mixtures and 1 reference PA mixture were developed in this research. For each mixture, 32 cylindrical test samples (Marshall samples) of 102 mm diameter, 65 ± 0.5 mm height and compacted at 50 blows per side by a Marshall compactor were manufactured and used in the tests performed in this research (Figure 2). A synthetic description of each PA mixture developed in this research can be seen in Table 4. The reference mixture was developed with a C<sub>V</sub>/C<sub>S</sub> relationship of 1, because according to IRAM 1542 and previous research [10,23,32], this dosage ensures the viscous behavior of the asphalt mastic, avoiding over- and under-filling effects, so it can be considered an optimal dosage of filler in the mixture.

**Figure 2.** Cylindrical asphalt sample (Marshall sample).**Table 4.** Matrix of experimental mixtures.

Mixture	Filler Type	Bitumen Content (% by Weight of Mixture)	Filler Content (C <sub>V</sub> /C <sub>S</sub> )
REF	Mineral Filler	5	1.00
CS0.50	Combustion Soot	5	0.50
CS0.75	Combustion Soot	5	0.75
CS1.00	Combustion Soot	5	1.00
CS1.25	Combustion Soot	5	1.25
CA0.50	Cellulose Ash	5	0.50
CA0.75	Cellulose Ash	5	0.75
CA1.00	Cellulose Ash	5	1.00
CA1.25	Cellulose Ash	5	1.25

## 2.5. Methods

All PA mixtures were tested to obtain their bulk density and air-void content by the geometrical–gravimetric method. Draindown tests were conducted for all the mixtures, but no draindown was detected (<0.1% b/w). Additionally, a series of mechanical and functional tests were used to assess the mixtures' performance.

### 2.5.1. Particle Loss (PL)

The particle loss test is a measurement of the raveling potential of the mixture. Particle loss in the PA mixtures was assessed by the Cantabro test, according to the European norm EN 12697-17 [47]. To perform this test, cylindrical specimens were introduced into a Los Angeles drum without steel balls for 300 gyrations. Comparing the sample weight before and after being introduced into the Los Angeles drum, it is possible to obtain the particle loss according to Equation (3).

$$PL = \frac{W_1 - W_2}{W_1} \cdot 100 \quad (3)$$

where  $PL$  is the particle loss of the sample (%),  $W_1$  is the initial sample weight (grams) and  $W_2$  is the weight of the sample after being tested (grams).

In order to also evaluate the influence of water in the particle losses of the tested PA mixtures, this test was performed in dry (four specimens) and wet (four specimens) conditions. In dry conditions, test samples were conditioned at 25 °C for 24 h prior to testing. In wet conditions, samples were conditioned in a water bath at 60 °C for 24 h; later, samples were conditioned in air for another 24 h at 25 °C prior to testing.

### 2.5.2. Water Sensitivity

The water sensitivity test aims to assess the influence of water on the aging of PA mixtures. The test was developed according to method A that is described in the European norm EN 12697-12 [48]. Cylindrical test samples were used and evaluated by the indirect tensile strength (ITS) test according to EN 12697-23 [49]. In total, 8 samples were tested per PA mixture: 4 in dry conditions and another 4 after being conditioned in a water bath at 40 °C for 72 h. Dry and wet test samples were conditioned in air for 24 h at 20 °C prior to testing. Once the ITS was obtained in both conditions (wet and dry), the indirect tensile strength ratio (ITSR) can be calculated by using Equation (4).

$$ITSR_{water} = \frac{\frac{\sum_{i=1}^N ITS_{wet_i}}{N}}{\frac{\sum_{j=1}^N ITS_{dry_j}}{N}} \cdot 100 \quad (4)$$

where  $ITSR_{water}$  is expressed as a percentage (%) and represents the percentage of the dry resistance to indirect tensile loads that is conserved after mixtures age.  $ITS_{dry}$  and  $ITS_{wet}$  are the resistances to indirect tensile loads in dry and wet conditions (respectively) and are expressed in MPa.

### 2.5.3. Freeze–Thaw Durability

Freeze–thaw durability was assessed through the ITS test, according to AASHTO T283 [50]. Four cylindrical specimens of 102 mm diameter and compacted by 50 blows per side were submerged and conditioned at −18 °C for 24 h. After that, specimens were placed in a water bath for 24 h at 60 °C and finally conditioned at 25 °C in the water bath prior to testing. After being conditioned, specimens were tested to obtain their ITS, which was compared with their ITS in dry conditions in order to obtain the  $ITSR_{freeze}$  according to Equation (5).

$$ITSR_{freeze} = \frac{\frac{\sum_{i=1}^N ITS_{freeze_i}}{N}}{\frac{\sum_{j=1}^N ITS_{dry_j}}{N}} \cdot 100 \quad (5)$$



where  $ITSR_{freeze}$  is expressed as a percentage (%) and represents the percentage of the dry resistance to indirect tensile loads that is conserved after mixtures age.  $ITS_{dry}$  and  $ITS_{freeze}$  are the resistances to indirect tensile loads in dry and freezing conditions, respectively, and are expressed in MPa.

#### 2.5.4. Marshall Test

The Marshall stability and fluency test is a basic laboratory test for asphalt mixtures that aims to assess the maximum load that can be applied over the pavement without causing plastic deformations (stability) and the maximum deformation that the pavement suffers at this maximum load (flow). Finally, the Marshall Quotient, also called the Marshall stiffness, is calculated by dividing the stability by the flow values in each mixture. The test was developed according to the European norm EN 12697-34 [51]. Four cylindrical specimens were manufactured for each mixture. Specimens were conditioned at 60 °C in a water bath for 40 min prior to testing. As the Marshall stability test is not mandatory for PA mixes in Chile and it is not used for mix design purposes, only stability, flow and stiffness were calculated.

#### 2.5.5. Resilient Modulus

The resilient modulus is the most critical variable to mechanistic design approaches for asphalt structures. It is the measure of asphalt response regarding dynamic stresses and their corresponding strains. The test is a non-destructive assay for investigating the recoverable strain of a mixture under repeated stress. Results with a high resilient modulus value indicate that the asphalt mixture is stiff and that the recoverable strain from repeated vehicle loads is smaller. The resilient modulus was assessed according to the method described in Annex C of the European norm EN 12697-26 [52]. This test aims to obtain the elastic stiffness of asphalt mixtures against cyclic diametral compressive loads of 250 ms duration and 3000 ms of relaxation at 22 °C. This temperature was selected because it is the typical air temperature in the south of Chile; hence, we considered it adequate for performing this test. The result of the test is the resilient modulus, expressed in MPa, and represents the maximum cyclic load which causes only elastic deformations in the mixture and hence is the maximum repeated load that can be applied over the pavement without causing plastic deformations. The test was conducted on four samples of each mixture, and one measurement was made for each sample.

#### 2.5.6. Permeability

The permeability test helps to assess the mixture's capacity to filter water and is a primary functional test for PA mixes. In this research, vertical permeability was assessed according to the European norm EN 12697-19 [53] by measuring the infiltrated water through the test samples. Four specimens were used per mixture, and one measurement was made for each sample. Specimens were placed in a constant head permeameter and perimetrically sealed. The water head was maintained at 30 cm over the specimen surface and the infiltrated water was collected for 60 s. The schematic of the test device is shown in Figure 3.

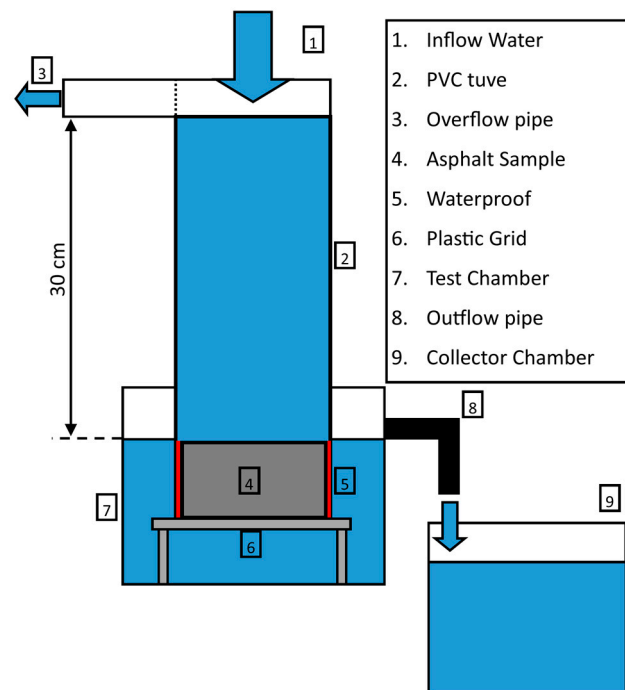
Vertical flow is calculated by using Equation (6), and the permeability coefficient was finally calculated according to Equation (7).

$$Q_v = \frac{(m_2 - m_1)}{t} \cdot 10^{-6} \quad (6)$$

$$K_v = \frac{4 \cdot Q_v \cdot L}{h \cdot \pi \cdot D^2} \quad (7)$$

where  $Q_v$  is the vertical flow through the sample expressed in ( $m^3/s$ );  $m_1$  and  $m_2$  are the weights of the collector chamber before and after the test, expressed in grams;  $t$  is the time taken for the water to infiltrate the sample, expressed in seconds;  $K_v$  is the permeability

coefficient, expressed in (m/s);  $L$  is the sample height in meters;  $h$  the water head over the sample surface in meters and  $D$  is the sample diameter in meters.



**Figure 3.** Schematic of the permeability test device.

### 2.6. Statistical Analysis

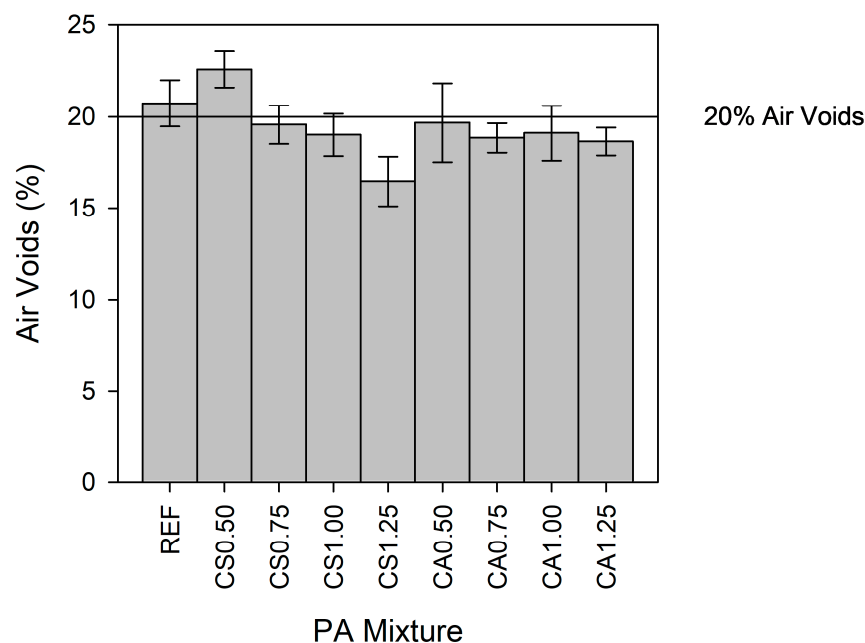
To validate the results obtained in the different tests performed in this research, a statistical analysis was carried out. On the one hand, test results were analyzed looking for the statistical significance of the differences observed among the tested mixtures by using ANOVA tests, Kruskal–Wallis H tests and Mann–Whitney U tests. Additionally, correlation analysis and ANCOVA tests were performed to assess the influence of air voids in particle loss results.

## 3. Results and Discussion

### 3.1. Air-Void Content

As can be observed in Figure 4, the average air-void content of the tested mixtures ranged from 16.5 to 22.6%, which are in agreement with previous research works [54–56]. The reference mixture showed average air voids of 20.8%, slightly higher than the values collected in the Chilean Roads Manual (5th Volume) [39] for PA mixtures (>20%). In the experimental mixtures, higher air-void contents were observed for the mixtures with lower filler dosages: CS0.50 and CA0.50, where the average air voids were 22.6% and 19.2%, respectively, but the differences were only statistically significant for mixture CS0.50, which showed significantly higher air voids than the rest of the mixtures (Sig = 0.000). As the filler dosage increases, the air-void content decreases, as expected, due to the progressive closeness of the porous matrix. In the case of CS, the variation in the filler dosage generates absolute differences in the void content of up to 6% between  $C_V/C_S$  ratios of 0.50 and 1.25; thus, the CS1.25 mixture showed significantly fewer air voids than the rest of the mixtures (Sig = 0.000). Interestingly, lower variations in the air-void content were found for CA mixtures, which showed significantly fewer air voids than the reference mixture for  $C_V/C_S$  relationships higher than 0.75 (Sig < 0.002). Absolute differences in the average void content for mixtures with the CA filler replacement between the lowest and the highest filler dosages were in the range of 2%, probably due to the higher density of CA and a lower  $C_S$  value, which produce lower volumetric variations in the mixture in relation to CS. The obtained results showed that experimental mixtures with  $C_V/C_S$  ratios higher than

0.75 lead to mixtures with air-void contents that fall below the limit of 20% and, as a result, need to be validated according to their permeability in order to be useful as a permeable surface layer in pavements.



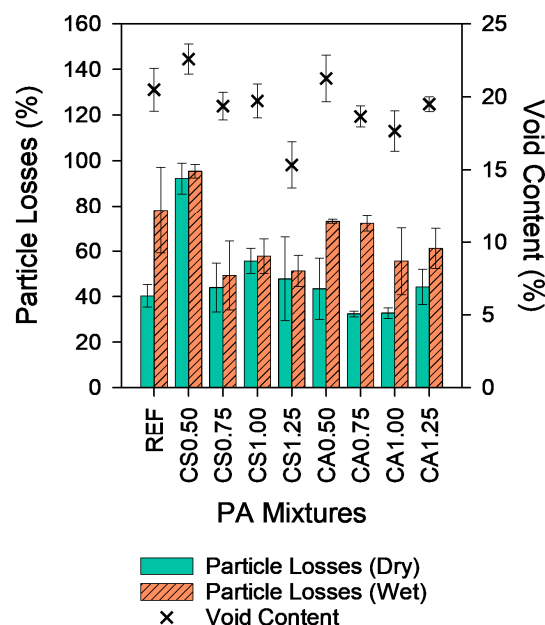
**Figure 4.** Void content for the tested PA mixtures, with error bars (N = 32).

### 3.2. Particle Losses

Particle losses in the experimental mixtures are shown in Figure 5, and the ratios of particle losses in dry and wet conditions for all the tested mixtures are presented in Table 5. As can be observed, dry particle losses in the tested mixtures ranged from 32 to 92% while wet particle losses ranged from 49 to 95%. The obtained values of particle losses are higher than those indicated in the fifth edition of the Roads Manual of Chile [39] for PA mixtures, where values lower than 20% in dry conditions and lower than 30% in wet conditions are required. These values are also slightly higher than those reported in previous research and are related to PA mixes made with a conventional bituminous binder [12,56–58] but in the range of those found in other works [59]. The low values of the Cantabro test result are probably related to the use of a conventional bitumen instead a polymer-modified one that is mandated for Chilean PA mixtures. Analyzing the obtained results, particle losses in the experimental mixtures (CS and CA) were shown to be similar to those obtained for the reference mixture, or even lower, especially in wet conditions, indicating the good performance of the filler materials used and their suitability as mineral filler replacements. A statistical analysis showed that the higher values of average particle losses in the experimental mixtures were not statistically significant in dry (Sig > 0.742) or wet (Sig > 0.557) conditions, so the particle losses are significantly similar to those obtained for the reference mixture. The wet-to-dry particle loss ratio was shown to be lower in mixtures with CS (100 to 112%) than in mixtures with CA (138 to 223%) or reference mixture (194%), indicating that the CS filler showed the best performance in protecting against the influence of water in raveling damage.

Comparing the effect of both filler materials, the results showed that CA seems to provide a better performance against raveling damage in dry conditions, leading to particle losses lower than those obtained for both the CS and reference mixtures, but these differences were not statistically significant (Sig > 0.235). Especially interesting are the results for the CA1.00 mixture, which showed 25% lower dry particle losses and 30% lower wet particle losses than the reference mixture. However, the statistical analysis revealed that these differences were not statistically significant (Sig > 0.760). In addition, CS showed a better performance in protecting against water action than CA, in that mixtures with CS

showed very similar average particle losses in wet and dry conditions for all the tested dosages, leading to a wet-to-dry particle loss ratio in the range of 100 to 112%. Additionally, in mixtures with CS, when the  $C_V/C_S$  ratio surpasses 0.75 (CS0.75, CS1.00 and CS1.25), the particle losses were shown to be very similar to those obtained by the reference mixture in dry conditions and up to 37% lower than that shown in wet conditions, even if the observed differences were not statistically significant (Sig > 0.463).



**Figure 5.** Average particle losses in dry (N = 4) and wet conditions (N = 4) and void content (N = 8), with error bars for the tested PA mixtures.

**Table 5.** Wet-to-dry particle loss ratios for the tested mixtures (N = 4).

Mixture	Wet-to-Dry Particle Loss Ratio (%)
REF	194.1
CS0.50	103.5
CS0.75	112.2
CS1.00	103.8
CS1.25	107.2
CA0.50	168.2
CA0.75	223.2
CA1.00	169.7
CA1.25	138.5

The generally reduced particle losses in wet conditions for mixtures with experimental fillers in relation to the reference mixture could be attributable to the differences in the characteristics and composition between experimental fillers and the conventional mineral filler. While CA and CS fillers were shown to be rich in calcite, which is highly alkaline, the mineral filler used was shown to be rich in silicon dioxide. This different chemical composition causes differences in filler acidity, influencing the affinity between the filler and both the bituminous binder and water, generating the observed performance. On the other hand, as the CA filler showed a lower critical concentration than CS, the filler/bitumen volumetric relationship in mixtures with the CA replacement is lower than their counterparts with CS. As a result, there is a higher amount of free bitumen in the bituminous mastic that can bond aggregate particles, which gives rise to a higher resistance to raveling damage in mixtures with the CA filler replacement.

On the other hand, the lower critical concentration and particle size of CA relative to CS and the mineral filler leads also to the higher stiffening capacity of CA compared with CS.

This can produce a higher dislodgement of the bituminous mastic and, in consequence, a higher strength against impact damage like that applied during the Cantabro test, reducing particle losses in dry conditions. This effect can also explain the higher particle losses for CA in wet conditions relative to CS. As the stiffness of the bituminous mastic is increased due to water action, in mixtures with CA, this can lead to an excessive stiffness that can ultimately result in higher particle losses in wet conditions than those observed in PA mixtures with a CS filler replacement.

Finally, analyzing the influence of air-void content in particle losses, a strong correlation between both parameters was observed. Higher air-void contents tend to bring about higher particle losses, especially in wet conditions, and these results were confirmed by a simple bi-variate correlation analysis, which showed a strong correlation between air voids and particle losses in dry ( $r = 0.580$ ) and wet ( $r = 0.675$ ) conditions. Additionally, a univariate ANOVA test showed that by considering the mixture type as a predictor of particle loss in the tested mixtures, the model performance reaches determination coefficients ( $R^2$ ) of 0.749 and 0.417 for dry and wet conditions, respectively. A further ANCOVA analysis showed that by considering air voids as a co-variable for particle loss prediction, the model performance increased to values of  $R^2 = 0.860$  and  $R^2 = 0.648$  for dry and wet conditions, respectively. This performance indicates the importance of air voids in particle loss and water damage over and above other design parameters or the used materials. Even if the statistical analysis showed significantly similar particle losses independently of the filler used due to the high dispersion of the obtained results and the low number of samples tested, these results indicate that both CA and CS, adequately dosed, can be a suitable replacement for a conventional filler, providing average particle losses similar to or even lower than those showed by the reference mixture in dry and/or wet conditions.

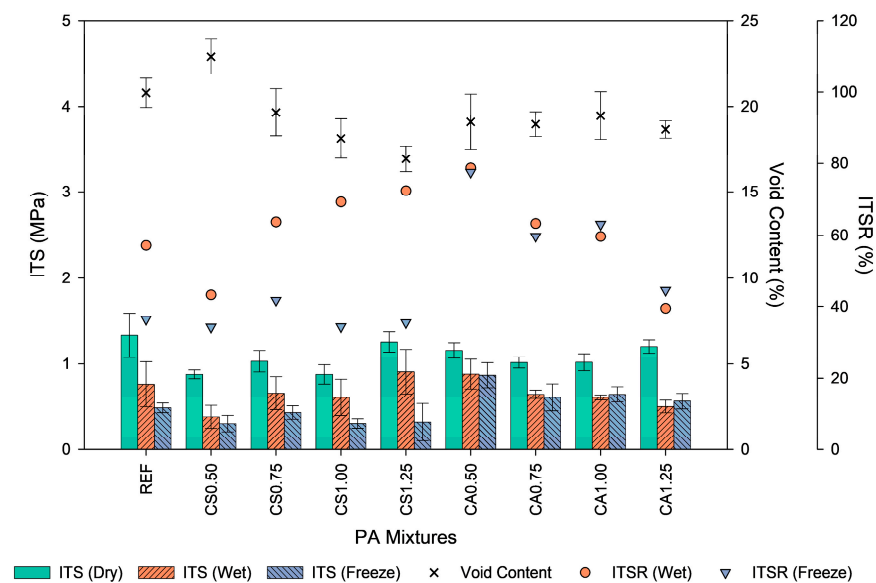
### 3.3. Water Sensitivity and Freezing and Thawing

As can be observed in Figure 6, the obtained  $ITS_{dry}$  values ranged from 0.87 to 1.33 MPa, which are in agreement with those found in the literature for PA mixes (0.21–1.80 MPa), some of which include polymer-modified bituminous binders, hydrated lime and/or cellulose fibers [56,58,60,61]. In spite of the relatively high values of ITS in relation to previous research, the ITSR values observed for all the mixes were shown to be slightly reduced relative to what has been reported by previous research [55,56,61], where ITSR values ranged from 73 to 96%, slightly higher than those obtained in this research, which ranged from 39% to 78%. Considering these results, the experimental mixtures developed in this research with the CA and CS fillers do not achieve the regulatory requirements in terms of the ITSR, whose minimum value is set at 85% in the Roads Manual of Chile (5th edition) [39]. The observed differences with previous research may be due to the different materials used, such as PMBB, different gradations and the inclusion of additives like fibers or hydrated lime, making it very difficult to perform a direct comparison with previous research results.

As shown in Figure 6, CS and CA filler materials can produce mixtures with similar  $ITS_{dry}$  results to the reference mixture, even if CA seems to generally produce slightly stronger mixtures in dry conditions. For both filler materials, the peak  $ITS_{dry}$  results were reached at dosages related to a  $C_V/C_S$  relationship of 1.25, while the averaged  $ITS_{wet}$  results were shown to be similar for both experimental filler materials. On the other hand, different performances were observed in relation to the filler dosage, depending on the filler material. The higher  $ITS_{wet}$  result for the CS filler was 18% higher than the result obtained for the reference mixture and was reached for a  $C_V/C_S$  relationship of 1.25 (CS1.25). In addition, for the CA filler, the peak  $ITS_{wet}$  result was 16% higher than that of the reference mixture and was obtained for a  $C_V/C_S$  relationship of 0.50 (CA0.50). These results are slightly different to those obtained for particle losses, probably due to the different types of efforts measured by each test. While the ITS test measures the resistance of the mixture against cracking failures and is an indicator of the adherence or cohesion among mixture components, the



Cantabro test is a durability test that indicates the toughness of the mixture; consequently, the results of both tests can be quite different.



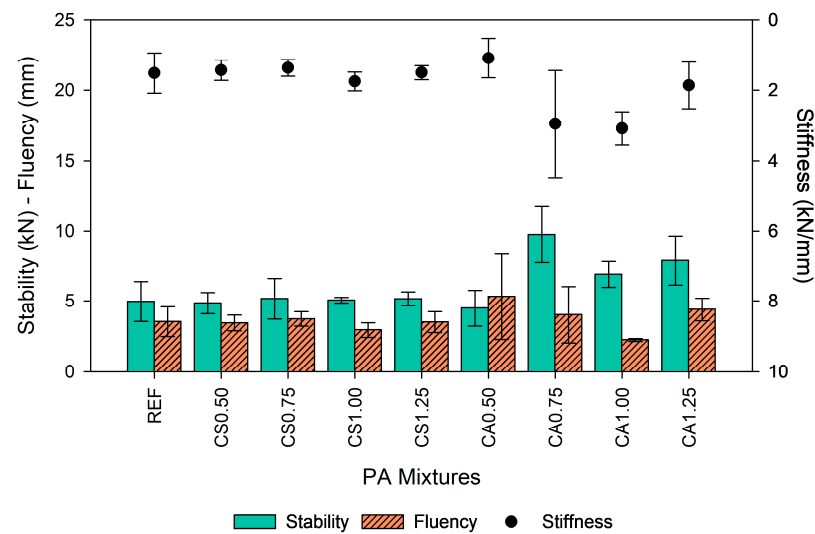
**Figure 6.** Water sensitivity ( $N_{dry} = 4$ ;  $N_{wet} = 4$ ), freezing and thawing ( $N_{freeze} = 4$ ) and void content ( $N = 12$ ) test results for the tested PA mixtures, with error bars.

Finally, even if mixtures with CA showed a better performance against freezing and thawing for all mixtures, showing  $ITS_{freeze}$  values very similar to  $ITS_{wet}$  values in all cases and 40% higher than those obtained for the reference mixture, mixtures with CS showed an average  $ITS_{freeze}$  value that was 30% lower than for the reference mixture, indicating a higher effect on these mixtures by freezing events.

Analyzing the  $ITSr$  values, it can be observed that the values obtained for the reference mixture were slightly lower than 60% in wet conditions and nearly 35% after being frozen. These values are very low but logical, considering that a conventional bituminous binder was used instead of a polymer-modified one. On the other hand, it was observed that experimental mixtures with both filler materials easily surpassed these values, reaching  $ITSr_{wet}$  values of up to 78% and  $ITSr_{freeze}$  values of up to 77% in mixtures with CA, both for mixture CA0.50, and up to 72% and 41% in mixtures with CS (mixtures CS1.25 and CS0.75, respectively). These values indicate that both filler materials can provide increased protection against water action and/or freezing relative to conventional filler materials. This is especially notable for the CA filler, which generates PA mixtures with higher ITS values in wet and freezing conditions, maintaining  $ITS_{dry}$  values in the range of the reference mixture, hence providing increased  $ITSr$  values against water and freezing.

### 3.4. Marshall Stability Test

The Marshall stability test results are shown in Figure 7 and show very similar strengths in mixtures with CS compared with those observed for the reference mixture. These were in the range of 5 KN, which were very low compared with previous research with PMBBs [62] but similar to those obtained in other works where a conventional bitumen was used [63]. The higher stability result for the CS filler was obtained by the CS1.25 mixture (5.15 KN), a value slightly higher than the result obtained for the reference mixture and the rest of the CS mixtures, but this was not statistically significant, according to the performed ANOVA test (Sig = 1.000). Something analogous occurred with the flow values and the Marshall stiffness, where very similar results were observed for the reference mixture and mixtures with CS (Sig = 1.000).

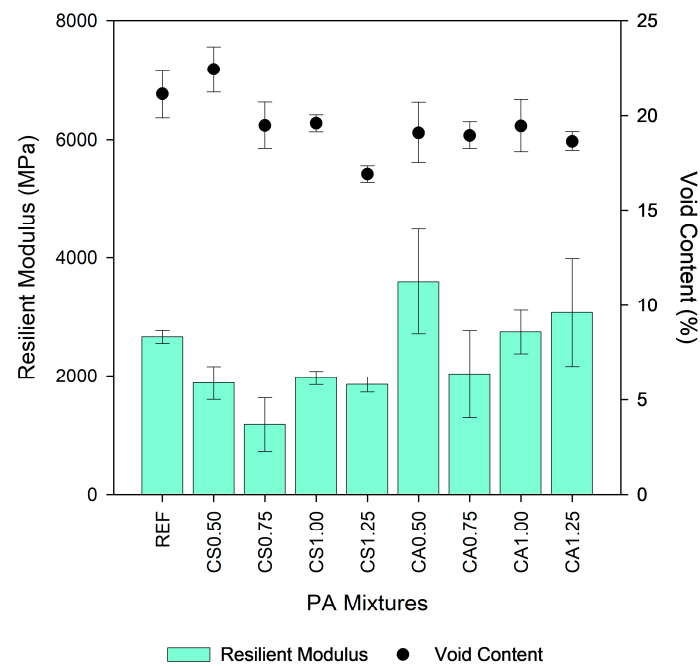


**Figure 7.** Stability, flow and stiffness obtained from the Marshall test (N = 4) for the tested PA mixtures.

On the other hand, mixtures with CA, especially those with filler dosages higher than 0.75 (CA0.75, CA1.00 and CA1.25), showed increased strengths, leading to stability values of up to 9.74 KN for mixture CA0.75, a value in the range of those obtained in previous works with PMBBs [64]. This value is 97% higher than the one obtained for the reference mixture and 90% higher than the strongest mixture with CS as the filler replacement (CS1.25 mixture). The statistical analysis revealed that the CS0.75 mixture showed significantly higher stability values than the reference and CS mixtures (Sig < 0.010). Furthermore, flow values seem to be higher also in mixtures with CA than in the reference mixture or in mixtures with the CS filler, but the statistical analysis showed that there are no significant differences among the mixtures with regard to their flow values (Sig > 0.625). Even if the Marshall stiffness is much higher in mixtures with CA, especially in the CA0.75 and CA1.00 mixtures, the statistical analysis did not show any statistically significant difference among the stiffness results (Sig > 0.222). The better performance of mixtures with a CA filler replacement relative to the rest of the tested mixtures could be due to the lower value of critical concentration for the CA filler and its lower particle sizes, which increase its stiffening potential in bituminous mastic, as this would ultimately produce mixtures with higher stability. Additionally, as the CA filler was shown to be alkaline in nature due to its composition being rich in calcite and calcium oxide, there is a better chemical affinity with the bituminous binder, which is acidic in nature, increasing the adhesion between the filler and the bituminous binder.

### 3.5. Resilient Modulus

The resilient modulus is the most critical variable to mechanistic design approaches for asphalt structures. It is the measure of the asphalt response to dynamic stresses and the corresponding strains. The results of this test for the analyzed mixtures are shown in Figure 8. As can be observed, the values of the resilient modulus ranged from 1187 to 3599 MPa, values in accordance to those found in previous research [57,65,66] for PA mixes using PMBBs.



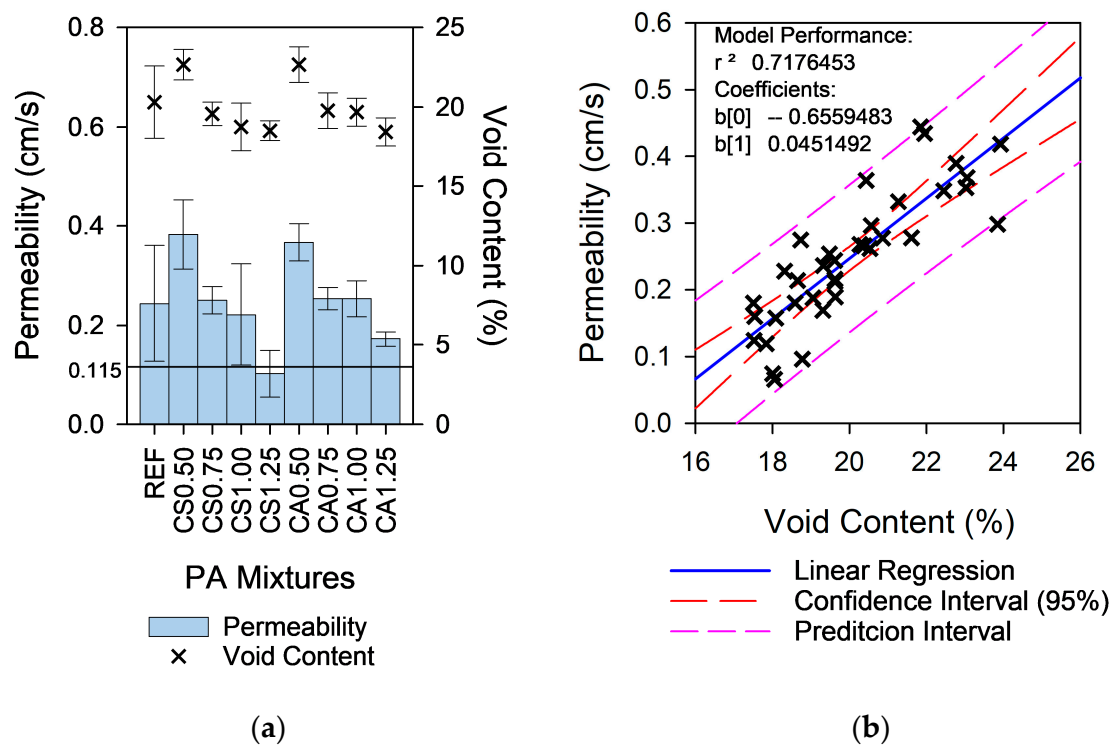
**Figure 8.** Resilient modulus test results (N = 4) for the tested PA mixtures.

Analyzing the influence of the filler material, it can be observed that CA provides higher average values of the resilient modulus than CS does for all dosages, indicating that CA produces a higher energy dissipation in the elastic range than mixtures with CS. Especially interesting are the results for  $C_V/C_S$  relationships of 0.50 and 1.25 in mixtures with the CA filler replacement (CA0.50 and CA1.25 mixtures), where the obtained resilient modulus was shown to be 35% and 15% higher (respectively) than the result obtained for the reference mixture, indicating a higher strength of these mixtures and lower deformations in the elastic range. However, the statistical analysis revealed that the observed differences with the reference mixture were not significant (Sig > 0.639), while the differences observed in relation to the CS mixtures are statistically significant (Sig < 0.050). On the other hand, the CS filler seems to reduce significantly the resilient modulus of PA mixtures relative to the reference mixture, especially mixture CS0.75, which showed significantly lower values of stiffness than the reference mixture (Sig = 0.045) indicating a lower stiffness of these mixtures and higher deformations for lower dynamic loads.

### 3.6. Permeability

Permeability values are shown in Figure 9. These were found to be strongly correlated with the void content in mixtures, as indicated by the simple linear regression analysis shown in Figure 9b. The higher the air-void content, the higher the permeability of the mixtures. As void content in the experimental mixtures was related to the filler dosage, important differences were observed for both filler materials, depending on the filler dosage, especially between mixtures with  $C_V/C_S$  relationships of 0.50 and 1.25 (Sig < 0.041). However, there are no differences in the permeability test results between both filler materials when each dosage was considered independently (Sig > 0.954).

Permeability values of the tested mixtures ranged from 0.101 cm/s and 0.382 cm/s, values in agreement with previous research [55,57,60,61], while the average void contents ranged from 18 and 22%. These values are in accordance with those obtained for PA mixtures by other authors in previous research [55,56], where permeability values normally range between 0.150 and 0.280 cm/s.



**Figure 9.** (a) Permeability test results for the tested PA mixtures ( $N = 4$ ), with error bars. (b) Regression analysis between permeability and void content in the tested PA mixtures ( $N = 36$ ).

Interestingly, even if some experimental mixtures showed air-void contents that fall below the limit of 20% for PA mixes in Chile according to the Roads Manual [39], permeability values of most of the tested mixtures were still above the limit of 0.115 cm/s (100 m/day), the value recommended for new PA mixtures offered by some road agencies and collected in the ASTM D7064-04 [67] standard.

#### 4. Conclusions

According to the results obtained in this research, both CA and CS filler materials were shown to be suitable replacements for mineral filler in PA mixtures, providing mechanical strength, functionality and durability results similar or higher than those obtained for reference PA mixtures. However, caution should be taken to consider the presence of iron oxide ( $\text{Fe}_2\text{O}_3$ ) in CS, as it can act as a catalytic chemical, leading to the stiffening of the bituminous binder as a result of its oxidative polymerization. This phenomenon can progress over time in asphalt mixtures (long-term aging), especially in PA mixes, due to their open structure, and lead to very quick damage in the form of cavities and long-term damage that must be further analyzed on the basis of the outputs of this research work.

Average dry particle losses were shown to be higher in mixtures with CS than in mixtures with CA and the reference mixture, while average wet particle losses were shown to be slightly lower in mixtures with CS than in mixtures with CA or the reference mixture. The statistical analysis revealed that no significant differences were observed. Among the tested dosages of each experimental filler material, mixtures CA1.00 and mixture CS0.75 stand out as they provide average particle losses similar to or lower than those obtained by the reference mixture in both dry and wet conditions.

Water sensitivity and freeze–thaw tests showed that mixtures with CA resulted in generally stronger mixtures than mixtures with CS and the reference mixture; as a result, they provide reduced susceptibility to cracking failure due to the action of water and freezing, especially the CA0.50 mixture.

The Marshall stability test results showed that the CA filler material generates higher stability and fluency values than the CS and reference mixtures, and hence provides a

higher cohesion in the mixture. The CA filler showed the best performance in this test, especially mixture CA0.75, a result in accordance with the obtained values of the resilient modulus, where PA mixes with CA showed higher resilient modulus values than the other PA mixes.

Permeability values in the tested PA mixes were generally higher than the minimum value of 0.115 cm/s suggested by international standards, with the exception of mixture CS1.25, even though the air-void content of the tested mixtures fell below the limit of 20% established by local regulations in Chile.

On the basis of the obtained results, the use of cellulose ash (CA) is recommended with a  $C_V/C_S$  relationship between 0.50 and 0.75 in order to produce PA mixtures with improved mechanical characteristics that maintain enough infiltration capacities according to the main international standards and high durability values.

**Author Contributions:** Conceptualization, V.C.A.-V. and O.M.-C.; methodology, V.C.A.-V. and O.M.-C.; validation, A.C.R., D.M.-Q., M.L.-V. and D.C.-F.; formal analysis, V.C.A.-V. and O.M.-C.; investigation, V.C.A.-V., D.M.-Q. and A.C.R.; resources, V.C.A.-V.; data curation, V.C.A.-V., O.M.-C. and D.M.-Q.; writing—original draft preparation, V.C.A.-V.; writing—review and editing, D.M.-Q., O.M.-C., A.C.R. and D.C.-F.; visualization, M.L.-V. and D.M.-Q.; supervision, D.C.-F., A.C.R. and O.M.-C.; project administration, V.C.A.-V.; funding acquisition, V.C.A.-V. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by the National Agency of Research and Development (ANID) of Chile through the FONDECYT Initiation program, grant number FONDECYT 11190909.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** Data sharing is not applicable to this article.

**Acknowledgments:** The authors would like to acknowledge BIOFOREST for providing the materials to perform this research.

**Conflicts of Interest:** The authors declare no conflict of interest.

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