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# Study of the Optimal Dosage of Celullose Ash as a Contribution Filler in Asphalt Mixtures Based on Its Adhesiveness under Moisture Conditions

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**Abstract:** Chile is the first Latin American country to begin an "*ecological overdraft*", as established by the Global Footprint Network (GFN). This implies that the country's ecological footprint has exceeded the global average bio-capacity. The consumption of natural aggregates for construction in Chile has grown by around 6.6% in the last year, with around 120 million tons being extracted. Given the above, it is important to seek alternatives that help to minimize the problem of resource scarcity, as well as the recovery of industrial by-products and/or waste. The Chilean forestry sector has also grown in recent years, generating approximately 4000 metric tons of waste in 2018, which was deposited in landfills or disposed of on forest roads. The present research is focused on the reuse and possible recovery of ash from the incineration of cellulose as a filler in bituminous mixtures. We analyze the adhesiveness of the filler/bitumen system in dry and wet states, based on the Cantabro wear loss test. The results obtained show that the limit of the relation between the volumetric concentration and critical concentration (*Cv/Cs*) is 1 for the addition of ash and that concentrations lower than or equal to this value present controlled losses, with 1.00 being the optimal (*Cv/Cs*) ratio that allows better behavior against the effect of water.

**Keywords:** cellulose ash; filler; bituminous mastic; adhesiveness; wear losses; damage moisture

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**Citation:** Movilla-Quesada, D.; Raposeiras, A.C.; Lagos-Varas, M.; Muñoz-Cáceres, O.; Andrés-Valeri, V.-C.; Troncoso, L. Study of the Optimal Dosage of Celullose Ash as a Contribution Filler in Asphalt Mixtures Based on Its Adhesiveness under Moisture Conditions. *Sustainability* **2021**, *13*, 854. https://doi.org/10.3390/su13020854

Received: 28 July 2020
Accepted: 13 January 2021
Published: 16 January 2021

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## 1. Introduction

In recent years, Chile has become one of fastest-growing Latin American countries in terms of population. According to the National Institute of Statistics (INE), in 2019, the country exceeded 19 million inhabitants, with 10.6% growth in the total population over the last 20 years [1]. This population increase has resulted in an increased demand for transportation and road traffic, particularly in the capital and the most populated regions, since a greater number of people are moving around daily. Specifically, in 2018, the National Automotive Association of Chile (ANAC) recorded a 10% increase in the vehicle fleet, indicating that there were a total of 5.5 million motor vehicles in the country [2]. However, this growth must be accompanied by government investment in road infrastructure, maintenance, and conservation, not to mention research, innovation, and development to promote new techniques and procedures focused on society, the economy, and the environment [3].

There are many factors that modify the current techniques used in road design, given the changes that are taking place not only in terms of technology and the economy, but also in the environment [4]. According to Greenpeace's 2020 annual report, Chile is the first Latin American country to fall into an "ecological overdraft", which indicates that no environmental saving capacity is being generated, clearly endangering the resources needed by future generations [5]. New alternatives that help to minimize the environmental problem and increase the useful life of pavements, improving their resistance to the action to the water, must be sought. An alternative to the filler from natural aggregates used in the design and construction of roads is proposed based on the valuation of a sub-product generated by the forestry sector (cellulose ash). It is important to point out that the forestry industry contributes approximately 3% to the country's gross domestic product (GDP), representing a sum of more than US$415 million [6].

The filler is the smallest portion of the aggregate that can pass through a sieve. In Chile, this has openings of 0.075 mm, occupying up to 12% by weight in the bituminous mix [7,8]. This material has a significant influence on the properties of the mixture, influencing its strength and mechanical properties and minimizing permanent deformation and cracking produced by various external factors [9,10].

The current regulations on the use of filler in bituminous mixtures establish the required properties of the material [7,8,11,12], such as the size of its particles and its plasticity, moisture level, and organic matter content, and indicate the amount of filler that should be used as a function of the filler/bitumen ratio (0.6–1.2 by weight) [13]. However, these parameters are not sufficient to obtain optimal performance of the asphalt mixture, since each by-product and waste material has specific properties, so it is necessary to perform other, more complex tests to determine the behavior of the mixture against external agents and conditions, such as water and temperature.

Several published studies have shown that the use of waste materials, such as biomass ash [13], fly ash [14], dust from electric arcing [15], marble dust [16], glass dust [17], polymer waste [18], and other industrial by-products, in optimal proportions yields good properties in terms of the performance of the bituminous mixture. In 2009, an investigation on the use of ash from municipal waste incineration and its performance in the stone matrix of the asphalt mixture was published. The results indicated that the use of this ash as a filler increased the water resistance of the mixture, in addition to improving its fatigue life [19]. It is important to note that not only has research on the behaviors of these filler materials in bituminous mixtures been conducted from a mechanical point of view, rheological parameters have also been analyzed. Zhang et al. published a study in which they compared three different types of filler (limestone, hydrated lime, and red mud) in a study with a rotary viscosimeter and a dynamic shear rheometer (DSR) to evaluate the adhesion strength of bituminous mastics. The results showed that the use of this residue increased the rigidity and elastic behavior of the mastic, improving its resistance to rutting [20].

Based on the results published in previous research conducted by the authors, ash from the incineration of cellulose can be used as filler in bituminous mixtures, not only to reduce economic costs but also to solve the environmental problem arising from the disposal of this ash in authorized landfills. Regarding the study conducted on the resistance of bituminous mixtures with cellulose ash to ageing, the results indicated that (when ash was used in an optimal dosage) this parameter increased by 3.3%, minimizing early cracking of the pavement. Subsequently, tests were conducted to study the thermal susceptibility in different climatic zones. The results indicated that based on volumetric criteria, this type of mix offers lower loss due to wearing (up to 35%) for temperatures between −10 and 60 °C compared with the reference mixes, demonstrating that cellulose ash increases the cohesiveness of the bituminous mixture [21,22].

In this research, the possibility of using cellulose ash as a filler in bituminous mixtures is analyzed. To eliminate the possible disadvantages of the disposal of cellulose ash in landfills, it is incorporated into the CA-24 binder and thick and fine aggregates to form the bituminous mixture. The dry and wet adhesiveness or wear losses produced in the bituminous mastic are obtained using the Cantabro test. The moisture damage, or the ease with which the bituminous mixture suffers degradation of its properties as a result of the effect of water, is determined from the indirect tensile strength test. Finally, the bituminous mixtures are made according to the volumetric and critical concentrations, which depend specifically on the nature of the filler.

## 2. Materials and Methods

The tests carried out in this research were divided into two parts. First, we carried out physical-chemical characterization of the materials. For cellulose ash, X-ray diffraction (XRD) tests were carried out to identify the compounds that form the ash. Secondly, a mechanical analysis of the bituminous mixtures was carried out by dry and wet abrasion tests (wear losses by the Cantabro test) and an analysis of water sensitivity by indirect tensile strength. The Cantabro method (UNE 12697-17:2018) allows evaluating the loss of adhesiveness between the mastic asphalt and the largest aggregate through mechanical wear of the asphalt mixture [23]. This test can be performed under different conditionings, including water conditioning, which allows inducing moisture damage since water has enough time to invade the filler-bitumen interface, and destroys the adhesion between the aggregate particles [24].

### *2.1. Aggregates and Bitumen*

A semi-dense IV-A-12 was used as the reference bituminous mix (Table 1). It was designated according to the Chilean Guide, Volume 5, and this nomenclature was determined according to the nominal maximum size of the aggregate, which was defined as the opening of the first sieve that retained more than 10% of the mass [23].

**Table 1.** Aggregate gradation distribution.

| Sieve (mm) | % Passed Through | % Accumulated |
|---|---|---|
| 25 | - | - |
| 20 | 100 | 0 |
| 12.5 | 80–95 | 12.54 |
| 10 | 70–85 | 22.54 |
| 5 | 43–58 | 49.54 |
| 2.5 | 28–42 | 65.09 |
| 0.63 | 13–24 | 81.63 |
| 0.315 | 8–17 | 87.63 |
| 0.16 | 6–12 | 91.00 |
| 0.08 | 4–8 | 94.00 |

The aggregates used were crushed limestone from a quarry located in the south of Chile. Table 2 shows the physical characteristics (water absorption and bulk specific density) of the aggregates. The results show that the water absorption in the different sizes analyzed did not vary significantly, showing that the largest change was 1.1% in the continuous interval of 0–5 mm. Since it is certain that aggregates have a certain amount of water in their pores, a bulk density calculation was performed, which showed an increase in water absorption as particle size increased with a maximum percentage difference of 4% between sizes of 2.5–10 mm.

**Table 2.** Physical characteristics of the aggregates.

| Size (mm) | Water Absorption (%) | Bulk Specific Density (g/cm$^3$) |
|---|---|---|
| 0–2.5 | 2.685 | 2.692 |
| 2.5–5 | 2.690 | 2.670 |
| 5–10 | 2.693 | 2.630 |
| 10–12.5 | 2.702 | 2.621 |
| 12.5–20 | 2.713 | 2.600 |

Based on the granulometry, the optimum binder content in the semi-dense mixture was determined to be 5% base binder (CA-24). According to Chilean standards, the acronym

"CA" corresponds to the definition "Asphalt Cement", and "24" means that its original viscosity will be greater than or equal to 2400 poises. The term "traditional" indicates that it is the most commonly used asphalt binder in Chile, due to its great versatility for use in the different conditions of temperature and load existing in the country [24]. Table 3 lists the traditional physical properties of the bitumen as well as the test method and limits according to the regulations. In particular, the CA-24 binder shows a penetration value similar to that of the conventional B50/70 binder used in Spain, with a consistency suitable for the applications and temperatures commonly found in Chile [23].

**Table 3.** Characteristics of the asphalt binder (CA-24).

| Property | Method | M.C. Specifications | Results for Binder Used |
|---|---|---|---|
| Absolute viscosity, 60 °C, 300 mm Hg (Pa·s) | M.C. 8.302.15 * | Min. 240.0 | 303.9 |
| Penetration, 25 °C, 100 g, 5 s (1/10 mm) | M.C. 8.302.3 * | Min. 40 | 59 |
| Ductility, 25 °C, 5 cm/min (cm) | M.C. 8.302.8 * | Min. 100 | Over 150 |
| Solubility in trichloroethylene (%) | M.C. 8.302.11 * | Min. 99 | Over 99 |
| Flash point (°C) | M.C. 8.302.9 * | Min. 232 | Over 232 |
| Softening point (°C) | M.C. 8.302.16 * | - | 50 |
| Penetration index | M.C. 8.302.18 * | −2.0 to +1.0 | −1.6 |
| Specific Gravity, 25 °C | 281-2 | - | 1.035 |

* M.C.: Chilean Roads Guide, Vol. 8 [24].

### *2.2. Cellulose Ash as a Contribution Filler*

The ash used as a filler is the product of the process of debarking and chipping pine and eucalyptus wood, which is incinerated at a high temperature (130–170 °C) in biomass boilers. The combustion of biomass generates high-pressure steam, which is then used to produce electricity for self-consumption in the plant, and the by-product of this process is ash, which is then deposited in authorized landfills.

To complement mechanical studies in asphalt mixtures, a non-destructive experimental test was carried out to characterize the microstructure of the cellulose filler. The test was performed using X-ray diffraction equipment (XRD) to identify the predominant minerals in the composition of the filler presented, and thus, to support the mechanical behavior of asphalt mixtures with ash.

#### 2.2.1. XRD Diffraction

X-ray diffraction (XRD) is a non-destructive method of solid-state characterization that allows the qualitative identification of the mineralogical composition of a crystalline sample. This technique is based on optical interference that is produced when monochromatic radiation crosses a slit of thickness comparable to the wavelength of the radiation [25].

The structural characterization of the samples was carried out with a Bragg–Brentano geometry with CuK$\alpha$ radiation ($\lambda$ = 1.5418 Å). Refinement of the crystalline structures was carried out by the Rietveld method [26] using a refinement program [27]. A pseudo-Voight function was chosen to generate the shape of the lines of the diffraction peaks. The following parameters were refined: scale factor, background coefficients, zero-point error, reticular parameters, pseudo-Voight correction for asymmetric parameters, and positio coordinates.

#### 2.2.2. Dosing of the Contribution Filler

In this study, a dosage based on the volumetric ratios of the filler to the asphalt binder was used [28]. Table 4 shows the amounts of aggregate filler and cellulose filler used for the different combinations, along with the percentage of asphalt binder added to the mix. All combinations included a total replacement of the contribution filler by cellulose filler or limestone filler. The amounts of aggregate (coarse + medium + fine aggregate, larger than the filler size) and binder remained constant for all combinations: 1083 g of aggregate (excluding the filler) and 57 g of asphalt binder.

**Table 4.** Combination of materials.

| Cv/Cs | Type of Filler | Quantity of Contribution Filler (g) | Asphalt Binder Added to Mixture (Mass %) |
|---|---|---|---|
| 0.50 | Limestone filler | 16.34 | 4.92 |
| 0.75 | Limestone filler | 25.95 | 4.88 |
| 1.00 | Limestone filler | 36.76 | 4.84 |
| 1.30 | Limestone filler | 51.66 | 4.78 |
| 1.50 | Limestone filler | 63.02 | 4.73 |
| 0.50 | Cellulose ash | 16.88 | 4.92 |
| 0.75 | Cellulose ash | 26.99 | 4.88 |
| 1.00 | Cellulose ash | 38.52 | 4.83 |
| 1.30 | Cellulose ash | 54.71 | 4.77 |
| 1.50 | Cellulose ash | 67.27 | 4.72 |

The intrinsic parameters of the cellulose ash and limestone filler, such as the critical concentration (*Cs*) and volumetric concentration (*Cv*), as well as the quantities of these contribution fillers to add to the mixture were obtained according to the Argentine standard IRAM1542 [29]. In this sense, the critical concentration (*Cs*) is an essential parameter when determining the quantity (volume) of filler to use in a mixture. Furthermore, other characteristics of the material were evaluated implicitly from this parameter, like its specific surface and surface texture [30,31]. The volumetric concentration has been defined by some authors as "the volume of filler per unit volume in the filler/bitumen system, designated by this parameter as *Cv*" [32]. 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].

### *2.3. Manufacturing Process of the Bituminous Mixtures*

A total of 160 samples were manufactured, corresponding to the ten combinations analyzed, with eight samples for the Cantabro test (four dry and four wet) and another eight for the water sensitivity test by indirect tensile strength (four dry and four wet) for each combination.

For this purpose, the aggregates were sieved and separated, washed, and dried in an oven until a constant mass was obtained. The filler (cellulose ash) also had to be sieved through sieve N°200, as it contained impurities from the rest of the wood that could impair the adhesiveness of the filler/bitumen matrix.

Once the material was prepared, the samples were manufactured according to the Marshall method by UNE EN 12697-30 [33]. The aggregates were heated in a forced convection oven to a temperature of 180 °C for 8 h, and the bitumen was heated to 155 °C for 2 h. The aggregates were then mixed with the asphalt binder until it was capable of coating all of the stone particles. Cellulose ash was added, and the process was repeated until a homogeneous mixture was achieved, maintaining the temperature of the mixture at 160 °C. Once the homogeneous mixture was obtained, it was placed in a Marshall mold (101.6 × 63 mm) and compacted at 75 strokes per face. The samples were allowed to cool down to room temperature and then removed from their molds [34].

#### Density and Air Void Spaces in the Bituminous Mixture

After the compacted Marshall test samples were cooled and demolded, their bulk density and void content in the aggregates and mix were calculated. To calculate the bulk density, the specimens were immersed in a water bath for 30 min to saturate the specimen (void fill). Afterwards, the test tubes were extracted from the water and weighed to determine the volume occupied by the voids.

In this study, the bituminous mixtures were designed for a wearing course so, according to the regulations, the void content had to be between 4% and 6%, and the actual density was calculated from method B for a dry saturated surface [33]. The void content of a specimen was calculated from the maximum density of the mixture and the apparent density of the specimen [34]. It was calculated using Equation (1):

$$V_m = \frac{\rho_m - \rho_b}{\rho_m} \cdot 100 \quad (1)$$

where $V_m$ is the air void content of the mixture (%), $\rho_m$ is the maximum density of the mixture (g/cm$^3$), and $\rho_b$ is the bulk density of the sample (g/cm$^3$).

### 2.4. UCL Method

The universal method for the characterization of asphalt binders (UCL) was developed by the Department of Transport Infrastructure of the Technical University of Catalonia (Spain). It focuses on the behavior of the asphalt binder, without taking the viscosity, composition, and/or consistency of the material into account, but considering functional parameters such as cohesion, adhesiveness, thermal susceptibility, and durability [28,30]. For the study of wear, the method applies the Cantabro test, in which the cohesion of the mixture is determined from the abrasion that occurs due to the effects of ageing, temperature, and water.

In the present research, the UCL method was modified to focus on the filler, rather than the asphalt binder. This methodology has proven to be conclusive in previous research both to differentiate the adhesiveness of the mastic asphalt depending on the type and dose of filler used under different thermal and aging conditions and to evaluate the durability of the asphalt mix [35–38]. Traditionally, the weight dosages are calculated according to size parameters and the plasticity of the filler, while in this study, the volumetric relations were calculated according to variables such as the density, porosity, and/or nature [31,32].

This research aimed, by means of the Cantabro test for loss due to wearing, to evaluate the adhesiveness of the bituminous mastic based on the losses produced in the mixture. An increase or decrease of losses would be directly related to the adhesiveness of the filler/bitumen system [32].

On the other hand, the Argentine Institute of Standardization and Certification IRAM 1542 stipulates that in order to maintain viscous deformation of the filler/bitumen system, it is necessary for the ratio of the volumetric concentration of filler (*Cv*) to its critical concentration (*Cs*) to be less than or equal to ($Cv/Cs \leq 1$).

#### Cantabro Test and Sample Conditioning Method

In order to test the adhesiveness of the samples (dry and wet), they were pre- conditioned in their entirety. To study adhesiveness under wet conditions, the specimens needed to be immersed in water for 24 h. They then had to be removed from the water and left to stand at room temperature for 24 h before being subjected to the Cantabro wear loss test. In the case of dry specimens, they were left to rest for 24 h at room temperature and then directly subjected to the Cantabro wear loss test. Based on this methodology, the aim was to compare the adverse climatic conditions of humidity to which the bituminous mix may be subjected and to evaluate the cohesion of the bituminous mastic by means of the wear test.

The Cantabro test by wear losses is described in UNE-EN 12697-17 [39]. This test allows for the estimation of resistance to separation that a bituminous mixture has upon being submitted to wear by the Los Angeles machine. According to the procedure outlined in the regulations, Marshall type samples must be put in the Los Angeles machine without any abrasives for 300 revolutions with a velocity of between 30 and 33 rpm (approximately 9 min per sample) (Figure 1).

**Figure 1.** Wear losses determined by the Cantabro test.

Finally, the losses obtained during this test were calculated using Equation (2):

$$P_c = \frac{P_i - P_f}{P_i} \cdot 100 \tag{2}$$

where $P_c$ is the wear losses due to the Cantabro test (%), $P_i$ is the initial mass of the sample (g), and $P_f$ is the final mass of the sample (g).

In order to correctly carry out this test, it was necessary to collect the material that fell away from each sample into the Los Angeles machine after each test, as this material could have acted as an abrasive in subsequent sample tests.

Additionally, it is possible to establish a proportion of loss of adhesiveness to the Cantabrian by effect of moisture by means of the following Equation (3):

$$WS_C = \left(1 - \frac{(100 - P_D) - (100 - P_W)}{100 - P_D}\right) \cdot 100 \tag{3}$$

where $WS_c$ is the proportion of material that remains adhered to the test tube in relation to the Cantabro losses in dry and wet conditions; $P_d$ is the proportion of Cantabro losses in dry conditions; and $P_w$ is the proportion of Cantabro losses in wet conditions.

*2.5. Water Sensitivity by Indirect Tensile Strength*

The moisture damage was determined in accordance with UNE-EN 12697-12 (2009). The indirect tensile strength of the two groups of identical bituminous mixtures was measured in a dry state and under saturated conditions in order to measure the effect of water on them. To do this, each group consisted of 10 identical samples, separated into two subgroups of five samples each: (1) the dry subset was maintained at a room temperature of 20 ± 5 °C, while (2) the wet subset had to go through a previous process in which the test samples were subjected to an absolute pressure of 50 mmHg in a vacuum vessel for 30 min. Subsequently, the atmospheric pressure slowly recovered, and then the samples were submerged for another 30 min in water. Finally, the specimens were placed in a container capable of maintaining a temperature of 40 °C for 72 h [40].

Subsequently, the resistance to indirect traction was determined according to the procedure established in the UNE-EN-12697-23 (2004) standard to obtain the maximum breaking load, above which the specimen stopped resisting and broke [41]. Subsequently, using Equation (4), which relates the geometric properties to the maximum breaking load, the diametric resistance to compression was obtained:

$$ITS = \frac{2 \cdot P}{\pi \cdot H \cdot d} \tag{4}$$

where *ITS* is the indirect tensile strength (GPa), *P* is the peak load (kN), *H* is the height of the sample (mm), and d is the diameter of the sample (mm).

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

where *ITSR* is the indirect tensile strength ratio (%), $ITS_w$ is the average indirect tensile strength of the wet specimens (GPa), and $ITS_d$ is the average indirect tensile strength of the dry specimens (GPa).

## 3. Results

### *3.1. Characterization of the Cellulose Ash*

This study was based on an analysis of the behavior of bituminous mixtures made with ash when faced with the effects of water. To ensure the correct use of the ash, an analysis of the material first had to be carried out, since, according to prior research, the use of different types of non-traditional filler materials can produce diverse effects on the short- and long-term behaviors of the bituminous mixture. In this case, the filler was associated with higher adhesiveness of the particles in the bituminous mixture, filling the voids created by both the larger and smaller aggregates present in the mixture. This effect could be beneficial, as it helps to shape a consistent framework within the mixture, giving it resistance. Conversely, miscalculation of the quantity of this material used could generate a "surplus of filler" effect, giving a rigid quality to the bituminous mastic, thus making a pavement that is more fragile and susceptible to critical breakage [13].

#### 3.1.1. XRD Diffraction

The analyzed sample was a grey crystalline powder. Figure 2 shows a laboratory XRD diagram obtained at room temperature. The main sample of calcite was indexed to the rhombohedral space group R-3c.

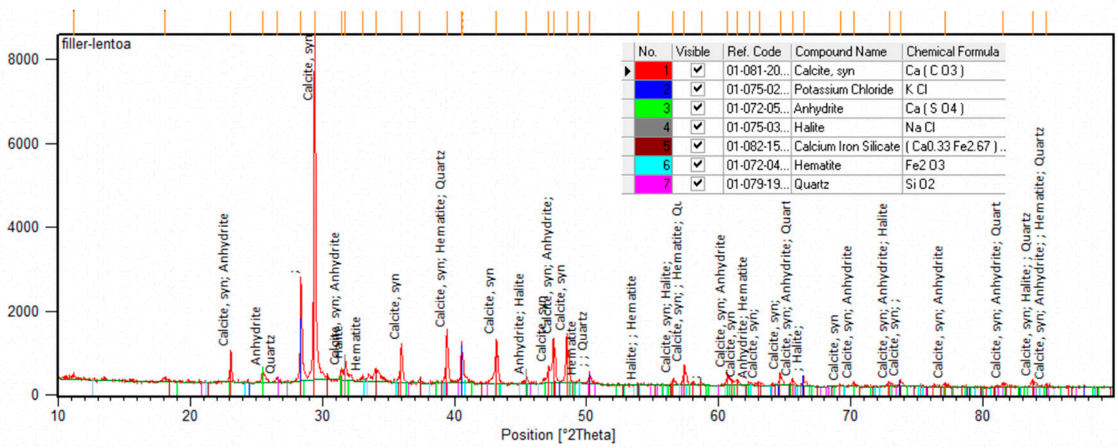


**Figure 2.** X-ray diffraction (XRD) pattern for Walter's filler sample. The majority phase of calcite was indexed in a rhombohedral space cell. Additionally, the phases of sylvite, anhydrite, halite, calcium iron silicate, hematite, and quartz were determined.

The phases found in the sample were refined from XRD data. In the model used for the main phase of calcite, the Ca atoms were distributed in site 6b (0 0 0), the C atoms were in position 6a (0 0 1/4), and the O atoms were in site Wyckoff 18e (x 0 1/4). The phases of sylvite, anhydrite, halite, calcium iron silicate, hematite, and quartz found in the diffraction pattern were refined as second phases.

Figure 3 illustrates the quality of the refinement, where the main phase of the sample was refined in the space group R-3C. The vertical markers correspond to Bragg's reflections (angles).

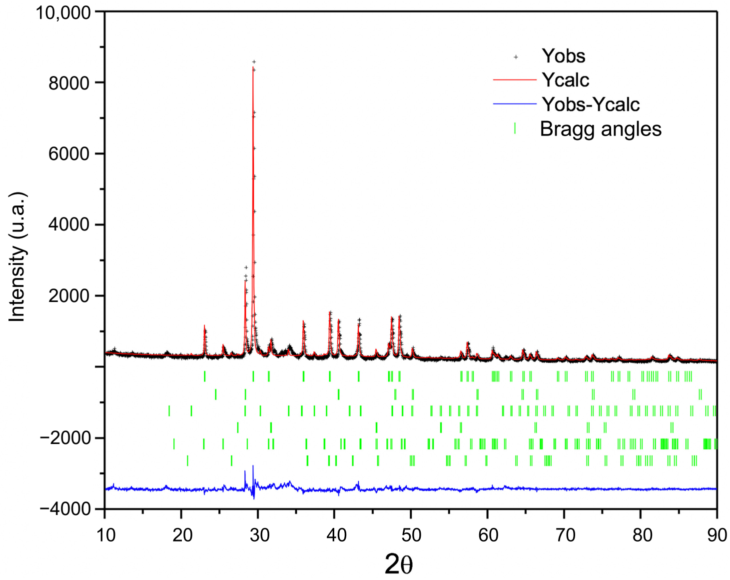


**Figure 3.** Observed profiles (red crosses), calculated values (full black line), and difference (blue) for the cellulose ash sample.

Table 5 presents the main atomic parameters after refinement.

**Table 5.** Structural parameters of the main phase of calcite from XRD data in the R-3C space group. The R factors of reliability are also provided.

| Parameter | $CaCO_3$ |
| --- | --- |
| a (Å) | 4.9927(2) |
| c (Å) | 17.0783(1) |
| V (Å3) | 368.68(3) |
| Bov (Å2) | 2.229(1) |
| Ca 6b(0 0 0) | |
| focc | 1.00 |
| C 6a (0 0 1/4) | |
| focc | 1.00 |
| O1 18e (x 0 1/4) | |
| x | 0.2564(7) |
| focc | 3.00 |
| $x^2$ | 3.80 |
| Rp (%) | 8.03 |
| Rwp (%) | 10.6 |
| RBragg (%) | 10.1 |

Table 6 shows the results obtained from the cellulose ash sample. The results show that it mainly contained calcite, sylvite, calcium iron silicate, halite, anhydrite, and quartz. The fundamental compounds such as calcite ($CaCO_3$; 79.3% of the total sample) and anhydrite ($CaSO_4$; 2.8%) generated an increase in the elasticity of the asphalt mixture, improving adhesion at the filler–bitumen interface due to the alkalinity of the lime.

It should be noted that the aggregate that gives greater durability to the traditional mix is limestone (composed mainly of carbonates), so the presence of calcite in the cellulose ash increases the resistance in the internal links of the asphalt mastic. However, the cellulose ash samples were made of 10.8% silica, which has been shown in studies to affect the performance of the asphalt mixture, leading to failures, such as rutting [10]. Among the materials mentioned, calcium iron silicate had the highest density in relation to other materials, but its percentage was negligible in reference to calcite and anhydrite, which had an average density of 2.71–2.88 g/$cm^3$.

**Table 6.** Percentage of phases found in the sample of cellulose ash.

| Phase | | Spatial Group | Volume (Å$^3$) | Density (g/cm$^3$) | % |
|---|---|---|---|---|---|
| $CaCo_3$ | Calcite | R-3c | 368.68(3) | 2.705 | 79.3(1) |
| KCl | Sylvite | Fm-3m | 249.09(2) | 1.988 | 10.8(0.2) |
| $Ca_3Fe_2(Si_3O_{12})$ | Calcium iron silicate | Ia-3d | 1636.32(5) | 4.102 | 3.4(0.2) |
| NaCl | Halite | Fm-3m | 179.50(1) | 2.163 | 3.1(0.1) |
| $Ca(SO_4)$ | Anhydrite | Amma | 305.09(1) | 2.877 | 2.8(0.2) |
| $SiO_2$ | Quartz | P3121 | 114.27(6) | 2.618 | 0.6(0.1) |

3.1.2. Volumetric Parameters

Table 7 shows the obtained density values and the critical concentration for each filler. The cellulose ash sample had a lower density than the natural filler which, in turn, is the reason the filler presented a higher critical concentration.

**Table 7.** Density and critical concentration of each used filler.

| Filler | Density (g/cm$^3$) | Critical Concentration |
|---|---|---|
| Cellulose Ash | 2.48 | 0.22 |
| Aggregate Filler | 2.67 | 0.20 |

### *3.2. Air Void Content and Density in Bituminous Mixtures*

Table 8 shows the results for the density and void content of each asphalt mixture. It should be noted that, for the definition of the groups of samples, these were divided so that the average density differed by no more than 0.015 g/cm$^3$ between groups, and the average height differed by no more than 5 mm. In this way, the results obtained in the different tests belonged to identical groups of samples. The results show that when the concentration of *Cv/Cs* increased, the percentage of voids increased, which occurred because a greater volume was generated that occupied the filler in the sample. Also, all samples of asphalt mixtures with cellulose ash content had higher void contents.

**Table 8.** Air void content and density in bituminous mixtures.

| Cv/Cs | Type of Filler | Density (g/cm$^3$) | Air Void Content (%) |
|---|---|---|---|
| 0.50 | Limestone filler | 2.319 | 4.18 |
| 0.75 | Limestone filler | 2.331 | 4.33 |
| 1.00 | Limestone filler | 2.340 | 4.49 |
| 1.30 | Limestone filler | 2.355 | 4.73 |
| 1.50 | Limestone filler | 2.368 | 4.96 |
| 0.50 | Cellulose ash | 2.282 | 4.98 |
| 0.75 | Cellulose ash | 2.301 | 5.17 |
| 1.00 | Cellulose ash | 2.289 | 5.09 |
| 1.30 | Cellulose ash | 2.268 | 5.76 |
| 1.50 | Cellulose ash | 2.251 | 5.92 |

On the other hand, since the amount of filler changes for each mixture (different *Cv/Cs* ratio) but the absolute value of binder quantity is 57 g for all mixtures, the percentage of binder in the mix (referred to mixture) changes for all cases; so, to calculate the void content, the real binder content for each case was considered—this being a subtle difference (±0.1%) between the mixes with higher and lower *Cv/Cs* ratios.

Based on the results shown in Table 7, we observed that all bituminous mixtures met the requirements of current regulations, which indicates that mixtures intended for wearing courses and heavy traffic must have a void content of between 4% and 6% [23].

### 3.3. Wear Losses by Cantabro Test (Adhesiveness)

To study the behaviors of mixtures with different *Cv/Cs* ratios, the Cantabro wear loss test was carried out on previously conditioned samples. It is interesting to note that none of the mixtures showed losses greater than 60%, which is the limit established for open grade (porous) mixtures.

Figure 4 shows two very different "state curves" when you compare the mixtures made with conventional filler and cellulose as the filler that were subjected to the Cantabro test of wear in the dry state. The lowest losses in the Cantabro test were obtained for *Cv/Cs* ratios close to 1.00, and the loss increased in both mixtures when these relations exceeded this value. This is indicative that overfillerization occurs in the filler–binder system, which can be dangerous in the short term, since the bituminous mastic is rigidified.

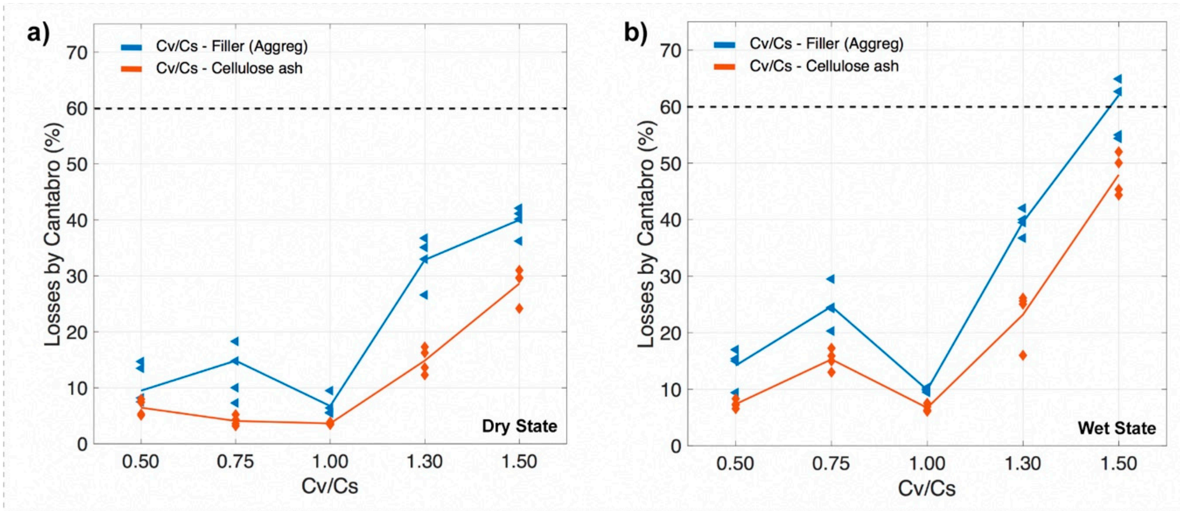


**Figure 4.** Wear losses by Cantabro test: (**a**) Dry state; (**b**) Wet state.

It is important to point out that there are significant differences in the use of the cellulose filler compared to the traditional one, since for all volumetric ratios tested, the losses in the Cantabro test were lower, showing differences as great as 20%.

This is consistent with the theory mentioned above. When the samples were subjected to the wet test, the cohesion between the particles in the mixture weakened—although the losses were kept under control, as long as the *Cv/Cs* ratio was equal to 1. On another note, it can be affirmed that a *Cv/Cs* ratio of 1.00 produced the lowest loss in both the dry and wet states, while mixtures with a *Cv/Cs* ratio of 1.5 had more wear.

The curves of the wet state (Figure 5b) presented a higher percentage of loss in comparison with the dry state. Furthermore, it is important to note that in the curve of the wet state with the *Cv/Cs* concentration being equal to 1.5, a loss of 59.2% was achieved, confirming that the mixture had started to lose its resistance to stress. It is important to point out that when the dry and wet state curves were compared for the mixtures made with cellulose ash, a loss of 10% in the Cantabro occurred in both cases, which indicates that the mixture with cellulose did not undergo greater changes due to water. In contrast, for conventional mixtures, the curve underwent a more drastic change, which may indicate some sensitivity of the mixture to the actions of water, and there may be losses in adhesiveness between the materials that make up the mixture.

Table 9 represents the index of Cantabro losses between dry and wet samples for the different combinations analyzed. The values represent the amount of material remaining in the sample for both types of conditioning, using Ec. (3).

**Table 9.** Cantabro wear losses wet/dry index vs. Cv/Cs for each type of filler.

| Cv/Cc | Aggregate Filler (%) | Cellulose Ash (%) |
|---|---|---|
| 0.50 | 96.38 | 99.03 |
| 0.75 | 86.22 | 88.31 |
| 1.00 | 96.63 | 96.81 |
| 1.30 | 90.00 | 90.22 |
| 1.50 | 67.81 | 72.92 |

### 3.4. Moisture Damage by Indirect Tensile Strength

Figure 5 shows the conserved strength (dry and wet) for each type of mix with aggregate filler and cellulose ash. In general, the dry and wet strengths are slightly higher for mixtures with cellulose ash, except for mixtures with *Cv/Cs* greater than 1, where the highest resistances occur in mixtures with traditional filler. The above is due to the effect of "overfiller" produced in the mixtures modified with ash once the optimum admissible percentage in the mixture is exceeded. The effect produced by the ash when the *Cv/Cs* exceeds 1 translates into an increase in the brittleness in the bituminous mastic, which is directly related to a much faster breakage of the mixture.

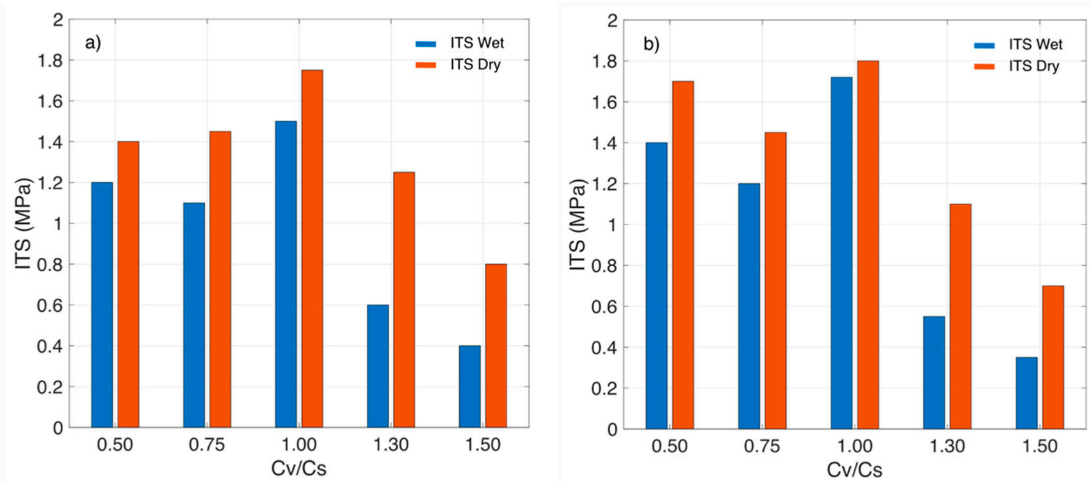


**Figure 5.** (**a**) Indirect tensile strength (ITS) natural filler; (**b**) ITS cellulose ash filler.

Figure 6 shows the conserved resistance (dry and wet) for each type of mixture, with aggregate filler and cellulose ash. In general, the dry and wet strengths were slightly higher for mixtures with cellulose ash, except for mixtures with *Cv/Cs* ratios above 1. The greatest strengths were found in mixtures with natural filler. It is important to emphasize that for this behavior, which was also displayed in the Cantabrian test, the samples that presented a *Cv/Cs* ratio of between 0.5 and 1.00 showed better mechanical behavior. The mixtures that exceeded a *Cv/Cs* of 1.00 had a stiffening of the asphalt mastic due to the large amount of filler volume. This caused the agglomeration of the larger particles to be reduced, due to the reduction of the visco-elastic behavior of the asphalt mastic, causing greater losses and less conserved resistance.

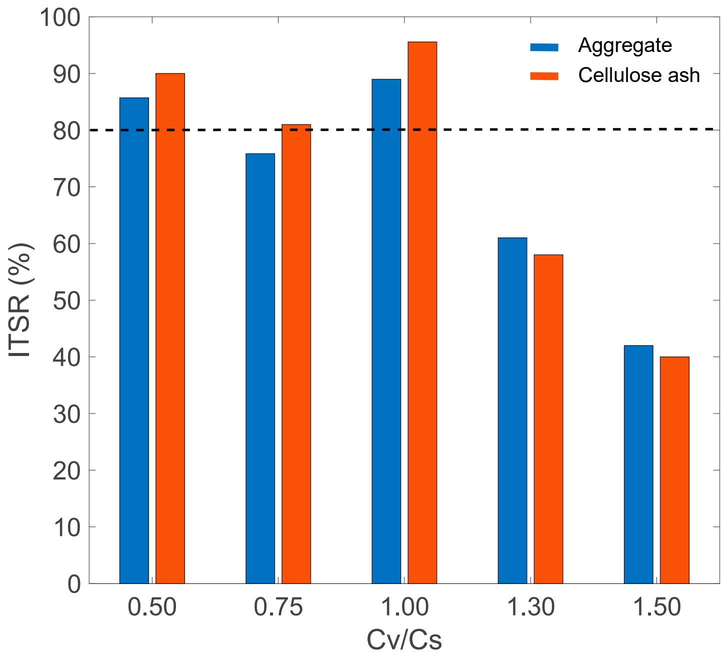


**Figure 6.** Indirect tensile strength ratio (ITSR) vs. *Cv/Cs* for each type of filler.

When comparing these materials with an analysis of variance (Table 10), we observed that there were no significant differences between the materials studied (p-value = 0.93.95) for dry samples, which generated similarity in the mechanical resistance between the aggregate and the cellulose filler blends. However, when evaluating the wet retained strength of the samples, it was found to be reduced by 13.54 percentage points ($p$-value = 0.8041), increasing the dispersion of the data of the bituminous mixtures in comparison. This was due to the "overfill" effect that occurred in the ash-modified mixtures once the optimal percentage admissible in the mixture was exceeded. The effect produced by the ash when the *Cv/Cs* exceeded 1 was an increase in the fragility of the bituminous mastic, which was directly related to a much faster breakage of the whole compound.

**Table 10.** ANOVA for bituminous mixtures with aggregate and cellulose filler.

| | Source of Variability | Sum of Squares | Degrees of Freedom | Mean Squares | F-Statistic | *p*-Value |
|---|---|---|---|---|---|---|
| Cantabro Dry | Materials | 0.001 | 1 | 0.001 | 0.01 | 0.9395 |
| | Error | 1.303 | 8 | 0.16287 | - | - |
| | Total | 1.304 | 9 | - | - | - |
| Cantabro Wet | Materials | 0.01764 | 1 | 0.01764 | 0.07 | 0.8041 |
| | Error | 2.14572 | 8 | 0.26821 | - | - |
| | Total | 2.16336 | 9 | - | - | - |
| ITSR | Materials | 12.05 | 1 | 12.054 | 0.03 | 0.8754 |
| | Error | 3680.46 | 8 | 460.058 | - | - |
| | Total | 3692.51 | 9 | - | - | - |

The *ITSR* index improves when *Cv/Cs* = 1 for both types of bituminous mixture, while for higher *Cv/Cs* ratios, it decreases for both types of filler. It should be noted that once the filler content is too high (*Cv/Cs* > 1), as shown in Figure 6 the two mixtures have an *ITSR* index lower than 80% and, therefore, according to current regulations, could not be used in either the intermediate layer or the base layer. However, satisfactory results are obtained when *Cv/Cs* = 1, since the standards for both types of mixtures are met. The final results of the ANOVA statistical analysis (Table 10) show that the data are similar between the aggregate and cellulose filler mixtures, with an 87.54% probability for the *ITSR* index, with the dry samples being the closest to each dosage studied. Also, it should be noted that the mixtures containing ash as filler have a higher *ITSR* index than conventional mixtures, indicating that not only does this type of material have better conditions of adhesion to the bitumen binder, but its pozzolanic reactions allow better performance against water.

## 4. Conclusions

The addition of cellulose ash as a contributing filler in semi-dense mixes made it possible to obtain similar results to a traditional bituminous mix in terms of adhesiveness between the filler and binder. In the present investigation, for a volumetric *Cv/Cs* ratio equal to 1.0, 60 g of mineral filler was considered in the reference mixture, and there was 38.6 g of cellulose ash in the modified mixtures, confirming that, to obtain similar behavior to the conventional mixture against water, a smaller quantity of cellulose ash is necessary.

From the analyses that were carried out, a *Cv/Cs* ratio of between 0.5 and 1 was determined to be necessary, with a value of 1.00 being optimal.

Furthermore, it was confirmed that what is stated by IRAM is relevant, since the volumetric concentration must be less than the critical concentration of filler used. This was demonstrated by the results obtained with the Cantabro wear loss test, in which the losses with volumetric ratios of less than 1 were controlled at a similar level to the mixtures used as a reference. In contrast, upon exceeding the said concentration, the losses rose considerably in both dry and wet conditions.

Wear losses did not exceed 60% in most cases, and only one of the concentrations reached this limit. Therefore, with the exception of mixtures that use this concentration, the rest can be applied in areas of high precipitation. On the other hand, the UCL method was found to be too sensitive to differentiate between the quantities of filler used and the adhesiveness between particles. The UCL method is also applicable to the study of semi-dense mixtures that are sensitive enough for its analysis, broadening the types of mixtures that this method had as its objective at the beginning (mixtures with open grading). Furthermore, the Cantabro test was effective for evaluating the adhesive properties of the bituminous mixtures, both dry and wet, resulting in the curves of the state presented by this research.

Also, the contribution of cellulose ash as a filler increases the resistance to indirect tensile strength in dry and wet conditions and reduces the amount of moisture damage that occurs compared to the conventional mixtures. However, it is important to indicate that using the optimal amount of filler influences the effectiveness of the mixture. Therefore, it is necessary to select the optimal filler content.

In short, while cellulose ash is an industrial by-product that can cause particular problems for the forestry company, it could be beneficial and valuable for use in road construction.

We demonstrated that when the optimal percentage of cellulose filler was used in asphalt mixtures, more adequate characteristics and adhesiveness were achieved in the filler/bitumen system compared with the reference mixture. Another advantage of the use of this filler (cellulose) was the reduced costs of transport and taxes for the company, since it is an industrial sub-product that must be deposited (in normal conditions) in authorized landfills. As an indication, in the year 2017, the cost for transfer to landfill was US$1075.00, and the cost for transportation was US$19,105.16 [42]. Also, currently, in the south of Chile, there is a great shortage of natural aggregates due to over-exploitation and poor regulation in recent decades, which has had serious consequences for the environment. The use of this type of "waste" not only helps to minimize this environmental problem but also improves the economy of companies.

**Author Contributions:** Conceptualization and methodology, D.M.-Q. and A.C.R.; tests development, D.M.Q., O.M.-C., and M.L.-V.; writing—original draft preparation, D.M.Q., V.-C.A.-V., and O.M.-C.; data analyses, D.M.Q., A.C.R., V.-C.A.-V., M.L.-V., and L.T.; funding acquisition and project administration, D.M.Q. and A.C.R.; writing—review and editing, D.M.Q. and M.L.-V. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by the project FONDECYT Regular into Research N° 1201029 (CONICYT), and the APC was funded by the InnovING 2030 project of the Faculty of Engineering Sciences UACh.

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

**Informed Consent Statement:** Not applicable.

**Acknowledgments:** The authors gratefully acknowledge the institutional support provided by the Department of R+D of the University Austral of Chile (DID UACh). The authors would also like to thank *ARAUCO*, *BITUMIX S.A.* and *Química Latinoamericana* for the material donations.

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

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