Thermal suspectability analysis of the reuse of fly ash from cellulose industry as contribution filler in bituminous mixtures

Movilla-Quesada, D., Muñoz, O., Raposeiras, A. C. & Castro-Fresno, D.

Author post-print (accepted) deposited by Coventry University's Repository

Original citation & hyperlink:

Movilla-Quesada, D, Muñoz, O, Raposeiras, AC & Castro-Fresno, D 2018, 'Thermal suspectability analysis of the reuse of fly ash from cellulose industry as contribution filler in bituminous mixtures' Construction and Building Materials, vol. 160, pp. 268-277. https://dx.doi.org/10.1016/j.conbuildmat.2017.11.046

DOI 10.1016/j.conbuildmat.2017.11.046

ISSN 0950-0618 ESSN 1879-0526

Publisher: Elsevier

NOTICE: this is the author's version of a work that was accepted for publication in Construction and Building Materials. Changes resulting from the publishing process, such as peer review, editing, corrections, structural formatting, and other quality control mechanisms may not be reflected in this document. Changes may have been made to this work since it was submitted for publication. A definitive version was subsequently published in Construction and Building Materials, 168 (2018) DOI: 10.1016/j.conbuildmat.2017.11.046

© 2017, Elsevier. Licensed under the Creative Commons Attribution-NonCommercial-NoDerivatives 4.0 International http://creativecommons.org/licenses/by-nc-nd/4.0/

Copyright © and Moral Rights are retained by the author(s) and/ or other copyright owners. A copy can be downloaded for personal non-commercial research or study, without prior permission or charge. This item cannot be reproduced or quoted extensively from without first obtaining permission in writing from the copyright holder(s). The content must not be changed in any way or sold commercially in any format or medium without the formal permission of the copyright holders.

This document is the author's post-print version, incorporating any revisions agreed during the peer-review process. Some differences between the published version and this version may remain and you are advised to consult the published version if you wish to cite from it.

AUTHOR'S POST-PRINT RELEASED WITH CREATIVE COMMONS ATTRIBUTION NON-COMMERCIAL NO DERIVATIVES LICENSE

Construction and Building Materials, vol. 160 (2018), pp. 268-277.

https://doi.org/10.1016/j.conbuildmat.2017.11.046

Thermal suspectability analysis of the reuse of fly ash from cellulose industry as contribution filler in bituminous mixtures

D. Movilla-Quesada^{a,*}, O. Muñoz^a, A.C. Raposeiras^a, D. Castro-Fresno^b

- ^a Civil Works Institute, Faculty of Engineering Sciences, Austral University of Chile, General Lagos Street, 2086 Valdivia, Chile
- ^b Department of Transports, Projects and Processes School of Engineering, University of Cantabria, Santander 39005, Spain

ABSTRACT

The increased population has accelerated pavement deterioration of and boosted production of residues, generating a constant environmental problem. One of the main problems of pavements is a failure to develop union between bituminous mastic ligand and aggregate. The following study evaluates the use of ash from cellulose incineration as a contribution filler to improve thermal susceptibility of pavements in different climatic zones. The applied methodology for dosage was based on volumetric criteria and state curves. The obtained results showed that reusing this residue in determined conditions (Cv/Cs 1) offers lower wear loss to 35% of temperatures between 10 and 60 C, due to the Cv/Cs = 0.75 ratio that maximizes the cohesive properties of the mixture. Therefore, we showed that this type of industry resi- due can be reused without complication in zones of certain climatic criteria and that it provides improved properties to the asphalt mix compared to conventional mixes.

Keywords: Cellulose ashes, highway pavements, temperature, filler, recycled materials

1. Introduction

Constant population increase and the ease of transportation have led to a parallel and gradual increase in transport demand. This growing demand leads to accelerated deterioration of road pavements, making improvements to roads and asphalt mixes with a longer service life essential. On the other hand, intensification of human activity has generated a progressive increase in dangerous substance and residue production, provoking an incessant environ- mental problem [1,2].

One of the main problems of asphalt pavements produced by climatic and environmental conditions is union failure between the ligand and surface aggregate [3]. These problems are mainly due to insufficient cohesion and adhesion characteristics which translate into cracks and pavement surfaces with loss of material [4–6]. One of the external agents that most affects asphalt mixture cohesion is the temperature of its service conditions. Asphalt mixes under extreme temperature conditions have features comparable to a fragile, elasto-plastic or sticky body [7]. The variation of mix- ture state with temperature can be controlled through cohesion characteristics and the low susceptibility supplied by bituminous mastic [8–11].

The concept of 'filler' has long been known, however, over the last years the use of this material in asphalt mixes has increased: lime and cement are the most widely used fillers. Within the mas- tic nucleus, it is common that studies and pavement jobs centre their attention on the ligand, given its absolute responsibility as a binding agent [12,13]. However, since the type of filler is the most variable mastic component (filler and bitumen), in charge of filling gaps in the mineral skeleton as well as waterproofing, densifying and modifying asphalt mix viscosity, it is of vital impor- tance to evaluate and define the characteristics, composition and quantity of filler to use when protecting or providing a higher mix- ture cohesion capacity against external agents such as water and temperature variations [5,6,8–10,14–16].

Numerous studies have evaluated the potential use of different materials as contribution fillers in asphalt mixtures. These materials include cement, calcium hydroxides, ash, recycled powder from building material, calcium carbonate, vegetable filler, among others [3,17–19]. Diverse authors such as Chuanfeng et al. (2014) [16] and Ke-Zhen et al. (2013) [20] evaluated the influence of the filler's characteristics on cohesive resistance at different tempera- tures and found that specific surface is a fundamental parameter when choosing a material, such as contribution filler, for the mix- ture [16,20]. Among the range of fillers, ash from industrial pro- cesses acquires added value due to its potential environmental contamination. Currently, solid industrial residues (SIR) constitute a critical environmental problem in the modern industrial society, making their management and valorisation essential [2,4,11,21].

This study focuses on analysing the influence of temperature on semi-dense asphalt mixture properties (functional and mechanic) through the sensibility or susceptibility concept. The evaluated mixtures were fabricated with ash, from cellulose incineration, as a contribution filler. Ash dosage was based on its volumetric con- centration (Cv), with special attention to parameters such as specific weight and critical concentration (Cs). This procedure is performed using the Argentinian standard IRAM 1542 (1983) [22–24]. Finally to obtain the results we used the universal ligand characterization method, UCL! [7]. Comprehension of functional and

mechanical properties of asphalt mixtures is essential at differ- ent temperatures, given the influence of this variable directly on the visco-elastic behaviour of materials.

2. Materials & methods

In this study we prepared 7 types of asphalt mixtures. Two of these corre- sponded to Semi-dense mixtures, according the standard [25]. In the other 5 mix- tures the mineral filler was completely replaced using ashes at different concentrations.

2.1. Aggregates and bitumen

The employed aggregate for this research was arid obtained by crushing natural gravel in the south of Chile, specifically in the Los Rios Region. The different fractions of arid used for mixture doses were combined so that particle size of the test samples adjusted to the "IV-A-12" type band, specified by the standard [25]. This particle size allowed obtainment of mixtures that can be used both in intermediate layers, such as asphalt layers, the reason for which these mixtures are the most widely used throughout Chile. Table 1 contains the values of the selected particle size bands.

Bitumen ligands or asphalt cements (AC) are presented as a bright and black mass with a consistency that varies with temperature. The Road Manual (2015) classifies AC according to their absolute viscosity at 60 C, not allowing projects to specify the use of classification of asphalt cements by penetration [26]. The ligands used for this research were the CA-24 ligand and an elastomer ligand "Stylink" modified with polymers. Table 2 contains the characteristics and specifications of the used ligands.

22 Filler

For thermal susceptibility analysis, this study centres its attention on the cohe- sive properties that bituminous mastic transfers to the mixture when it is subjected to temperature variations. The ash used as bituminous mastic filler come from the production process of the cellulose plant. Mainly, they are the residues from burn- ing bark and wood-chips for the generation of energy from high pressure vapor gen- erated by biomass boilers, where combustion occurs [21]. Currently, theses ashes are disposed in RSI authorized landfills [27]. The ash used was sifted through a sieve N 200 (0.08 mm) since by nature this is mixed with other solid residues such as car- bon, sand and wood-chips (Fig. 1) [21].

2.3. Material dosage

Due to the importance of filler selection and dosage for a bituminous mixture, material dosage for fabrication of the sample mixtures was based on a volumetric concentration (Cv) of the filler in regards to the ligand [23,28].

It is important to obtain and characterize in detail the ash to be used as filler. The methodology used to characterize ash meets the Argentinian standard IRAM 1542 [22]. To evaluate exclusively the effect of ash on asphalt mixture cohesion under variable temperatures, without taking into account the effect of nature, the quantity and type of arid and ligand, the retained material in each sieve, as well as ligand contents, these remained at fixed values during the fabrication processes, therefore the nature and ash content of the mixture were the only variables.

The bitumen content corresponds to 5% (aprox.) of ligand in the mixture (5% bS/ M). Fig. 2 contains the arid particle size used in the mixtures.

2.3.1. Real density of the filler

To determine ash density in kerosene, we performed a total of 30 tests with 50 gram samples, following a previously described method from the Argentinian stan- dard [22]. Once the results of kerosene density were obtained for each of the 30 samples, we calculated the average.

2.3.2. Critical concentration of the filler

To calculate filler volumetric dosage (Vc), we must first determine critical con- centration (Cc). This value relates intrinsic characteristics of the analysed filler, since it depends (among others) on fineness and surface characteristics of the filler [13,24]. The critical concentration, in theory, assures mastic viscosity behaviour, a fun- damental characteristic when evaluating mixture cohesion. This value is obtained by accommodation and sedimentation of filler dispersion particles at rest. The continuous medium used for this purpose is kerosene due to its chemical similarity with the ligands [22,23]. The calculation for ash critical concentration is determined by the following expression (a):

 $C_s = m / (V \times q)$

where:

Cs: Ash critical concentration,

m: Ash weight, (gr.),

V: Sediment volume, (cm3),

q: Dry ash density, (gr./cm3).

Table 3 shows the values obtained from ash characterization.

2.4. UCL method

The universal characterization method of ligands or simply UCL method, devel- opment by Dr. Miró Recasens and Dr. Pérez Jiménez (1994) is based on the study and assessment of the functional properties that the binder provides the mixture [7]. Among these properties are cohesion and thermic susceptibility; the first is related to binder and material disintegration capacity, meanwhile the second is related to mechanical behaviour variability of mixtures at different temperatures [29].

To assess these properties, the method will evaluate the disintegration resist ance of samples submitted to different conditions (dry, after immersion, at different temperatures and time periods) through the Cantabrian test of wear loss at 25 C [30].

2.4.1. Thermal susceptibility

Through the elaboration of graphs of Cantabrian losses in function of tempera- ture, called state curves, we evaluated thermic susceptibility of binder or bitumi- nous mastic as well as demonstrate their fragile behaviour through high cantabrian losses, elastoplastic, low cantabrian losses and its inconsistency through a sharp increases in losses [7]. The flatter the curve, the less susceptible the binder (or mastic), meanwhile concave curves indicates higher susceptible to temperature variations.

The filler's effect on the variability of thermic susceptibility in mixtures is eval- uated by determining cantabrian losses at different temperatures for different filler concentrations. If we maintain a fixed temperature and focus our attention on filler volume concentration in the filler-binder system, we can determine the maximum amount that can be added without affecting the deformation resistance by overfill- ing. This phenomenon produces an increase in mixture ridgity which behaves as a rigid solid [23].

The Argentinian standard IRAM 1542 suggests that to maintain the viscous deformation capacity of the filler-binder system the concentration of volume filler (Cv) in the system should be equal or less than the critical concentration (Cs), Cv/Cs=1 [22]. The calculation for filler volumetric concentration (Cv) is the following:

2.5. Experimental design

2.5.1. Design and preparation of mixtures

To complete the experimental stage of the study we fabricated and tested a total of 186 samples, the quantity necessary to satisfy the conditions in Table 4. We manufactured 6 series of 27 samples and one of 24 samples. The first of the series corresponds to a reference mixture (REF), without addition of ash, and the filler was comprised of material recovered by sieving the aggregate at a proportion of 4– 8% of the total aggregate mass and a CA-24 type binder. In the other 5 sample series we completely substituted the recovered filler (Rec) for industrial ash residue (RSI) whose dosage was determined from the Cv/Cs ratio of the designs described in Table 5. Finally, the mixture from the series of 24 samples (MOD) was similar to the REF mixture, apart from the binder. In this case, the binder was a modified bin- der "STYLINK 60/80" supplied by Quimica Latinoamericana S.A. All mixtures possed IV-A-12 semi-dense particle size and approximately 5% of binder on top of the total mixture mass [25].

2.5.2. Manufacturing process

Elaboration (mixing and compacting) the samples was performed using the Marshall methodology (UNE-EN 12697–35) [31]. Previous to fabrication, sample materials were conditioned. For this, the different aggregate sizes of the mixture were washed and dried, along with the filler, in an oven until they reached a con- stant mass. Once dry, each aggregate and binder test sample fraction (1100 gr. aprox.) that produced the required granule size (Fig. 3) were weighed and were introduced into the oven with the Marshall mould during 8 h at a minimum tem- perature of 170 C. The binder was also conditioned in the same way during 2 h at 155 C.

The manual mixture process starts by homogenizing the aggregate, wrapping it and then incorporating the necessary amount of binder and filler to the sample. The mixing process culminates when the mixture is homogenous and all the components are covered by the binder.

Compactation of the material within the mould is performed with a compactor or Marshall hammer, whose technical characteristics are specified in the UNE-EN 12697–30 normative, with a total of 75 hits on each side during a controlled period of time. Once the samples are compact, they are left to cool at room temperature (25 C) and then unmolded [32].

2.5.3. Mixture density and holes

To determine the real density of the compacted samples we followed the pro- cedure described by the standard [26]. This density considers mass per unit of sam- ple volume, including accessible and non-accessible pore volume (holes) at a known temperature. Given that the designed samples have between 4 and 6% of holes in the mixture due to their projection as pavement, the method used was "Method B: Samples with Dry and Saturated Surface" of the previous paragraph.

The content of holes in the mixture was obtained in agreement with the UNE- EN-12697-8 standard, through the difference between real mixture volume (sam- ple) and the theoretical volume occupied by the binder and aggregates [33].

2.6. Sample conditioning

To evaluate loss of cohesion and thermic susceptibility of mixtueres fabricated with ash, the samples were submitted to temperatures at 30, 10, 10, 25, 40, 60 and 80 C. Previous to the cantabrian test, each of the fabricated sample series were divided into 6 groups of 4 samples and one group of 3 samples. These groups were maintained during 6 h at room temperature before entering the Los Angeles drum at 25 C (Fig. 4). This methodology reflects the adverse climatic temperature condi- tions that

fluctuate from values below the binder fragility point to values superior to the softening point. Temperatures the asphalt mixtures can face during their ser- vice life [7].

2.7. Cantabrian test

The Cantabrian test of wear loss is carried out according to the UNE-EN-12697- 17 standard [30]. This test estimates disintegration resistance of the bituminous test mixtures. A preconditioned Marshall sample was introduced into the Los Ange- les drum, without abrasive charge, where it was subjected to wear (abrasion and impact) during 300 revolutions in an estimated time of 8–9 min (30–33 rpm) (Fig. 5).

Finally, cantabrian loss values produced by wear during temperature conditions were obtained by the weight difference of the sample vía the following expression (c):

 $P_C = (P_1 - P_2) \times 100/P1$

Where:

P_C: Cantabrian loss, (%), P₁: Initial sample weight, (gr.), P₂: Final sample weight, (cm³).

The final sample weight ŏP2Þ corresponds to the heaviest portion after the test, this alteration is relevant when samples are submitted to extreme temperature conditions (30 C and 80 C) where cantabrian losses are of greater magnitude and in some cases reach portions of similar and significant sizes.

It is convenient to remove all worn particles from the interior of the drum after each test since these may play an important role on the abrasive charge of the fol-lowing tests.

From the obtained results and the elaboration of the state curve we can evalu- ate the suitability of ash (RSI) from the cellulose industry as a constitutive element of bituminous mastic.

3. Results and discussion

3.1. Mixture density and holes

Table 5 shows the results obtained from density assays and the voids content of the bituminous mixtures used in this study. The corresponding sample groups, at each of the tested temper- atures for the different series of mixtures, were divided so that the average density of and height of each group was not superior to 0.015 gr/cm3 and 5 mm respectively. This way the results obtained belong to an identical sampling group. On the other hand, since the amount of binder is constant (57 gr) for each of the Cv/Cs ratios, mixture content (bS/M) was not 5% in all cases. Therefore to calcu- late the percentage of holes we considered the real content of bin- der in each case, being subtle (±0.1%) between mixtures of higher and lower Cv/Cs ratio.

This scarce variation in binder content will practically have no effect on wear loss for each mixture. Based on the results described in Table 6, we observed that all mixtures meet the hole% requisites for mixtures destined to be pavements and for heavy traffic, corre-sponding by definition to 4–6% [25,34].

To determine thermic susceptibility of the fabricated mixtures with different Cv/Cs ash ratios, Table 6 shows the values obtained from the Cantabrian test for wear loss applied to samples, previous to conditioning, at possible service temperatures. In the range of test temperatures, the mixture with lower losses corresponds to those fabricated with the modified binder "STYLINK". However, this mixture is not our reference parameter due to its high cost in comparison to mixtures fabricated with unmodified binder, it is practically unused in the construction of roads in Chile.

In order to visualize the behaviour of different mixtures a state curve is presented for each one (Fig. 6). In the state curve graph different minimum cantabrian losses are observed for different mix- tures. These minimum losses occur, in majority, between 40 and 60 C except for the Cv/Cs = 1.5 mixture, where minimum losses occur at 25 C.

The shift of the curve towards lower temperatures with an increased filler-ligand ratio acquires a special feature, since an increase in filler content produces an increase in mixture viscosity [13], therefore to obtain inconsistent behaviour higher temperatures should be applied compared to mixtures that contain lower amounts of filler. In the case of ash, as the Cv/Cs ratio increases, along with the filler, the inconsistency temperature tends to become smaller. In different analysed volumetric ratios, we observed promising results for mixtures with ratios below the crit- ical concentration i.e., Cv/Cs mixtures 0.5, 0.75 and 1.0 where the highest losses, over the temperature range previous to inconsis- tency, did not surpass 50%. These mixtures with Cv/Cs ratios between 0.5 and 1 have a similar behaviour to the reference mix- ture (REF) in particular the Cv/Cs = 0.75 mixture, which at 25 C had losses of 0.1 points in comparision to the REF mixture (4.5% and 4.6% respectively). The largest differences between mix- tures with the Cv/Cs ratio lower than the critical concentration and the REF mixture are found in extreme temperatures of 30 C and 80 C, where these reach the order of 27 points for Cv/Cs = 1.0 (49.6% and 22.3%) at 30 C and 29 points for Cv/Cs = 0.75 (82.2% and 53.1%) at 80 C, favouring in both cases the mixture REF. In regards to mixture behaviour with volumetric ratios superior to the

critical concentration (Cv/Cs = 1.3 and Cv/Cs = 1.5) we can affirm that these lose the capacity to absorb deformation forces due to an increase of excessive ash in the bituminous mastic. This overfilling effect is clearly evident in Cantabrian losses (shifting of the curve upwards, Fig. 6) of these mixtures, that quickly rise and reach minimum losses of 34.2%.

The lowest losses between mixtures fabricated with ash as a fil- ler correspond to the Cv/Cs = 0.5 mixture (16.91 gr of ash) with a 0.5% loss at 60 C and the highest loss for mixtures with Cv/Cs = 1.5 (67.40 gr of ash) with an 83.5% loss at 80 C.

Given the similarity of the mixtures with volume ratios less than one unit (Cv/Cs 1.0) with the reference mixture, it is impor tant to indicate that mixtures with addition of ash are slightly more susceptible to temperature variations. The slope –a measure of thermic susceptibility- of the corresponding curves to the Cv/Cs ratios 0.5, 0.75 and 1.0 is higher than the pendent of the REF mix- ture, in particular for temperatures lower than 10 C, which makes them thermally more susceptible. Between 10 C and 60 C these slopes are practically equal, except from the Cv/Cs ratio = 1.0 which at 60 C is slightly higher.

When exceeding 60 C drastic changes can be produced in the slope of the studied mixtures which demonstrates the softening temperature of the system, as well as its inconsistent behaviour.

Even though the test distinguishes the fragile, viscous-elastic and inconsistent behaviour of the mixtures, it is important to pay attention to the behaviour of these at elevated temperatures, since at 60 C the samples were highly deformed and could be kneaded with pressure from the fingers, which could alter Cantabrian results [7]. When samples were subjected to a temperature of 80 C they reached their collapsing point.

3.3. Determining optimum filler content

To further elucidate the consequences of overfilling and to determine the Cv/Cs ratio in function of temperature, we analysed the results from another perspective. Fig. 7 outlines the Cantabrian losses of mixtures with different concentrations of ash, varying the Cv/Cs ratio.

Losses at 80 C are maintained practically invariable over the concentration range used in this study, for which the overfilling effect cannot be appreciated. This is due to the fact that the sam- ples at this temperature reach their collapsing point and have no cohesive property or resistance to disintegration. For the rest of the sample temperatures we observed a similar behaviour where losses were controlled from Cv/Cs = 0.5 to Cv/Cs = 1.0, with varia- tions not superior to 9 points however, from the latter concentra- tion onwards the losses began the increase rapidly, reaching differences of up to 30 points (5.5% and 36%) between consecutive volumetric ratios suggesting that it is at this point where overfill- ing begins to occur, confirming the statements of the Argentinian standards [22]. For low temperatures (between 30 and 10 C), optimum ash concentration is reached at Cv/Cs = 0.75, for moder- ate temperatures (between 10 C and 25 C) at Cv/Cs = 1.0, mean- while in conditions with elevated temperatures (between 40 C and 60 C) at Cv/Cs = 0.5.

Cantabrian losses between 25 C and 60 C for mixtures with a Cv/Cs ratio of 0.5, 0.75 and 1.0 are very similar (less than 5 points) obtaining similar results when using any of these ratios.

Fig. 8 shows the distribution of wear loss in function of pave- ment temperatures (pavement) at service conditions [35].

To determine the approximate temperatures at service conditions we used the records of the Metrologic Office of Chile from the last 20 years (Fig. 9) and the formula (d):

T1 = 0:9545 x (Tmax - 0.00618 x Lat² + 0.2289 x Lat. + 42.2) -17.78 °C T2 = 0.859 x Tmin +1.7 °C

where

T1: Approximate maximum temperature of the pavement 20 mm deep, (°C)

T2: Approximate minimum temperature of the pavement, (°C)

Tmax: Maximum absolute temperature of the air in a period of 20 years, (°C)

Tmin: Minimum absolute temperature of the air in a period of 20 years, (°C)

Lat: Geographic latitude of the place of study, (sexagesimal degrees).

These mathematical expressions have been deduced from heat flow models, and adjusted in base of pavement temperature mea surements in North America [35]. These expressions are used to approximately narrow the service temperatures to which the mixtures will be exposed, highlighting the extreme temperatures of fragility and inconsistency hardly found in Chile.

Fig. 9 obtains the range of pavement temperatures in service conditions along Chile found between 10 C and 60 C. Of the samples fabricated with ash as filler and submitted to testing temperatures that are within the service temperature range,

96% of them were below the maximum allowed value by the Spanish regulation for mixtures destined to be used as pave- ments (Fig. 8). This criteria of 60% loss is used since the Chilean

3.4. Volumetric dosage

As observed in previous points, the dosage criteria for the filler used in this study is based in the critical concentration and volume concentration by the Cv/Cs ratio. If we compare the volume dosage tion for filler dosage, in a dense, semi-dense mixture, a

heavy traf- fic T00 to T2 category and a pavement for any summer heat zone, we can appreciate differences in the quantity of filler used in both methods [34].

According to the Spanish regulation, the optimum filler/bitu- men (binder) weight ratio for the previously described traffic and summer zone conditions is between 1.1 and 1.2. Table 7 shows the ratios between volume concentrations Cv/Cs used in this study and the filler/bitumen weight ratio. We can appreciate that the optimum weight ratio (1.1–1.2) is equivalent to using a volume ratio of Cv/Cs = 1.5.

Fig. 10 shows the consequences of Cantabrian losses when dos- ing ash according to the weight criteria compared to the higher performance volume ratio in this study corresponding to Cv/Cs = 0.75. For any pavement temperature within the service tempera- ture range, weight dosage produces Cantabrian loss factors higher than one unit, and therefore a lower performance than dosage mix- tures with volume criteria.

3.5. Predictive linear model

To perform a prognostic of Cantabrian losses and determine the optimum ash content for each particular case and therefore pro- vide in advance in the compliance of mixture disintegration resis- tance, this study presents a general regression model for Cantabrian losses of mixtures with addition of ash. In the develop- ment of this model we have omitted Cantabrian losses at 80 C since the inconsistency of the mixture produces an alternation of the results, affecting the model's confidence level.

Previous to elaboration of the general models of temperature loss, we analysed variables to determine their normality and homoscedasticity. To study normality we used the Kolmogorov-Smirnov test, meanwhile for homoscedasticity we used the Levene test. The significance of some samples was slightly inferior to 0.05 so once the model was generated we paid special attention to its precision. Table 8 shows the correlation coeficients of the mode,

a measure of how well the regression fits the data. The R squared value indicates that 77.5% of the variation of losses is due to the considered variables.

Table 9 shows the existing relationship between exogenous variables (Cv/Cs, temperature and density) and endogenous variables (loss%). Given that this critical level of significance (Sig) is zero, we can establish that both variables (exogenous and endoge- nous) are linearly related.

Table 10 shows the values of partial regression coeficients (B) that define the predictive lineal model equation as well as the relative importance of each independent variable (exogenous) within the equation (Beta value).

In agreement with the previous, the predictive lineal model is (e):

 $P_p = 20.987 \text{Rel.} - 0.493 \text{T}^{\circ} - 416.342 \text{d} + 965.544$

where:

Pp: Predicted Cantabrian losses, (%),

Rel:: Volume ratio Cv/Cs,

T°: Approximate pavement temperature, (°C).

d: Real density of compacted samples, (gr./cm³).

This model is valid for pavement temperatures inferior to 80 C, the point where mixtures become inconsistent.

4. Conclusions

The addition of ash from the cellulose industry as a filler in bitu- minous mastic offers similar results as mixtures fabricated with fil- ler of the same nature as granular material when used in volume ratios below the unit (Cv/Cs 1). Therefore is we consider its incorporation in weight, we need to use a lower quantity of ash to obtain the same results.

The results obtained in regards to Cantabrian losses for the mix- tures with addition of ash in variable temperature conditions were satisfactory; we did not obtain an equal performance as the mix- tures fabricated with modified ligand under these conditions. The mixtures that contained a Cv/Cs ratio of 0.75 presented a similar behaviour for all the temperature ranges analysed, in some cases improved behaviour compared to the reference mixture, in partic- ular at 25 C. Therefore of all the Cv/Cs ratios studied, 0.75 was the optimum ash ratio to improve cohesive capacities and low thermic susceptibility of the mixtures. For extreme temperatures of 30 and 80 C the reference mixture had an improved performance compared to mixtures with the addition of ash, which provoked a curve with a slightly stretched state and therefore a decreased thermic susceptibility.

The mixtures with a volume ratio that surpassed the unit (Cv/ Cs > 1) presented higher losses due to a stiffening state caused in the mixture, losing its capacity to absorb stress by deformation. These results corroborate the studies undergone by the Argentinian Institute of rationalization of materials that indicate that the volume concentration cannot surpass the critical filler concentration.

The temperatures of pavement service conditions in the Chilean summer correspond to 40 and 60 C, which produce lower losses in mixtures fabricated with ash.

Dosage weight criteria are not completely valid when a non- conventional filler is used, such as cellulose ash, given that this does not allow optimization of mixtures presented as an optimum amount of ash equivalent to the Cv/Cs ratio=1.5 that would amplify the losses since this corresponds to a state of overfilling.

The Cantabrian test was sufficiently sensitive at measuring cohesion and thermic susceptibility of the mixtures with semidense granulometry and this way amplifying the usage range, being limited to open mixtures. Through the UCL method it is possible to characterize the effect of non-conventional filler, allowing detection of mastic variations via the state curve with the temperature and ash content, showing its brittle, visco-elastic and incon- sistent behaviour; characteristics that are transferred to the mixture as a unit. The filler used, a pollutant and industrial residue normally disposed in landfills, is a good candidate to be used as an active part of the mastic in asphalt mixtures.

Acknowledgements

The authors of this study acknowledge the support provided by the Research and Development Division of the Universidad Austral of Chile (DID) and the National Commission for Scientific and Tech-nological Research (CONICYT) that without awarding the FONDECYT Initiation Project FONDECYT 2013 (No. 11130309) this research would not have been able to have been conducted.

References

- [1] S. Köfteci, P. Ahmedzade, B. Kultayev, Performance evaluation of bitumen modified by various types of waste plastics, Constr. Build. Mater. 73 (2014) 592–602, https://doi.org/10.1016/j.conbuildmat.2014.09.067.
- [2] K. Jnyanendra, K. Sanjaya, S. Basarkarc, Concrete using agro-waste as fine aggregate for sustainable built environment A review, Build. Environ. 96 (2016) 170–177, https://doi.org/10.1016/j.ijsbe.2016.06.003.
- [3] Y. Xue, N. Haobo, S. Zhu, J. Zha, Utilization of municipal solid waste incineration ash in stone mastic asphalt mixture: pavement performance and environmental impact, Constr. Build. Mater. 23 (2009) 989–996, https://doi.org/10.1016/j.conbuildmat.2008.05.009.
- [4] A. Mousaad, Atmospheric boundary-layer simulation for the built environment: past, present and future, Build. Environ. 75 (2014) 206–225, https://doi.org/10.1016/j.buildenv.2014.02.004.
- [5] H. Wang, I. Al-Qadi, A.F. Aheem, H. Bahia, S. Yang, G. Reinke, Effect of mineral filler characteristics on asphalt mastic and mixture rutting potential, J. Trans. Res. Board 2208 (2011) (2011) 33–39, https://doi.org/10.3141/2208-05.
- [6] J. Zhu, J. Zhong, S. Wu, D. Wang, Experimental investigation on related properties of asphalt mastic containing recycled red brick poder, Constr. Build.
- Mater. 25 (2011) 2883–2887, https://doi.org/10.1016/j.conbuildmat. 2010.12.040.
- [7] R. Miró, Metodología para la caracterización de ligantes asfálticos mediante el empleo del ensayo Cántabro, Barcelona, ETSECCP. 270 p. 1994.
- [8] M. Zeng, W. Chaofan, Effects of type and content of mineral filler on viscosity of asphalt mastic and mixing and compaction temperatures of asphalt mixture, J. Trans. Res. Board 2051 (2008) 31–40, https://doi.org/10.3141/2051-05.
- [9] R. Recasens, A. Martínez, F. Pérez-Jiménez, H. Bianchetto, Effect of filler on the aging potential of asphalt mixtures, J. Trans. Res. Board 2005 (1901) 10–17, https://doi.org/10.3141/1901-02.
- [10] A. Hüseyin, G. Cahit, Laboratory performance comparison of the elastomer- modified asphalt mixtures, Build. Environ. 42 (2007) 1921–1930, https://doi.org/10.1016/j.buildenv.2006.03.012.
- [11] R. Melotti, E. Santagata, M. Bassani, M. Salvo, S. Rizzo, A preliminary investigation into the physical and chemical properties of biomass ashes used as aggregate fillers for bituminous mixtures, Waste Manage. 33 (2013) 1906–1917, https://doi.org/10.1016/j.wasman.2013.05.015.
- [12] E. Carnielo, M. Zinzi, Optical and thermal characterisation of cool asphalts to mitigate urban temperatures and building cooling demand, Build. Environ. 60 (2013), https://doi.org/10.1016/j.buildenv.2012.11.004. 56–55.
- [13] H. Bianchetto, R. Miró, F. Pérez-Jiménez, Mezclas Asfálticas, Resistencia al envejecimiento de las mezclas bituminosas en caliente: beneficios y limitaciones de la incorporación de fílleres comerciales. Primera parte: estudios en base al método UCL. Infraestructura vial. Argentina. N 17: 13-24, 2007.
- [14] A. Modarres, M. Rahmanzadeh, Application of coal waste powder as filler in hot mix asphalt, Constr. Build. Mater. 66 (2014) 476–483, https://doi.org/ 10.1016/j.conbuildmat.2014.06.002.
- [15] K. Sobolev, I. Vivian, R. Saha, N. Wasiuddin, N. Saltibus, The effect of fly ash on the rheological properties of bituminous materials, Fuel 116 (2014) 471–477, https://doi.org/10.1016/j.fuel.2013.07.123.
- [16] Z. Chuanfeng, L. Dan, Q. Yong, B. Heng, L. Keyao, H. Junfei, Analysing the effects of the mesoscopic characteristics of mineral powder fillers on the cohesive strength of asphalt mortars at low temperatures, Constr. Build. Mater. 65 (2014) 330–337, https://doi.org/10.1016/j.conbuildmat.2014.04.123.
- [17] B. Huang, X. Shu, X. Chen, Effects of mineral fillers on hot-mix asphalt laboratory-measured properties, Int. J. Pavement Eng. 8 (2007) 1–9, https://doi.org/10.1080/10298430600819170.
- [18] S. Wu, M. Chen, J. Lin, Potential of recycled fine aggregates powder as filler in asphalt mixture, Constr. Build. Mater. 25 (2011) 3909–3914, https://doi.org/ 10.1016/j.conbuildmat.2011.04.022.
- [19] Y. Xue, S. Wu, J. Cai, M. Zhou, J. Zha, Effects of two biomass ashes on asphalt binder: dynamic shear rheological characteristic analysis, Constr. Build. Mater. 56 (2014) 7–15, https://doi.org/10.1016/j.conbuildmat.2014.01.075.
- [20] Y. Ke-zhen, X. Hong-bin, Z. Heng-larga, Effect of mineral filler on properties of warm asphalt mastic containing Sasobit, Constr. Build. Mater. 48 (2013) 622–627, https://doi.org/10.1016/j.conbuildmat.2013.07.085.
- [21] C. Sarah, G. Taylor-Lange, J. Stewart, M. Juenger, J. Siegel, The contribution of fly ash toward indoor radon pollution from concrete, Build. Environ. 56 (2012) 276–282, https://doi.org/10.1016/j.buildenv.2012.03.009.
- [22] IRAM 1542, Determinación de la concentración crítica y densidad del filler, Instituto argentino de racionalización de materiales, Argentina, 1992.

- [23] A. Synnefaa, T. Karlessia, N. Gaitania, M. Santamourisa, D. Assimakopoulosb, C. Papakatsikasc, Experimental testing of cool colored thin layer asphalt and estimation of its potential to improve the urban microclimat, Build. Environ. 46 (2011) 38–44, https://doi.org/10.1016/j.buildenv.2010.06.014.
- [24] C. Ruíz, Concentración crítica de fíller, su origen y significado en la dosificación de mezclas abiertas. Dirección de Vialidad Prov. de Buenos Aires. Argentina. Publicación N 11, 1960.
- [25] Ministerio de Obras Públicas, Manual de carreteras. Especificaciones técnicas generales de construcción. Vol. 5. Chile. 690 p., 2015.
- [26] Ministerio de Obras Públicas, Manual de carreteras. Especificaciones y Métodos de Muestreo, Ensaye y Control. Vol. 8. Chile. 1112 p., 2015.
- [27] G. Nesibe, A. Bappy, M. Said, R. Srinath, Mechanical performance and durability of treated palm fiber reinforced mortars, Build. Environ. 2 (2013) 131–142, https://doi.org/10.1016/j.ijsbe.2014.04.002.
- [28] S. Tapkin, Mechanical evaluation of asphalt-aggregate mixtures prepared with fly ash as a filler replacement, Can. J. Civ. Eng. 35 (2008) 27–40, https://doi.org/ 10.1139/L07-082.
- [29] G. Valdes, R. Miró, A. Martínez, A. Calabi, Effect of the physical properties of aggregates on aggregate-asphalt bond measured using the UCL method, Constr. Build. Mater. 73 (2014) 399–406, https://doi.org/10.1016/ j.conbuildmat.2014.09.098.
- [30] AENOR. Spanish Association for standardization and certification. Bituminous mixtures test methods for hot mix asphalt Part 17: Particle loss of porous asphalt specimen, UNE EN 12697-17:2006. Madrid: AENOR, 2006.
- [31] AENOR. Spanish Association for standardization and certification. Bituminous mixtures test methods for hot mix asphalt Part 35: Laboratory mixing, UNE EN 12697-35:2006. Madrid: AENOR, 2006.
- [32] AENOR. Spanish Association for standardization and certification. Bituminous mixtures test methods for hot mix asphalt Part 30: Specimen preparation by impact compactor, UNE EN 12697-3:2006. Madrid: AENOR, 2006.
- [33] AENOR. Spanish Association for standardization and certification. Bituminous mixtures test methods for hot mix asphalt Part 8: Determination of void characteristics of bituminous specimen, UNE EN 12697-8: 2006. Madrid: AENOR, 2006.
- [34] Pliego de prescripciones técnicas generales para obras de carreteras y puentes, Art. 542. Mezclas bituminosas tipo hormigón bituminoso, España. 514 p., 2015.
- [35] S. Borghini, V. Flachsland, N. Sucias, Elaboración del mapa de temperaturas viales de la república oriental del Uruguay para la selección de ligantes asfálticos de acuerdo al método superpave, XVIII Congreso Ibero Latinoamericano del Asfalto. Bariloche, Argentina, 2015.



Fig. 1. Ash used was sifted through a sieve N°200 (0.08 mm).

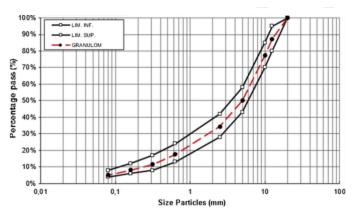


Fig. 2. Granulometric analysis used in the mixtures.



Fig. 3. Aggregates and filler for the mixture's preparation.



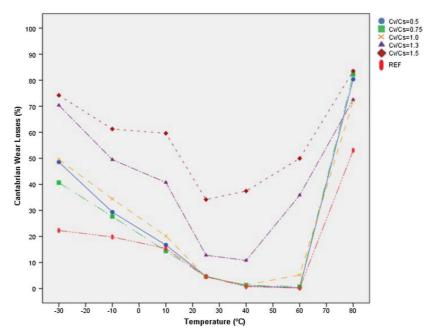


Fig. 6. Cantabrian losses vs. temperature.

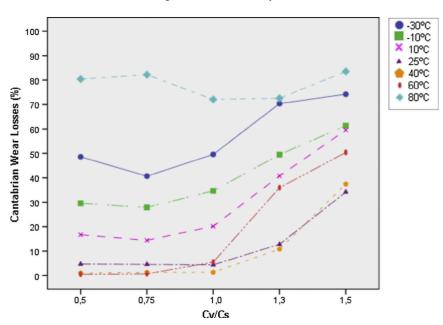


Fig. 7. Cantabrian losses vs. Volumetric Concentration.

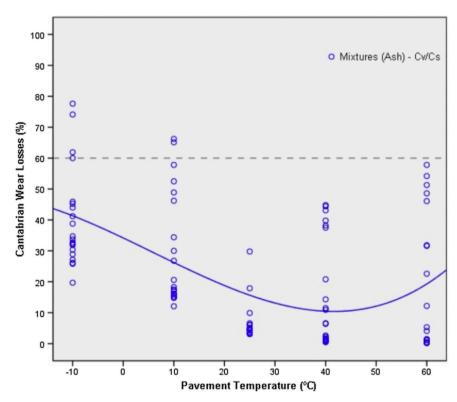
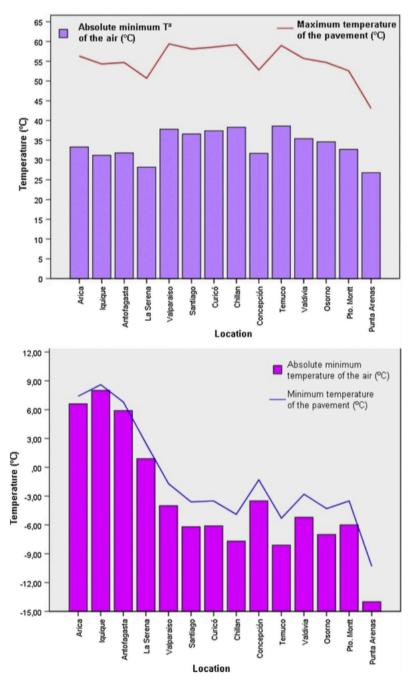


Fig. 8. Cantabrian wear losses vs. pavement temperature at service condition.



 $\textbf{Fig. 9.} \ \ \textbf{Range of pavement temperatures in service conditions along Chile.}$

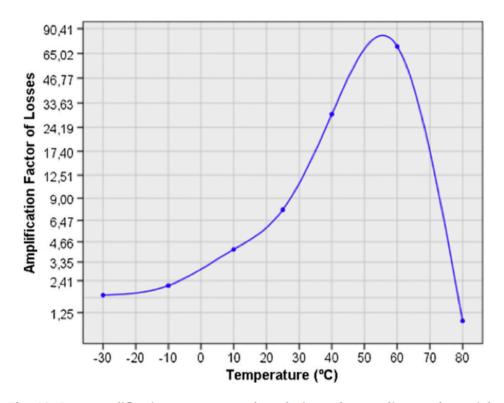


Fig. 10. Loss amplification parameter when dosing ash according to the weight criteria (Cv/Cc = 0.75).

Table 1 Granulometric analysis used for IV-A-12 mixture.

Mixture	25	20	12.5	10	5	2.5	0.63	0.315	0.16	0.08
IV-A-12	-	100	80-95	70-85	43-58	28-42	13-24	8-17	6-12	4-8

Table 2 Specific characteristics of bitumen.

Test	Method	Type of bitumen		
		CA-24	STYLINK	
Penetration 25 °C (0.1 mm)	NLT124	59	79	
Softening Point (°C)	NLT125	50	69.5	
Brittle Point of Fraas (°C)	NLT182	-	-18	
Absolute viscosity (60°)	NLT181	3039	-	

Table 3Volumetric characterization of the filler.

Filler	Dry Density (gr./ cm³)	Critical concentration (Cs)
Ash	2.48	0.22

Table 4 Design parameters of bituminous mixtures.

Materials	Properties	Condition	Quantity (gr.)
Aggregate	Semi	IV A 12	1034
Filler	Rec	%	60
	Ash	0.50	16.91
		0.75	27.04
		1.00	38.60
		1.3	54.81
		1.5	67.40
Bitumen	CA-24 Stylink	5% (about mixture)	57.00

Table 5Density and voids content of the bituminous mixtures.

Mixtures	Density (gr./cm ³)	Voids content (%)
Cv/Cs = 0.5	2.285	4.96
Cv/Cs = 0.75	2.304	5.10
Cv/Cs = 1.0	2.292	5.00
Cv/Cs = 1.3	2.271	5.76
Cv/Cs = 1.5	2.254	5.91
REF.	2.343	4.43
MOD.	2.332	4.57

Table 6Cantabrian wear losses by different temperatures (300 rev.)

Temperature (°C)	Type of mixt (Cv/Cc)						
	0.5	0.75	1.0	1.3	1.5	REF.	MOD.
-30	48.6	40.7	49.6	70.4	74.2	22.3	13.8
-10	29.6	27.9	34.7	49.5	61.3	20.0	8.5
10	16.7	14.4	20.2	40.8	59.5	15.4	3.7
25	4.7	4.5	4.4	12.8	34.2	4.6	0.8
40	1.1	1.3	1.4	10.8	37.5	0.7	0.2
60	0.5	0.7	5.5	36.0	50.0	0.3	0.2
80	80.4	82.2	72.1	72.6	83.5	53.1	-

Table 7Relation between volume concentrations.

	Volume	tric concen	tration (Cv/	Cs)	
	0.5	0.75	1.0	1.3	1.5
Weight relation (F/b)	0.297	0.475	0.678	0.960	1.181

Table 8Correlation coefficients of the mode.

Model	R	R squared	R squared (corrected)	Error típ.
% Losses	0.873	0.761	0.755	11.686

Table 9Relationship between exogenous variables.

Model	Squared suma	gl	Quadratic medium	F	Sig.
% Losses Regression	50558.62	3	16852.87	123.42	0.000
Residual	15839.84	116	136.55		
Total	66398.46	119			

Table 10Values of partial regression coefficients.

	Non standa	ırd values	Tip. values			
Model	В	Error típ.	Beta	t	Sig.	
% Losses (Constante)	965.544	176.317		5.476	0.000	
Cv/Cs	20.987	4.089	0.322	5.132	0.000	
Temperature (°C)	-0.493	0.036	-0.630	-13.889	0.000	
Density (gr./cm3)	-416.342	75.942	-0.344	-5.482	0.000	