

Moisture damage resistance of grave emulsion manufactured with waste biomass fly ashes

A.R. Pasandín ^{1,*}, I. Pérez ¹, B. Gómez-Meijide ², A. Ramírez ³, M.M. Cano ³

¹ School of Civil Engineering, Universidade da Coruña (UDC), Campus de Elviña s/n 15071, La Coruña, Spain.

² European Asphalt Pavement Association (EAPA). Rue du Commerce 77 – 1040 Brussels – Belgium

³ Sacyr Construcción, S.A., Madrid, Spain

* Corresponding author. Tel.: +34-881011438. E-mail address: arodriguezpa@udc.es

Abstract

Purpose

Biomass combustion is an economic and environmentally friendly source of energy with a growing demand. Nevertheless, the environmental management of fly ashes generated during energy production must be improved. In this regard, this study analyses the feasibility of using biomass fly ashes from the paper industry (BioFAPI) as filler in cold asphalt mixture (type grave emulsion GE1).

Materials and methods

The chemical and mineralogical composition of BioFAPI samples were analysed by means of X-ray fluorescence spectroscopy (XRF) and X-ray diffraction (XRD). A qualitative evaluation of their morphology was performed using a scanning electron microscope (SEM). In addition, the grain-size distribution was obtained by means of light-scattering analysis techniques. Finally, immersion-compression tests were conducted in order to analyse the stripping potential of the grave emulsion manufactured using BioFAPI as filler. These results were compared with those of a control mixture.

Results

In this case, the use of BioFAPI as filler led to mixtures with adequate moisture damage resistance. Also, the compression strength of the mixture containing BioFAPI was higher than that of the control mixture.

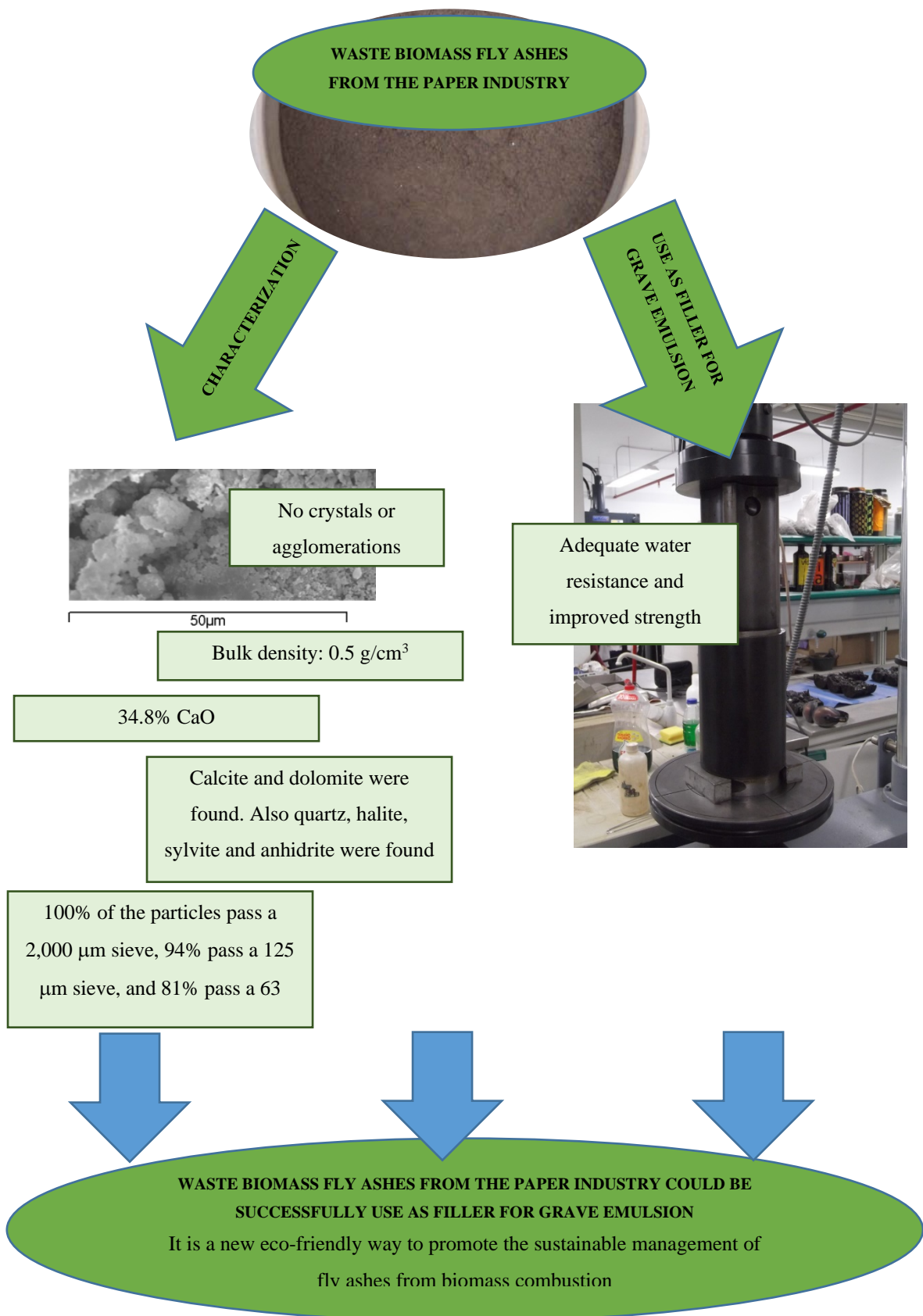
Conclusions

Thus, BioFAPI could be successfully used as filler for grave emulsion. It is a new eco-friendly way to promote the sustainable management of fly ashes from biomass combustion.

Keywords

Waste biomass fly ash; paper industry; filler; asphalt mixture; grave emulsion. It is a new eco-friendly way to promote the sustainable management of fly ashes from biomass combustion.

Graphical abstract



Statement of novelty

Biomass combustion is a low-cost source of energy that contributes to the energetic valorisation of organic wastes and helps to decrease CO₂ emissions from fossil fuels. Nevertheless, the environmental management of biomass fly ashes from the biomass combustion is one of its main drawbacks. In this regard, to solve the aforementioned problem, some researchers have analysed the feasibility of using biomass fly ashes as filler for bituminous mixtures. Most of them, used this waste as filler for hot-mix asphalt (HMA), showing, that depending of the type of fly ashes, the mixtures could display adequate or inadequate water resistance. This research describes a laboratory research on the successful and eco-friendly valorization of waste biomass fly ashes from the paper industry as filler for cold asphalt mixtures (CAM) type grave emulsion.

Introduction

Biomass combustion seems to be a sustainable way of heat and electricity production because it is a renewable energy source and helps to decrease CO₂ emissions from fossil fuels [1]. Biomass combustion is also a low-cost source of energy [2] that contributes to the energetic valorisation of organic wastes [3]. Despite all these advantages, biomass combustion also has some drawbacks. One of the main inconveniences associated to using biomass combustion as an energy source is the environmental management of biomass fly ashes [4].

Biomass fly ashes, or bioashes, can be defined as solid byproducts generated during the combustion of biomass [5]. Depending on the biomass used and the incineration processes, the composition of bioashes can vary significantly [6]. Therefore, the reuse or recycling of biomass fly ashes needs detailed analysis. It must be noted that the advantages of using biomass as a source of energy lead to a growing demand for its use. Consequently, it is necessary to explore new ways of reusing biomass fly ashes to avoid landfilling, as they are solid waste.

In recent decades, there has also been a growing need for using alternative raw materials in the pavement industry. This is due to the risk of depletion of natural resources [7] and the economic and environmental impact of their exploitation [4].

As is well known, the filler plays a crucial role in the asphalt mixtures properties and its performance [8-10]. In fact, the filler has a high specific surface area, which conditions the binder content of the mixture. It also forms a mastic with the binder that gives cohesion to the mixture and takes part in the adhesion

phenomena. Finally, the filler influences the percentage of air voids in the mixture and, therefore, the impermeability and the resistance of the mixture.

Thus, to solve the aforementioned problem, some researchers have analysed the feasibility of using biomass fly ashes as filler for bituminous mixtures. Melotti et al. [11] conducted a preliminary study on the potential use of 27 types of biomass fly ashes as filler in bituminous mixtures. They concluded that after a sieving process, these ashes may be considered as a valid alternative to natural fillers.

Mirhosseini et al. [12] analysed the water resistance of bituminous mixtures made with date seed ash (DSA) as filler and concluded that these mixtures showed better moisture damage resistance than conventional ones.

Tahami et al. [5] analysed the feasibility of using rice husk ash (RHA) and DSA as fillers in hot-mix asphalt (HMA). They concluded that these fillers lead to HMA with improved thermal sensitivity and, therefore, higher resistance to permanent deformation and higher fatigue resistance than that obtained using conventional filler. Also, these mixtures showed higher Marshall stability and higher stiffness modulus. The authors stated that the optimum replacement percentages were 75% of the HMA filler for RHA and 100% of the HMA filler for DSA.

Mistry et al. [13] also concluded that the use of RHA as filler in HMA leads to bituminous mixtures with higher stiffness. Regarding moisture damage resistance, the authors stated that the use of 4% RHA obtained better results than conventional fillers. However, this did not occur for higher percentages.

Only one investigation dealing with the use of biomass waste fly ashes from the paper industry (BioFAPI) as filler for HMA has been found in the literature [14]. The authors indicate that the use of BioFAPI as filler leads to HMA with inadequate moisture damage resistance.

The aim of the present research is to find new eco-friendly ways to promote the sustainable management of BioFAPI. For this purpose, this study attempts to deepen the knowledge of the use of this material as filler in cold asphalt mixtures (CAM). Because, according to the literature, the main drawback associated with the use of this alternative filler seems to be low resistance to moisture damage, the present investigation is focused on this property.

Materials and methods

Bituminous mixtures

The aggregate gradation of the CAM (**Fig. 1**) chosen in this investigation is a GE1 grave emulsion according to the requirements of the Spanish Technical Association of Bituminous Emulsions (ATEB) [15].

Aggregates

A cornubianite – a metamorphic quarry siliceous aggregate - was used to manufacture the CAM. The properties of the aggregate are shown in table 1. According to ATEB [15] requirements, the bulk specific gravity (ρ_a), the water absorption (W_{24}), the sand equivalent (SE), the Los Angeles (LA) abrasion coefficient, and the flakiness index (FI), were determined for the aggregates. These properties fulfil the ATEB requirements for all heavy-traffic categories.

Filler

A cornubianite filler with a bulk density of 0.77 g/cm^3 (**Fig. 2**) was used for the manufacture of CAM as control filler. BioFAPI (**Fig. 3**) was also examined as filler. Its bulk density is 0.5 g/cm^3 . Both fillers comply with the limits of $0.5\text{--}0.8 \text{ g/cm}^3$ established by ATEB [15].

Binder

For the manufacture of GE1, a slow-setting cationic bitumen emulsion, C60B5 GE, with 60% bitumen content was selected.

Fly ash characterization

As noted, the mineralogical and chemical compositions of biomass fly ash play a crucial role in its performance. In this regard, BioFAPI samples were analysed by means of X-ray fluorescence spectroscopy (XRF) (Bruker S4 Pioneer Fluorescence Spectrometer) and X-ray diffraction (XRD) (Siemens D5000 X-ray diffractometer). Also, the shape of the biomass fly ashes could affect their performance as filler. Therefore, a qualitative evaluation of their morphology was performed using a scanning electron microscope (SEM). In addition, the grain-size distribution of biomass fly ashes was

obtained by means of light-scattering analysis techniques (Saturn Digisizer II 5205).

Mixture design

The optimum emulsion and water content of GE1 was obtained as follows. Firstly, the optimal total water content was obtained from the Modified Proctor test following UNE 103-501-94 [20]. Secondly, cylindrical samples with different bitumen/emulsion contents were tested in order to analyse their water resistance by means of the immersion–compression test, according to the Spanish standard NLT-162 [21]. Residual binder contents of 2%–6% and water contents of 6%–12% of the total weight of the dry aggregates were tested. The optimal contents were 3% bitumen and 6% water, giving air voids content (Va) of 11.3% [22].

Moisture damage resistance

The resistance of CAM to water damage was studied with the immersion–compression test, according to the Spanish NLT-162 standard [21] (**Fig. 4**). In this test, ten cylindrical specimens (101.6 mm diameter and 101.6 mm height) were manufactured at the optimum water and residual-binder content by using BioFAPI as filler. A control mix made with natural cornubianite as filler was also analysed. The test was conducted once in each case. Thus, a total of 20 samples were tested.

In this case, the wet subset was composed of specimens that were immersed in water at a temperature of 49 °C for 96 h, while the other five specimens (dry subset) were maintained at room temperature in a dry state. Finally, the specimens were subjected to unconfined compression (Axial Compression test, NLT-161 [21], and the average value of the strength in each subset was determined. The retained strength ratio (RSR) was calculated as follows:

$$RSR (\%) = \frac{USC_W}{USC_D} \cdot 100 \quad (2)$$

where USC_W is the unconfined compression strength of the group immersed in water at 49 °C (MPa) and USC_D the unconfined compression strength of the group that was not submerged.

According to ATEB [15] this index must be higher than 75% for GE1, whereas UCS must be higher than 1.5 MPa for the non-immersed group and 1.2 MPa for the immersed group for T2 heavy-traffic category and higher (annual average daily heavy traffic higher than 200).

Results and discussion

BioFAPI characterization

Fig. 5 shows the chemical composition of BioFAPI (XRF results). As can be seen, the main chemical constituent is CaO (34.8%), followed by the loss on ignition (LOI) (15.8%) and SiO₂ (11.6%). Therefore, the SiO₂ percentage, which is usually associated with poor adhesion, is somewhat lower than the CaO percentage, which, conversely, is associated with adequate adhesion.

It is interesting to compare the BioFAPI chemical composition with the chemical composition of other biomass fly ashes. In this regard Sarkinenn et al. [6] analysed two biomass fly ashes and obtained percentages of SiO₂ (37.43% and 20.38%), CaO (10.96% and 40.13%) and LOI (2% in both cases) different from those obtained for the BioFAPI. Melotti et al. [11] obtained that the main component was calcium oxide (CaO) (in 13 out of 21 ashes), silicon dioxide (SiO₂) (in 7 out of 21 ashes) and potassium oxide (K₂O) (in 1 out of 21 ashes). These authors found CaO percentages ranging from 0% to 65% and SiO₂ percentages ranging from 5% to 95%. For these 21 fly ashes, the LOI of ignition was between 0.1% and 12.4%. That is, as stated before, it must be noted that it exists a great variability into the chemical composition of the biomass fly ashes, depending on its origin.

Regarding its mineralogical composition (XRD results) it must be noted that calcite (CaCO₃) and dolomite (CaMg(CO₃)₂) were found in BioFAPI. These two compounds generally have good bitumen adhesion [23]. Portlandite (Ca(OH)₂), that is, hydrated lime, is also part of the mineralogical composition of BioFAPI. As is well known, hydrated lime has beneficial effects on the stripping potential of bituminous mixtures [24]. By contrast, some compounds associated with poor adhesion [23], such as quartz (SiO₂), halite (NaCl), sylvite (KCl), or anhydrite (Ca(SO₄)), were also found.

Therefore, the analysis of the chemical and mineralogical composition of BioFAPI is not conclusive in terms of stripping potential [14].

Fig. 6 shows a SEM image of BioFAPI. As can be seen, no crystals or agglomerations were found. Thus, it is not expected that the morphology of these filler particles could negatively affect their performance [14].

In the case of biomass fly ashes, **Fig. 7** shows that a 100% of the particles pass a 2,000 µm sieve, 94% pass a 125 µm sieve, and 81% pass a 63 µm sieve.

Resistance to water damage of CAM containing BioFAPI

As noted, the mineralogical composition of BioFAPI includes Portlandite ($\text{Ca}(\text{OH})_2$), which is chemically hydrated lime. This compound is especially effective in improving water resistance if it comes into contact with siliceous and wet aggregates owing to the type of chemical reaction generated at the aggregate–binder interface.

For this reason, it seemed to be a good idea to use BioFAPI as filler for CAM made with siliceous aggregates. In this regard and as mentioned above, grave emulsion GE1 manufactured with cornubianite was selected.

As shown in table 2, an RSR of 81.6% was obtained for the control mixture; for the mixture made with BioFAPI, a similar RSR of 81.8% was obtained. These results exceed the 75% specified by ATEB for this type of cold mix for roadways with heavy-traffic category T2 or higher (annual average daily heavy traffic higher than 200). In both cases, the dry strength (2,914 KPa for the control mixture and 3,434 KPa for the BioFAPI mixture) was higher than the 1,500 kPa required by the aforementioned specifications, and the wet strength (2,379 Kpa for the control mixture and 2,810 KPa for the BioFAPI mixture) was higher than the required 1,200 kPa. However, it should be noted that the resistance of the mixture containing BioFAPI was higher than that of the control mixture in both dry and wet conditions.

According to these results, it could be stated that BioFAPI can be successfully used as filler for CAM.

Conclusions

This study investigated the moisture damage resistance of cold asphalt mixtures (CAM) manufactured using waste biomass fly ashes (BioFAPI) as filler. A grave emulsion (type GE1) was studied. As a result, the following conclusions were drawn from this research:

- Grave emulsion GE1 was manufactured using BioFAPI as filler and siliceous (cornubianite) aggregates. This mixture complied with the Spanish specifications for water resistance.
- The compression strength of the grave emulsion in the dry and wet states was higher than that obtained for the control mixture.
- The main reason for this adequate performance seems to be that BioFAPI includes portlandite (Ca(OH)_2) in its mineralogical composition. Portlandite, or hydrated lime, is especially effective in improving the water resistance of bituminous mixtures if it comes into contact with siliceous and wet aggregates.
- Thus, it can be concluded that BioFAPI may be used as alternative eco-friendly filler for the manufacture of CAM.

This is a preliminary research with encouraging results. However, deeper analyses on the use of waste biomass fly ashes as alternative filler in CAM should be conducted in order to guarantee adequate performance.

References

- [1]. van Eijkm, R.J, Obernberger, I & Supancic, K (2012). 30102040-PGR/R&E 11-2142. IEA Bioenergy Task 32. Deliverable D4. Options for increased utilization of ash from biomass combustion and co-firing. Retrieved from http://www.ieabcc.nl/publications/Ash_Utilization_KEMA.pdf. Accessed 22 June, 2018.
- [2]. Balata, G., & Tola, A. (2018). Cost-opportunity analysis of the use of *Posidonia oceanica* as a source of bio-energy in tourism-oriented territories. The case of Alghero. *J. Clean Prod.*, 172, 4085-4098. <https://doi.org/10.1016/j.jclepro.2017.02.072>
- [3]. Rodrigues, S., Almeida, A., Ribeiro, A., Neto, P., Ramalho, E., & Pilão, R. (2020). Gasification of cork wastes in a fluidized bed reactor. *Waste Biomass. Valor.* , 11(3), 1159-1167. <https://doi.org/10.1007/s12649-018-0335-5>
- [4]. Rajamma, R., Ball, R. J., Tarelho, L. A., Allen, G. C., Labrincha, J. A., & Ferreira, V. M. (2009). Characterisation and use of biomass fly ash in cement-based materials. *J. Hazard. Mater.*, 172(2-3), 1049-1060. <https://doi.org/10.1016/j.jhazmat.2009.07.109>
- [5]. Tahami, S. A., Arabani, M. & Mirhosseini, A. F. (2018). Usage of two biomass ashes as filler in hot mix asphalt. *Constr. Build. Mater.*, 170, 547-556. <https://doi.org/10.1016/j.conbuildmat.2018.03.102>.
- [6]. Sarkkinen, M., Kujala, K., Kemppainen, K., & Gehör, S. (2018). Effect of biomass fly ashes as road stabilisation binder. *Road Mater. Pavement Des.*, 19(1), 239-251. <https://doi.org/10.1080/14680629.2016.1235508>
- [7]. Varma, S., Jamrah, A., Kutay, M. E., Korkmaz, K. A., Haider, S. W., & Buch, N. (2018). A framework based on engineering performance and sustainability to assess the use of new and recycled materials in pavements. *Road Mater. Pavement Des.*, 1-20. <https://doi.org/10.1080/14680629.2018.1474789>
- [8]. Nazary, M., & Kofteci, S. (2018). Effect of filler types and asphalt penetration grade on properties of asphalt mastic. *Pet. Sci. Technol.*, 36(18), 1503-1509. <https://doi.org/10.1080/10916466.2018.1501385>
- [9]. Choudhary, J., Kumar, B. and Gupta, A. (2018). Effect of filler on the bitumen-aggregate adhesion in asphalt mix. *Int. J. Pavement Eng.* 1-9. <https://doi.org/10.1080/10298436.2018.1549325>
- [10]. Zhang, J., Li, X., Liu, G., & Pei, J. (2019). Effects of material characteristics on asphalt and filler interaction ability. *Int. J. Pavement Eng.* 20(8), 928-937. <https://doi.org/10.1080/10298436.2017.1366765>

- [11].Melotti, R., Santagata, E., Bassani, M., Salvo, M., & Rizzo, S. (2013). A preliminary investigation into the physical and chemical properties of biomass ashes used as aggregate fillers for bituminous mixtures. *Waste Manage.* 33(9), 1906-1917.
<https://doi.org/10.1016/j.wasman.2013.05.015>
- [12].Mirhosseini, S.F., Khabiri, M.M, Kavussi, A. & Kamali, M.J. (2016). Applying surface free energy method for evaluation of moisture damage in asphalt mixtures containing date seed ash. *Constr. Build. Mater.* 30 (125), 408–416. <https://doi.org/10.1016/j.conbuildmat.2016.08.056>
- [13].Mistry, R., Karmakar, S., & Kumar Roy, T. (2018). Experimental evaluation of rice husk ash and fly ash as alternative fillers in hot-mix asphalt. *Road Mater. Pavement Des.*, 1-12.
<https://doi.org/10.1080/14680629.2017.1422791>
- [14].Pasandín, A.R., Pérez, I., Ramírez, A., and Cano, M.M. (2016). Moisture damage resistance of hot-mix asphalt made with paper industry wastes as filler. *J. Clean Prod.*, 112, 853-862.
<https://doi.org/10.1016/j.jclepro.2015.06.016>
- [15].ATEB, Technical Association of Bituminous Emulsions (no date). Gravel-emulsion specifications (in Spanish). Retrieved from http://ateb.es/images/pdf/PLI_GRAVA.pdf. Accessed 22 June, 2018.
- [16].AENOR (2014). Asociación Española de Normalización y Certificación. UNE-EN 1097-6 “Tests for mechanical and physical properties of aggregates. Determination of particle density and water absorption”. Madrid, Spain (in Spanish).
- [17].AENOR (2012b). Asociación Española de Normalización y Certificación. UNE-EN 933-8 “Tests for geometrical properties of aggregates. Assessment of fines. Sand equivalent test”. Madrid, Spain (in Spanish).
- [18].AENOR (2012a). Asociación Española de Normalización y Certificación. UNE-EN 933-3 “Tests for geometrical properties of aggregates. Determination of particle shape. Flakiness index”. Madrid, Spain (in Spanish).
- [19].AENOR (2010). Asociación Española de Normalización y Certificación. UNE-EN 1097-2 “Tests for mechanical and physical properties of aggregates. Methods for the determination of resistance to fragmentation”. Madrid, Spain (in Spanish).
- [20].AENOR (1994). Asociación Española de Normalización y Certificación. UNE 103501:94. “Unbound and hydraulically bound mixtures. Test methods for laboratory reference density and water content. Proctor compaction”. Madrid, Spain (in Spanish).
- [21]. MOPT, 2002. MOPT (Public Works and Transportation Ministry). NLT Standards. Road Test. General Directorate of Highways (second ed.). Madrid, Spain (in Spanish).

- [22]. Gómez-Meijide, B., & Pérez, I. (2014). Effects of the use of construction and demolition waste aggregates in cold asphalt mixtures. *Constr. Build. Mater*, 51, 267-277.
<https://doi.org/10.1016/j.conbuildmat.2013.10.096>
- [23]. Bagampadde, U. (2004). On investigation of stripping in bituminous mixtures. Doctoral Thesis. Karlstad University, Sweden. Retrieved from <https://www.diva-portal.org/smash/get/diva2:7644/FULLTEXT01.pdf>. Accessed 20 June, 2018.
- [24]. Little, D.N. and Epps, J.A. 2001. The benefits of hydrated lime in hot mix asphalt. Report prepared for National Lime Association. Retrieved from <http://citeseerx.ist.psu.edu/viewdoc/download?doi=10.1.1.508.3233&rep=rep1&type=pdf>. Accessed 01 July, 2018.

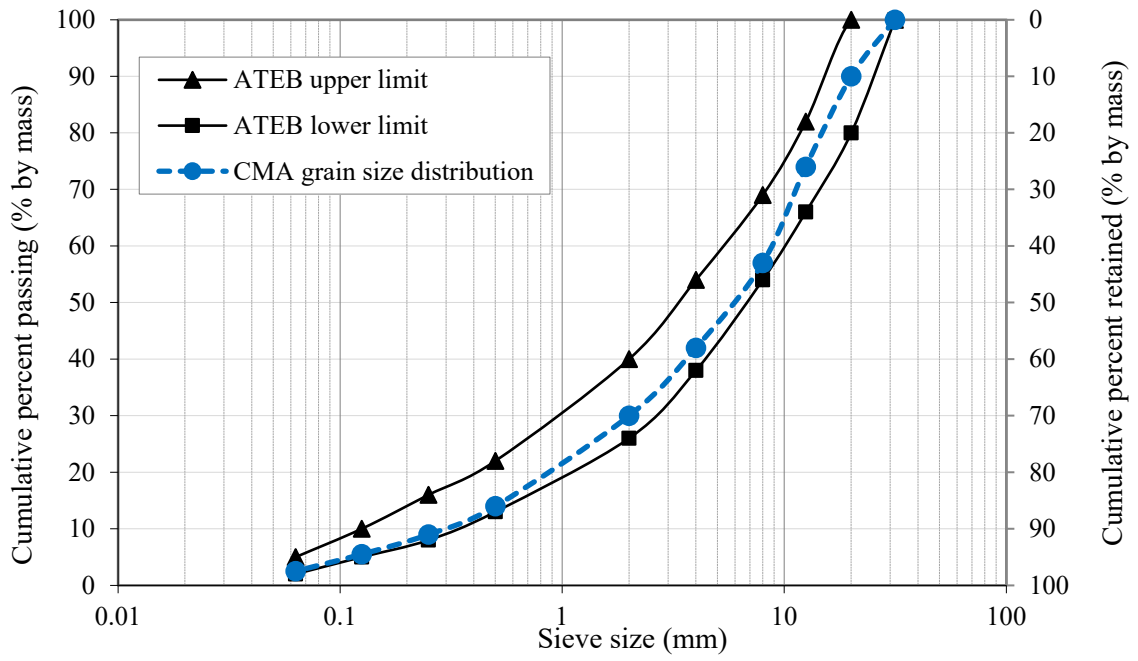


Fig. 1 Grain size distribution of grave-emulsion GE1



Fig. 2 Example of bulk density test (cornubianite)



Fig. 3 Waste biomass fly ashes from the paper industry



Fig. 4 Immersion-compression test

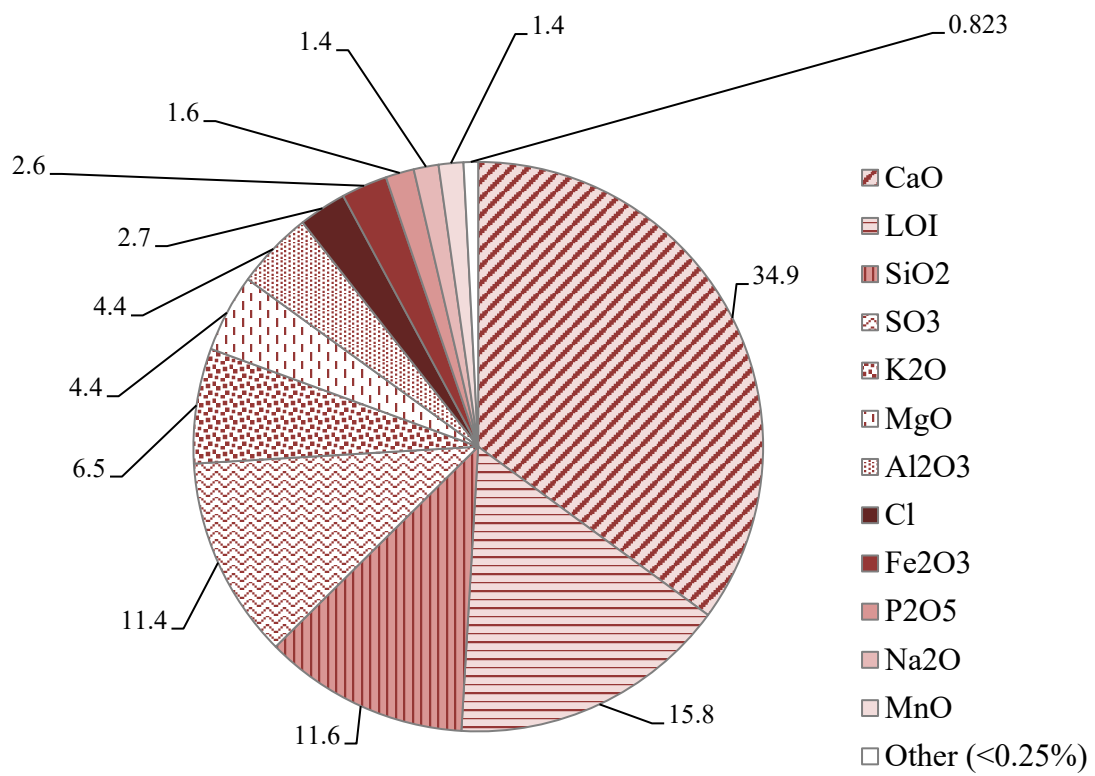


Fig. 5 Chemical composition of biomass fly ashes from the paper industry (XRF results)

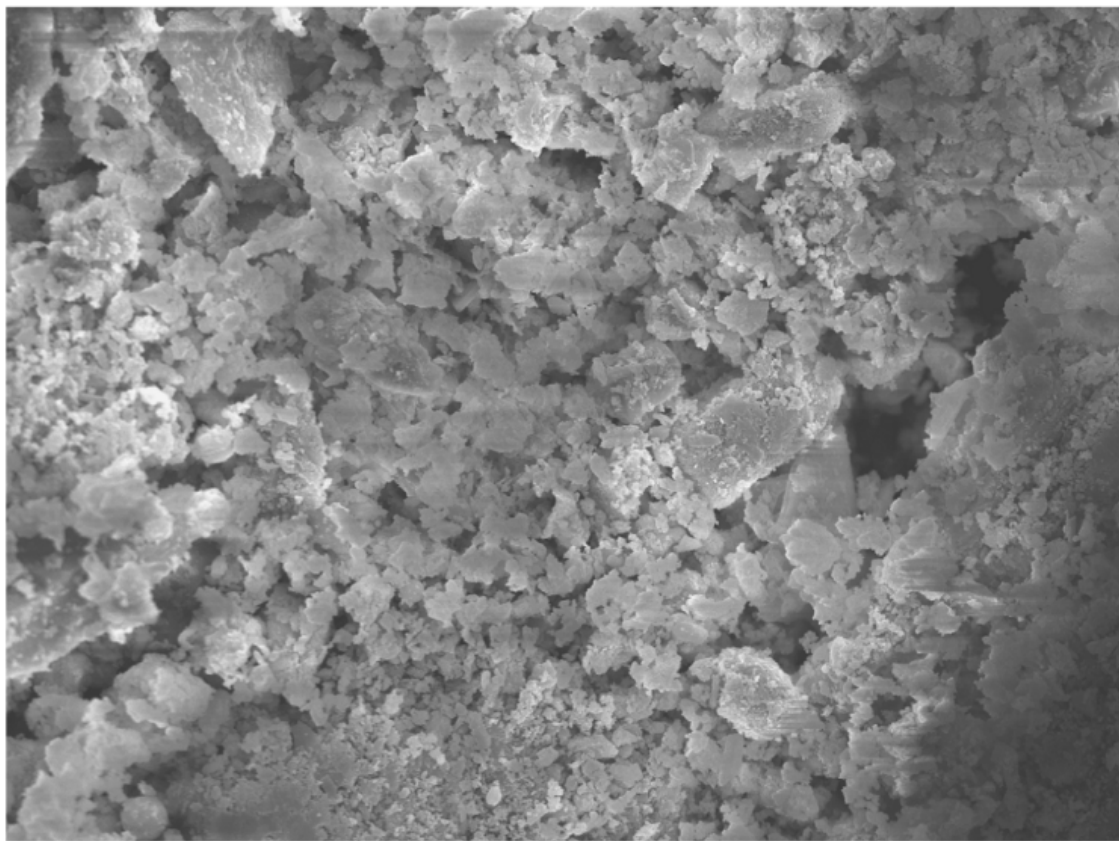


Fig. 6 SEM picture of the biomass fly ashes from the paper industry

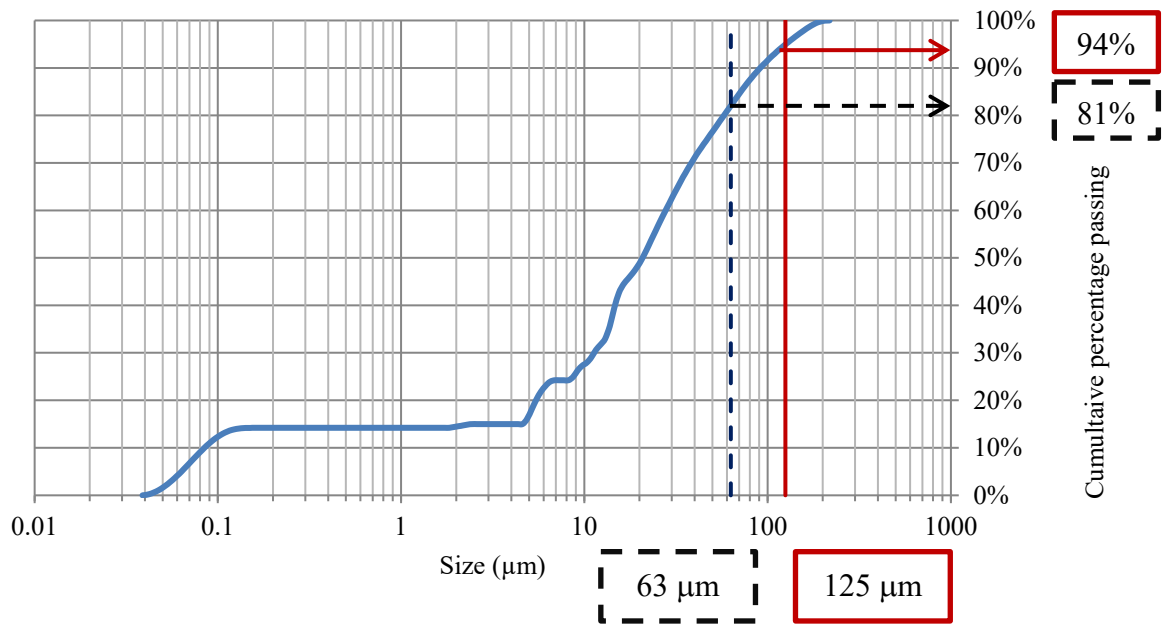


Fig. 7 Grain size distribution of the biomass fly ashes from the paper industry

Property	Cornubianite	ATEB Specifications (*)							
		T00	T0	T1	T2	T31	T32	T4	
ρ_a (g/cm ³)	EN-1097-6 [16]	2.78	-	-	-	-	-	-	-
WA ₂₄ (%)	EN 1097-6 [16]	0.5	-	-	-	-	-	-	-
SE (%)	EN 933-8 [17]	78	≥ 50	≥ 50	≥ 50	≥ 50	≥ 50	≥ 50	≥ 50
FI (%)	EN 933-3 [18]	19.8	≤ 20	≤ 25	≤ 25	≤ 25	≤ 25	≤ 30	≤ 30
LA abrasion (%)	EN 1097-2 [19]	14	≤ 25	≤ 25	≤ 25	≤ 30	≤ 30	≤ 30	-

(*) Traffic category T00 refers to AADHT (Annual Average Daily Heavy Traffic) ≥ 4,000

Traffic category T0 refers to 4,000 > AADHT ≥ 2,000

Traffic category T1 refers to 2,000 > AADHT ≥ 800

Traffic category T2 refers to 800 > AADHT ≥ 200

Traffic category T31 refers to 200 > AADHT ≥ 100

Traffic category T32 refers to 100 > AADHT ≥ 50

Traffic category T4 refers to AADHT < 50

Table 1. Aggregates characterization

GE1	Residual binder content (%)	Water content (%)	USC _{DRY} (Kpa)	USC _{WET} (Kpa)	RSR (%)
Control	3.0%	6.0%	2,914	2,379	81.6
Biomass fly ashes as filler			3,434	2,810.1	81.8

Table 2. Moisture damage resistance results for GE1