MATERIALES DE CONSTRUCCIÓN
Vol. 67, Issue 328, October–December 2017, e138
ISSN-L: 0465-2746
http://dx.doi.org/10.3989/mc.2017.07516

# Using petrographic techniques to evaluate the induced effects of NaCl, extreme climatic conditions, and traffic load on Spanish road surfaces

A. P. Pérez-Fortes[a] ✉, M. J. Varas-Muriel[a,b], P. Castiñeiras[a]

[a] Dept. de Petrología y Geoquímica, Facultad de Ciencias Geológicas. Universidad Complutense (UCM) (Madrid, Spain)
[b] Dept. de Geomateriales. Instituto de Geociencias (IGEO-CSIC) (Madrid, Spain)
✉ appfortes@gmail.com

Received 23 June 2016
Accepted 21 December 2016
Available on line 5 October 2017

**ABSTRACT:** The asphalt surface layer is the most exposed to weather and traffic conditions on roads, especially those subjected to winter maintenance. Therefore, a deep knowledge of the mechanisms which can damage this layer is necessary to improve its design, construction and long-term use. With this purpose, two types of asphalt mixtures used on roads from NW Spain were subjected to durability tests (freezing-thaw and thermal-stress) with a saturated NaCl solution. After the durability tests, a wheel tracking test was performed on the samples, and the resultant material was analyzed by optical polarized light and fluorescence microscopy. This analysis showed that the binder-aggregate low adhesion was the main responsible of the asphalt mixture damage. This damage was concentrated in the aggregates because the binder acted as an impermeable wall. Consequently, the NaCl solution penetrated and degraded the aggregates quickly and strongly.

**KEYWORDS:** Aggregate; Asphalt surface; Winter maintenance; Traffic; Petrography

**Citation/Citar como:** Pérez-Fortes, A.P.; Varas-Muriel, M.J.; Castiñeiras, P. (2017) Using petrographic techniques to evaluate the induced effects of NaCl, extreme climatic conditions, and traffic load on Spanish road surfaces. *Mater. Construcc*. 67 [328], e138. http://dx.doi.org/10.3989/mc.2017.07516

**RESUMEN:** *Utilización de técnicas petrográficas para evaluar los efectos inducidos del NaCl, condiciones climáticas extremas y el paso del tráfico en las superficies de las carreteras españolas*. Las capas de rodadura son las más expuestas al clima y al efecto del tráfico, especialmente en carreteras sometidas a mantenimiento invernal. Por este motivo, es importante conocer mejor los mecanismos de deterioro de dichas capas a la hora de mejorar su diseño, construcción y uso a largo plazo. Con este fin, dos tipos de mezclas asfálticas utilizadas en el NO de España fueron expuestas a ensayos de durabilidad (heladicidad y choque térmico) con una solución saturada de NaCl. Después, se realizó el ensayo de pista y el material deteriorado y deformado resultante fue analizado mediante microscopia óptica de luz polarizada y de fluorescencia. Dicho análisis mostró que el principal responsable de la degradación de la mezcla fue la baja adhesión árido-betún. Esta degradación se concentró en el árido debido a que el ligante actuó como una pared impermeable. En consecuencia, la solución de NaCl pudo penetrar y degradar rápida e intensamente el árido.

**PALABRAS CLAVE:** Árido; Capa de rodadura; Mantenimiento invernal; Tráfico; Petrografía

**ORCID ID:** A. P. Pérez-Fortes (http://orcid.org/0000-0003-0068-7722); M. J. Varas-Muriel (http://orcid.org/0000-0003-2378-9414); P. Castiñeiras (http://orcid.org/0000-0002-1835-3192)

**Copyright:** © 2017 CSIC. This is an open-access article distributed under the terms of the Creative Commons Attribution License (CC BY) Spain 3.0.

## 1. INTRODUCTION

Currently, the investment in the maintenance of the Spanish roads is low because of the economic crisis. As a result, roads are in a poor state of conservation (1), specifically the surface layer, which is the most exposed to traffic, weather, and other effects of external agents. For these reasons, it is indispensable to improve the design and construction criteria of this layer in order to produce more durable and, thus, more environmentally friendly pavements. In this regard, it is important to consider the deterioration processes taking place when those pavements are exposed to extreme conditions, such as the use of salts during winter maintenance practices, to better understand the pavement damaging mechanisms.

More than 25,000 km of Spanish roads are exposed to winter maintenance, and NaCl is the most common deicer applied (2). However, this product has a negative impact on motor vehicles, transportation infrastructure, as well as on the environment (3). Particularly evident is the effect of this compound on the durability of asphalt pavements. Freeze–thaw cycles cause the expansion of water, which results in the decrease of the pavement's indirect tensile strength but, combined with salts, especially chlorides, it is also responsible of the hardening of the binder and, therefore, the loss of pavement elasticity. In addition, the combination of freeze-thawing and salting causes either the interfacial damage between binder and aggregate or the fracture of the binder, which produces a greater increase of weight loss (4–5). The latter is considered the main cause of asphalt pavement deterioration, and it is directly related to the low adhesion between the aggregate and the binder in asphalt mixtures (6–9). However, the mechanisms which produce the low adhesion between the aggregates and the binder are still unclear.

The so-called stripping phenomenon, or asphalt-aggregate bond failure, can be produced by a large number of factors, including composition, porosity and roughness of the aggregates, wettability between bitumen and aggregates, chemical composition at the interface (10), exposure aggregate history (e.g., freshly crushed versus days of exposure to environmental weathering after crushing), asphalt viscosity or traffic cyclic loading (11). However, the most accepted hypothesis is that the aggregate surface chemistry is the main responsible of the stripping process (7, 11). Fromm (12) reported that stripping is high in mixes with acidic granites with hydroxylated $SiO_4$. Water vapor, which is emitted during the production of asphalt mixtures, can form strong hydrogen bonds with siliceous surfaces, forming silanols. This hydrated silica molecules can cause the replacement of the bitumen polar parts (13). Vuorinen and Hartikainen (14) showed a similar behavior in $Al_2O_3$. In addition, mixtures of aggregates containing alkali metallic elements, like Na and K, exhibit relatively high stripping sensitivity. In contrast, metallic elements, such as Ca, Mg, and Fe, have a good aggregate-binder adhesion (15).

Despite the importance of the aggregate properties in the asphalt mixture durability, they are usually underestimated during road pavement design and production (2). Aggregates are considered the most stable component of the mixture (high resistance to deformation, durability and chemical inertia, 16). In contrast, asphalt binder is chemically more complex and expensive (6€/tone of aggregate versus 500€/tone of asphalt binder, 2), and consequently it gets more attention by producers, technicians and researchers. However, it is important to better understand the influence of the aggregate properties in the asphalt mixture behavior. In this regard, petrographic techniques, such as optical and fluorescence microscopy, can provide adequate information about the process of asphalt mixture damaging, and the interaction between the aggregates and the binder, especially from the aggregate point of view (2).

Optical polarized light microscopy has been widely applied in the study of the physical-mechanical properties of the aggregates (17–21). In addition, fluorescence microscopy has been extensively performed to estimate the damage of building materials, because it can detect the cracks produced by external agents, such as water or extreme temperature values (22, 23). This technique has been applied on concrete (24–29), but it has barely been used in the study of asphalt mixtures. Eriksen (30) used optical and fluorescence microscopy on asphalt mixtures to estimate their homogeneity of compaction. Broekmans (31) evaluated the effect of studded tires on asphalt pavements with fluorescence microscopy, obtaining different failure patterns depending on the type of aggregates used in the mixture. Poulikakos and Partl (32, 33) studied the microstructure of asphalt mixtures by fluorescence microscopy and its relationship with the mechanical effects of compaction and fatigue. The microscopic observations, supported by field data, concluded that the performance quality of mixes depends on the shape of voids, and interlocking and binder adhesion of the aggregates.

The aim of this paper is to evaluate with petrographic techniques the effect of the stripping phenomenon (low binder-aggregate adhesion) on the durability of asphalt mixtures, paying especial attention to the interaction between the aggregates and the binder of the mixtures, and to discuss the use of optical and fluorescence microscopy in their characterization. It is important to point out that Spanish roads are subjected to extreme climatic conditions, very cold winters with frequent maintenance operations, and high temperatures during the summer. Lately, these temperatures can often reach up to 40° C owing of the recurring heat waves registered in the Iberian Peninsula (34, 35). In addition, the most common asphalt mixtures used on surface layers in Spain are open-graded. The presence of voids (up to 12%, 36) and the low rate of rainfalls, produce the retention of the NaCl spread during winter inside the asphalt mixture. NaCl then begins to precipitate from the solution to form crystals within the pores (37, 38), and this crystal growth is capable of producing tensile hoop stresses above 2500 psi (17.2 MPa) (39, 40). For these reasons, the effect of low and high temperatures (winter and summer seasons, respectively), both combined with NaCl, has been taken into account in this paper as they have been poorly studied (41, 42).

## 2. MATERIALS AND METHODS

With the purpose of estimating the combined effect of the NaCl, extreme climatic conditions and traffic load, two types of asphalt mixtures used at NW Spain were prepared in the laboratory. The mixtures where exposed to freeze-thaw and thermal-stress cycles with a saturated solution of NaCl to simulate the effect of the seasonal thermal variations and the winter maintenance practices on road surfaces. After the durability tests, the samples were tested with the wheel tracking method to simulate the effect of the traffic load.

Finally, the samples treated and tested in the laboratory were analyzed by optical and fluorescence microscopy so as to evaluate the mechanisms of the damage caused. In addition, the surface properties (morphology and composition), as well as the mechanical resistance of the aggregates used in the asphalt mixtures, were also determined to round off the results collected from the petrographic analysis.

### 2.1. Materials

Two open-graded asphalt mixtures with different types of coarse aggregates, amphibolite (AAM) and a 1:1 blend of schist and paragneiss (SAM) were used in this research (Table 1). The metasediments are mixed in the quarry because of the complex geological setting of the quarry, which makes it difficult the separation of the lithologies during the extraction process (2).

The asphalt mixtures were prepared in the laboratory of a quarry located close to Santiago de Compostela (Galicia, Spain), which exploits amphibolite and schist-paragneiss aggregates. This quarry has supplied asphalt mixtures produced by these aggregates to several civil works of the area, especially roads and highways (2).

Both mixtures show a good aggregate gradation (Figure 1). Micro-porosity is detected in both materials (Figure 1), especially related to the binder-aggregate limits. The schist aggregate has a rounded shape (Figure 1a) while the amphibolite coarse aggregate exhibits an angular shape (Figure 1b).

### 2.2. Coarse aggregates characterization

The mineralogical analysis of the coarse aggregates was obtained by X-ray powder diffraction. The device used was a Philips Analytical PW 1752 diffractometer operated at 40 kV and 30 mÅ, equipped with a copper K-alpha anode tube, a graphite monochromator. The interpretation was carried out with the PC-ADP diffraction software.

The surface morphology was measured using the optical roughness tester TRACEiT (InnowepGmbH, Würzburg, Germany) with a testing field of 5x5 mm and a resolution of 1.5 µm in the Z direction. The roughness parameter Rz or the ten-point height, which is the average of the five highest peaks and the five lowest valleys along the assessment length

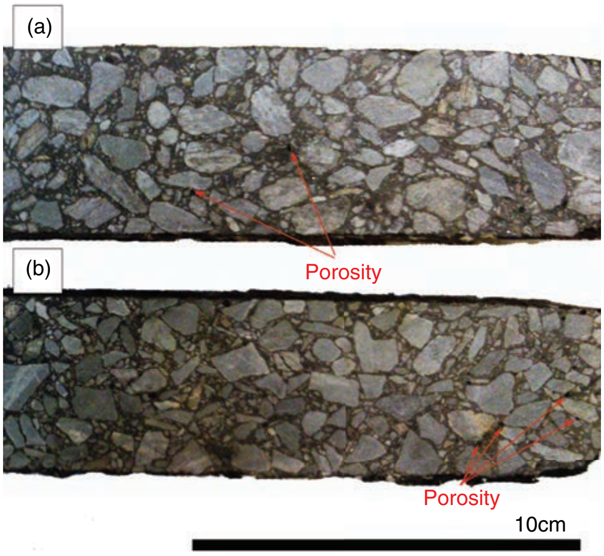


FIGURE 1. Photographs of (a) asphalt mixture with schist aggregate, SAM, and (b) asphalt mixture with amphibolite aggregate, AAM.

TABLE 1. Asphalt mixture characteristics

| Asphalt mixture | Mixture type | Aggregate | Filler | Binder | Binder content [%] | Air voids [%] | Density [g/cm$^3$] |
|---|---|---|---|---|---|---|---|
| AAM | BBTM 11b | Amphibolite | Limestone | PMB 45/80-65 | 4.8 | 12 | 2.63 |
| SAM | BBTM 11b | Schist | Limestone | PMB 45/80-65 | 5.0 | 8 | 2.53 |

of the profile (43), was calculated from the measured topographic data (44, 45).

Finally, the water absorption coefficient (46), and the mechanical resistance (Los Angeles test, 47) of the coarse aggregates used in the asphalt mixtures were determined in the laboratory following their respective current standards (46, 47).

### 2.3. Asphalt mixture sample preparation

A roller compactor with a maximum load of 30 kN was used to produce 10 wheel tracking samples with 40 mm of thickness, as referred to in the current standard (48, Figure 2a). Five samples per type of asphalt mixture, AAM and SAM. One control sample per type of mixture were used as reference materials to evaluate the damages produced by the different tests in the laboratory.

### 2.4. Effect of NaCl and extreme climatic conditions: Durability tests

To simulate the effect of the NaCl and extreme climatic conditions, the asphalt mixture samples were subjected to durability tests in the laboratory. Two samples per each type of asphalt mixture were exposed to 70 daily freeze-thaw cycles. During 16 hours, the samples were initially exposed to -21 °C in a freezer, and following that, they were immersed during 8 hours in a solution with 23.3 wt % of NaCl at 20 °C.

The remaining two samples per each type of mixture were subjected to 40 daily thermal-stress cycles. Firstly, the samples were exposed to 50 °C in a heater during 6 h and, secondly, they were immersed in a solution with 23.3 wt % of NaCl at 20 °C during 18 h (2, 49, 50).

### 2.5. Effect of the traffic: Wheel tracking test

The wheel tracking test was performed, as referred to in the current standard (51), to evaluate the effect of the traffic in the asphalt mixtures, both untreated and exposed to durability tests. A small wheel tracking device was used (52, Figure 2b) and the test consisted in repeatedly rolling a small wheel with a load of 700 N at 60 °C across the prepared sample, and consequently measure the deformation originated.

### 2.6. Evaluation of the induced effect of NaCl, extreme climatic conditions and traffic: Petrographic analysis

To evaluate the damage produced by the induced effect of NaCl and extreme climatic conditions, thin sections were cut longitudinally from the surface of the asphalt mixtures exposed to the durability tests. At the same time, the additional effect of the traffic was studied from thin sections cut perpendicularly to the deformation area produced by the loaded wheel after both tests (wheel tracking and durability). All the thin sections obtained were injected with epoxy resin doped with artificial fluorescein (53) to carry out the fluorescence microscopy analysis.

Thin sections were studied using an optical polarized light microscopy Olympus BX51 with an Olympus DP12 (6V/2.5 A) camera. This microscope is also equipped with a mercury lamp Olympus U-RFL-T to perform the fluorescence study of the samples.

## 3. RESULTS

### 3.1. Coarse aggregate characterization

The mineralogy and surface morphology of the aggregates used to produce the asphalt mixtures studied are shown in Table 2. Amphibolite is composed of $Ca^{2+}$, $Fe^{2+}$ and $Mg^{2+}$ bearing minerals, such as actinolite or plagioclase, while schist has minerals with $Na^{+}$, $Al^{3+}$ or $K^{+}$, such as albite, muscovite and biotite (Table 2). Surface roughness in the amphibolite aggregate is lower than in the schist aggregate (Table 2).

The water absorption coefficient and the Los Angeles values of the aggregates are exposed in Table 3. The schist aggregate had a higher water absorption coefficient and a lower mechanical resistance than the amphibolite aggregate (Table 3).

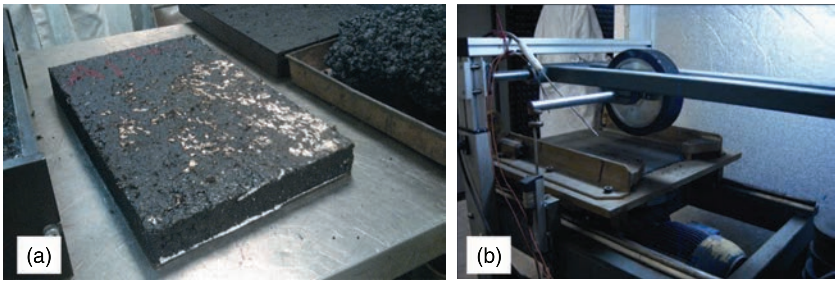


FIGURE 2. Photographs of (a) the wheel tracking asphalt mixture sample and (b) the small wheel tracking testing device (52).

TABLE 2. Semi-quantitative analysis by XRD and surface morphology of the aggregates used in the asphalt mixtures

| | | Mineralogy (%) | | | | | | | | | | | Surface morphology |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Mixture | Aggregate | Qz | Ab | Pl | Bt | Ms | Grt | Act | Hbl | Ep | Ttn | Sul | Rz (μm) |
| AAM | Amphibolite | | | 35 | | | | 56 | <5 | <5 | 6 | | 38.36 ± 7.81 |
| SAM | Schist | 33 | 31 | | 28 | 7 | <5 | | | | | <5 | 42.15 ± 8.27 |

*Qz = Quartz; Ab = Albite; Pl = Plagioclase; Bt = Biotite; Ms = Muscovite; Grt = Garnet; Act = Actinolite; Hbl = Hornblende; Ep = Epidote; Ttn = Titanite; Sul = Sulphide minerals*

TABLE 3. Water Absorption Coefficient and Los Angeles data

| Aggregate | Wa (%) | LA |
|---|---|---|
| Amphibolite | 0.50 | 9 |
| Schist | 0.95 | 18 |

*Wa = Water absorption coefficient; LA = Los Angeles Test Index*

## 3.2. Effect of the traffic: Wheel tracking test

After the wheel tracking test, the amphibolite asphalt mixture (AAM, Figure 3a) was less deformed by the effect of the traffic than the schist asphalt mixture (SAM, Figure 3b). In both cases, the deformation was higher when the mixtures were exposed to the effect of NaCl and low temperatures (Figure 3). By contrast, the deformation was lower when the mixtures were subjected to the effect of NaCl and high temperatures (Figure 3).

## 3.3. Evaluation of the induced effect of NaCl, extreme climatic conditions and traffic: Petrographic analysis

The control sample of AAM under optical polarized light and fluorescence microscope (Figure 4a) showed a good aggregate gradation, but the adhesion between the binder and the aggregate was poor. This low adhesion produced inter-particle porosity in the aggregate-binder limits of the mixture (Figure 4a). Intra-particle porosity was also observed, owing to the cracking of small amphibolite and quarz aggregates (Figure 4a). In general, the maximum length of the porosity observed was 0.5 mm, and the maximun thickness was 50 μm. Some inter-particle porosity was also caused by air bubbles trapped during the manufacture of the asphalt mixture.

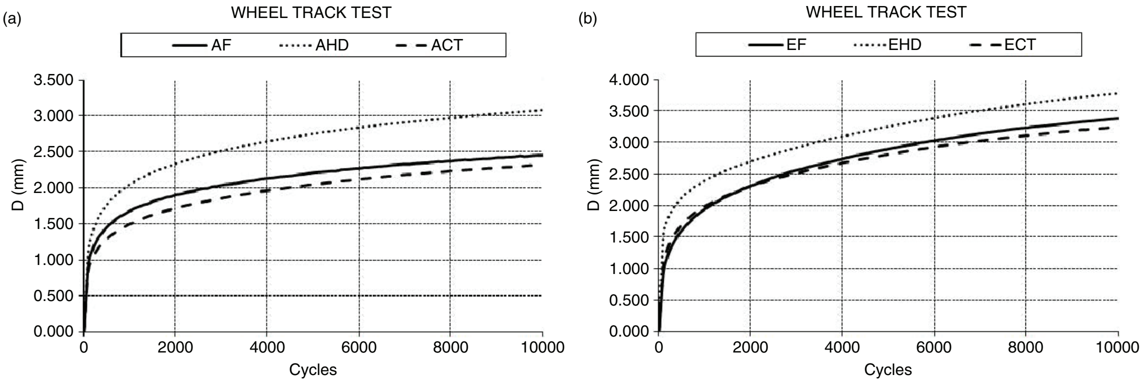
The control sample of SAM showed a good aggregate gradation and adhesion between the binder and the aggregate (Figure 4b). Intra-particle porosity was due to the cracking of quartz and mica aggregates (Figure 4b). The maximum length of these cracks was 1 mm, and the maximum thickness was 40 μm. The same inter-particle porosity related to air bubbles trapped detected in the AAM was seen in this mixture.

The induced effects of NaCl and extreme climatic conditions were also characterized with optical and fluorescence microscopy (Figure 5).

After the freeze-thawing test, the amphibolite aggregate displayed some cracks, especially in the limits between the aggregate and the binder (Figure 5a), suggesting inter- and trans-particle cracking after the test. The maximum crack length observed was 1 mm, and the maximum thickness was less than 0.5 μm. In the schist asphalt mixture, the coarse aggregate was also the most damaged component. Inter- and intra-particle cracks were detected with a maximum length of 2 mm and 0.5 μm of maximum thickness. In both asphalt mixtures, trans-particle cracks were found, related to the damage of previous porosity in the binder. This porosity was mainly related to the air bubbles produced during the manufacture of the asphalt mixture, especially those in contact with the coarse aggregates (Figure 5b).

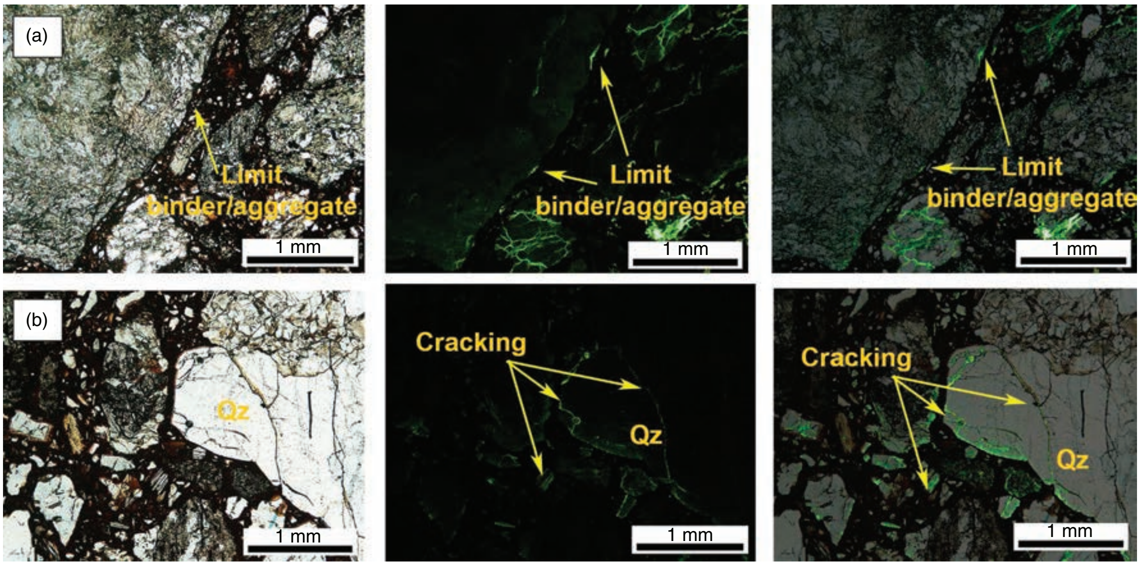
After the thermal-stress test, the main damage in the mixture was located in the coarse aggregates from both asphalt mixtures. Inter- and trans-particle cracks were observed in the amphibolite aggregates because of their low adhesion with the binder. Additionally, new inter-particle porosity was found in the mixture produced by the binder loss during the test (Figure 5c). The cracks detected in the amphibolite mixture were up to 1 mm in length, and their maximum thickness was 1 mm. In the schist mixture, the test caused the intra-, inter- and trans-particle cracking of the coarse aggregate. The intra-particle cracking was more abundant and it was related to the mica domains of the rock (Figure 5d). These cracks were up to 5 mm in length and 1 mm in thickness. The same binder loss process observed in the amphibolite mixture was found in the schist mixture. Thus, new inter-particle porosity was produced after the thermal-stress test.

Finally, the effects produced by the freeze-thaw and thermal cycles were aggravated by the wheel tracking test. Inter- and trans-particle cracking was produced, especially in the schist aggregate (Figure 5e). Additionally, the deformation and deterioration of the air bubbles from the mixture caused the cracking of the aggregates and the binder (Figure 5f). The cracks observed had up to 3 mm in length and they reached 1 mm in thickness.



AF = amphibolite asphalt mixture untreated; AHD = amphibolite asphalt mixture after freeze-thawing; ACT = amphibolite asphalt mixture after thermal-stress; EF = schist asphalt mixture untreated; EHD = schist asphalt mixture after freeze-thawing; ECT = schist asphalt mixture after thermal-stress.

FIGURE 3. Graphs showing the wheel tracking test results for (a) AAM and (b) SAM.



Qz= Quartz

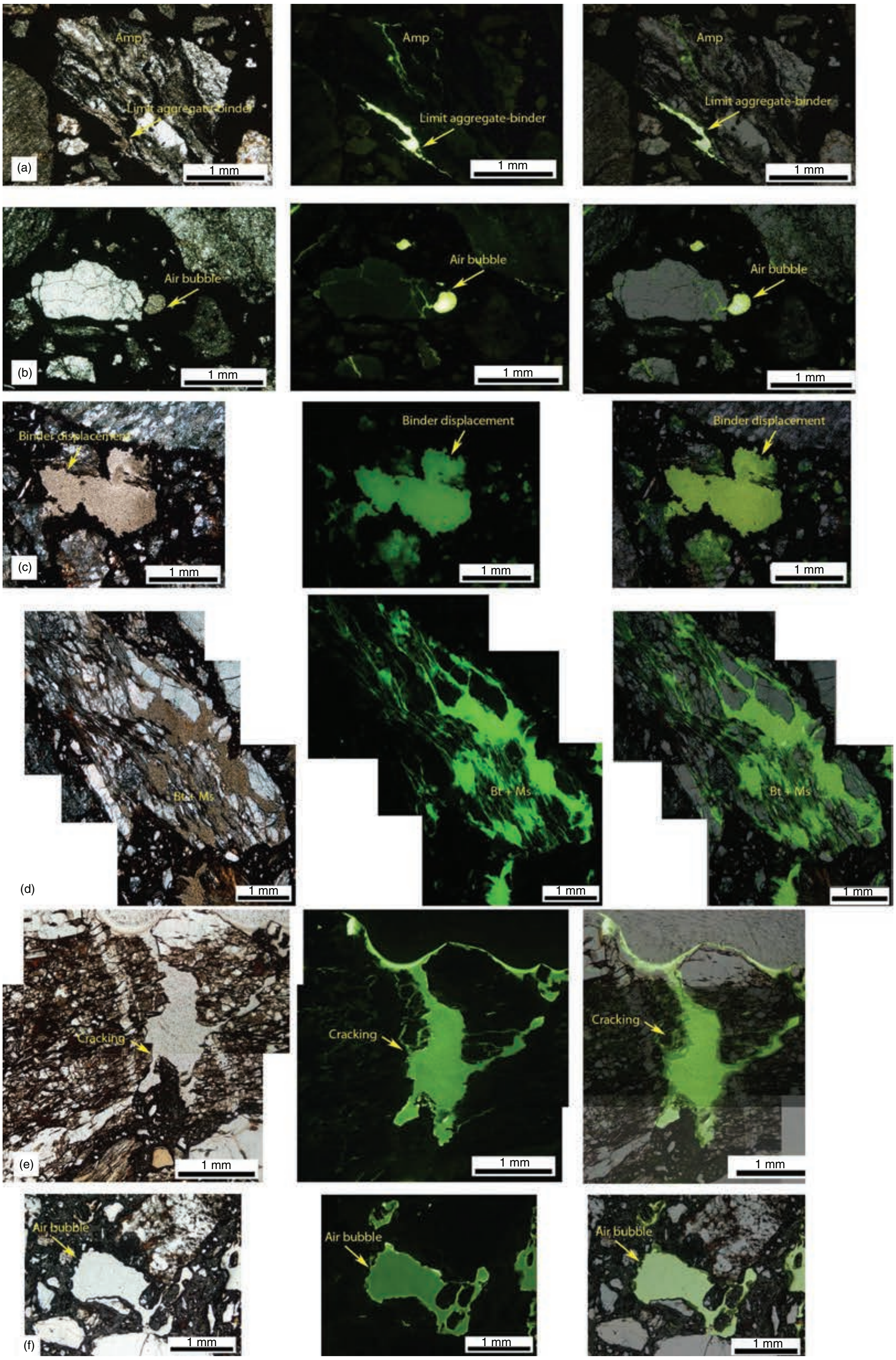
FIGURE 4. Images taken with optical microscopy (parallel nicols, left image), fluorescence microscopy (center) and a composition of both techniques (right) of (a) AAM and (b) SAM.

It can be affirmed that after wheel tracking test, the damage was higher than after the durability tests only. Owing to the different orientation of the thin sections obtained from the asphalt mixtures depending on the test performed (longitudinally from the surface after the durability tests, and perpendicularly after the wheel tracking test), the degradation after the durability tests was performed on the first 1 mm of exposed area, while after the wheel tracking test, the degradation penetrated up to 3 mm inside the mixture.

## 4. DISCUSSION

The data collected from the petrographic analysis and the mechanical tests performed in the laboratory showed that the surfaces of the asphalt mixtures were damaged by the induced effect of NaCl, extreme climatic conditions, and traffic load.

Both tests, freeze-thawing and thermal-stress with NaCl, produced the deterioration by cracking of the asphalt mixture coarse aggregate with a similar mechanism. The binder acted as an impermeable wall. Thus, NaCl solution could only penetrate in the mixture through the aggregate, producing an intense and quick damage (Figure 5). The penetration of the NaCl solution inside the aggregate was possible because of the presence of previous or initial porosity in the mixture. This porosity was mainly caused by the low adhesion between the aggregate and the binder (Figure 5a), the presence of air bubbles in the binder originated during the mixture production (Figure 5b) and the internal structure and mineral composition of the coarse aggregate (Figure 5d). In addition,



*Amp = Amphibole; Bt = Biotite; Ms = Muscovite*

FIGURE 5. Images with optical polarized microscopy (parallel polars, left), fluorescence microscopy (center) and a composition of both techniques (right). (a) Low adhesion between the amphibolite aggregate and the binder producing cracking processes after freeze-thawing. (b) Cracking of the coarse aggregate related to the damaging of an air bubble after freeze-thaw cycles. (c) Binder loss causing the aggregate deterioration after thermal-stress. (d) Mica cracking on schist aggregates after thermal-stress. (e) Cracking of the aggregate after freeze-thaw cycles and wheel tracking test. (f) Air bubble deformation and causing the mixture damage after thermal-stress and wheel tracking tests.

the effect of the traffic aggravated the deterioration of the asphalt mixtures exposed to both, NaCl and extreme climate conditions. The deformation of the internal structure of the mixture (Figure 3) caused the cracking of its components, especially the coarse aggregates, which were weakened by the effect of the NaCl and extreme climatic conditions.

In spite of the similar damaging mechanism exhibited by both durability tests, the effect of thermal-stress (high temperature) was more harmful on the mixture surface (Figure 5c). This test affected not only the aggregate, but also the binder. It was observed that the wheel tracking tests after thermal-stress decreased the deformability of the mixtures analyzed (Figure 3). This phenomenon could result from the possible aging of the binder after its exposure to high temperatures (54–56). In addition, petrographic analysis showed the surface loss of the binder (Figure 5c), causing a higher exposure of the aggregate surface to the effect of the NaCl solution and increasing the intensity of the cracking, in contrast to the lower amount of cracks detected after the freeze-thaw cycles.

Even though the effect of NaCl used during winter on asphalt pavements has been widely studied by researchers and Transportation Agencies (3–5, 57–59), no current standards consider their effects in asphalt mixtures. In addition, the effect of that salt during the summer weather conditions has been poorly studied (41, 42). For these reasons, it is recommended to consider both, winter and summer weather conditions, in future studies of road surface durability, especially in countries as Spain, with extreme temperature changes between winter and summer.

The low adhesion between the aggregates and the binder was one of the main degradation mechanisms of the asphalt mixtures. The amphibolite aggregate, with an “a priori” higher quality than the schist aggregate because of its lower water absorption coefficient and higher mechanical resistance (Table 3), showed an intense degradation by the effects of the NaCl, extreme climatic conditions and traffic because of its lower adhesion with the binder. In spite of the importance of this process in the durability of the mixture, the causes of this low adhesion are unclear. The most accepted explanation is that rocks with high silica content have low adhesion with asphalt binders (12–13, 15, 60, 61). Other compositional factors that hinder an optimal adhesion include minerals with K and Na (albite, biotite or muscovite) because of their higher solubility (Table 2; 10, 13). Conversely, minerals rich in non-soluble Ca and Mg, such as actinolite or plagioclase (Table 2) favor the adhesion with the binder (62). However, the amphibolite aggregate showed lower adhesion than the schist aggregate, giving rise to a challenging conundrum. It follows that, in this case, the low adhesion with the binder cannot be explained only by the presence of minerals with soluble ions which can rapidly react with water molecules displacing the asphalt binder (10, 13). Therefore, additional properties from the aggregate surface should be taken into in account in this process.

It is argued that these additional properties comprise the surface roughness (Table 2) and the water absorption coefficient (used as a proxy of the connected porosity), as both parameters are much higher in the schist than in the amphibolite aggregate (see Tables 2 and 3, respectively). This surface roughness and “porosity” would improve the penetration of the binder inside the aggregate contributing to its mechanical interlocking when it hardens (6, 63). In addition, the angularity of the aggregates can also influence the adhesion with the binder. Angular shapes on aggregates increase the potential for asphalt film rupture producing trapped porosity in the mixture, as well as low adhesion between the binder and the aggregate (6, 64). The amphibolite aggregate shape, more angular, could be pivotal in the low adhesion of this material with the binder. For this reason, it is recommended to study parameters such as surface roughness and shape, combined with the mineralogical and chemical composition of the aggregates to better understand the causes of low adhesion with the binder.

Other important factors responsible of the asphalt mixture deterioration were the mineral habit and the fabric of the coarse aggregates. While the cracking of amphibolite coarse aggregates was generally related to this low adhesion (Figure 3a), the schist aggregate damaging was related to the delamination and cracking of its mica domains (Figure 3c). The foliated structure of the schist aggregate was also responsible of the deeper deterioration caused by the traffic effect in the schist asphalt mixture compared to the amphibolite mixture (Figure 3e). Therefore, both factors must be taken into account as selection criteria to improve the mechanical resistance and durability of asphalt mixtures.

Finally, the petrographic analysis (65) is not included in Spanish or European current standards to evaluate the quality of the aggregates used on roads surface (16, 66). However, the optical polarized light microscopy and the fluorescence microscopy, as it has been showed in this paper, are good techniques to evaluate the durability and the quality of the asphalt mixtures, especially the coarse aggregates.

## 5. CONCLUSIONS

The main conclusions of this study can be summarized as follows:

- The effect of NaCl and extreme climatic conditions damaged the studied asphalt mixtures, especially at high temperatures (thermal stress caused in summer conditions).
- Coarse aggregates were the main deteriorated component of the mixtures analyzed, while the

binder seemed to be unchanged after the durability tests. This deterioration was increased by the effect of the traffic.

- The low adhesion between the aggregate and the binder, the presence of air bubbles in the binder originated during the mixture manufacture, and the internal structure and mineral composition of the coarse aggregate were the main factors responsible of asphalt mixture deterioration.
- The mineralogical composition of the aggregates is not the only parameter related to the aggregate-binder low adhesion phenomenon; the surface roughness and the shape of the aggregate are also important factors to be taken into account when studying this process.
- The combined use of both, optical and fluorescence microscopy, can provide important information about the damaging mechanisms of the mixtures. This information can help to improve the road surface design, construction and long-term use. For this reason, these techniques should be considered in future roads surface pavement standards and requirements.

## ACKNOWLEDGEMENTS

This research was partly funded through the Community of Madrid under the project S2013/MIT-2914 (GEOMATERIALS-2CM) and the Grant Program from the CEDEX Research Agency (BOE 27/06/2009). Authors thank to Dr. Herminia Cano Linares, Technical Coordinator of Geotechnical Laboratory (CEDEX) and Associate Professor at the Technical University of Madrid (UPM) her support during this project. We also thank to the company Construcciones Francisco Gómez y Cía., S.L, the provision of the materials to develop this research.

## REFERENCES

1. Potti, J. J.; de Asefma, P. E. (2012). Los mensajes sobre la conservación y la realidad de la conservación de nuestros firmes de carreteras. *Carreteras: Revista técnica de la Asociación Española de la Carretera*, (186), 24–33.
2. Pérez-Fortes, A. P. (2015). *Quality and durability of metamorphic crushed stone aggregates used in Galician asphalt pavements subjected to the effect of NaCl and extreme climates*. PhD thesis, Universidad Complutense de Madrid, 452 pp.
3. Shi, X.; Akin, M.; Pan, T.; Fay, L.; Liu, Y.; Yang, Z. (2009). Deicer impacts on pavement materials: Introduction and recent developments. *Open Civil Engineering Journal*, 3, 16–27. https://doi.org/10.2174/1874149500903010016
4. Hassan, Y.; Abd El Halim, A. O.; Razaqpur, A. G.; Bekheet, W.; Farha, M. H. (2002). Effects of runway deicers on pavement materials and mixes: comparison with road salt. *J. Transp. Eng.* 128(4), 385–391. https://doi.org/10.1061/(ASCE)0733-947X(2002)128:4(385)
5. Feng, D.; Yi, J.; Wang, D.; Chen, L. (2010). Impact of salt and freeze–thaw cycles on performance of asphalt mixtures in coastal frozen region of China. *Cold. Reg. Sci. Technol.* 62(1), 34–41. https://doi.org/10.1016/j.coldregions.2010.02.002
6. Tarrer, A. R.; Wagh, V. (1991). *The effect of the physical and chemical characteristics of the aggregate on bonding* (No. SHRP-A/UIR-91-507). Washington, DC: Strategic Highway Research Program, National Research Council. 31 pp.
7. Bagampadde, U.; Isacsson, U.; Kiggundu, B. M. (2004). Classical and contemporary aspects of stripping in bituminous mixes. *Road. Mater. Pavement., 5*(1), 7–43. https://doi.org/10.1080/14680629.2004.9689961
8. Airey, G. D.; Collop, A. C.; Zoorob, S. E.; Elliott, R. C. (2008). The influence of aggregate, filler and bitumen on asphalt mixture moisture damage. *Constr. Build. Mater*., 22(9), 2015–2024. https://doi.org/10.1016/j.conbuildmat.2007.07.009
9. Cui, S.; Blackman, B. R.; Kinloch, A. J.; Taylor, A. C. (2014). Durability of asphalt mixtures: Effect of aggregate type and adhesion promoters. *International J Adhesion*, 54, 100–111. https://doi.org/10.1016/j.ijadhadh.2014.05.009
10. Horgnies, M.; Darque-Ceretti, E.; Fezai, H.; Felder, E. (2011). Influence of the interfacial composition on the adhesion between aggregates and bitumen: Investigations by EDX, XPS and peel tests. *J Adhesion*, 31(4), 238–247. https://doi.org/10.1016/j.ijadhadh.2011.01.005
11. Abo-Qudais, S.; Al-Shweily, H. (2007). Effect of aggregate properties on asphalt mixtures stripping and creep behavior. *Constr. Build. Mater*, 21(9), 1886–1898. https://doi.org/10.1016/j.conbuildmat.2005.07.014
12. Fromm, H.J., (1974). The mechanisms of asphalt stripping from aggregate surfaces. *J. Assoc. Asphalt. Pav.*, 43, 191–219.
13. Bagampadde, U. (2004). *On investigation of stripping propensity of bituminous mixtures*. Licentiate thesis. Division of Highway Engineering. 102 pp.
14. Vuorinen, M.; Hartikainen, O. P. (2001). A new ultrasonic method for measuring stripping resistance of bitumen on aggregate. *Road. Mater. Pavement*, 2(3), 297–309. https://doi.org/10.1080/14680629.2001.9689905
15. Bagampadde, U.; Isacsson, U.; Kiggundu, B. M. (2005). Influence of aggregate chemical and mineralogical composition on stripping in bituminous mixtures. *Int. J. Pavement. Eng.,* 6(4), 229–239. https://doi.org/10.1080/10298430500440796
16. UNE-EN 13043 (2003). *Aggregates for bituminous mixtures and surface treatments for roads, airfields and other trafficied areas*. Spanish Association for Normalization and Certification (AENOR), Madrid.
17. Ersoy, A.; Waller, M. D. (1995). Textural characterization of rocks. *Eng. Geol.*, 39(3), 123–136. https://doi.org/10.1016/0013-7952(95)00005-Z
18. Tuğrul, A.; Zarif, I. H. (1999). Correlation of mineralogical and textural characteristics with engineering properties of selected granitic rocks from Turkey. *Eng. Geol.*, 51(4), 303–317. https://doi.org/10.1016/S0013-7952(98)00071-4
19. Přikryl, R. (2001). Some microstructural aspects of strength variation in rocks. *Int. J. Rock .Mech. Min.,* 38(5), 671-682. https://doi.org/10.1016/S1365-1609(01)00031-4
20. Räisänen, M. (2004). Relationships between texture and mechanical properties of hybrid rocks from the Jaala–Iitti complex, southeastern Finland. *Eng. Geol.*, 74(3), 197–211. https://doi.org/10.1016/j.enggeo.2004.03.009
21. Tandon, R. S.; Gupta, V. (2013). The control of mineral constituents and textural characteristics on the petrophysical & mechanical (PM) properties of different rocks of the Himalaya. *Eng. Geol.*, 153, 125–143. https://doi.org/10.1016/j.enggeo.2012.11.005
22. Freire-Lista, D.M.; Fort, R.; Varas-Muriel, M.J. (2016). Thermal stress-induced microcraking in building granite. *Engineering Geological*, 206, 83–93. https://doi.org/10.1016/j.enggeo.2016.03.005
23. Freire-Lista, D.M.; Fort, R.; Varas-Muriel, M.J. (2015). Freeze-thaw fracturing in building granites. *Cold. Reg. Sci. Technol*, 113, 40–51. https://doi.org/10.1016/j.coldregions.2015.01.008
24. Rodríguez Rey A.; Montoto San Miguel M.; Calleja Escudero L.; Gómez Ruiz de Argandeña V.; Suárez del Río L. M. (1987). Aplicación de la microscopía óptica de fluorescencia al estudio textural del clínker de cemento

Pórtland. *Mater. Construcc.*, 37(205), 17–22. https://doi.org/10.3989/mc.1987.v37.i205.875
25. Larbi, J. A.; Heijnen, W. M. M. (1997). Determination of the cement content of five samples of hardened concrete by means of optical microscopy. Heron, 42, 125–138.
26. Jakobsen, U. H.; Laugesen, P.; Thaulow, N. (1999). Determination of water-cement ratio in hardened concrete by optical fluorescence microscopy. *ACI Special Publication*, 191, 27–42.
27. Elsen, J. (2006). Microscopy of historic mortars—a review. *Cem. Concr. Res.*, 36(8), 1416–1424. https://doi.org/10.1016/j.cemconres.2005.12.006
28. Binal, A. (2008). The determination of gel swelling pressure of reactive aggregates by ASGPM device and a new reactive-innocuous aggregate decision chart. *Constr. Build. Mater*., 22(1), 1–13. https://doi.org/10.1016/j.conbuildmat.2007.07.021
29. Grantham, M. G. (Ed.). (2011). *Concrete Repair: A Practical Guide*. CRC Press. 328 https://doi.org/10.1201/b12433
30. Eriksen K. (1993). *Microscopical analysis of asphalt–aggregate mixtures related to pavement performance*, Report No. 245, Danish Road Institute, Denmark.
31. Broekmans, M. A. (2007). Failure of greenstone, jasper and cataclasite aggregate in bituminous concrete due to studded tyres: similarities and differences. *Materials Characterization*, 58(11), 1171–1182. https://doi.org/10.1016/j.matchar.2007.05.012
32. Poulikakos, L. D.; Partl, M. N. (2009). Evaluation of moisture susceptibility of porous asphalt concrete using water submersion fatigue tests. *Constr. Build. Mater*, 23(12), 3475–3484. https://doi.org/10.1016/j.conbuildmat.2009.08.016
33. Poulikakos, L. D.; Partl, M. N. (2010). Investigation of porous asphalt microstructure using optical and electron microscopy. *J. Micros.c*, 240(2), 145–154. https://doi.org/10.1111/j.1365-2818.2010.03388.x
34. Yagüe, C.; Martija, M.; Torres, J.; Maldonado, A. I.; Zurita, E. (2006). Análisis estadístico de las olas de calor y frío en España. *XXIX Jornadas Científicas de la AME*, 20–26.
35. Izquierdo, A.; Manzano, M.; Martín, Q.; Montero, J.; Salazar, J. (2012). Evolución temporal de las olas de calor en la meseta central española, entre 1961 y 2010. *Sociedad Española de Climatología*, 441–448.
36. UNE-EN 12697-8. *Mezclas bituminosas. Métodos de ensayo para mezcla bituminosa en caliente. Parte 8: Determinación del contenido de huecos en las probetas bituminosas*. Asociación Española de Normalización y Certificación (AENOR), Madrid, 2003.
37. Scherer, G. W. (2004), Stress from Crystallization of Salt. *Cem. Concr. Res.*, 34, [9] 1613–1624. https://doi.org/10.1016/j.cemconres.2003.12.034
38. Yang, Q.; X. Wu; Huang, S. (1997). Concrete Deterioration Due to Physical Attack by Salt Crystallization. In *10th International Congress on the Chemistry of Cement*. Gothenburg, Sweden.
39. Flatt, R. J. (2002) Salt Damage in Porous Materials: How High Supersaturations Are Generated. *J. Cryst. Growth.,* 242, 435–454. https://doi.org/10.1016/S0022-0248(02)01429-X
40. Sumsion, E. S.; Guthrie, W. S. (2013). *Physical and Chemical Effects of Deicers on Concrete Pavement: Literature Review* (No. UT-13.09). Utah Department of Transportation. 50pp. http://www.udot.utah.gov/main/uconowner.gf?n=8081525197623431
41. Obika, B.; Freer-Hewish, R. J.; Fookes, P. G. (1989). Soluble salt damage to thin bituminous road and runway surfaces. *Q. J. Eng. Geol. Hydroge.,* 22(1), 59–73. https://doi.org/10.1144/GSL.QJEG.1989.022.01.05
42. Obika, B.; Freer-Hewish, R. J.; Woodbridge, M.; Newill, D. (1995). Prevention of salt damage to thin bituminous surfacings: design guidelines. In *Transportation Research Board Conference Proceedings* (No. 6). 12 pp.
43. DIN-EN ISO 4287 (2010–07). *Geometrical product Specifications (GPS). Surface texture: Profile method. Terms, definitions and surface texture parameters*. Germany.
44. López-Arce, P.; Gomez-Villalba, L. S.; Pinho, L.; Fernández-Valle; M. E.; de Buergo, M. Á.; Fort, R. (2010). Influence of porosity and relative humidity on consolidation of dolostone with calcium hydroxide nanoparticles: Effectiveness assessment with non-destructive techniques. *Materials Characterization*, 61(2), 168–184. https://doi.org/10.1016/j.matchar.2009.11.007
45. Vázquez-Calvo, C.; de Buergo, M. A.; Fort, R.; Varas-Muriel, M. J. (2012). The measurement of surface roughness to determine the suitability of different methods for stone cleaning. *J Geophys. Eng.,* 9(4), 108–117. https://doi.org/10.1088/1742-2132/9/4/S108
46. UNE-EN 1097-6 (2014). *Tests for mechanical and physical properties of aggregates - Part 6: Determination of particle density and water absorption*. Spanish Association for Normalization and Certification (AENOR), Madrid.
47. UNE-EN 1097–2 (2010). *Tests for mechanical and physical properties of aggregates - Part 2: Methods for the determination of resistance to fragmentation*. Spanish Association for Normalization and Certification (AENOR), Madrid.
48. UNE-EN 12697–33 (2008). *Bituminous mixtures - Test methods for hot mix asphalt - Part 33: Specimen prepared by roller compactor.* Spanish Association for Normalization and Certification (AENOR), Madrid.
49. Pérez Fortes, A. P.; Varas Muriel, M. J.; Cano Linares, H; Castiñeiras García, P., Pardo Santayana, F. (2013). Petrofísica y durabilidad de las anfibolitas de Touro (A Coruña, España) para su uso en obra civil. *Boletín Geológico y Minero*, 124(3), 367–369.
50. Pérez Fortes, A. P.; Varas Muriel, M. J.; Cano Linares, H.; Marfil Pérez, R.; Castiñeiras García, P. (2014). Durabilidad de las cuarcitas de A Fonsagrada (Lugo, España) explotadas como rocas industriales. *Ingeniería civil*, 173, 5–14.
51. UNE-EN 12697–22 (2008). *Bituminous mixtures - Test methods for hot mix asphalt - Part 22: Wheel tracking*. Spanish Association for Normalization and Certification (AENOR), Madrid.
52. Navarro Gómez, M. J. (2009). *Análisis del nuevo procedimiento de compactación de probetas de mezcla bituminosa para los ensayos de pista y de fatiga*. Tesina Universitat Politècnica de Catalunya, 183 pp. http://upcommons.upc.edu/handle/2099.1/8464
53. Varas-Muriel, M.J. (2012). Técnicas de caracterización petrográfica (II): Microscopía Óptica de Fluorescencia (MF) y Microscopía Electrónica de Barrido (MEB). En: *La conservación de los geomateriales utilizados en el patrimonio. Instituto de Geociencias (UCM-CSIC)*. Eds. E.M. Pérez-Monserrat y R. Fort. 31–37.
54. Lesueur, D. (2009). The colloidal structure of bitumen: Consequences on the rheology and on the mechanisms of bitumen modification. *Adv. Colloid. Interfac.*, 145(1), 42–82. https://doi.org/10.1016/j.cis.2008.08.011
55. Mateos, A.; Ayuso, J. P.; Cadavid Jáuregui, B. (2012). Evolution of asphalt mixture stiffness under combined effects of damage, aging, and densification under traffic. *Transportation Research Record: Trans. Res. B*, 2304, 185–194. https://doi.org/10.3141/2304-21
56. Miró, R.; Martínez, A. H.; Moreno-Navarro, F.; Rubio-Gámez, M. C. (2015). Effect of ageing and temperature on the fatigue behaviour of bitumens. *Mat. Des.*, 86, 129–137. https://doi.org/10.1016/j.matdes.2015.07.076
57. Iwata, H.; Watanabe, T.; Saito, T. (2002). Study on the performance of porous asphalt pavement in winter road surface conditions. In *Proceedings of the XIth PIARC International Winter Road Congress, Sapporo, Japan*, 162–165.
58. Doré, G.; Konrad, J. M.; Roy, M. (1997). Role of deicing salt in pavement deterioration by frost action. *Transportation Research Record: Trans. Res. B*, 1596, 70–75. https://doi.org/10.3141/1596-11
59. Doré, G.; Konrad, J. M.; Roy, M. (1999). Deterioration model for pavements in frost conditions. *Transportation Research Record: Trans. Res. B*, 1655, 110–117. https://doi.org/10.3141/1655-15
60. Scott, J. A. N. (1978). Adhesion and disbonding mechanisms of asphalt used in highway construction and maintenance. In Association of Asphalt Paving Technologists Proceedings, 47, 19–43.
61. Judele, L. (2011). The mechanism of bitumen adhesion to aggregates: the influence of mineralogic nature.

*Buletinul Institutului Politehnic din lasi. Sectia Constructii, Arhitectura*, 57(2), 175–181.

62. Buckley, J. S.; Takamura, K.; Morrow, N. R. (1989). Influence of electrical surface charges on the wetting properties of crude oils. *SPE Reservoir Engineering*, 4(3), 332–340. https://doi.org/10.2118/16964-PA
63. Majidzadeh, K.; Brovold, F. N. (1968). *State of the art: Effect of water on bitumen-aggregate mixtures*. Highway Research Board Special Report, 98. 88.
64. Gzemski, G. F.; McGlashan, W. D.; Dolch, L. W. (1968). *Thermodynamic Aspects of the Stripping Problem. HR Circular*, 78. Committee MC-A6 Characterisitcs of Aggregates and Fillers for Bituminous Construction. National Research Council (U.S.). Highway Research Board, Washington DC. 10 pp.
65. UNE-EN 12407 (2007). *Natural stone test methods - Petrographic examination*. Spanish Association for Normalization and Certification (AENOR), Madrid.
66. PG-3 (2007). *Pliego de Prescripciones Técnicas Generales para Obras de Carreteras y Puentes*. Dirección General de Carreteras, Ministerio de Fomento. Ediciones Liteam, Madrid. http://www.fomento.es/MFOM/LANG_CASTELLANO/DIRECCIONES_GENERALES/CARRETERAS/NORMATIVA_TECNICA/PPTG/PG3/