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Towards an Experimental Protocol for the Study of Induction Heating in Asphalt Mastics

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ABSTRACT

The development of asphalt mixtures with improved electrical and thermal properties is crucial in terms of producing suitable mixtures for the induction heating without losing their durability. The main scope of this research is to evaluate experimentally the impact of filler-sized electrically conductive additives on the induction heating efficiency and the rheological performance of asphalt mixtures. Within this framework, an experimental assessment protocol of structural and non-structural important parameters of induction heated asphalt mastics – asphalt mixtures without stone aggregates and sand – was developed. It was observed that by adding iron powder as filler-sized conductive additive to asphalt mastics the electrical and thermal properties improve. Moreover, the rheological investigations of different conductive asphalt mastics show the importance of adding iron powder after replacing the amount of mineral filler in order to maintain the workability of mastics. The micro-morphological observation of asphalt mastics using scanning electron microscopy illustrates the impact of filler-size particles – minerals and conductive additives – on the skeleton of asphalt mastics.

1 INTRODUCTION

2

3 Today, the rapid growth of the transportation infrastructure around the world and the need to focus more on 4 environmental friendly solutions for the construction and maintenance of pavements is leading the asphalt 5 paving industry to explore novel technological improvements. With the impending European and global 6 regulations on greenhouse gas emissions, fumes and energy conservation, these demands are becoming 7 increasingly challenging (1-3). Meanwhile a lot of effort is on developing sustainable asphalt mixtures with 8 non-structural properties by integrating new functionalities without losing their durability. One of these non-9 structural functionalities is induction healing. Induction healing asphalt mixtures have attracted considerable 10 attention as conductive mixtures capable to restore their mechanical properties under induction energy (4-10).

With regard asphalt pavement maintenance, there are various techniques that can be used to restore the mechanical characteristics of mixtures during their lifespan. The induction heating is one of these and a promising technique to prolong the service life of asphalt pavements by speeding up the self-healing process of asphalt. In order to increase the efficiency of induction heating, new mixtures with electrically conductive additives need to be developed. The contribution of these additives is to create asphalt mixtures with improved electrical and thermal properties, suitable for induction heating.

17 To study the new asphalt mixtures for induction heating, it is important to have in-depth understanding of 18 the interaction between the conductive additives and other asphalt components. Because of the fact that the 19 improved macroscopic response of an asphalt pavement has a direct link with the durability of the bonding 20 components in the asphalt mixtures, much research is focused on the behavior of asphalt mastics (binder and 21 filler-sized particles) and mortars (binder, filler-sized particles and sand). Particularly, the influence of filler-22 to-binder interaction on mastic performance (11-12) and the volumetric concentration of different types of 23 fillers (13) are studied at mastic level. On the other hand, asphalt mortars have been studied extensively in 24 (14-15).

25 To develop conductive asphalt mixtures suitable for induction heating, many efforts were concentrated on 26 adding fiber-type conductive particles (e.g. steel fibers or steel wool) in order to improve the durability of 27 mixtures and increase the induction heating efficiency. However, mixtures with steel fibers require a strong 28 mixing effort and longer mixing time to disperse steel fibers uniformly. Especially, the longer steel fibers 29 easily produce clusters inside the asphalt mixtures, causing inhomogeneity and reducing the mechanical 30 response (9, 10). Apart from the performance degradation because of the large amounts of fiber-type 31 additives, this type of additives can result significant increase of cost (17). For this reason and in order to 32 resolve the problems resulted by the fiber-type particles, conductive asphalt mixtures can be produced by 33 adding filler-sized conductive particles.

34 The effective properties of asphalt mixtures vary considerably according to the type and the characteristics 35 of filler-sized conductive additives. Higher conductivity of additives results in higher conductivity of the 36 asphalt concrete (18). Moreover, the volumetric concentration of particles affects on the effective 37 conductivity of asphalt mixture. Carbon black (18) and graphite powder (4, 18, 19) are among the examples 38 of filler-sized particles that were used to improve electrical conductivity of asphalt mixture. Also it is known 39 from previous researches that carbon black and graphite powder appear to have excellent compatibility with 40 asphalt binder imparting in parallel easy mixing. However, no extended research has focused on other types 41 of filler-sized conductive additives and subsequently on additives for developing asphalt concrete mixtures 42 for induction heating application.

In this paper, iron powder is selected as filler-sized additive with very high electrical conductivity and its interaction with the conventional components of asphalt mastic is studied. For a certain asphalt binder, asphalt mastics with different volumetric properties are developed and characterized following a new experimental protocol designed for this purpose. Initially, the evaluation of physical properties of mineral fillers and iron powder is required before the development of conductive asphalt mastics. To study in detail the micro-morphology of different conductive asphalt mastics scanning electron microscopy (SEM) is utilized. As mentioned above, the performance of asphalt mastic is associated with the skeleton of filler particles inside and for this reason the micro-morphology of mastic surface is examined. Furthermore, electrical, thermal and rheological properties of conductive asphalt mastics are determined by using a digital multimeter, a thermal sensor and dynamic shear rheometer (DSR), respectively. The finding of the current research will contribute to understand the influence of filler-sized conductive additives on the mechanical performance of asphalt mastics designed for induction heating applications.

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56 INDUCTION HEATING APPROACH OF CONDUCTIVE ASPHALT MIXTURES

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58 Induction heating is adapted as a maintenance technique for asphalt pavements and requires the development 59 of conductive asphalt mixtures. For this reason, conductive additives are mixed within the mixtures and an 60 alternating magnetic field is applied. This field induces eddy currents in the additives and consequently heats them according to the principles of Joule's law, see Figure 1.c. The generated heat in the additives increases 61 62 the temperature of the asphalt mixture around them, through the temperature rise the bitumen is melting, the 63 micro-cracks are healed and the pavement is treated. In particular, induction power heats locally the mastic 64 part of asphalt concrete and because of diffusion and flow of bitumen the cracks are healed without damaging 65 the stone skeleton. According to Faraday's law, the electromotive force from the magnetic field is:

66

$$\varepsilon = -\frac{d\phi B}{dt} \tag{1}$$

67

68 where ε is the electromotive force, $\frac{d\phi}{dt}$ is the momentary angular velocity and B is the magnetic flux.

Based on Joule's first law, the alternating electric currents generate heat in the additives. Consequently,
 power dissipation occurs locally on the mixture and it is expressed in terms of exposure time t as:

 $Q = I^2 R t \tag{2}$

72

73 where I is the amount of current and R is the effective electrical resistance of the conductive mixture with 74 additives.

75 The alternating electric current through the conductive mixture with effective resistance R is:

76

 $I = \frac{\varepsilon}{R} \tag{3}$

77

and by substituting the above equation for the current into one or both factors of current in Joule's law, thepower dissipated on the asphalt mixture can be rewritten in the equivalent form:

80

$$Q = \frac{\varepsilon^2}{R} \cdot t \tag{4}$$

81

82 The induction heating efficiency depends on the operational parameters, such as frequency, power, and the 83 effective properties of asphalt mixtures. In this paper, as previously mentioned, the induction heating 84 efficiency is investigated of additives on the asphalt mastics under constant operational conditions.







FIGURE 1 Infrared image (a) during induction heating of an asphalt pavement (A58 near Vlissingen,
the Netherlands), (b) of heated asphalt pavement surface at high resolution and (c) the schematic of
induction heating of an asphalt mixture (c.1) induced by eddy currents and (c.2) heat generation in the

90 mixture based on the Joule's law91

92 MATERIAL AND PREPARATION

93

Firstly, the selected fillers and filler-sized conductive additive are analyzed. Scanning electron microscopy
 (SEM), BET (Brunuer, Emmett and Teller theory) and Ultrapycnometer have been utilized in order to
 determine the shape, specific surface area and density, respectively. Figure 2 shows the SEM images of the

97 filler-sized particles; weak limestone (WL) filler, produced limestone (PL) filler and iron powder (IP). It can 98 be seen that the angular shape and size of filler limestone – WL and PR – is similar comparing iron powder 99 (IP) where it presents smaller size and smoother shape than mineral fillers. Moreover, the physical properties 100 of filler-sized particles of conductive mastics were determined and given in Table 1.

101 In order to investigate the impact of iron powder as filler-sized conductive particle within the asphalt 102 mastics, two mastic preparation processes are used. The first one is by adding iron powder with replacing an 103 equivalent volumetric amount of mineral fillers and the other one is without replacing the mineral fillers. It is 104 important to note that the adding order of filler-sized particles, the mixing time and the mixing temperature 105 affect on the well-dispersion of asphalt mastics. In the current research, the mixing process is separated in 106 two stages; (1) adding and mixing filler-sized particles together for 90 sec and (2) adding asphalt binder 107 which is SBS polymer modified and mixing it together with particles for 120 sec. Mixing is carried out at 180 108 $^{\circ}$ C for 180 sec. The compositions of the different conductive asphalt mastics (MA_F()_P()) are given in Table 109 1. The notation MA indicates mastic, F represents filler, P represents iron powder. The values between 110 brackets indicate the corresponding volume of the components.

111





112 FIGURE 2 High magnification SEM SEI images of filler-sized particles; (a) WL, (b) PL and (c) IP

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	н	•	

Specific surface area (m ² /g)		Mir	neral filler (WL)	Mineral filler (PR)	Iron powder (IP)	
		(m^2/g)	10.2650	1.9765	1.0066	
	Density (kg/m ³)		2780	2698	7507	
	Type of	Density of	Mineral filler	WL Mineral filler	PR Iron powder	
	Asphalt mastic	Asphalt mastic	(% m/m)	(% m/m)	(% m/m)	
	MA_F100_P0	1.594	50.40	7.10	0.00	
	MA_F95_P5	1.646	47.88	6.75	7.79	
	MA_F90_P10	1.683	45.36	6.39	15.58	
	MA_F85_P15	1.730	42.84	6.04	23.37	
	MA_F80_P10	1.844	40.32	5.68	31.16	
	MA_F75_P25	1.957	37.80	5.33	38.95	
	MA_F50_P50	2.243	25.20	3.55	77.90	
	MA_F25_P75	2.455	12.60	1.78	116.85	
	MA_F0_P100	2.796	0.00	0.00	155.80	
	MA_F100_P25	2.361	50.40	7.10	38.95	
	MA F100 P50	3.006	50.40	7.10	77.90	

4 TABLE 1 Physical properties of filler-sized particles and composition of conductive asphalt mastics

MA: asphalt mastic, F: mineral filler, P: iron powder, bitumen (% m/m): 42.5

116

117 EXPERIMENTAL METHODS

118

119 SEM Imaging

120

Micrographs of the conductive asphalt mastics are captured using a scanning electron microscope (SEM).
 The micrographs are obtained from a JEOL JSMM 6500F using an electron beam energy of 15 keV and beam
 current of approx. 100 pA. The backscattered electron image mode (BSE) is selected for the images
 acquisition.

Aluminum cylinders with a height of 18 mm and a diameter of 31 mm are used as sample-substrates for SEM scanning. A thin film of mastic is applied on a glass plate at 140 °C in order to form a very smooth area at one side after which the sample is stored at room temperature for 24 hours. Then, the sample is gently cut and placed on the aluminum cylinders. The study of micro-morphology of conductive asphalt mastic is performed in the environmental mode.

130

131 Electrical and Thermal Properties

132

After mixing the components, the hot conductive asphalt mastic is poured in a silicon-rubber mould to obtain rectangular samples with dimension $125 \times 20 \times 25$ mm. Electrical resistivity is determined with the twoelectrode method at room temperature of 20 °C. The short ends of specimen are cut by 1mm in order to avoid the problem of binder concentration at the surface and to have better contact with the electrodes. The electrodes are made of copper, placed in the right and left sides of the moulds and with the samples inside the

138 mould the electrical volumetric resistance is measured using a digital Multimeter.

139 The geometry and the electrical resistivity of the material are the only parameters that influence the 140 resistance. The difference in potential value between the electrodes and their total charge do not play a role 141 for this material property. Therefore, the electrical resistivity is obtained from the second Ohm-law:

142

$$\rho = \frac{RS}{L} \tag{5}$$

143

144 where ρ is the electrical resistivity, measured in Ω mm, L is the internal electrode distance, measured in mm, S 145 is the electrode conductive area measured in mm² and R is the measured resistance, in Ω .

Thermal conductivity measurements are performed by using the C-Therm TCi thermal analyzer. The sensor is based on the Modified Transient Plane Source Method to determine the thermal resistivity and effusivity of the conductive asphalt mastic. The specimen has a diameter of 17 mm to cover the entire sensor. The sensor is heated with a small current and its responses are monitored while in contact with the specimen. The thermal resistivity and effusivity of the specimen are measured and obtained directly from the sensor. From the inverse of the resistivity the thermal conductivity is obtained. Using the effusivity concept other thermal properties such as heat capacity and diffusivity can be derived. The effusivity is given by:

153

$$Effusivity = \sqrt{k \cdot \rho \cdot c_p} \tag{7}$$

154

where k is the thermal conductivity [W/m·K], ρ is the density [kg/m³] and c_p is the heat capacity [J/kg·K]. The thermal conductivity is defined from the Fourier law as:

157

$$q = -k \cdot \frac{dT}{dx} \tag{8}$$

158

where q is the heat flux (the amount of thermal energy flowing through a unit area per unit time), $\frac{dT}{dx}$ is the temperature gradient and k is the coefficient of thermal conductivity, often called thermal conductivity. The heating, reading and cooling process was repeated 6 times per specimen and the average value was used for the analysis.

164 Frequency Sweep Test

165

Dynamic Shear Rheometer (DSR) was utilized to obtain the rheological properties of the conductive asphalt mastic. Frequency sweep tests are carried out over a temperature range from -10 °C to 60 °C and the complex modulus and phase angle can be determined. By shifting these mechanical properties to a reference temperature (i.e. 30 °C), the master curves of the complex modulus and phase angle are built up for all conductive asphalt mastics. Before starting frequency sweep tests, a stress sweep test was conducted in order to identify the material linear viscoelastic range (LVR). The LVR is characterized as the 10% stiffness reduction criterion and was used to filter the linear and non-linear viscoelastic region.

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174 Multiple-Stress Creep Recovery Test

175

Apart from the frequency sweep analysis, DSR is also used to conduct the Multiple Stress Creep Recovery Test (MSCRT) at high service temperature. This test has been developed by FWHA as result of refinements in the repeated creep and recovery test and it basically consists of applying subsequent loading-unloading cycles monitoring the accumulated strain levels at each cycle (20). The presence of the elastic response of the

180 asphalt mixtures is defined by determining the percentage recovery and non-recoverable compliance.

181 Notably, the non-recoverable creep compliance denotes the rutting resistance or the permanent deformation

182 sensitiveness of asphalt mixture under repeated loading and that can be determined using the MSCR test (21,
183 22).

According to AASHTO TP 70-10 standard, the conductive asphalt mastics are loaded at a constant stress for 1 s and then allowed to recover for 9 s. Ten creep and recovery cycles are run at 0.1 kPa creep stress followed by ten more cycles at 3.2 kPa creep stress. The stress and strain are recorded at least every 0.1 seconds for the creep cycle and at least every 0.45 seconds for the recovery cycle during the test. The percent recoveries and the non-recoverable compliance were obtained at the end of each cycle and the average values were used at each loading level.

Here, multiple stress creep and recovery tests were carried out at 64 °C and the tests were performed with the parallel plate geometry with diameter 25 mm and 1 mm gap. The asphalt mastic samples were allowed to reach constant temperature for 10 minutes (within +/- 0.1 °C tolerance). Two replicates of each mastic were used for analysis and the rutting potential of each was evaluated at high temperatures. It should be noticed that the test described above is normally done on pure binders, so the results are only for comparison the different mastics under the given loading conditions.

196197 **RESULTS**

198

199 Micro-Morphological Images

200

201 The surface micro-morphology of asphalt mastic with iron powder is presented in Figure 3.a. The different 202 conductive asphalt mastics with different amounts of iron powder as described in Table 1 are investigated. 203 The grey particles represent the mineral fillers and the brightest parts of the images are the iron powder. By 204 comparing images 3 and 5 in Figure 3.b, it is obvious that the conductive asphalt mastics without substituting 205 the mineral filler - see image 3 - appear to have a surface morphology with less dark space than asphalt 206 mastics produced with substituting mineral filler with iron powder, see image 5. The spacing among the 207 filler-sized particles is reducing with increasing the amount of iron powder without substituting relative 208 volumetric amount of mineral filler, see images 1 to 3. Qualitative observation of conductive asphalt mastics 209 surfaces with SEM shows that the morphology of asphalt mastics after adding iron powder has a direct link 210 with the volumetric concentration of filler-sized particles – iron powder and mineral fillers. It should be noted 211 that the current micro-morphological results agree with the rheological results of conductive asphalt mastics 212 which will be explained in the Frequency Sweep Test subsection of the current paper.



(b)

FIGURE 3 SEM BSE (a) image of a conductive asphalt mastics with iron powder and (b) images of conductive asphalt mastics demonstrating the influence of replacing mineral filler with iron powder on the micro-morphology: (1) MA_F100_P0, (2) MA_F100_P25, (3) MA_F100_P50, (4) MA_F75_P25 and

- 217 (5) MA_F50_P50
- 218 219

19 Electrical and Thermal Properties

220

The electrical resistivity decreases with increasing iron powder content with or without replacing an equivalent proportion of mineral filler, see Figure 4. In Figure 4.a, a reduction of the electrical resistivity is observed when iron powder is mixed proportionally within the asphalt mastic by substituting mineral filler.



FIGURE 4 Effect of the volume content of iron powder on (a) the electrical resistivity of conductive asphalt mastics after replacing mineral filler with iron powder and (b) the electrical resistivity of conductive asphalt mastics without replacing mineral filler with iron powder

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239

Finally, the conductive asphalt mastics without replacing of mineral fillers with iron powder show a lower electrical resistivity than those developed after replacement, see Figure 5. This observation happens because the filler-sized particles form a highly density skeleton with very short spacing between the particles when extra iron powder is added in the asphalt mastic.



240

241

FIGURE 5 Effect of developing conductive asphalt mastics with and without replacing part of mineral
 filler with iron powder on electrical conductivity

245 The thermal conductivity and heat capacity of asphalt mastics produced, with and without substituting part 246 of the mineral filler with iron powder, are presented in Figure 6. It was found that the thermal conductivity of 247 asphalt mastic increased after adding iron powder. This increasing tendency can be explained by the thermal 248 properties of iron powder which is added into the asphalt mastic. It is known that the thermal conductivity of 249 iron powder is considerably higher than the conductivity of the other asphalt components. Hence the increase 250 of the amount of iron powder leads to an increase of the effective thermal conductivity of the conductive 251 asphalt mastic. This can be seen in Figure 6.a1&a2 showing that the thermal conductivity of sample 252 MA F85 P15, which represents the conductive asphalt mastic at the electrical percolation threshold, was 253 0.56 W/mK is higher than the thermal conductivity of pure asphalt mastic sample MA_F100_P0 which was 254 0.487 W/mK. On the other hand, Figure 6.b1&b2 demonstrates a reduction of the heat capacity of asphalt 255 mastics when iron powder is added.





FIGURE 6 Effect of the volume content of filler-size additives on (a.1) the thermal conductivity and (b.1) heat capacity of conductive asphalt mastics after substituting mineral filler with iron powder, (a.2) the thermal conductivity and (b.2) heat capacity of conductive asphalt mastics without substituting mineral filler with iron powder

Finally, the produced conductive asphalt mastics without substitution of mineral filler-sized particles had a higher thermal conductivity and lower heat capacity, see Figure 7. At higher filler-sized particles concentration, the interaction among the particles is increasing within the asphalt mastics. Thus, the spacing among the particles and the coating role of asphalt binder around the particles reduces having as consequence this thermal observation for the conductive asphalt mastics.

267



FIGURE 7 Effect of developing conductive asphalt mastics with and without substitution of mineral filler with iron powder on (a) thermal conductivity and (b) heat capacity

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271 Frequency Sweep Test

Before the frequency sweep tests, the stress sweep test was conducted from -10 °C to 60 °C with a shear stress range from 0.01 to 10 Pa and at 1 Hz in order to identify the linear viscoelastic range (LVR). The LVR is characterized as the 10% stiffness reduction criterion and was used to filter the linear and non-linear viscoelastic region. Afterwards, the frequency sweep test was carried out over a temperature range from-10 °C to 60 °C. At a reference temperature of 30 °C, the master curves as given in Figure 8 show the rheological behavior for all the conductive asphalt mastics. The test stress sweep and frequency sweep were run on 8 mm parallel plates with a 2 mm gap for mastics at all the testing temperatures.

280 It can be observed that the asphalt mastic without adding iron powder is obviously much stiffer than the 281 conductive mastics produced after replacing mineral filler with iron powder. This happens due to the fact that 282 iron powder is spherical and finer particle than the other mineral fillers and is easily rolling under shear stress 283 when is added in the mastic by replacing mineral filler. However, the asphalt mastics appear to have a higher 284 complex modulus and lower phase angle when iron powder is added without replacing the mineral filler. The 285 reducing visco-elastic properties at higher concentrations of filler-sized particles and when particles are added 286 without substitution are linked with the interaction of particle-particle. Increasing the concentration of filler-287 sized particles leads to lower the spacing among the particles and asphalt mastics with lower viscosity and 288 higher stiffness are obtained. Consequently, the lower workability of mastic during mixing process is 289 resulted.

- 290
- 291



FIGURE 8 (a) Complex modulus and (b) phase angle master-curves for conductive asphalt mastic produced with and without substitution part of filler with iron powder

295 Multiple-Stress Creep Recovery Test

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MSCR test was used to quantify the rutting sensitivity of conductive asphalt mastics and for this reason the percent recovery and the non-recoverable compliance were determined at two different stress levels. Figure 9.a shows that the percent recovery of the conductive asphalt mastics experienced a slight reduction from 97.5% to 95% for MA_F100_P0 and MA_F0_P100 respectively, at lower stress level. This slight reduction indicates that the conductive asphalt mastics can recover a lower portion of the total strain at the end of each loading-unloading cycle for the lower load level. Similarly, reduction of the percentage recovery shows the same tendency for the higher stress level for the same mastics. This observation of lower percent recoveries indicate that conductive mastics appear marginally higher prone to rutting when iron powder substitutes mineral filler. Moreover, conductive mastics demonstrate reduction of the percent recovery as well when iron powder was added without replacing part of mineral filler.

307





(b)

308FIGURE 9 (a) Recovery (%) and (b) non-recoverable creep compliance (kPa⁻¹) of conductive asphalt309mastics

310

The non-recoverable compliances of conductive asphalt mastics are illustrated in Figure 9.b. High compliance values of mastic imply that the rutting performance is weak. It can be observed that significant

decrease of the creep compliance is found in case of producing conductive asphalt mastics by adding iron powder (MA F100 P25). This means that mastic MA F100 P25 can accumulate plastic deformations by

heavy traffic loads sufficiently. However, as noticed in Frequency Sweep results subsection, the visco-elastic

316 properties of mastics produced with adding iron powder, such as MA_F100_P25, were reduced (lower

317 viscosity and higher stiffness) and subsequently the workability of asphalt mixture lowers. About the mastics

318 produced by replacing mineral filler with iron powder, these appear a minor increase at 0.1 kPa stress level

319 when 25% of iron powder was added. The creep compliance shows similar performance for both low and 320 high stress level such as the percent second stress of meeting.

high stress level such as the percent recovery response of mastics.

322 CONCLUSIONS

323

As it is mentioned several times on this paper, the type of filler-sized particles, the concentration of particles and the interaction among particles and asphalt binder have direct influence on structural and non-structural performance of asphalt mastics. Here an experimental protocol was proposed with main objective to explore the impact of filler-sized particles on the performance asphalt mastics produced for induction heating applications.

For these purposes, as well as for the purpose of improving the electrical and thermal properties of asphalt mastics, iron powder was selected as filler-sized additive. During this research, it became clear that understanding the conductive additives-mineral fillers interaction within the binder matrix provides the necessary framework not only to control the electro-thermal properties but also to adjust the workability of mastic at desired levels. Viscosity, effective electrical and thermal conductivity of mastics were assessed as the most valuable parameters to manufacturing more durable asphalt mixtures with induction heating capabilities.

Future studies should include more fundamental parameters of filler-sized particles of mastics, such as chemical and electrochemical studies on the particles-particles and the particles-binder interactions. Moreover, the evaluation of moisture and chloride induced damage of conductive asphalt mastics and subsequently of asphalt concrete mixtures has been assessment crucial to predict the proper time of induction heating maintenance of asphalt pavement.

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343

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