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ORIGINAL ARTICLE

Induction heating of mastic containing conductive fibers and fillers

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Abstract The objective of this research is to examine the induction heating of mastic through the addition of electrically conductive fillers and fibers (graphite and steel wool), and to prove that this material can be healed with induction energy. The effect of fibers content, sand–bitumen ratio and the combination of fillers and fibers on the induction heating of mastic was investigated. It was found that there is an optimum content of fibers for each sand–bitumen ratio, above which mastic cannot be heated any more. This optimum seems to coincide with the optimum electrical conductivity of the mixture shown in [1]. It was found that the maximum temperature reached within a certain time period was a function of the sand–bitumen ratio (s–b) and of the volume content of fibers. The mastic could be heated with the addition of a very low volume of conductive fibers. The fastest heating power was obtained with the mix with the maximum electrical conductivity. Gel-Permeation Chromatography (GPC) was also used to show that there is not ageing of bitumen during the heating process.

Keywords Induction heating · Conductive mastic · Steel wool · Graphite · Self-healing

1 Introduction

Asphalt concrete is one of the most common types of pavement surface materials used in the world. It is a material that consists of a mixture of asphalt binder, aggregate particles and air voids. This material must resist in good conditions all the traffic loads under many different climatic conditions for a prolonged time. In order to maintain these characteristics during its lifetime, asphalt concrete wearing courses should be constantly maintained and repaired. Little cracking on highway runway can mean the start of some big distress. The objective of this research is to investigate how mastic can be heated through the addition of different volumes of electrically conductive particles.

It is well known that asphalt concrete is a self healing material. As Little and Basin explain [2], healing occurs only after a stress or strain is induced,

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which is sufficiently large to generate damage. Immediately after the load that generated the damage has been removed, and both faces of the crack are in contact, the diffusion of molecules from one face to the other starts. This process will happen, while there are not more loads, until the crack has completely disappeared and the repaired material has the level of strength of the original one. It is also well known that the amount of healing increases when the material is subjected to a higher temperature during the rest period [3, 4]. The problem comes because it is difficult to stop traffic circulation on a road to allow enough self healing recovery at ambient temperature.

Conductive asphalt concrete may be defined as the mixture of bitumen, aggregates and electrically conductive components to obtain high electrical conductivity. In [1] it was shown how to make electrically conductive mastic by adding conductive fillers and fibers. It was discovered that it is much more effective to add electrically conductive fibers than to add fillers. In addition, it was observed that there is an optimum volume of fibers for each mixture. Below this volume, the conductivity of the material suddenly drops to that of a non conductive material. Above this volume the fibers are difficult to mix and the conductivity increases very slowly. In this research it was also demonstrated how this electrically conductive mastic could be heated very fast with induction energy.

If it would be possible to heat mastic on site, self healing rates will increase and cracks will be closed much faster. To study this, the research presented in [1] is continued and the effect of mastic with different volumes of electrically conductive fibers and fillers and sand–bitumen ratios on the heating rate is investigated. To get a better understanding of the ageing effects of this technology on the mastic, Gel-Permeation Chromatography (GPC) is used.

2 Experimental method

2.1 Materials

Mastic specimens were prepared with different sand–bitumen ratios and volumes of conductive particles. Five different sizes (<0.120, 0.120–0.250, 0.250–0.5, 0.500–1.0, 1.0–2.0 mm) of natural silica mineral, with density 2.67 g/cm^3, were mixed to have uniform grading. The virgin bitumen used was 70/100 pen, obtained from Kuwait Petroleum, with density 1.032 g/cm^3.

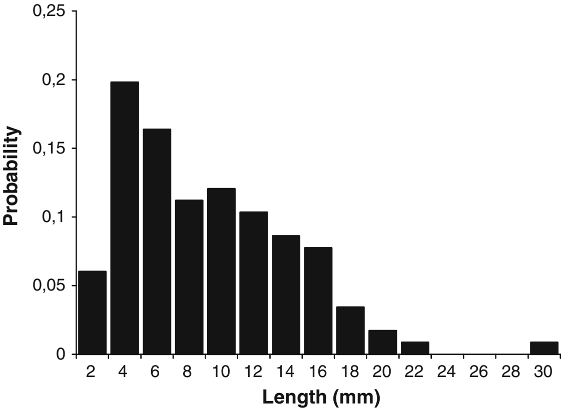

Fig. 1 Chopped steel wool lengths distribution

With regard to the electrically conductive particles, two different types were used, conductive fibers and filler. The first one was steel wool, of type 000, with diameters between 0.00635 and 0.00889 mm and approximate density 7.6 g/cm^3, chopped by hand, always by the same operator. To find the size, more than 100 fibers were checked by taking photographs under the optical microscope and measuring their length with an image processing program, obtaining the distribution showed in Fig. 1. The conductive filler used was graphite with a particle size of less than 20 μm, and a carbon content of more than 99.0%. Its electrical resistivity was 10^{-4} Ω cm.

Finally, the material proportions for the mastic used to test the ageing with GPC were, by total percentage of weight in the mixture: 74.5% of sand without filler (aggregates with size less than 0.120 mm were eliminated), 18% of bitumen and 7.5% of fibers.

2.2 Methods

Conductive fillers and fibers, aggregates and bitumen were blended during 15 min at 285 r.p.m. and 150°C of temperature. Later, the mass was hand-compacted in silicon-rubber moulds, obtaining rectangular specimens of 125 × 25 × 15 mm.

Electrical resistivity measurements were done at 20°C. The electrodes were made of nickel and placed at both ends of the test sample to measure the electrical volumetric resistance. Dry graphite powder

(<20 μm) was used to fill the gaps between the electrodes and the specimens and to ensure a perfect contact between them. The total contact resistance between the electrodes and the graphite was less than 0.1 Ω, which is negligible with respect to the great resistances studied (higher than 20 Ω in the samples). A digital multimeter was used to measure the resistance below 36×10^6 Ω. A resistance tester was used to measure the resistance higher than this value. From the resistance data, the electrical resistivity was obtained from the second Ohm-law:

$$\rho = \frac{RS}{L} \tag{1}$$

where ρ is the electrical resistance, L is the internal electrode distance, S is the electrode conductive area and R is the measured resistance. The electric field is assumed constant and the end-effects considered negligible.

The temperature changes were measured with a 320 × 240 pixels, full colour infrared camera. The induction heating experiment was performed by using an induction heating system with a capacity of 50 kW and at a frequency of 70 Hz (Fig. 2). The coil used was rectangular and its position was horizontal, 3 cm above the sample, which was bellow one of the external spirals of the coil. The temperature recorded is obtained from the mean temperature on the sample. During the heating, it could be observed that the corners of the specimen were at a higher temperature than the rest of the specimen. In future research, larger specimens will be used to avoid these effects.

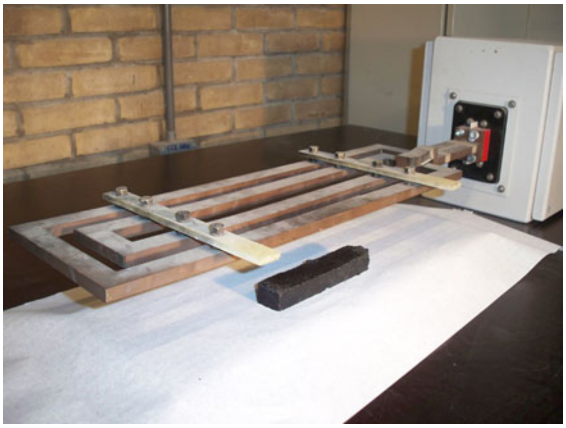

Fig. 2 Test setup used in the research

Although the system was not fully optimized, it had not influence on the research objectives: demonstrate how asphalt mortar can be heated and healed through induction energy.

Furthermore, Gel-Permeation Chromatography (GPC), a chromatographic method in which particles are separated based on their hydrodynamic volume, was performed to analyze the molecular weight distribution change of induction heated samples at 60, 110, 160 and 200°C at a frequency of 70 Hz and a power of 50 kW. From each one of these specimens, more than 10 tests were done taking material from different positions in the sample. In total, 62 GPC tests were performed.

3 Induction heating

Induction heating is a process which is used to bond, harden or soften metals or other conductive materials. When an alternating electrical current is applied across a conductive coil, an alternating magnetic field with the same frequency as the alternating current causing it is created [5]. According to Faraday's effect, if a magnetically susceptible and electrically material is located within the magnetic field, an electric current (eddy current) will be induced with the same frequency than the magnetic field (Fig. 3).

Bitumen is not an electrically conductive material, but conductive closed loops must be present in the material for eddy currents to be induced, for that reason, electrically conductive particles were added to the mixture. The eddy currents heat up the material according to the well-known Joule effect:

$$P = RI^2t \tag{2}$$

where R is the material resistance, I the current and t the time of exposure to the magnetic field.

In Fig. 4 the heating curve of the mastic samples used to test the ageing of bitumen through induction heating is shown. In this Figure it can be observed that the curve shape is almost parabolic. Unfortunately, the resolution of the infrared camera used was not so high to find out the heating mechanism. Anyway, based on the researches by Ahmed et al. [6], it is possible to elucidate that the predominant mechanism of heating will be the fiber heating, although other mechanisms such as junction heating due to the dielectric hysteresis

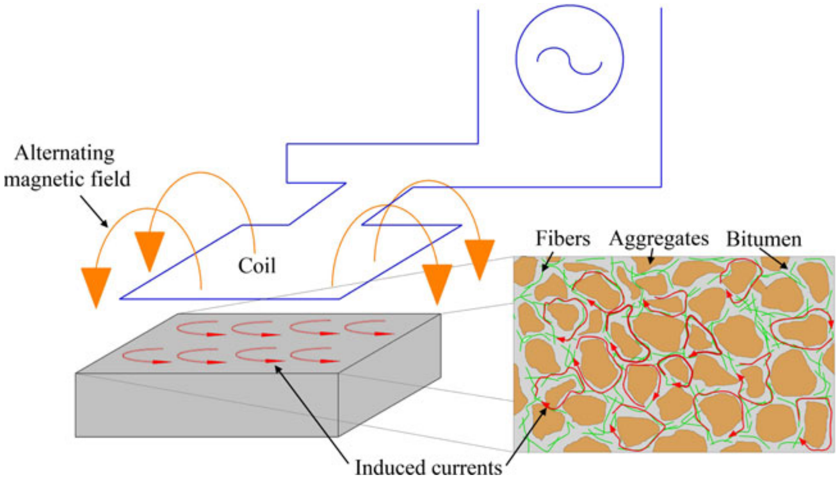

Fig. 3 Induction heating scheme

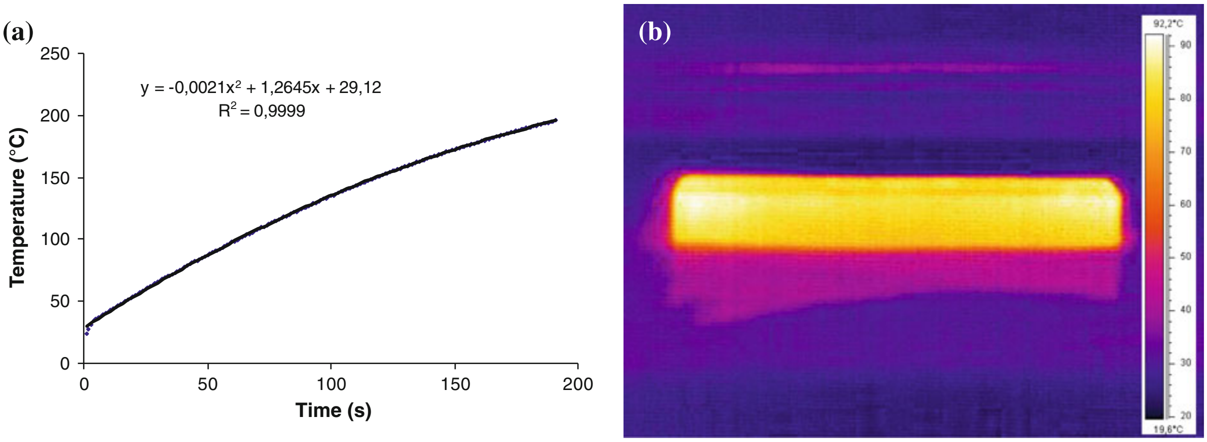

Fig. 4 **a** Heating curve for a sample with 5.66% steel wool (related to the volume of bitumen) and sand–bitumen 1.60. **b** Infrared Image of one sample during the induction heating

heating and junction heating due to the contact resistance heating may happen. In addition, it has been observed how, as the material heats, the viscosity of the sample lowers and a squeeze-out of the bitumen happens. As pointed by Yarlagadda et al. [7], this can result in higher fibre contact.

The amount of heat generated in the specimen is proportional to the power induced on it [6]. The relationship between the source and the power generated in the sample can be expressed as:

$$P = \frac{(2\pi f \mu_r H(I) A)^2}{R} \qquad (3)$$

where f is the field frequency, μ_r the magnetic permeability of the material studied, $H(I)$ is the magnetic field intensity, which is dependent on the current of the equipment, A the cross-sectional area of the conductive loop in the workpiece and R is the material resistance.

Other fundamental factor in the induction heating of a conductive material is the current frequency. It has been demonstrated that the time to heat a composite to a certain temperature decreases quadratically with the increasing frequency [8]; but the higher the frequency, the lower the heating depth. That is why it would be important to compensate both factors. Nevertheless, for this research, as the purpose was to demonstrate that it is possible to heat conductive mastic with induction energy, these factors were not considered.

4 Results and discussion

4.1 Effect of sand–bitumen ratio

The volume resistivity variation against different sand–bitumen ratios for samples with volumetric conductive particles–bitumen ratio 6.54% is displayed in Fig. 5. In this Figure it can be clearly seen how the volume resistivity is greatly influenced by the sand–bitumen ratio (s–b): 8.76% for s–b 0.77, 6.54% for s–b 2.00 and 5.83% for s–b 2.50 (percentages related to the total volume of bitumen in the mixture) (Table 1). In addition, it is very remarkable that a sudden increase in the volume resistivity takes place when reducing the sand–bitumen ratio below the optimum. It can also be observed that the resistivity in the optimum decreases almost exponentially with less volume of sand in the mixture: 82.50×10^{-2} Ωm, 17.54 Ωm and 420.00 Ωm for sand–bitumen ratios 0.77, 2.00 and 2.50, respectively. Besides, if the sand–bitumen ratio is increased above the optimum, the electrical resistivity increases exponentially. Samples with sand–bitumen ratio above the percolation threshold were difficult to mix, with clusters of fibers that grew when increasing the volume of sand in the mixture. In addition, samples looked porous and weak. Finally, in the 5.83% steel wool curve it was found that if the sand–bitumen ratio is increased above a certain limit (in this case, 2.74), the resistivity of the sample drops to that of a non-conductive material.

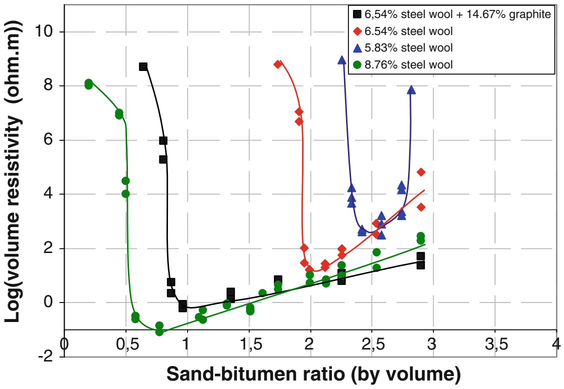

Fig. 5 Effect of sand–bitumen ratio on the electrical resistivity of the system for different conductive particles–bitumen ratios [1]

Table 1 Sand–bitumen ratios, % of fibers and electrical resistivities of the three mixtures studied at the optimum

s–b	0.77	2.00	2.5
% of fibers	8.76	6.54	5.83
Resistivity (Ωm)	82.50×10^{-2}	17.54	420.00
Maximum temperature after 120 s heating (°C)	≈150	≈163	≈150

In Fig. 6a–c the maximum temperatures reached, at three different heating times, for samples with different sand–bitumen ratios and three fixed volumes of conductive fibers (8.76, 6.54 and 5.83%) are shown. Comparing Fig. 6a and b with the curves in Fig. 5 with 6.54 and 8.76% of steel wool, it is clear that the maximum temperature reached in the mastic after a fixed heating time is directly related to its maximum electrical conductivity. It can also be observed that it is not necessary to have an electrically conductive mastic to heat it. Even below the percolation threshold in the conductivity (6.54% of fibers for s–b ratio 2 and 8.76% of fibers for s–b ratio 0.77), very similar temperatures to the ones reached in samples with the optimum amount of fibers are obtained.

Besides, in Fig. 6c the maximum temperatures reached by samples with conductive additives content 5.83% are shown. In this case, the optimum for heating does not coincide with the optimum for the conductivity in Fig. 5. The reason for this is that in these mixtures, the volume of bitumen was so low that it was difficult to mix, and many clusters of fibers appeared, they were not well distributed in the samples and they could not percolate. Finally, in Fig. 6a–c, it can be observed how at 60 s and at 120 s heating, the maximum temperatures reached are very similar, but at 180 s heating, the maximum temperature reached seems to be related to the volume of fibers in the mixture. This is a point for more study, because it is not clear which is the main parameter for heating: the number of contact between fibers, their electrical resistance or their volume in the mixture.

4.2 Effect of mixing steel wool and graphite

The effect of adding graphite to the mixture is examined in Fig. 5 and in Fig. 6d. In Fig. 5, volume resistivity variation against different sand–bitumen ratios for samples with volumetric conductive particles–bitumen ratio 20.54% (6.54% steel wool + 14.67%

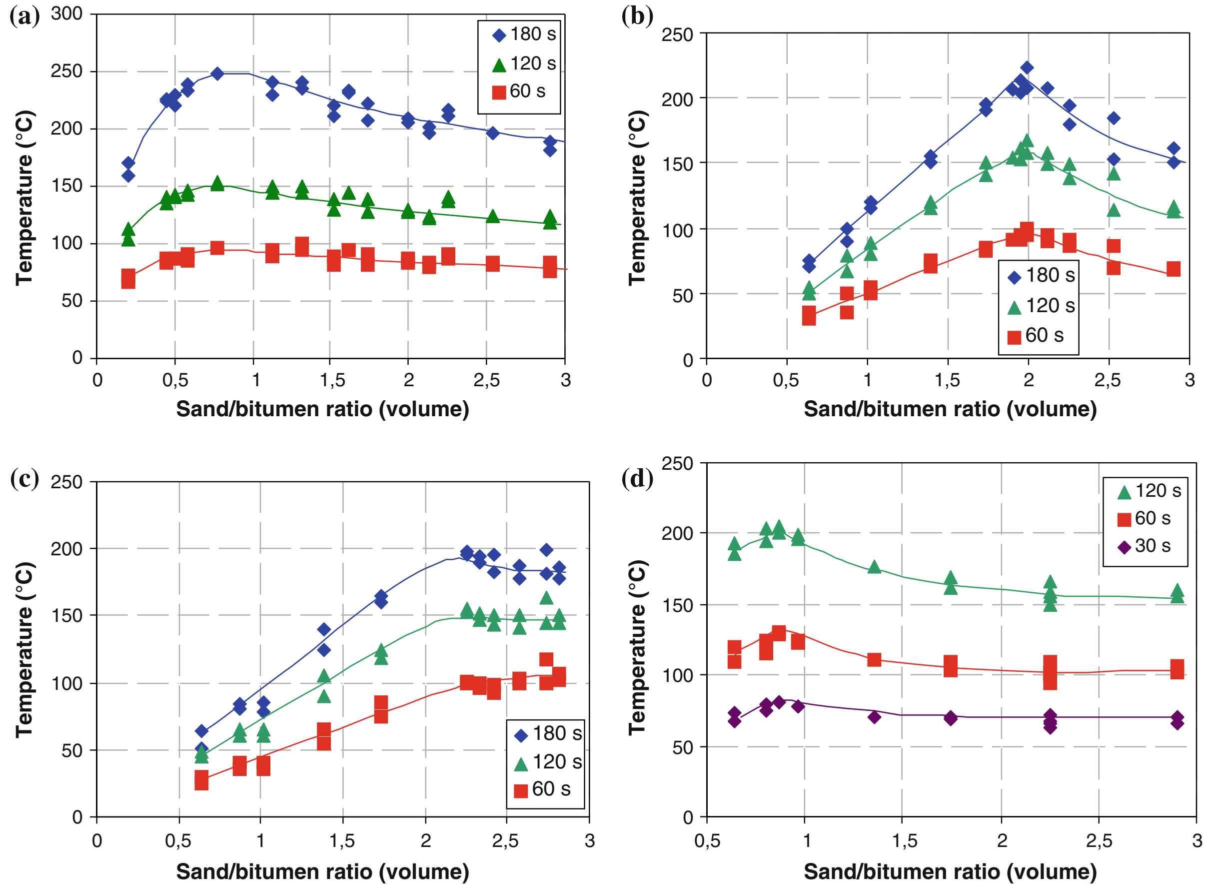

Fig. 6 Maximum reachable temperatures at three different heating times for mastic with different conductive additives content and different sand/bitumen ratios. **a** Conductive additives content 8.76% (steel wool). **b** Conductive additives content 6.54% (steel wool). **c** Conductive additives content 5.83% (steel wool). **d** Conductive additives content 20.54% (6.54% steel wool + 14.67% graphite)

graphite) is presented. Comparing this curve with the 6.54% steel wool one, it can be observed how the optimum conductivity is now shifted to the left: It is necessary to have a lower sand–bitumen ratio in order to make asphalt concrete conductive. Besides, the conductivity in the optimum is much higher than when only fibers were added.

In Fig. 6d the maximum temperatures reached at three different heating times for different sand–bitumen ratios are shown. The same than in the previous section, the maximum temperature coincides with the point where the electrical conductivity is maximum. In this Figure, for the three times studied, the temperatures reached are higher than when only steel wool is added to the mixture, so adding electrically conductive powders could be a solution to increase the system efficiency, although this is something that will be studied in the future.

4.3 Effect of fiber volume content

Volume resistivities versus fibers content (conductive fibers–bitumen ratio) with a fixed sand–bitumen ratio 2.25 are displayed in Fig. 7a. As explained in [1], for small amounts of fibers, the electrical resistivity is very similar to that of a plain mastic, exhibiting insulating behaviour. For the type of fibers and the sand–bitumen ratio used, after more than 6.02% of fibers have been added, mastic becomes suddenly conductive. Adding more fibers does not improve the electrical conductivity of mastic, but make mastic difficult to mix and clusters of fibers appear during the mixing process.

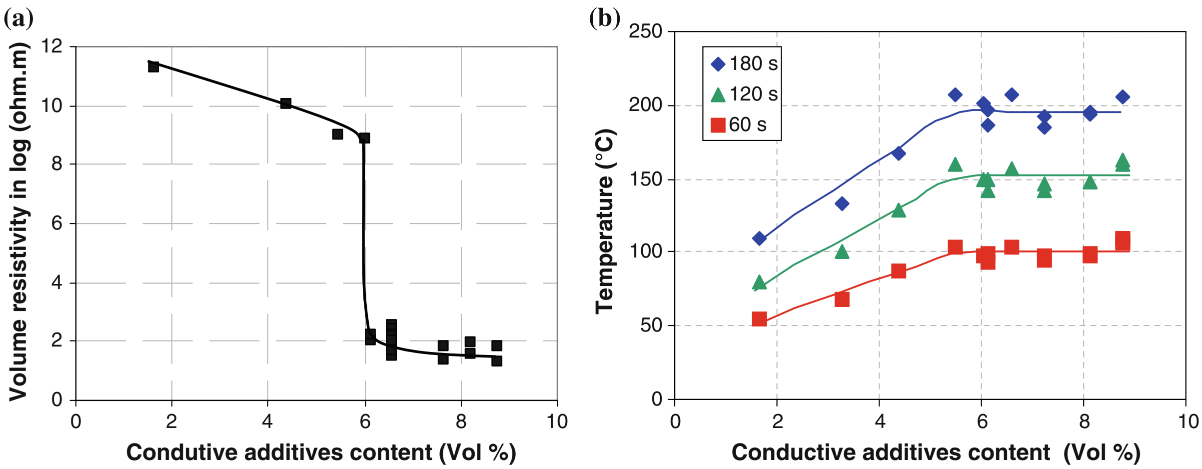

Fig. 7 **a** Volume resistivity versus conductive particles content for mastic with sand–bitumen ratio 2.25. **b** Maximum reachable temperatures at three different heating times for different volumes of fibers

In Fig. 7b the maximum temperatures reached at three different heating times for different volumes of fibers are shown. In this Figure it can be seen how the maximum temperature increases with the volume of fibers until a certain point, where the temperature does not increase any more, independently of the volume of fibers added. For example, with approximately 6% of fibers in the mixture, the maximum temperature after 120 s heating is close to 150°C, and with 8% of fibers, the maximum temperature reached after 120 s heating is also close to 150°C. This optimum volume of fibers seems to coincide with the 6.02% of fibers in the mixture needed to have electrically conductive mastic.

4.4 Analysis and discussion

In Figs. 6 and 7 it has been proven that mastic does not need to be electrically conductive to increase its temperature via induction heating. Fibers are like small heaters inside the mastic (Fig. 3). To be heated with induction energy they need to be connected in small closed-loop circuits. When few fibers are added to the mixture (related to the total amount of bitumen in it), very few circuits will be formed and the increase of temperature will be relatively low. When more fibers are added to the mixture, the maximum temperature reached will increase (so as the electrical conductivity of the sample). Eventually, there will be so many fibers that all the possible spaces where they could be (they can only be in the volume occupied by bitumen) will be saturated By increasing the volume further beyond this point, clusters of fibers will appear, there will not be enough bitumen to cover all the fibers and they will be exposed to air (with the consequent oxidation and loss of properties). It was tried to heat the steel wool with induction heating. Its temperature did almost not increase, independently of the heating time, so it can be deduced that the fibers lose the heat very fast into the surrounding environment. Which means that if they are not completely covered with bitumen, they will not increase the temperature of the mixture. Finally, if their volume is above the optimum of fibers, and clusters are present, the temperature increase will not be uniform.

If the 180 s curves in Fig. 6a–c, are compared, it can be seen that there is a dependence between the sand–bitumen ratio and the volume of fibers: There is an optimum of fibers for each sand–bitumen ratio where the temperature is maximum. In the optimum, the fibers are easy to mix and the electrical conductivity of the mixture is the highest. Outside the optimum, the heating speed is lower, the resistivity increases exponentially (Fig. 5) and the fibers are difficult to mix. Each mixture is different and the optimum volume of fibers should be found for each sand–bitumen ratio or for each aggregate type.

Besides, it can be also observed that when there are too many fibers for each sand–bitumen ratio, the maximum temperature reached is not as high as it could be if few fibers were added. Otherwise, as stated in the percolation theory [9, 10], in Fig. 7a it is possible to observe that the electrical resistivity decreases with the increase of fiber content once there is enough volume of fibers in the sample. In [1] it is stated that the changes in the resistivity under conductive additive content variations can be divided in four phases: Insulated Phase (1), where the resistivity of the system is presumably similar to that of plain asphalt concrete, Transition Phase (2), where both sides of the test samples are connected with fibers, their resistivity drops very fast and the material becomes electrically conductive, Conductive Phase (3), where the number of fibers is at its optimum; and the Excess of Fibers phase (4), where the increase of fibers in the mixture has none or negative effect. These phases are clearly visible in Fig. 7a, and have their projection in Fig. 7b: during the Insulated Phase; the temperature increases with the volume of conductive particles from 20°C to its maximum at the Conductive Phase. In addition, as explained in the paragraph below, during the Excess of Fibers phase, where clusters of fibers appear and the mixture looks porous and weak, the temperature does not increase any more and for some volumes of fibers, it is even reduced.

Finally, in [1] it was explained how small additions of graphite can stabilize the electrical conductivity of the mastic and increase its conductivity if the volume of fibers added is at the optimum. Mixing small volumes of graphite in the mixture, will avoid the electrical conductivity dropping from the Conductive Phase through the Transition Phase to that of a non conductive material due to small errors during the production process that could change the conductivity locally. Although this is not very important for the heating temperatures due to the smooth transition between the Insulated Phase and the Conductive Phase, as seen in Fig. 6d, graphite additions would increase the heating speed and the efficiency of the system. The problem of graphite is that it reduces the mechanical properties of the material [11], so it should be limited to a minimum, just to avoid the conductivity dropping when the volume of fibers is not enough.

4.5 Chemical changes analysis

In GPC, the sample components are eluted in order of decreasing molecular weight. An example of a GPC chromatogram is shown in Fig. 8a and, for all the measurements done, the average molecular weight has been studied as a good parameter to evaluate ageing [12]. It has been calculated in the following way:

$$M_w = \frac{\sum \left(n_i \cdot M_i^2\right)}{\sum \left(n_i \cdot M_i\right)} \tag{4}$$

where M_w is the weight-average molecular weight (g/mol) M_i is the molecular weights and n_i are the number of molecules of molecular weight M_i.

To show what happens with this value during the induction heating, in Fig. 8b, the empirical cumulative distribution function $(i - 0.5)/n$, where i is the

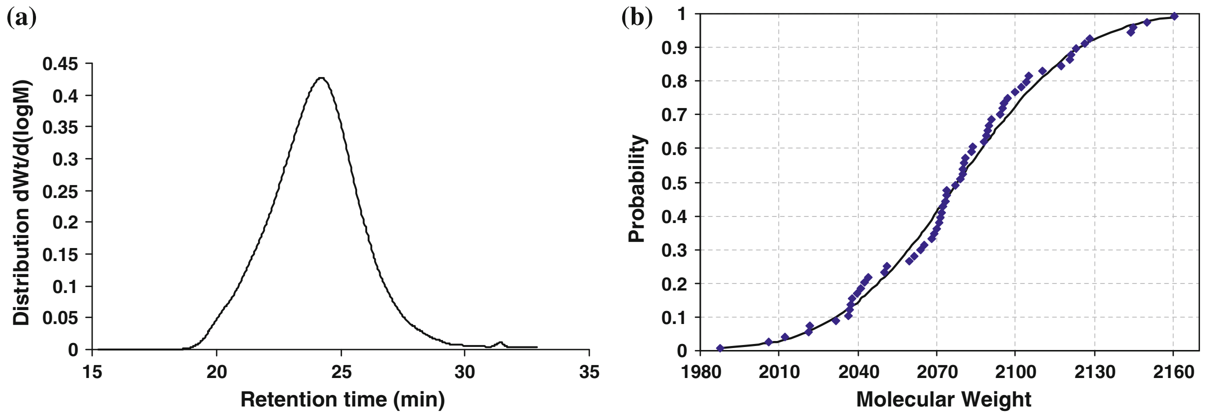

Fig. 8 **a** Molecular weight distribution plot of an induction-heated bitumen at 110°C. **b** P–P plot of all the GPC samples, heated at four different temperatures, the original bitumen and the non heated samples

position of the value in the series and n is the number of elements in the series, is represented together with the theoretical normal cumulative distribution function. It can be observed how both plots fit quite well, which means that, the molecular weight average does not change due to the induction heating.

5 Conclusions

This paper represents the continuation of the research showed in [1]. This is the first paper that explains how to heat mastic with induction energy. It has been proven that to heat mastic with induction, it is necessary to add electrically conductive fibers and fillers and that sand–bitumen ratio is a key factor in the design of these mixtures that cannot be considered separately from the volume of conductive particles added. There is an optimum volume of conductive fibers and fillers for each sand–bitumen ratio, above which the heating speed does not increase any more, the electrical resistivity remains constant or is reduced and clusters of fibers start appearing in the mixture, which makes the heating non uniform. This optimum volume of conductive particles coincides with the volume needed to have the maximum conductivity in the asphalt. Below this optimum value, the mastic electrical resistivity drops to that of a non conductive material, but mastic can still be heated. Otherwise, it is necessary to have a minimum amount of bitumen around the aggregates to have a good mixture; if this minimum film thickness is not enough, then it is impossible to mix the fibers in the asphalt concrete and clusters of fibers appear, which reduce the total heating speed.

Although in [1] it was stated that a small increment or a small decrement of the sand–bitumen ratio can force an increase in the resistivity, induction heating behaves differently. The Transition Phase does not exist and, when the volume of fibers is below the optimum, the heating rates simply decrease until there are not more fibers in the mixture or the sand–bitumen ratio approaches to 0. In the case of the heating rates, each mixture is different; it basically depends on the electrical conductivity of mastic, which comes from the type of aggregates used, the sand–bitumen ratio, the type of conductive fibers, its length and diameter, the type of conductive filler used, etc. In this case, due to the lack of Transition Phase, the combination of fibers and filler does not have an especial effect on the mixture: electrically conductive fillers just increase the heating rates as any other conductive addition. To find the optimum volume of conductive particles needed, each mixture should be analyzed separately by increasing the volume of fibers added until the optimum of fibers (percolation threshold) is found. Finally, it has been demonstrated that the bitumen is not aged by the induction heating.

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