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Influence of the Microwave Heating Time on the Self-Healing Properties of Asphalt Mixtures

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Featured Application: A new generation of advanced self-healing asphalt pavements is coming. The asphalt mixture presented in this study could be potentially used to build long-life roads with crack-healing properties through electromagnetic heating, e.g., induction and microwaves.

Abstract: This paper aims to evaluate the influence of the microwave heating time on the self-healing properties of fibre-reinforced asphalt mixtures. To this purpose, self-healing properties of dense asphalt mixtures with four different percentages of steel wool fibres were evaluated as the three-point bending strength before and after healing via microwave heating at four different heating times. Furthermore, the thermal behaviour of asphalt mixtures during microwave heating was also evaluated. With the aim of quantifying the efficiency of the repair process, ten damage-healing cycles were done in the test samples. In addition, self-healing results were compared with the fibre spatial distribution inside asphalt samples evaluated by CT-scans. Crack-size change on asphalt samples during healing cycles was also evaluated through optical microscopy. It was found that the heating time is the most influential variable on the healing level reached by the asphalt mixtures tested by microwave radiation. CT-Scans results proved that fibre spatial distribution into the asphalt mixtures play an important role in the asphalt healing level. Finally, it was concluded that 40 s was the optimum heating time to reach the highest healing levels with the lowest damage on the asphalt samples, and that heating times over 30 s can seal the cracks, thus achieving the self-healing of asphalt mixtures via microwave heating.

Keywords: self-healing; asphalt mixture; metallic fibres; microwave heating; heating time

1. Introduction

Asphalt mixtures are composite materials made of aggregates and bitumen. They are widely used in the pavement industry around the world. However, mechanical resistance and durability of asphalt mixtures can be reduced due to their permanent exposure to traffic loading and environmental conditions [1]. Ageing [2], moisture damage [3] and thermal cracking [4] are the main factors that affect the durability of asphalt pavements. With the aim of improving the behaviour of flexible pavements against these factors, different types of fibres can be added to the asphalt mixtures [5], such as: cellulose and mineral fibres [6]; polymeric fibres like polypropylene and polyester [7]; and steel wool and other waste fibres [8]. In particular, due to the higher tensile strength of metallic fibres compared with asphalt mixtures they can improve the tensile resistance and cohesive force when added to the mixture [5]. Additionally, these fibres can help to prevent the origin and propagation of cracks [9]. Also, asphalt mixtures with metallic fibres are known for presenting improved resistance, fatigue and ductility characteristics [10]. Therefore, asphalt mixtures reinforced with metallic fibres can have good resistance to ageing, moisture damage and cracking [11].

Moreover, metallic fibres can be added to asphalt mixtures with the aim of increasing their thermal and electrical conductivity [12]. Hence, metallic fibres have been used to develop new flexible pavements with energetic [13] and self-healing purposes via heating processes by electromagnetic induction [12,14–17] or microwave radiation [17–20]. It is known that asphalt pavements have self-healing properties when they reach high temperatures during summer, when the cracks in the road can be closed by themselves [12]. This is due to the fact that the bitumen viscosity is related with the temperature, and consequently when the bitumen reaches a temperature threshold (between 30 °C and 70 °C) it can flow by capillarity through the micro-cracks opened in the pavement [21].

There are currently different heating technologies that can accelerate the healing process of asphalt mixtures. One of them is the electromagnetic induction heating [22]. This method consists on adding ferrous materials (like metallic fibres) to the asphalt mixture and exposing it to an alternating electromagnetic field with a frequency of Kilohertz [23], using an induction heating device. This process induces an electrical current in the ferrous particles increasing their temperature due to the Joules principle. Thus, the thermal energy disperses in the asphalt mixture and increases the temperature of the bitumen [24].

A second technique is microwave heating [17], where asphalt mixtures are exposed to an alternating electromagnetic field with a frequency in the order of Megahertz [20], using an electromagnetic radiation device like a microwave. Microwave heating affects the bitumen, producing a change in the orientation of polar molecules, which results in internal friction and an increase of the mixture temperature [25]. Additionally, when ferrous materials are added to the mixtures, they can reflect the microwave radiation and accelerate the temperature increase procedure [25]. In this context, metallic fibres can be used to increase the heating rates of asphalt mixtures considering that they can absorb more thermal energy than the aggregates and bitumen [19]. For all the above, fibres addition can have influence on the mechanical and self-healing properties of the new asphalt mixtures, although it is still not clear how this influence is produced.

The main objective of this study is to evaluate the influence of the microwave heating time on the self-healing behaviour of asphalt mixtures. With this purpose, four different asphalt mixtures have been studied under laboratory conditions, using the same aggregates gradation and bitumen content, but varying the amount of fibres added to the mixture: 2%, 4%, 6% and 8% by total volume of bitumen. Self-healing properties of asphalt mixtures have been measured using the three-point bending test before and after microwave healing at four different heating times: 10 s, 20 s, 30 s and 40 s, during ten cracking-healing cycles.

Finally, the main results of this research concluded that the heating time is the most influential variable on the healing level reached by the asphalt mixtures tested by microwave radiation, over the effect of the amount of fibres. Besides, CT-Scan results proved that fibre spatial distribution into the asphalt mixtures play an important role in the asphalt healing level.

Furthermore, it was concluded that the optimum microwave heating time is 40 s, to reach the highest healing levels with the lowest damage on the asphalt. In short, the study of the crack-sizes revealed that heating times over 30 s can also seal the cracks, thus achieving the self-healing of asphalt mixtures via microwave heating.

2. Materials and Methods

2.1. Materials

A standard IV-A-12 dense asphalt mixture was used in this research, since it is the mainly used in asphalt pavement building in Chile [1]. Table 1 shows the composition and specifications of the materials that form the asphalt mixture used. Fluvial type aggregates were used, and they were classified in three fractions: coarse aggregate or gravel (size between 5 and 12.5 mm), fine aggregates or sand (size between 0.08 and 2.5 mm), and filler (size < 0.08 mm). Bitumen used was a CA24 type, according to Chilean Specifications. The penetration grade of the CA24 bitumen used for mixture preparation was 80/100 mm at 25 °C. In addition, metallic fibres made from low-carbon steel were added to the asphalt mixtures. The length of the fibres was measured using an optical microscope, as the average value of 120 fibres randomly selected from the total sample, and the diameter was measured in 10 fibres using a digital calibrated micrometre. Resulting in an average diameter of 0.157 mm with an average aspect ratio of 30 and an initial length in the range of 2–8 mm.

Finally, four different percentages of fibres were added to the asphalt mixtures: 2%, 4%, 6% and 8%, by total volume of bitumen (see Table 1). Hence, 5 different types of asphalt mixtures were manufactured: 1 reference mixture without fibres and 4 asphalt mixtures with fibres, using the same aggregates gradation and bitumen content but varying the amount of fibres added to the mixture.

Table 1. Composition of the asphalt mixtures used in the study.

	Sieve Size (mm)	**Cumulative Mass (% Retained)**	**Mass (g)**	**Density (g/cm^3)**
Aggregates	12.5	16	176	2.779
	10	29	143	
	5	53	264	
	2.5	69	176	2.721
	0.63	86	187	
	0.315	90	44	
	0.16	93	33	
	0.08	95	19	
	<0.08	100	58	2.814
Bitumen	**Type**	**Amount (%mass/Aggr.)**	**Mass (g)**	**Density (g/cm^3)**
	CA24	5.3	58.3	1.039
Steel Wool Fibres	**Amount (% vol./Bitumen)**	**Length Range/ Avg. Diameter (mm)**	**Mass (g)**	**Density (g/cm^3)**
	2	2–8/0.157	8	7.180
	4		16	
	6		24	
	8		32	

2.2. Test Specimens Preparation

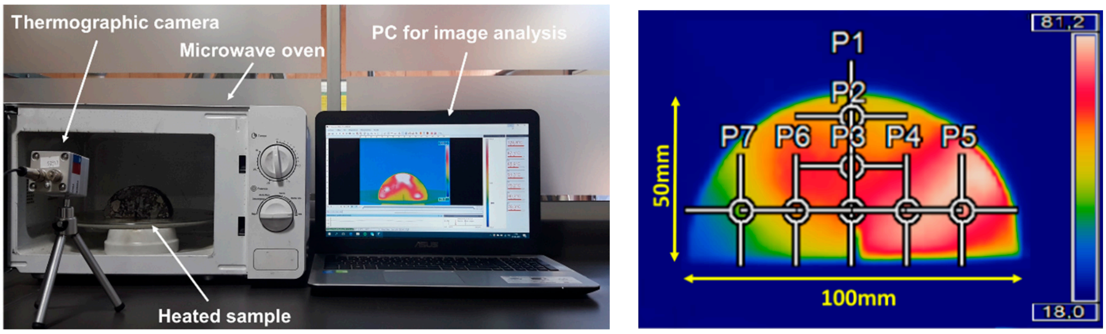
A total of 40 Marshall specimens were manufactured using the following procedure:

- Before the mixing process, the aggregates were heated during 24 h at 150 °C, while the bitumen and the fibres were heated at 150 °C during 2 h.
- Raw materials were added to the mixer in a specific order: bitumen and fibres, coarse aggregate, fine aggregate and at last, filler.
- Raw materials were mixed in a metallic bowl during approximately 3.5 min at a speed of 100 rpm and keeping a constant temperature of 150 °C.
- After mixing, asphalt mixtures were poured into a previously heated Marshall mould (10 cm diameter, 6 cm height) and compacted using a Marshall hammer applying 75 blows on each face.
- Marshall specimens were left to cool at ambient temperature during 24 h and then extracted from the mould.

After manufacturing, bulk density and air void content of all Marshall specimens were determined. Finally, each Marshall specimen was sawed into 4 semi-circular samples obtaining 160 semi-circular samples in total (with dimensions 10 cm diameter, 5 cm height and 2.5 cm thickness), 80 of which were used in the heating tests, while the other 80 were used in the crack-healing cycles. In both cases, 4 samples for each mixture type and heating time were tested.

2.3. Asphalt Heating by Microwave Radiation

With the aim of knowing the temperatures reached by the asphalt mixtures, and their heating-cooling behaviour when submitted to microwave radiation, semi-circular specimens of asphalt mixture with and without fibres were heated in a microwave oven during four different heating times (10 s, 20 s, 30 s and 40 s). All the heating tests were carried out using a microwave oven with 700 W power, and a working frequency of 2.45 GHz that corresponds to a wavelength of 120 mm [20]. In total, 80 samples were tested, starting at approximately 20 °C (see Figure 1a). The temperature variation on the surface of the samples was measured using a thermographic camera PCI PI160 connected to a computer. A real-time video record of the surface temperature was obtained with PI Connect software. The video register started just after the heating ended (and the microwave door was opened, see Figure 1a), and its duration was variable depending on the cooling time (required to reach the initial temperature of 20 °C) of each asphalt sample. Additionally, the surface temperature on 7 points distributed on the front surface of the samples was also real-time measured. Thus, the maximum surface temperature was calculated as the average value of all the measured points of the sample in the moment when the highest temperatures were registered (see Figure 1b).

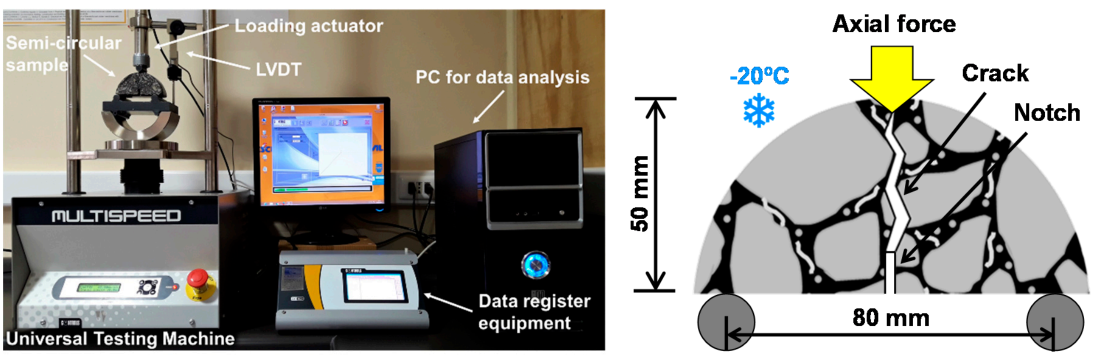


(a) (b)

Figure 1. (**a**) Microwave heating test; and (**b**) surface temperature measurement points, example for an asphalt mixture with 4% of fibres heated during 30 s.

2.4. Flexural Strength of the Test Samples

Flexural strength of asphalt mixtures was measured in semi-circular samples using the three-point bending test (see Figure 2). With the purpose of locating the initial point of cracking, a notch of 4 mm thickness and 10 mm height was made centered in the bottom side of the samples, and oriented in the load direction (see Figure 2b). Moreover, all the samples were pre-conditioned at a temperature of −20 °C during 24 h before testing, to ensure that a brittle fracture was produced.



(a) (b)

Figure 2. (**a**) Three-point bending test on asphalt mixture semi-circular samples; and (**b**) schematic representation of the broken test sample.

To do the flexural tests, the semi-circular samples at −20 °C were placed on two cylindrical supports separated 80 mm, and then a vertical load at a speed of 0.5 mm/min was applied centered on the top part of the samples using a loading actuator (see Figure 2a,b). The equipment used was a CONTROLS Multispeed static universal testing machine, with a load cell of 50 kN and controlled by computer (see Figure 2a).

2.5. Self-Healing Level of Asphalt Mixtures by Microwave Heating

The evaluation of the self-healing level of asphalt mixtures with fibres using microwave heating has been carried out according to the following methodology [17]: first, each sample was submitted to a three-point bending test (see Section 2.4) at −20 °C, until it breaks in two parts. After this, the broken samples were conditioned during 2 h at a temperature of 20 °C, until the surface moisture due to defrosting was completely dried. Then, the two parts of the sample were put together and heated using microwave radiation during four different heating times (10 s, 20 s, 30 s and 40 s). After heating, the samples were left to cool at ambient temperature to later be conditioned at a temperature of −20 °C during 24 h and submitted again to the three-point bending test, thus completing a cracking-healing cycle. Hence, the healing level reached by each tested sample (*HL*) can be defined as the quotient between the maximum force resisted by the sample initially tested (F_i) and the maximum force resisted by the sample after the healing process (F_r).

$$HL = \frac{F_r}{F_i} \quad (1)$$

Finally, to quantify the efficiency of the healing methodology, 10 cracking-healing cycles were carried out for each asphalt sample.

2.6. X-ray Computed Microtomography

With the aim of evaluating the spatial distribution of the fibres inside the asphalt mixture specimens, X-ray micro computed tomography was employed. With this purpose, cylindrical samples of approximately 25 mm in diameter and 12 mm in height were cut from sections of semi-circular asphalt samples. The X-ray micro-tomography scans were obtained using a Bruker SkyScan 1272 scanner. The scanner was operated at 100 kV and 100 µA and images were reconstructed at a spatial resolution of 26.8 µm (voxel side). Thus, classification of voxels as phases present in the mixture (i.e., voids, bitumen, aggregate and fibres) was achieved by segmenting based on voxel intensity values, which are proportional to average density of the specimen within the volume mapped by each voxel. Finally, reconstructions of the spatial distribution of the fibres were prepared by segmenting the materials found in a specific volume, based on simple thresholding [26]. The software used for the reconstruction of the CT scans images was ImageJ®.

2.7. Morphological Study of the Cracks before and after Healing

To qualitatively evaluate the effect of the heating time on the healing level of the asphalt mixtures, the width of cracks has been measured by taking photographs of a square area with sides of approximately 1 cm (see Figure 3), under an optical microscope Leica EZ4 with 35× magnification. After the flexural test, the crack width of all samples was calculated as the average value of four measurements equidistantly distributed along the image. In addition, the crack width was also measured after healing by microwave heating at different times in all the tested samples, using the same procedure. The software used for the crack-size measurements by optical images was ImageJ®.

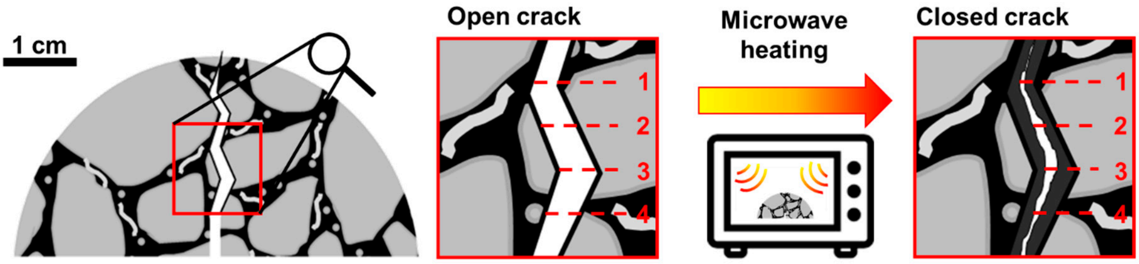


Figure 3. Schematic diagram of the measurement of the crack size before and after microwave heating.

3. Results and Discussion

3.1. Influence of Heating Time on the Thermal Behaviour of Asphalt Mixtures with Fibres

Table 2 shows the average results of the heating-cooling test of asphalt mixtures. The values presented in this Table are: T_{max}, maximum surface temperature reached by the samples; and CT, cooling time until the samples reached the temperature before heating (20 °C).

Regarding to heating of asphalt mixtures, it can be observed that the surface temperature of the samples (T_{max} in Table 2) increased with the increase of the microwave heating time, and that this temperature was higher in samples with higher fibres contents. Thus, samples heated during 40 s registered the highest temperatures, followed by samples heated during 30 s, 20 s and 10 s, respectively. Additionally, it can be seen that asphalt mixtures with 6% of fibres registered the highest surface temperatures, followed by mixtures with 4%, 8%, 2% and 0%, in that order (reaching surface temperatures of 98.86 °C, 87.41 °C, 78.71 °C, 59.76 °C and 50.46 °C, respectively) when they were heated during 40 s. Also, a similar trend was observed with the other three heating times.

Moreover, using short heating times (10 and 20 s), asphalt mixtures with different contents of fibres presented similar temperature increases (see Table 2). For example, samples with 0% and 8% of fibres registered temperature increases of 7.91 °C and 10.33 °C respectively, when they were heated during 10 s. However, in the case of using long heating times (30 and 40 s), the asphalt mixtures with different contents of fibres registered big differences regarding to their temperature increase, with the same microwave heating time. Specifically, samples with 2% and 6% of fibres presented an increase on their surface temperature of 39.76 °C and 78.86 °C respectively, when they were heated during 40 s. This reveals that 10 s and 20 s are too short heating times to induce a significant heating of the fibres that can distribute the temperature through the asphalt mixture and melt the bitumen, resulting in those similar temperature increases in the samples regardless their fibre content.

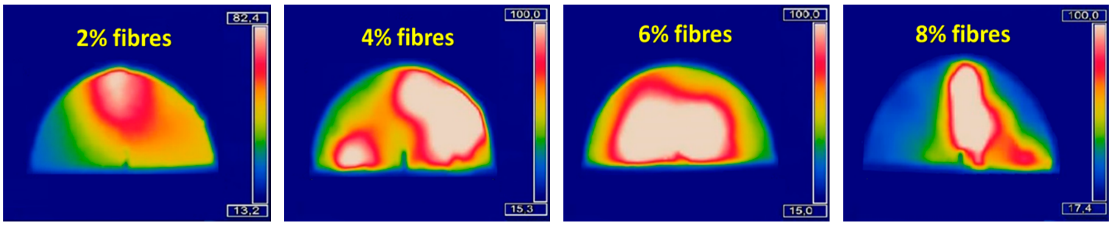
Therefore, it does not seem likely that asphalt mixtures can heal their cracks using heating times of 10 s and 20 s, due to the fact that the bitumen did not reach the temperature threshold required to reduce its viscosity and flow through the cracks opened in the pavement, sealing it [21].

Table 2. Average results of the heating-cooling test on asphalt mixtures.

Fibres Content (% vol. Bitumen)	Microwave Heating Time (s)							
	10		20		30		40	
	T_{max} (°C)	CT (min)	T_{max} (°C)	CT (min)	T_{max} (°C)	CT (min)	T_{max} (°C)	CT (min)
2%	28.11	55	40.76	60	47.33	63	59.76	56
4%	28.13	55	45.56	81	64.90	85	87.41	54
6%	29.19	63	55.71	90	65.30	87	98.86	63
8%	30.33	65	47.19	88	61.93	81	78.71	92
Ref. 0%	27.91	48	40.08	22	45.26	52	50.46	44

Furthermore, in view of the results presented in Table 2, it can be concluded that asphalt mixtures with higher contents of fibres reached, in general, the highest surface temperatures. Nevertheless, in the case of the samples with 8% of fibres, they did not register the highest surface temperature values. This was attributed to the bad distribution of the fibres inside the asphalt mixtures with 8% of fibres, which generates located heating in certain zones of the samples with presence of fibre clusters, see Figures 4d and 5d, respectively. Then, the located heating is distributed to the rest of the sample, as it can be observed in the sample with 2% of fibres, see Figure 4a. Thus, samples with 6% (Figure 4c), and 4% (Figure 4b) of fibres reached the highest surface temperatures (Table 2) for the heating time of 40 s, since they present a better distribution of the fibres in the asphalt mixture matrix. This result can also be seen in the CT-Scan images shown in Figure 5a–d.

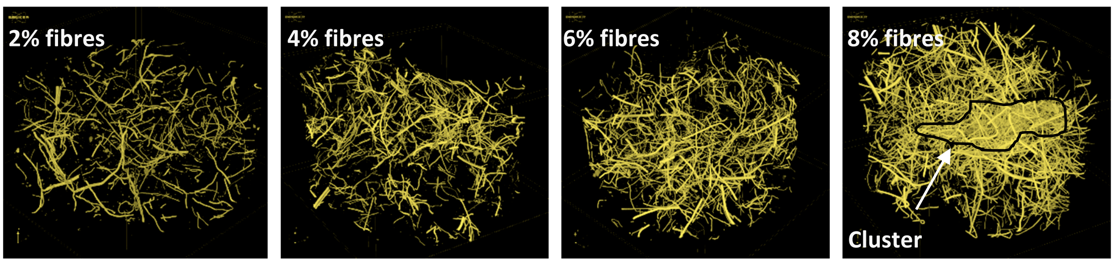
Additionally, Table 3 presents the results of bulk density and air voids content of the asphalt mixtures with different fibres content. As it can be observed, bulk density slightly decreased with the increase of fibres, while the air voids content increased with the increase of the amount of fibres added to the mixture. Therefore, the heat distribution in the samples with a high content of air voids is not the optimum (see Figure 4d), which is attributed to the heat dissipation through the air voids inside these asphalt mixtures.



(**a**) (**b**) (**c**) (**d**)

Figure 4. Thermographic images of asphalt mixtures with different fibre content after 40 s of microwave heating: mixtures with (**a**) 2%; (**b**) 4%; (**c**) 6%;(**d**) 8% of steel wool fibres.

Conversely, regarding the cooling results shown in Table 2, it can be observed that in general the longest cooling times were recorded after the heating times of 20 s and 30 s. In contrast, the shortest cooling times were registered after 10 s and 40 s of heating. Additionally, these results are related with the temperatures reached by the samples. Thus, samples without fibres reached lower temperatures and consequently needed a shorter time to cool down, while samples with 6% and 8% of fibres reached the highest surface temperatures and took longer times to cool down.



(**a**) (**b**) (**c**) (**d**)

Figure 5. CT-scan 3D reconstructions of fibre spatial distribution inside asphalt mixture samples with different fibre contents: (**a**) 2%; (**b**) 4%; (**c**) 6%; (**d**) 8% of steel wool fibres. Bounding box dimensions are 25 × 25 × 12 mm, approximately.

Finally, it can be deduced from Table 2 that, from a practical point of view, the best solution is to heat the samples with 4% or 6% of fibres during 40 s. This is because these samples presented the highest surface temperature increases and, also, they last 54 min and 63 min to cool down, respectively. These times are in the range of the cooling times obtained with a heating of 10 s. Besides, regarding the application of these asphalt mixtures in road pavements, they could be microwave heated during 40 s, reaching surface temperatures over 85 °C, implying that the traffic should be stopped during approximately 1 h to heal the cracks and return to the service temperature.

Table 3. Bulk density and air void content of asphalt mixtures (Average ± Stand. Deviation).

Fibres Content (% vol. Bitumen)	Bulk Density (g/cm^3)	Air Voids Content (%)
2%	2.33 ± 0.02	7.45 ± 0.98
4%	2.32 ± 0.02	8.70 ± 0.98
6%	2.31 ± 0.02	9.64 ± 0.92
8%	2.28 ± 0.03	11.43 ± 0.97
Ref. 0%	2.35 ± 0.02	6.70 ± 0.85

3.2. Influence of Fibres and Heating Time on the Asphalt Self-Healing Properties

Figure 6a–d shows the results of the healing levels reached by the asphalt mixtures with fibres depending on the number of healing cycles, using microwave heating at 4 different times: 10 s, 20 s, 30 s and 40 s. In these Figures, it can be observed that microwave heating helps to the partial resistance recovery of cracked asphalt mixtures with fibres. In addition, it can be observed that the healing levels reached by the mixtures increased with the increase of the microwave heating time. Thus, the highest healing levels reached by the samples are the obtained using 40 s as the heating time, followed by those obtained using 30 s, 20 s and 10 s, respectively (see Figure 6). This was produced because the longer the heating time, the higher the temperature reached by the samples (see Table 2), which allowed to reach higher healing levels.

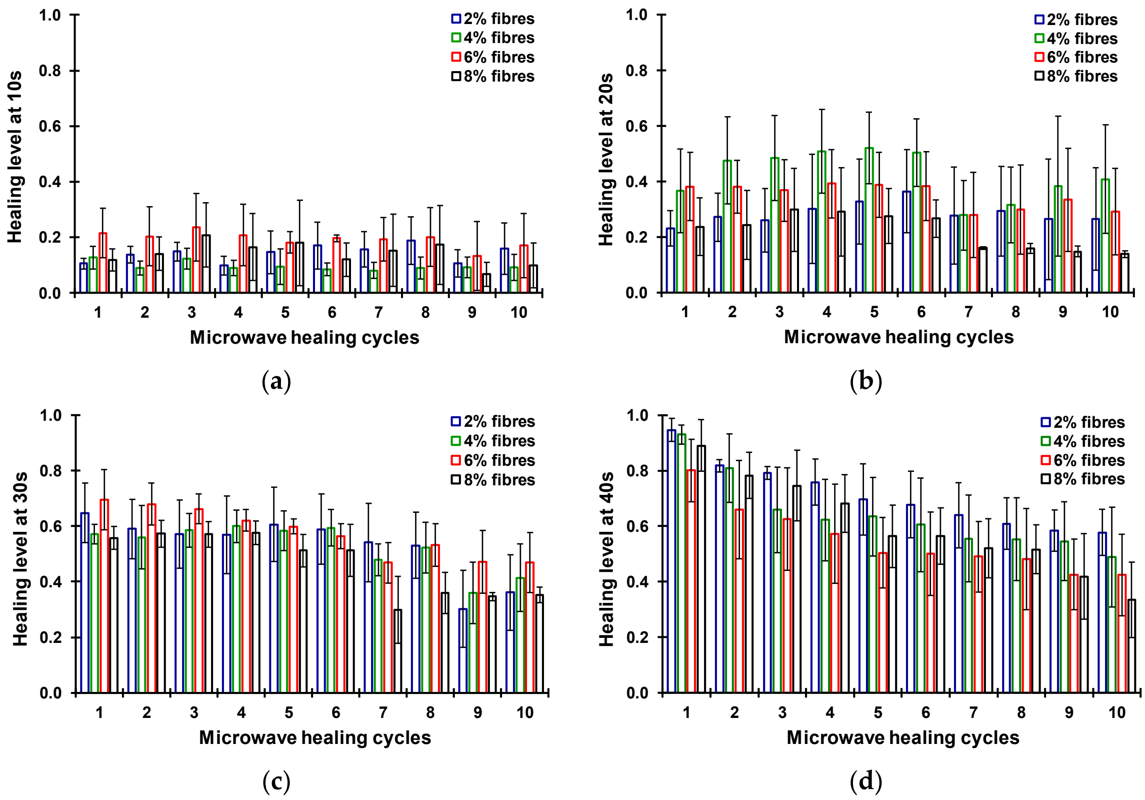


Figure 6. Healing levels of asphalt mixtures with different content of fibres, depending on the number of healing cycles, heated in a microwave at 4 different times: (**a**) 10 s; (**b**) 20 s; (**c**) 30 s and (**d**) 40 s.

Additionally, it can be seen that in the cases of short heating times (10 and 20 s), the number of healing cycles has not a significant influence on the healing level of the samples. For example, in the case of 20 s heating times (see Figure 6b), samples with 6% of fibres reached healing levels of 38% and 29% after the first and tenth cycles, respectively. In contrast, with longer heating times (30 and 40 s), it can be observed that the healing levels reduced with the increase of the number of healing cycles. In the case of samples with 4% of fibres heated 40 s (see Figure 6d), healing levels of 93% and 49% are reached after the first and tenth healing cycles, respectively. The variation can be attributed to the high temperatures reached by the asphalt samples when heated for long times (see Table 2).

This behaviour was also observed in previous researches [17] that demonstrated that microwave heating can change the internal structure of asphalt mixtures, modifying the aggregates position inside the samples and thus changing the air void content distribution of the mixtures. In this way, the mechanical resistance of asphalt mixtures and consequently their healing levels are reduced after each cycle. In addition, using these heating times (30 and 40 s), the bitumen can also be damaged in each cycle (see Figure 7).

Furthermore, in cases of long heating times it can be observed that healing levels also depend on the fibres content added to the asphalt mixtures. In general, mixtures with 6% and 2% of fibres registered the highest healing levels in the case of 30 s heating (see Figure 6c), followed by mixtures with 4% and 8% of fibres, respectively. Therefore, higher healing levels are not necessary attributed to higher mechanical resistance and higher fibres contents, as can be deduced from the obtained results of the maximum flexural force (force initially resisted by the samples before submitted to cracking-healing cycles) in Figure 8a. This result can be due to the temperature reached by the samples when heated during 30 s (see Table 2). Thus, the samples that reached high temperatures (6% of fibres) presented high healing levels (60% on average).

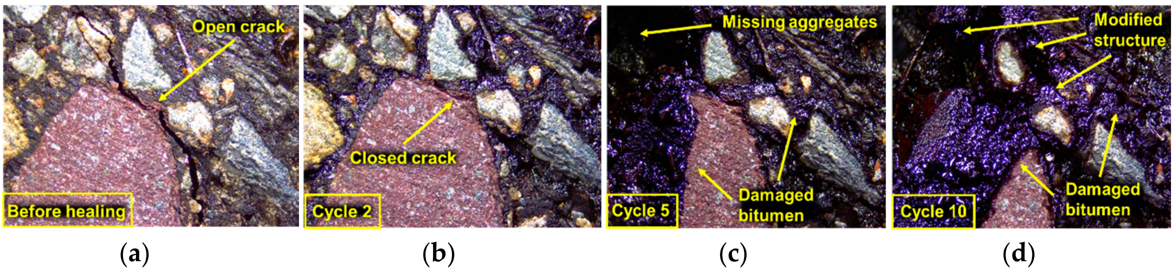


Figure 7. Optical microscope images of the crack produced on an asphalt sample with 4% of fibres, in different stages of the cyclic healing process: (**a**) before healing; (**b**) after the 2nd healing cycle; (**c**) after the 5th healing cycle; (**d**) after the 10th healing cycle. Image dimensions are 8 × 10 mm, approximately.

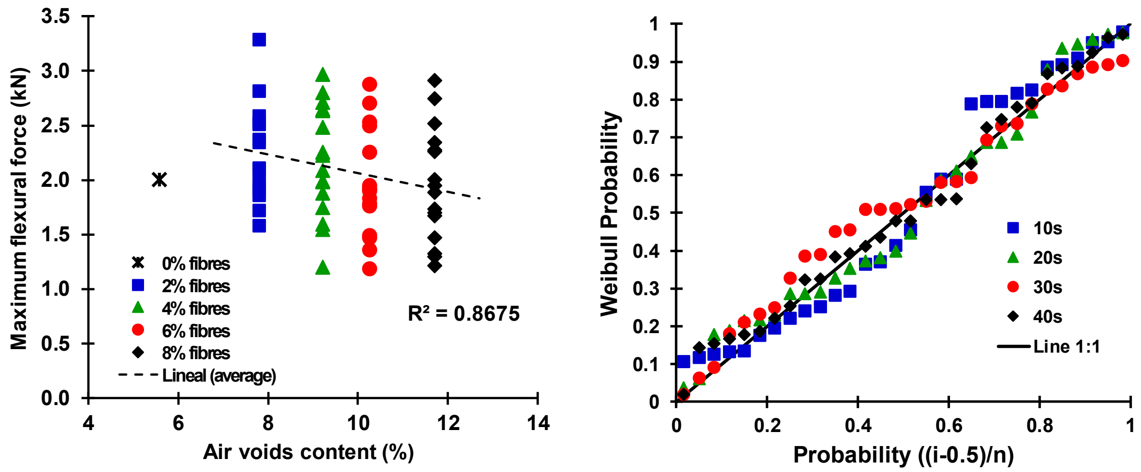
In addition, mixtures with 2% and 4% of fibres, registered similar values on their healing levels (55% on average), which can be related with the fact that their air void content and fibres distribution inside them are similar. Finally, mixtures with 8% of fibres presented the lowest healing levels (40% on average) after microwave heating during 30 s. This can be mainly attributed to the fact that higher fibres content produce higher air void contents in the mixtures, that consequently produced a decrease on their flexural resistance (see Figure 8a), and an increase on the formation of fibre clusters (see Figures 4d and 5d), which means that the use of asphalt mixtures with elevated contents of fibres is not recommended.

Moreover, analysing the error bars obtained from the healing level results (see Figure 6), it can be observed that, regardless the healing cycle, the variability of the results obtained from samples with different fibres content was similar for all the heating times. This suggests that there is not a significant difference between healing levels of asphalt mixtures with different fibre contents. However, the influence of the fibres content on the healing levels reached by the asphalt mixtures has not been proved. Due to that, the Cycle Reduction Ratio (*CRR*) has been defined in this study with the purpose of evaluating if an increase on the fibres content has an effect on the healing level of asphalt mixtures, according to the following Equation:

$$CRR = \left[\frac{HL_i}{HL_{max}}\right]_n \tag{2}$$

where n is the studied healing cycle, HL_i is the healing level of an asphalt sample with i% of fibres and HL_{max} is the maximum healing level within the cycle n (considered as the average healing level obtained from the samples with the fibres content that reach the maximum value). This ratio represents the variation of the healing level of asphalt mixtures within a specific cycle, with different fibres contents.

With the aim of demonstrating that the difference between *CRR* values is due to the scatter of the results (see Figure 6), the Weibull probability of all *CRR* data has been calculated and presented versus the data percentiles in a probability-probability plot, see Figure 8b. This probability function was used considering that the cyclical process of cracking-healing fits to a fracture process of materials, idealized by means of a damage function of Weibull type [26]. Figure 8b represents all the *CRR* data regardless the healing cycle and fibres content, for all the studied heating times. The results show that *CRR* values are aligned to a straight line with a slope 1:1, thus fitting to the Weibull distribution function. This means that the differences between healing levels reached by the asphalt mixtures within a specific cycle can be attributed to statistical variations, which implies that the maximum healing levels in a cycle are not statistically significant and that these values are not produced by the influence of the fibres or the number of cycle. Consequently, it can be stated that the variable that has influence on the healing level is the heating time.



(**a**) (**b**)

Figure 8. (**a**) Maximum flexural force versus the air void content of asphalt mixtures with different fibres content; (**b**) Weibull probability-probability plot for all Cycle Reduction Ratio data.

Besides, the goodness of fit of the CRR data series to the Weibull distribution function can be observed in Figure 8b, for the different heating times. In this context, it can be stated that the better is the data fitting, the higher is the influence of heating time on the healing levels of asphalt mixtures. As can be seen, *CRR* data corresponding to 30 s and 40 s heating times fit better to the probability distribution function, while *CRR* data corresponding to 10 s and 20 s heating times are more dispersed, see Figure 8b. On the one hand, the worse fitting to the Weibull distribution function of *CRR* data corresponding to heating times of 10 s and 20 s is related with the high scatter and randomness of healing levels of asphalt mixtures heated during short times. On the other hand, in the case of asphalt samples heated during 30 s and 40 s, they have enough time to increase their temperature and distribute it. Based on these results, it is necessary to define an optimum heating time of asphalt mixtures via microwave radiation.

In this context, Figure 9 presents the healing levels of asphalt mixtures with 4% of fibres, at 7 different heating times (from 10 s to 100 s). In this Figure, the heating times of 60 s, 80 s and 100 s have been obtained from a previous work of Norambuena-Contreras and García [17]. As it can be seen in Figure 9 there are 2 possible optimum times, 40 s and 100 s, since they allow asphalt mixtures to obtain the highest healing levels. However, heating time of 100 s was discarded against 40 s due to the fact that heating asphalt mixtures by microwave during 100 s melted the bitumen, thus damaging the composite material. In addition, the effect of heating the samples during 100 s can be observed in Figure 9, as the reduction on the healing levels after each healing cycle. Therefore, it can be stated that using a 700 W microwave with a working frequency of 2.45 GHz, the optimum heating time is 40 s.

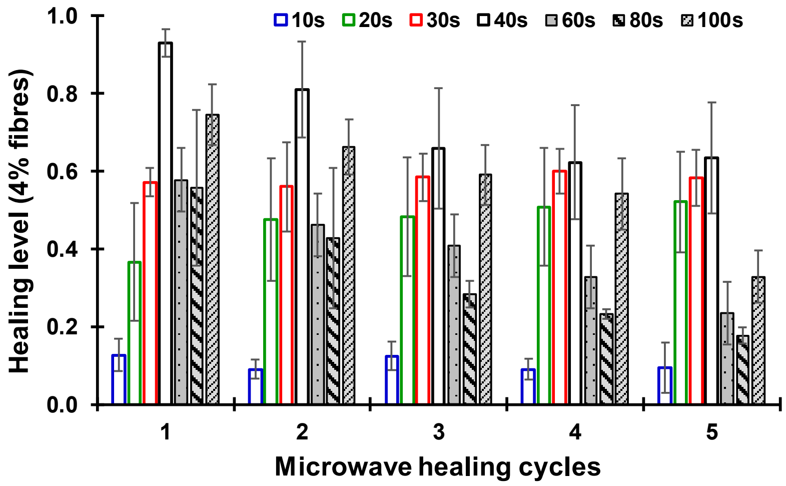


Figure 9. Healing levels of asphalt mixtures with 4% of fibres, depending on the heating time (includes results of healing level at 60 s, 80 s and 100 s from the previous research of Norambuena-Contreras and García [17]).

3.3. Crack-Size of Asphalt Samples Healed by Microwave Radiation

Figure 10a shows the frequency histogram for the average crack size measured using an optical microscope of all the semi-circular samples after the first three-point bending test. It can be seen that the average size of the open cracks before healing is 0.431 mm, and that the values ranged from 0.141 mm and 0.781 mm. Additionally, with the aim of knowing if the amount of fibres added to asphalt mixtures have an influence on the crack size, a probability analysis has been developed. In this way, Figure 10b presents the Normal probability-probability plot for all the crack size data obtained from the samples regardless their amount of fibres added to the mixtures. It can be seen that the relationship between the Normal probability and the data percentiles align in a straight line with a 1:1 slope, which means that it can be concluded that the amount of fibres added to the asphalt mixture has not a significant influence on the size of the cracks produced in the semi-circular samples by the three-point bending test.

Moreover, Figure 11a shows the box-plot graph of the crack size measured in all the samples before (shown as 0 s) and after healing by microwave heating at different times (10 s, 20 s, 30 s and 40 s), regardless the amount of fibres added to the mixtures. Observing this Figure, it can be concluded that the longer is the heating time, the smaller are the crack sizes measured on the samples. In addition, with shorter heating times (10 s and 20 s), the measured cracks presented more dispersed values, that can be related with the dispersion of the healing level results, see Figure 6a,b. In this way, crack sizes measured on the healed samples ranged from 0.035 mm to 0.38 mm, and from 0.032 mm to 0.206 mm, in the case of samples heated during 10 s and 20 s, respectively. While samples heated during 30 s and 40 s presented crack apertures ranged from 0.018 mm to 0.072 mm, and from 0.014 mm to 0.058 mm, respectively.

Furthermore, a crack has been considered healed when its aperture after healing is half of the minimum aperture measured before healing, which is under 0.07 mm. In view of that, it can be concluded that only the cracks of the samples heated during 30 s and 40 s were healed, which reaffirms the results obtained from the measurements of the self-healing levels of asphalt mixtures, see Figure 6c,d. In contrast, although samples heated during 10 s and 20 s can heal some of their cracks, they are more likely to obtain wide crack sizes, not being able to heal most of the open cracks and consequently losing a high percentage of the initial mechanical resistance, see Figure 11b.

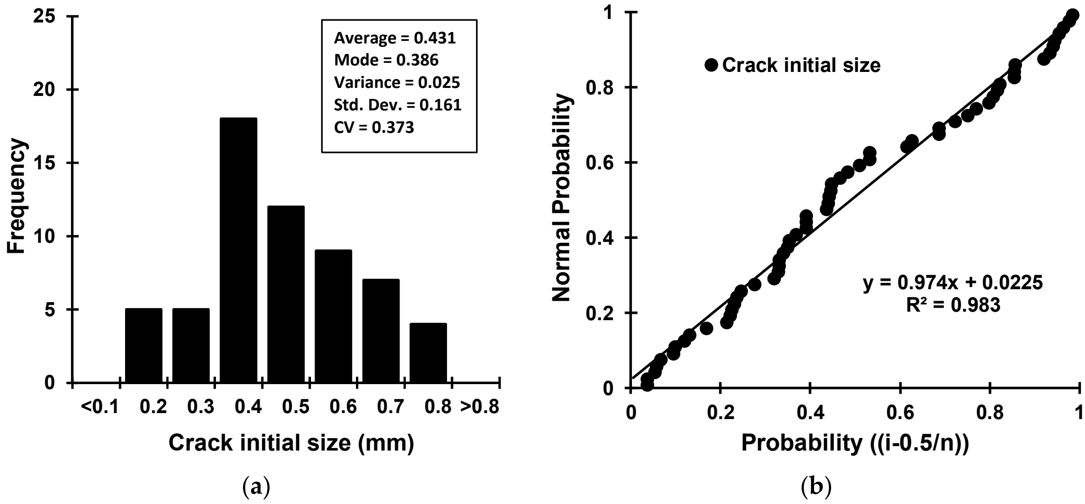


Figure 10. (**a**) Frequency histogram and statistical parameters of the crack initial size data; and (**b**) Normal probability-probability plot for the crack sizes.

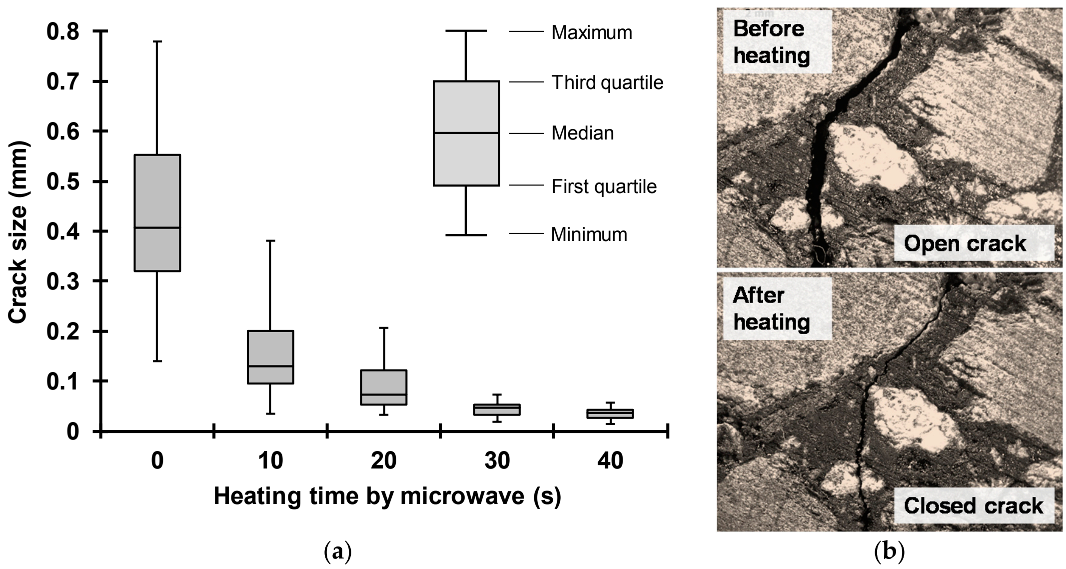


Figure 11. (**a**) Box plot representation of the crack size before healing (heating time = 0 s) and after healing of asphalt mixture samples at different heating times; (**b**) Optical images of a cracked asphalt sample before and after heating by microwave radiation at 40 s (Image dimensions are 8 × 10 mm, approximately).

4. Conclusions

This paper has studied the effect of the microwave heating time on the self-healing properties of asphalt mixtures containing metallic fibres. To do this, an experimental study including heating-cooling tests and cracking-healing cyclic tests was carried out on asphalt mixtures with four different contents of steel wool fibres, evaluating the (1) thermal behaviour under microwave heating; (2) self-healing properties at different heating times; and (3) crack-sizes in the asphalt mixtures before and after healing. The main conclusions of this article are summarised as follows:

- It was proved that the longer the microwave heating time and the higher the amount of fibres, the higher the reached temperatures. However, the influence of the metallic fibres amount on the asphalt mixtures heating occurs in the case of long times (30 s and 40 s).
- Additionally, it was found that asphalt mixtures with 6% of fibres reached the highest surface temperatures followed by mixtures with 4% of fibres, while mixtures with 8% of fibres presented difficulties during heating due to the bad distribution of the fibres, proved by CT scans analysis.
- In addition, cooling times after heating the asphalt mixtures during 40 s are in the range of those obtained with a heating of 10 s (between 54 min and 63 min). Consequently, the best solution is to heat the samples with 4% or 6% of fibres during 40 s, thus achieving that the application of these asphalt mixtures in road pavements should imply that they could be microwave heated during 40 s, implying that the traffic should be stopped for approximately 1 h to heal the cracks and return to the service temperature.
- Moreover, it was found that the healing level of the asphalt mixtures increased with the increase of the microwave heating time, due to the higher temperatures reached by the samples when heated longer times. However, using long heating times (30 s and 40 s), the mechanical resistance of asphalt mixtures and consequently their healing levels were reduced after each healing cycle.
- It was also found that the temperature achieved as a result of the heating time is the most influential variable on the self-healing properties of asphalt mixtures, while the fibres amount did not have a significant influence on the healing levels of asphalt mixtures.
- In addition, based in the results of this and previous research, it was concluded that the optimum microwave heating time is 40 s, since the highest healing levels are reached without an early damage of the bitumen.
- Finally, the probability analysis of the morphological study of the cracks revealed that the presence of fibres did not have influence on the obtained crack-size. Additionally, in view of the crack-sizes obtained with the different microwave heating times, it was demonstrated that the cracks can be sealed using long heating times (30 s and 40 s), consequently achieving the self-healing of asphalt mixtures via microwave heating.

The results contained in this paper are part of a research about self-healing asphalt mixtures including metallic waste materials that is being developed in Chile. The second part of the study, currently under development, is the evaluation of the effect of adding different metallic waste to asphalt mixtures, e.g., steel shavings, analysing their self-healing properties under microwave radiation.

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Conflicts of Interest: The authors declare no conflict of interest. The founding sponsors had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

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