



# Production of self-healing asphalt with steel short fibres and microwave heating: pilot study

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**ABSTRACT.** This pilot study aims to develop a self-healing asphalt through microwave heating, to reduce processing time, save energy and allow real commercial applications in a near future. Asphalt based composites were produced with different percentages of steel short fibers, obtained from the cutlery industry, to serve as a heat source for microwave radiation. Structural characteristics, morphology, and thermic behavior of the short steel fibers were characterized through X-ray Diffraction, X-ray Fluorescence, Scanning Electron Microscopy and Thermogravimetric Analysis, that was also used to verify the relation between mass variation and temperature of the asphalt-based composite. Temperature was monitored during micro wave heating to determine the heating rate of the composites with different percentage of fibers. Brookfield viscosity, penetration, ductility, softening point and density tests were carried out to characterize physical properties of the most representative asphalt-based composite. Results show the feasibility to use microwave heating in the production of the composite, indicating potential for future application as self-healing asphalt and pavement repair.

**Keywords:** waste; bitumen; characterization; cutlery; temperature.

## Produção de asfalto auto cicatrizante com fibras curtas de aço e aquecimento por micro-ondas: estudo piloto

**RESUMO.** Este estudo piloto visa desenvolver um asfalto auto cicatrizável através de aquecimento por micro-ondas, buscando reduzir tempo de processamento, economizar energia e permitir aplicações reais em um futuro próximo. Os compósitos foram produzidos com diferentes porcentagens de fibras curtas de aço obtidas da indústria da cutelaria, que foram introduzidas no asfalto com o objetivo de servir como fonte de calor, permitindo assim o aquecimento por micro-ondas. Características estruturais, morfologia e comportamento térmico das fibras curtas de aço foram obtidos por meio de difração de Raios-X, Fluorescência de Raios-X, Microscopia Eletrônica de Varredura (MEV) e Análise Térmica (TGA), que também foi utilizada para verificar a variação da massa do compósito com a temperatura. A temperatura do compósito foi monitorada durante o aquecimento para determinação da taxa de aquecimento em função da porcentagem de fibras. Ensaios foram realizados para determinar viscosidade Brookfield, penetração, ductilidade, ponto de amolecimento e densidade, permitindo assim a caracterização das propriedades físicas da amostra mais representativa do compósito. Os resultados mostram a viabilidade do aquecimento por micro-ondas para produção do compósito, indicando potencial para futuras aplicações como asfalto auto cicatrizável e reparo de pavimentos.

**Palavras-chave:** resíduo; betume; caracterização; cutelaria; temperatura.

### Introduction

Inclement weather and growing tire pressures, originated by increased traffic levels due to larger and heavier trucks, have aggravated the conditions upon the highway system, resulting in rutting, stripping, fatigue cracking and reflective cracking (Golestani, Nam, Nejad, & Fallah, 2015). Severe demands due to low traffic speed and frequent braking and acceleration in urban roads tend to accelerate pavement deterioration (Santos, Lucena,

Lucena, Silva, & Costa, 2015). In general, asphalt coatings in Brazil are designed for a service life of 10 years (Bernucci, Motta, Ceratti, & Soares, 2007) but frequently present premature damage. Structurally sound pavements are commonly repaired by means of crack sealing or filling, surface treatments such as fog seal, seal coat, double chip seal, slurry seal as well as micro surfacing, or even cold-mix asphalt, spray injection patching, and hot-mix asphalt, in case of pothole and patching repair (Johnson, 2000).

Asphalt pavement repair in Brazil often occurs in a rather rudimentary way. The aggregate is usually launched in irregularly shaped cavities, followed by a slight compaction. Such time and material wasting procedure does not ensure quality besides leading to fast pavement deterioration and security hazards.

Researches have concentrated efforts on the development of innovative materials and techniques for asphalt pavement repair, many of them related to crack healing (Bhasin, Little, Bommavaram, & Vasconcelos, 2008; Shan, Tan, Underwood, & Kim, 2010; Nan, 2015; Sangadji & Schlangen, 2013; Barrasa et al., 2014; Ayar, Moreno-Navarro, & Rubio-Gómez, 2016; Menozzi, Garcia, Partl, Tebaldi, & Schuetz, 2015; Zanko, Hopstock, & Derocher, 2016). On the other hand, several studies have demonstrated that micro-wave radiation may be applied for crack healing purposes (Bosisio, Spooner, & Granger, 1974; Osborne & Hutcheson, 1987; Sangadji & Schlangen, 2013; Norambuena-Contreras, Serpell, Valdés Vidal, González, & Schlangen, 2016; Zanko et al., 2016) as well as on the production and recycling of asphaltic pavements (Bosisio, et al., 1974; Osborne & Hutcheson, 1987; Norambuena-Contreras et al., 2016; Wang, Zhao, Yang, & Wang, 2011). Microwave industrial devices have already been designed especially for this application (Eliot, 2013). Besides being effective on the heating of asphalt mixtures, microwave heating is less time consuming and considerable reduces efforts required in terms of energy supply. In addition, microwave heating of asphalt may also reduce potential risks related to human health during its production cycle (Benedetto & Calvi, 2013). However, electrical and thermal conductivities of asphalt need to be modified to allow microwave heating, which can be achieved, for instance with the addition of a metallic material (Norambuena-Contreras et al., 2016). Thus, despite microwave heating has shown potential for the crack healing of asphalt mixtures, it has not been deeply explored (Benedetto & Calvi, 2013) and there is a lack of knowledge about the huge variety of metallic materials that could be used as asphalt matrix reinforcement.

A modified asphalt used during original pavement placement and heated by microwave actuates as a self-healing material, in other words, a material able to heal itself, which may result very reliable and durable asphalt pavements. In such case, the modified asphalt would be able to heal its opened cracks during the service life. A modified asphalt used during original placement and heated by microwave heating would also be convenient for

asphalt pavement recycling purposes. This technique may also be used for rapid patches or even crack healing of conventional asphalt pavements. Crack healing with microwave heating begins with the removal of loose debris and/or blow water from the crack and the placement of the modified asphalt. A microwave device placed over the cracks heats the modified asphalt leading to crack healing in a few minutes (Zanko et al., 2016). In the case of self-healing, the microwave device is placed directly over the existing pavement and there is no need to add modified asphalt before heating.

This paper reports results obtained through a pilot study on the development of a new asphalt based composite material reinforced with short steel fibres for microwave heating purposes, aiming at to improve the efficiency of the existing asphalt pavement repair techniques.

#### **The use of microwave radiation for asphalt mixtures heating**

The most common frequency used for microwave heating is 2.45GHz that corresponds to a microwave photon energy of 0.0016eV. Electron binding energies of H-OH and CH<sub>3</sub>-CH<sub>3</sub>, for instance, are 5.2 and 3.8eV, respectively (Krug, 2008). Thus, it is natural that the microwave energy is too low to break chemical bonds and then microwaves cannot induce chemical reactions (Kappe, 2004).

Bosisio et al. (1974) showed that it is possible to use microwave heating to seal road cracks. Field applications in Montreal and Quebec proved that microwave radiation with a frequency of 2.45 GHz could be effectively coupled into bituminous pavements to a depth of 12 cm without overheating the top layer of the asphalt road. Such frequency is usual in asphalt pavement microwave maintenance vehicles and microwave radiation heaters (Wang et al., 2011). A patent of Osborne and Hutcheson (1987) registered the potential of a modified asphalt composite with dispersed granular material, to allow microwaves absorption, as feasible for reconditioning and asphalt repair. Sangadji and Schlangen (2013) proposed the use of malten microcapsules as healing agent. Such capsules embedded in asphalt mixtures proved to increase the self-healing rates of asphalt concrete and thereby, the lifetime of the road. A pilot study conducted by Benedetto and Calvi (2013), on the reduction of worker health risks and environmental damages caused by the conventional heating of asphalt mixtures, proved the microwave heating potential of both aggregates and asphalt.

Microwave heating techniques have also been applied to estimate the initial asphalt curing time or even to improve mechanical properties of the matrix. The carbonyl iron powder, a magnetic, metallic and good microwave absorber material, may be used for this purpose, contributing to boost the energy efficiency of the system. Microwave heating of asphalt and carbonyl iron powder mixtures may improve aggregate adhesion as well as mechanical properties, such as Marshall stability and dynamic stability (Wang et al., 2011). On the other hand Zanko et al. (2016) showed that magnetite or magnetite-containing aggregate can enhance microwave absorption and therefore be used in asphalt mixtures for in-place pothole/pavement repair/recycling, with a high-power (50kW) vehicle based (truck-mounted) microwave system. In a recent study of Norambuena-Contreras et al. (2016) steel wool fibres were applied in asphalt mixtures with crack-healing purposes by microwave radiation. Despite being beneficial to increase the heating rates of the asphalt mixtures, steel wool fibers did not improve particle loss resistance, stiffness modulus and cracking resistance of asphalt mixtures.

## Material and methods

### Raw Materials Characterization

The short steel fibers are an industrial waste originated by the cutlery production process which main constituent is the carbon steel. In order to analyze structural characteristics, thermal behavior and morphology of the short steel fibers, the following techniques were employed: X-ray Diffraction, X-ray Fluorescence (XRF), Scanning Electron Microscopy (SEM)

and Thermogravimetry Analysis (TGA). X-ray diffraction patterns were obtained using a Shimadzu 6000 X-ray diffractometer, with Cu K $\alpha$  radiation ( $\lambda = 1.5418\text{\AA}$ ) and 30 kV / 30 mA (without graphite monochromator) in a range of 5-80° (2 $\theta$ ). Chemical analysis was realized in a Shimadzu EDX-720 Energy Dispersive X-ray Fluorescence Spectrometer with measuring range from Na (Z = 11) to U (Z = 92), Rh target X-ray generator, voltage 5 to 50kV, and a 10 mm collimator. The waste thermal behavior was analyzed by thermogravimetry (TG/DTG) using a SHIMADZU TGA-60 thermogravimetric analyzer. Topographic images were obtained through a SHIMADZU S550 Scanning Electron Microscope. It was possible to identify the short steel fibers shape and the morphology.

The asphalt cement CAP 50/70 (Penetration-Graded Asphalt) follows the requirements of the Brazilian National Agency of Petroleum, Natural Gas and Biofuels for asphalt pavement materials (Agência Nacional do Petróleo [ANP] 19, 2005). The asphalt cement thermal behavior was analyzed by thermogravimetry using a SHIMADZU TGA-60 thermogravimetric analyzer. Viscosity, penetration, density, softening point, and ductility were determined according to the recommendations of ASTM D4402-15 (2015), ASTM D5-13 (2013), ASTM D70-09 (2009), ASTM D36-14 (2014), and ASTM D113-07 (2007), respectively.

### Preparation of Asphalt Based Composite Specimens

The waste was first dried in a bench oven for 24h at 100°C. Then, it was refined in a 100 mesh sieve (0.149 mm) and weighed to compose the percentages (5, 10, 15, 20, 25, and 30%) to be added to each sample (Figure 1).



**Figure 1.** (a) Waste aspect after drying; (b) Waste refinement.

The bitumen was heated in a bench oven for 24h at 80°C and then it was kept in an oil bath at 60°C, avoiding direct contact with the flames and extreme temperature, which could cause bitumen oxidation, making it inappropriate for this application. Samples were manually mixed and named according to the amount of waste added to 100 g of bitumen, i. e., CAP50/70 0, CAP50/70 5, CAP50/70 10, CAP50/70 15, CAP50/70 20, CAP50/70 25 and CAP50/70 30. Mixtures were homogenized through 20 clockwise and 20 counterclockwise movements with the bitumen at 120°C. Mixtures were then placed in silicon molds able to handle up to 220°C.

### Asphalt Based Composite Characterization

The asphalt based composite was heated in a domestic microwave oven, with internal dimensions 18x20x20 cm, 2.45GHz and maximum 1.2KW power output. Samples were heated at the maximum microwave oven power output. The temperature change in the asphalt based composite samples was measured every 10s, up to a maximum of 60s, by a Skil-Tec SKTI-550 infrared thermometer with measuring range of -50 to 550°C. Measurements were performed at a distance of 10 cm from the samples surface with emissivity 0.95 and spectral response 8 to 14  $\mu\text{m}$ . Such measurements are not real time but are rapid enough to reduce thermal losses. Samples heating was also visually monitored, to avoid smoke and sparks inside the microwave oven. Temperature is a difficult parameter to measure in the microwave processing once conventional thermocouples are not suitable for it due to their metallic nature, as reported by Akbarnezhad, Kuang, and Ong (2011).

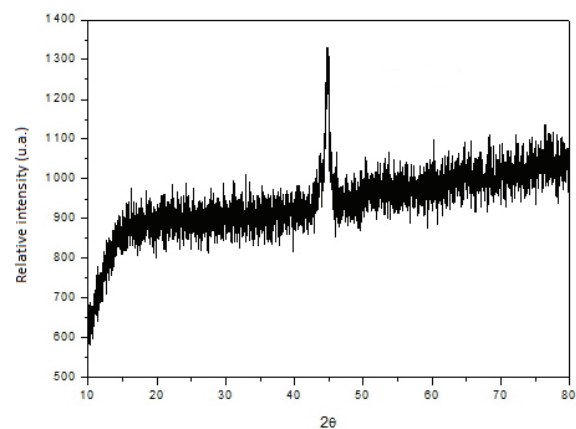
Resistance to flow test was performed in a Brookfield DVII+ viscometer with a Brookfield Thermosel System, according to ASTM D4402-15 (2015). The consistency of the asphalt based composite was determined through the penetration test, according to the recommendations of ASTM D5-13 (2013). Density, softening point and ductility of the asphalt based composite were determined according to ASTM D70-09 (2009), ASTM D36-14 (2014), and ASTM D113-07 (2007), respectively.

### Results and discussion

The quantitative chemical analysis obtained through X-ray fluorescence identified the following chemical elements in the waste: Fe (91.4%), Al (7.2%), Mn (0.66%), Cr (0.18%), and Si (0.56%). Fe is the major component due to the nature of the waste and the amount of Al is associated to the abrasive particles of the grinding wheel, which

consists essentially of  $\text{Al}_2\text{O}_3$ , as described in ISO 525-13 (2013). The mentioned chemical composition represents the whole amount of waste material provided the cutlery industry, which is composed of different types of carbon steel.

Figure 2 shows the XRD diffractogram of the waste, obtained through X-ray diffraction. The peak at about 45° refers to the (110) family of planes of Body-Centered Cubic (BCC 06-0696). Other peaks, including the ones of the abrasive, cannot be observed due to the high noise caused by the fluorescence of Fe, since the diffractometer uses  $\text{Cu K}\alpha$  radiation and there is no Graphite monochromator on the secondary optical system.



**Figure 2.** XRD diffractogram of the waste.

No mass loss was observed in the waste sample as well as in the asphalt based composite sample with 30% of waste up to 200°C, as shown in Figure 3. It leads to the conclusion that short steel fibres do not accelerate thermal degradation of the bitumen matrix in the temperature range for asphalt paving operations.

Figure 4 shows a mass gain at temperatures above 400°C, attributed to Fe oxidation. No mass variation can be observed up to 200°C (temperature range for asphalt heating).

In Figure 5 morphological characteristics of the waste can be observed. Due to the nature of the waste (abrasive wear caused by a grinding wheel during grinding operations), such fibres present variable diameter/length combinations. Fibres length ranges from a few tens of micrometers to millimeters. Fibres superficial morphology and the shear bands can be seen in Figure 5 (b).

The morphology of the short steel fibres, with irregularities, edges, and variable sizes and lengths, as showed in Figure 5, explains the fibres interlocking capacity and the tendency to form clusters. Such behaviour, also reported by



Norambuena-Contreras et al. (2016) in steel wool fibres may difficult the distribution of fibres within asphalt matrix, leading zones where fibres pack together forming dense clusters.

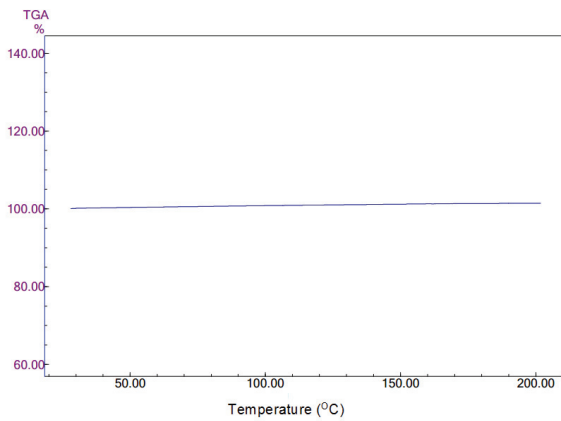
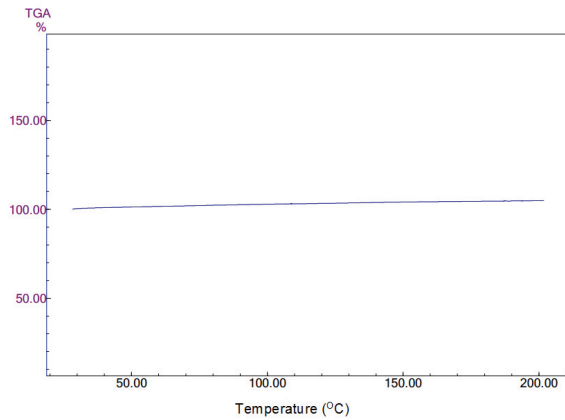


Figure 3. TGA: (a) CAP 50/70 0%; (b) CAP 50/70 30%.

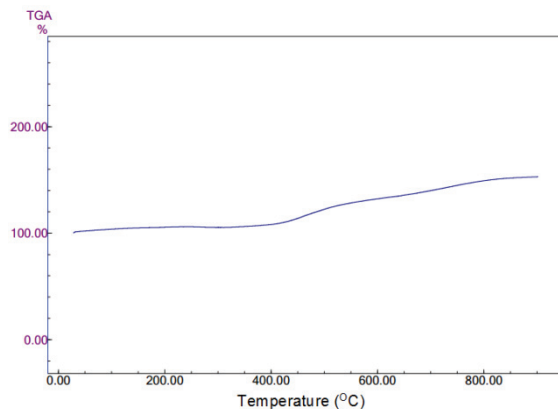


Figure 4. Thermogravimetric analysis of short steel fibres.

Figure 6 shows temperatures of the asphalt based composites obtained during microwave heating, indicating the maximum temperature registered during the testes. First measurements were made at ambient temperature (approximately 25°C) and samples were exposed to microwave radiation for

60s. Preliminary tests resulted smoke release after 60s, indicating bitumen thermal degradation.

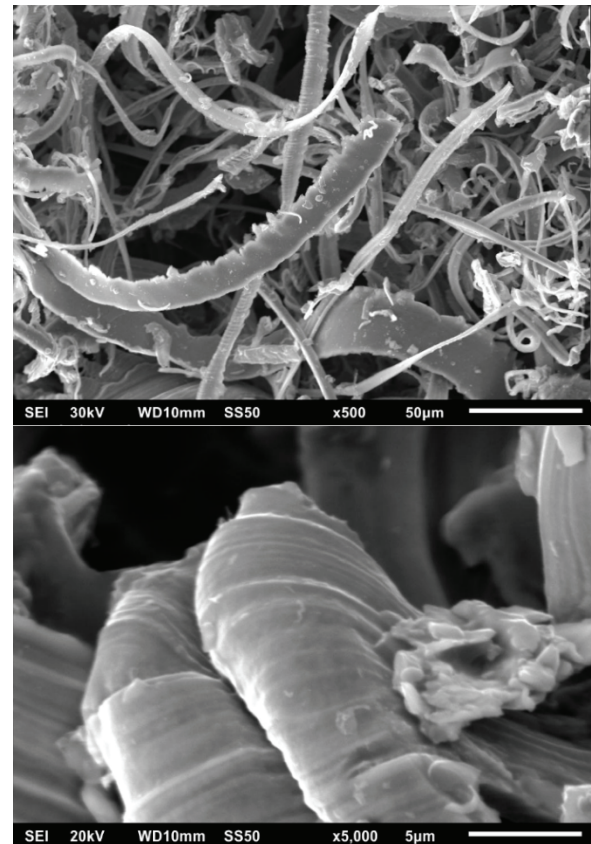


Figure 5. SEM micrograph of short steel fibres: (a) 100x; (b) 5.000x.

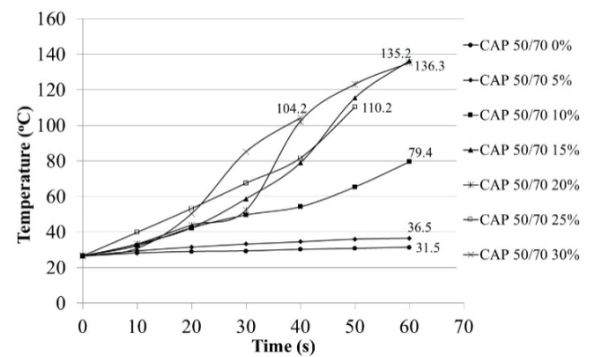
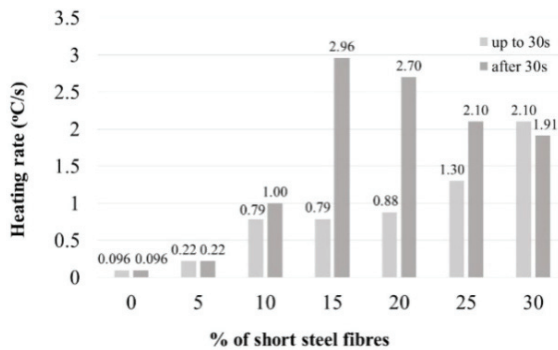


Figure 6. Heating curves of asphalt based composites.

It can be observed in Figure 6 that after short steel fibers addition, surface temperature of the different asphalt based composites distinctively increases. Composites with 15 and 20% of short steel fibres reached the highest temperatures. Heating of samples with 35 and 30% of short steel fibers was interrupted at 50 and 40s, respectively, to avoid bitumen deterioration, once smoke was detected inside the microwave oven.

Figure 7 shows samples heating rate, as a function of the percentage of short steel fibers and the exposure time to microwave radiation. Heating rate of samples with more than 10% of short steel fibres increased considerably after 30s of exposition to microwave radiation, reaching 2,96°C/s for CAP 50/70 15%, which is 30 times higher than that for ordinary asphalt. Heating rate increases the higher the fiber content, up to a level of 15%. Samples with more than 20% of fibres do not behave the same way and when 30% of fibers is used the heating rate decreases after the first 30s of microwave heating.



**Figure 7.** Heating rate of asphalt based composites.

Results show that the asphalt based composite is suitable for microwave heating purposes, once it can easily reach usual temperatures for asphalt pavement repair. However, it is clearly demonstrated that the use of more than 20% of steel short fibers is not suitable for microwave heating purposes due to deterioration of the asphalt after the first 30s of exposition.

Microwave heating results much more effective when compared to conventional heating. Preliminary tests indicated that 50 g of CAP 50/70 15% take 25 minutes to reach 135 at 100°C in a bench oven whereas this temperature is achieved in 60 seconds by microwave heating. Thus, the possibility of using microwave heating may contribute to reduce energy consumption as well as to contain most of the health risks related to the production cycle of asphalt and to eliminate toxic emissions or air pollution in the environment, as highlighted by Benedetto and Calvi (2013). Additionally, microwave heating is simple and may dispense with transportation of hot asphalt and its drawbacks. Due to the upper power limit of the domestic microwave oven used in this pilot study, which depends on microwave frequency and electromagnetic intensity (Wang et al., 2011), it was not possible to reach higher temperatures even with higher

exposure times. The use of an industrial microwave may overcome such limitation.

Results confirm the findings of previous studies with different materials mixed with bitumen for microwave heating purposes (Bosisio et al., 1974; Wang et al., 2011; Norambuena-Contreras et al., 2013; Zanko et al., 2016).

Additional tests were realized to characterize physical properties CAP 50/70 15% (Table 1), which was considered the most promising asphalt based composite regarding heating rate.

Values of softening point, penetration and ductility (12.5% higher, 25.92% lower, and 78.5% lower when compared to CAP 50/70) resulted from the stiffening effect of the short steel fibres. Brookfield viscosity of the asphalt based composite with 15% of short steel fibres is 131.57, 86.04, and 199.21% higher when compared to CAP 50/70 at 135, 150, and 177°C, respectively. Such results exceed the limits presented in the Brazilian Standard DNIT 095/2006 – EM (2006) that fixes the characteristics of petroleum asphalt cement for direct use in pavement construction: 5 to 7 mm for penetration, 46°C for softening point, 60 cm minimum for ductility and, 214, 97 and, 28 to 214 cP for Brookfield viscosity at 135, 150, and 177°C, respectively. However, results proved that the asphalt based composite allows microwave heating, which suggests that it could be tested when directly added in asphalt mixtures.

Under this overall perspective, it is necessary to carry out laboratory tests to verify the influence of the fibres on the physical and mechanical properties of asphalt mixtures on a scale compatible with the real one, which will allow confirming and improving the results of the present experimental study.

## Conclusion

Promising findings on the possibility of producing self-healing asphalt with waste from the cutlery industry to allow microwave heating were presented. Results contribute to the diffusion of microwave technology in road engineering applications and confirm several literature findings about the advantages of microwave heating compared to conventional heating.

**Table 1.** Results of physical tests.

	Brookfield Viscosity (cP)			Density (g cm <sup>-3</sup> )	Softening Point (°C)	Penetration (0.1 mm)	Ductility (cm)
	135°C	150°C	177°C				
CAP 50/70	475	322.5	127	1.012	48	54	100
CAP 50/70 15%	1100	600	380	1.127	54	40	21.5

Results of thermal analyzes showed that microwave heating does not result bitumen degradation. SEM images of short steel fibers showed the presence of irregularities and edges that allow the interlocking effect. Asphalt based composite with 15% of short steel fibers was considered the most representative and suitable for microwave heating purposes.

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