



Incorporation of synthetic macrofibres in Warm Mix Asphalt

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The reinforcement of asphalt concrete with fibres is not a common practice in the pavement field. Although some research papers refer to the use of microfibres, the behaviour of asphalt concrete incorporating macrofibres has not been studied. Warm Mix Asphalt (WMA) is environmentally friendly because it is possible to reduce emissions and fuel consumption. WMA technologies allow the reduction of mixing temperatures. The WMAs performances must be better than or similar to traditional Hot Mix Asphalts (HMAs). This work explores the benefits of incorporating synthetic macrofibres in WMA. Important improvements in rutting performance were observed when comparing to WMA without fibres, outperforming even HMA. Improvements in fracture behaviour at low to medium temperatures represent another benefit of macrofibre incorporation, confirming the advantages of the synergic combination of WMA and synthetic macrofibres.

Keywords: fibre reinforced asphalt concrete; Warm Mix Asphalt; synthetic macrofibres; fracture; rutting

1. Introduction

In the pavements field, fibres have not been extensively used in asphalt concretes. Although many researchers have reported improvements in the behaviour of Fibre Reinforced Asphalt Concretes (FRAC) including improved rutting, fatigue and cracking behaviour, most of them refer to reinforcement with microfibres (Chen & Xu, 2010; Guo, Li, Cheng, Jiao, & Xu, 2015; Kumar, Sikdar, Bose, & Chandra, 2004; Kutay, Gibson, & Youtcheff, 2008; Liu, Schlangen, van de Ven, van Bochove, & van Montfort, 2012; Mahrez & Karim, 2010; Mohammed & Hasan, 2016; Moreno Navarro, Sol-Sánchez, Tomás-Fortún, & Rubio-Gámez, 2016; Park, El-Tawil, Park, & Naaman, 2015; Qadir, Gazder, & Ali, 2018; Qian, Ma, Feng, Yang, & Huang, 2014; Sagnol, Quezada, Chazallon, & Stöckner, 2018; Serin, Morova, Saltan, & Terz, 2012; Wang, Wu, & Zhang, 2017; Xiong, Fang, Xu, Guan, & Liu, 2015; Yoo & Kim, 2014).

At present, at least to the authors' knowledge, no studies incorporating polymer macrofibres are available in the literature and even less for field applications. The difference between micro and macro fibres is related to the maximum aggregate size and the interaction inside the asphalt mixture. Microfibres modify mastic behaviour because their length is shorter than the maximum aggregate size. Meanwhile, the incorporation of macrofibres (length > 25 mm) can lead to improvements not only in rutting behaviour, but also in enhancing the fracture properties of asphalt mixtures when exposed to low temperatures. The macrofibres act as a bridge in the

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cracks and transfer the stress. Recent studies have verified these benefits in FRACs which incorporated glass macrofibres (Morea & Zerbino, 2018). Synthetic macrofibres could also improve the performance of the mixture, in particular the fracture behaviour. Nevertheless, it is evident that polymer macrofibres suffer degradation when exposed to Hot Mix Asphalts (HMA) mixing conditions.

On the other hand, Warm Mix Asphalt (WMA) was developed with the aim of minimising CO₂ emissions in the production and placement process of conventional HMA by reducing mixing and compaction temperatures as shown in the Federal Highway Administration's report (D'Angelo et al., 2008). Herein, it is mentioned that WMA not only allows minimising the amount of energy required, but also reduces emissions and odours and improves the welfare of workers. Other important beneficial aspects of WMA are its paving advantages, including the ability to pave in cooler temperatures, to haul the mix longer distances, to compact the mixture with less effort and the possibility to pave and open to traffic in a shorter period of time. Nevertheless, some problems in asphalt concrete performance were associated with the temperature reduction, such as worsened rutting behaviour and moisture damage. Su, Maekawa, and Hachiya (2009), when comparing their results to HMA, found lower rutting performances and increased moisture damage of WMA with a chemical synthetic wax incorporated into the mixture. Hurley and Prowell (2006) found increments in the potential rutting as mixing and compaction temperatures decrease.

A worse rutting performance of WMA prepared with conventional asphalt binders with respect to control HMA was found by Morea, Marcozzi, and Castaño (2012). Zelelew, Paugh, Corrigan, Belagutti, and Ramakrishnareddy (2013) concluded that both additive dosage rates and production temperatures affected the performance of WMA. Vargas-Nordbeck and Timm (2012) indicate that the use of WMA technologies tends to increase permanent deformations, while Luo, Zhang, Cheng, and Zhang (2017) remark that long-term mechanical properties of WMA are lower than HMA.

Regardless, the doubts about WMA rutting performance in the past were based on laboratory results. In a recent report, the National Cooperative Highway Research Programme studied and compared the long term field performance of WMA and HMA (NCHRP 843). This report concluded that pavements containing various WMA technologies exhibited long-term field performances comparable to those of the companion HMA pavement in terms of transverse cracking, wheel-path longitudinal cracking, and rutting. However, the rutting of WMA must be carefully studied in the laboratory during design to avoid problems in the field. For instance, in Argentinian standards, rutting tests are an obligatory step in the mixture's design process. The incorporation of fibres could help to improve the performance of mixtures in general, and WMA in particular.

Finally, and regarding the combination of WMA technologies and fibre reinforcement, Fazel, Samin, Pirmoun, and Dabiri (2016) incorporated polyolefin-aramid microfibres in WMA to improve rutting and fracture behaviour.

This experimental work analyses the potential advantages of the synergetic contribution of synthetic macrofibres with WMA technologies to improve asphalt pavement performance. The rutting behaviour and the fracture response at low temperatures are compared with similar tests performed on hot and warm asphalt concretes without fibres.

2. Experimental

The objective of this exploratory work was to study the improvements in WMA performance prepared with conventional asphalt binders by means of polymer macrofibres. The results

of wheel tracking tests (at 60°C), fracture tests of notched beams and indirect tensile tests at low temperatures (0°C and 10°C) were analysed in Fibre Reinforced Warm Mix Asphalt (FRWMA) and compared with those obtained from HMA and WMA without fibres as references. A volumetric and mechanical characterisation of each mixture from Marshall tests was also done.

2.1. Materials and mixtures

The study was developed from a common dense grade asphalt mixture. This was made using two fractions of coarse aggregates (6–20 mm and 6–12 mm), crushed sand (0–6 mm) and a conventional asphalt binder (CA30 Argentinian standard IRAM 6835; PG 64-16 of ASTM D6373). Table 1 shows the mixture proportions and the asphalt binder characteristics. This control mixture was designed following the Marshall method to obtain the optimum asphalt content of the mixture. This asphalt content was kept constant during the research with the aim of evaluating how the incorporation of fibres affects the mixture properties without including other variables. Nevertheless, as should be done with mixtures with fibres, a volumetric correction in gradation was done to consider their incorporation.

Three FRWMA (F2, F4, and F6) were prepared incorporating polypropylene fibre dosages of 0.2, 0.4 and 0.6% in weight of mixture; Figure 1 shows the fibres and Table 2 their main properties. As polypropylene fibres have a melting point near the temperature needed to work with traditional asphalt concrete, see Table 2, it is expected that fibres could be degraded in HMA. The use of warm mixture technology allows reducing the temperature while maintaining the mixture workability and compactability and prevents the degradation of fibres. A commercial tensoactive additive that came in pellets was incorporated into the asphalt binder in 1.6% by weight of binder to elaborate the WMA. The additive reduces the asphalt surface tension and decreases the contact angle with the aggregate acting as a lubricant to improve the workability of the mix. This was incorporated into the asphalts prior to preparing the mixtures. The asphalt was heated for two hours in an oven at temperatures that ensured a proper blend. Then, the additive was incorporated and blended by means of a pallet stirrer.

To prepare the mixtures, the fibres were mixed with the aggregates for a minimum of 30 s to enhance fibre dispersion and then the asphalt binder was added and mixed until the aggregates and fibres were coated. Figure 2 shows the distribution of macrofibres during the mixing process. A visual inspection of the mixture post mixing was done to ensure the integrity of the fibres.

Two mixtures without fibres (H and W) were prepared as references. Mixture H is conventional HMA elaborated at 160–150°C (mixing and compaction temperatures, respectively), while mixture W is a WMA elaborated at 130–120°C (mixing and compaction temperatures, respectively).

Table 1. Base asphalt concrete characteristics.

	Mixture proportions			
	Coarse aggregate 1 6–20 mm	Coarse aggregate 2 6–12 mm	Crushed sand 0–6 mm	Asphalt binder
Weight %	23.8	10.5	60.9	4.8
	Binder properties			
	Viscosity at 60°C [Pa.s]	Penetration [dmm]	Softening point [°C]	
CA-30	335	47	54.8	

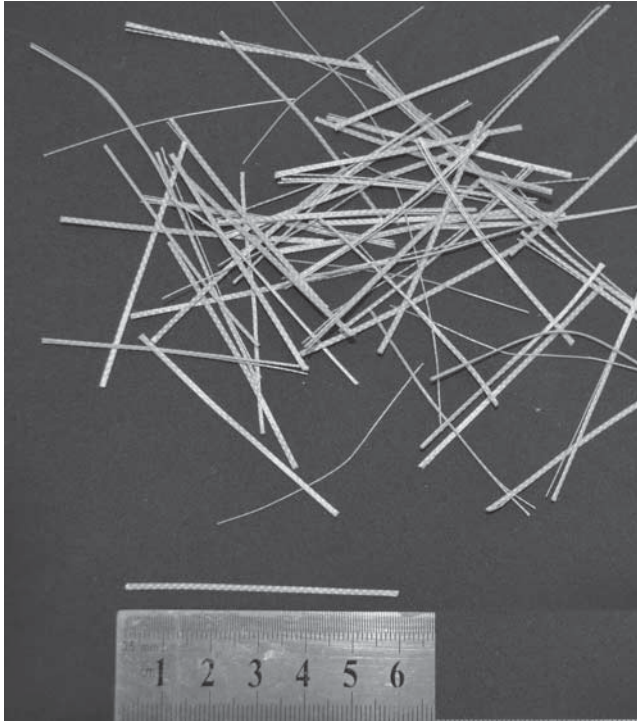


Figure 1. Synthetic macrofibres used.

Table 2. Fibre properties.

		Synthetic Fibre
Length	[mm]	54
Number of fibers per Kg	[-]	37,000
Surface texture	[-]	Continuously embossed
Density	[g/cm ³]	0.90–0.92
Tensile Strength	[MPa]	640
Modulus of elasticity	[GPa]	10
Melting point	[°C]	159–179
Ignition point	[°C]	> 450

Asphalt concrete slabs (300 × 300 × 50 mm) were cast to perform wheel tracking tests and notched beam bending tests as described in the next section. They were compacted with a roller compactor according to EN 12697-33. As well, Marshall specimens were done to evaluate indirect tensile strength at low temperatures, and also to compare volumetric and mechanical properties with those of the control mixtures (H and W).

2.2. Test procedures

2.2.1. Marshall test

Marshall Samples according to ASTM D6926 were done on all mixtures. The density, air voids, Stability and Flow were measured in accordance to ASTM D2726, ASTM D3203, and ASTM D6927.



Figure 2. FRAC mixing. Fibre incorporation and mixture aspect.

2.2.2. Wheel tracking test

The rutting performance was evaluated through the Wheel Tracking Test (WTT). The device is held in a chamber to maintain the sample at the test temperature required. The testing temperature was 60°C and at least two samples were tested for each mixture.

The test procedure was configured according to Argentina standard IRAM 6850 (similar to EN 12697-22 “small size device procedure B” standard). The rut depth was measured on the sample at one minute intervals through a LVDT. The collected data were used to obtain the curve of permanent deformation versus cycles and fitted with a potential model, Equation 1, in accordance with the Argentina standard.

$$D_n = a.n^b \quad (1)$$

where D_n : permanent deformation; n : wheel cycles; a and b : model constants.

The wheel tracking slope (WTS) and the proportional rut depth (PRD), Equations 2 and 3, respectively, were calculated as WTT results. WTS was calculated from a period that represents the shear resistance behaviour of mixture against rutting. This parameter is considered a better tool for characterisation of the rutting performance of mixtures. The PRD was used as a comparative parameter.

$$\text{WTS} = \frac{D_{10000} - D_{5000}}{5} \left[\frac{\text{mm}}{10^3 \text{ cycles}} \right] \quad (2)$$

$$\text{PRD} = \frac{D_{10000}}{h} 100[\%] \quad (3)$$

where D_{5000} and D_{10000} : permanent deformation at 5000 and 10,000 cycles, respectively; h : sample height.

If during the test a third flow is reached, the data must be fitted with another model, other than the potential model, and the WTS parameter cannot be calculated with Equation 2.

2.2.3. Notched beam bending test

Bending tests of notched beams are usually adopted to evaluate the fracture behaviour of different materials. In these experiences, beams of 50 × 75 × 300 mm were cut from slabs of

50 × 300 × 300 mm. The beams were notched at their centre; the depth of the notch (15 mm) was long enough to ensure adequate stress intensity at the notch tip to initiate a crack, but short enough to ensure a ligament adequate to prevent crack initiation under self-weight (Wagoner, Buttlar, & Paulino, 2005). A three-point bending load configuration was used and the test was controlled with a clip gage that registered the crack mouth opening displacement (CMOD). The CMOD was used as a control for managing the applied load to provide a stable post peak fracture. A constant CMOD rate of 0.9 mm/min was used. Tests were done at 0°C and 10°C with at least three samples for each temperature and mixture type. Figure 3 shows the scheme of the test setup and a sample after being tested. Also, in Figure 3 a typical stress-CMOD curve can be seen, obtained from the test. The main idea of the test was to observe the post peak fracture response of the material, mainly its residual stress capacity post peak tension that maintain the material. This is related to the capacity to resist fracture and still support loads. Meanwhile, as the residual stress is higher, so is the fracture resistance. As test results, the peak stress (f_p) and two residual stresses (f_1 and f_3) for CMODs of 1 and 3 mm were obtained. In addition, a toughness parameter (T_3) was calculated as the area under the stress-CMOD curve up to a CMOD limit of 3 mm.

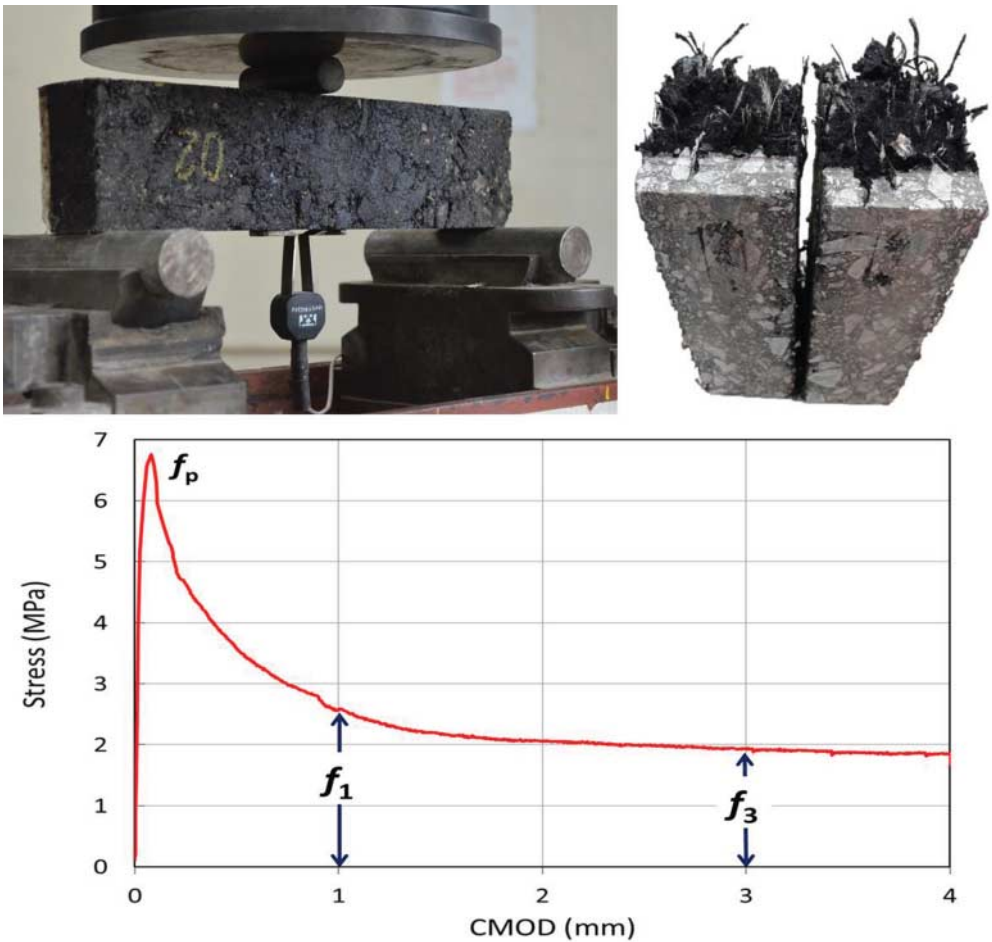


Figure 3. Bending test of notched beam. Left: test set up. Right: view of fracture surfaces after bending tests. Below: Typical test curve.

2.2.4. Indirect tensile strength test (ITS)

The indirect tensile strength (ITS) was also used to evaluate the fracture behaviour of the mixtures at low temperature. This test is capable of showing the effect of fibres during the fracture process of the mixture. The ITS was obtained by loading Marshall specimens across its vertical diametric plane with a loading rate of 50 mm/min according to ASTM D6931. Two test temperatures (0°C and 10°C) were evaluated. The loading caused a tensile deformation perpendicular to the loading direction, which yielded a tensile failure. The ITS can be calculated knowing the diameter and height dimensions of the test specimen. The ITS versus loading deformation curve was registered. Toughness was calculated as the area under the stress-loading deformation curve up to a strain limit of 3 mm. At least three samples for each mixture and temperature were tested.

3. Results

The main objective of this work was to explore the rutting and fracture performance of Fibre Reinforced Warm Mix Asphalts. The selected macrofibre used in these exploratory experiments are those commonly used to reinforce portland cement concretes, mainly for improvements in fracture behaviour. Because of this, it is important to note that the fibres were developed to optimise their geometry, material and adherence to maximise their efficiency in portland cement concretes. Despite this, the results shown herein are promising, as can be seen in the following lines. In summary, Marshall, rutting and fracture tests were performed on F2, F4 and F6 as well as on the control mixtures without fibres (H and W).

Table 3. Volumetric and mechanical parameters obtained from Marshall tests.

	Density [g/cm ³]	Air Voids [%]	Estability [kN]	Flow [mm]
H	2.428	4.4	17.9	4.2
W	2.407	5.0	15.3	3.7
F2	2.380	5.3	13.9	3.7
F4	2.356	5.8	12.4	5.0
F6	2.323	7.1	12.8	5.6

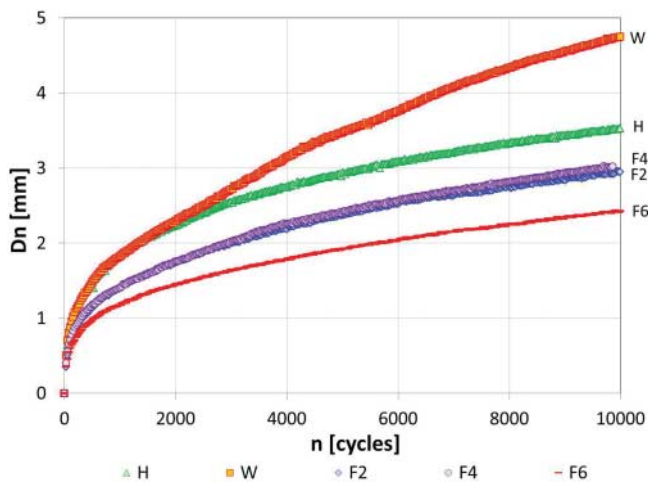


Figure 4. Permanent deformation versus cycles for FRAC with synthetic macrofibres.

Table 4. WTT result for FRAC with macrofibres.

	WTS (mm/10 ³ cycles)		PRD (%)		D ₁₀₀₀₀ (mm)	
	Mean	Min/Max	Mean	Min/Max	Mean	Min/Max
H	0.132	0.131 / 0.133	7.0	6.9 / 7.0	3.58	3.57 / 3.59
W	0.222	0.164 / 0.283	9.0	7.4 / 10.7	4.61	3.77 / 5.46
F2	0.119	0.115 / 0.122	5.7	5.7 / 5.8	2.96	2.95 / 2.97
F4	0.115	0.098 / 0.144	5.8	5.0 / 6.9	2.96	2.53 / 3.50
F6	0.091	0.082 / 0.101	4.7	4.5 / 4.8	2.39	2.32 / 2.47

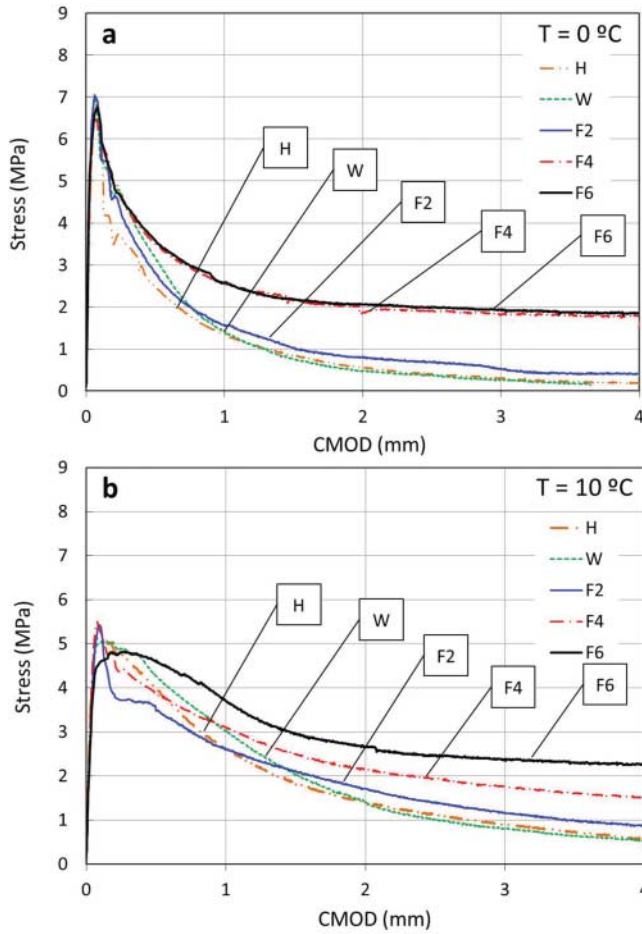


Figure 5. Stress versus CMOD curves of some tested samples in fracture bending tests. (a) T = 0°C, (b) T = 10°C.

Table 3 shows the mean values of density, Air Voids, Stability and Flow measured in the Marshall tests. It can be observed that the density of F2, F4, F6 and W were lower than H. In addition, the densities of FRWMA decrease as the dosage of fibres increases. Stabilities of FRWMA and W were lower than H, while flow values of F4 and F6 were slightly higher than H. This can be explained considering the lower densities reached as compared to the average H density.

Table 5. Stress and toughness parameters from bending tests at different temperatures.

		0°C				10°C			
		f_p	f_1	f_3	T_3	f_p	f_1	f_3	T_3
T_{test}		(MPa)	(MPa)	(MPa)	(J/m ²)	(MPa)	(MPa)	(MPa)	(J/m ²)
H	Mean	6.60	1.40	0.32	634.7	4.95	2.85	0.94	1033.7
	Min	6.30	1.34	0.29	603.4	5.10	3.03	0.82	994.6
	Max	6.90	1.45	0.35	665.9	4.80	2.91	1.04	1072.7
W	Mean	7.00	1.42	0.26	621.9	4.95	2.97	0.93	979.5
	Min	6.90	1.39	0.25	621.9	5.10	3.03	0.81	969.6
	Max	7.10	1.45	0.27	630.0	4.80	2.90	1.04	989.4
F2	Mean	6.95	1.42	0.60	657.3	5.05	2.61	1.06	965.4
	Min	6.90	1.28	0.53	650.9	5.40	2.59	1.16	962.4
	Max	7.00	1.56	0.67	663.7	4.70	2.62	0.96	968.3
F4	Mean	6.50	2.49	1.89	1066.0	5.15	3.24	1.85	1168.5
	Min	6.50	2.42	1.82	1016.6	5.50	3.10	1.77	1135.7
	Max	6.50	2.55	1.95	1115.4	4.80	3.38	1.93	1201.2
F6	Mean	7.05	2.50	1.77	1150.2	5.45	3.14	1.84	1196.1
	Min	6.80	2.41	1.41	1065.8	6.10	2.57	1.29	1059.4
	Max	7.30	2.58	1.85	1234.6	4.80	3.71	2.38	1332.8

Figure 4 shows the results of wheel tracking tests. It can be observed that mixture W shows the worst rutting performance. Table 4 presents the calculated parameters from the WTT. It can be seen that WTS of W double the value of H (indicating lower shear resistance). The incorporation of macrofibres, even in low contents, has a positive effect in reducing rutting and even leads to a better performance than H.

Figure 5 shows the stress versus CMOD curves of fracture tests performed on notched beams at 0°C and 10°C (Figure 5a and b, respectively). It is important to mention that each curve is representative of one of the tested samples for each type of mixture. In Figure 5a (0°C), it can be seen that F2 presents similar behaviour as H and W. However, F4 and F6 show better fracture behaviour with higher residual capacities after the peak stress and also greater load capacity at wider crack openings, indicating that a minimum fibre content is necessary to modify the fracture performance. A similar behaviour, but on a smaller scale, was observed in tests performed at 10°C, Figure 5b, where FRWMA shows higher residual capacity than H and W.

Table 5 presents the stress values (f_p , f_1 and f_3) and the toughness obtained from the bending tests. It can be seen that the main differences appear in tests performed at 0°C and in the residual stresses of the FRWMA with the highest fibre contents (F4, F6).

Figure 6 shows the stress versus loading strain curves of ITS tests performed at 0°C and 10°C (Figure 6a and b, respectively). It is important to mention that each curve is representative of one of the tested samples for each type of mixture. It can be observed that fibre mixtures present a better fracture behaviour with higher residual load capacity than control mixtures. Figure 7 shows a sample of F4 after the indirect tensile strength test; the macrofibres can be seen bridging both parts of the broken sample.

4. Discussion

This study demonstrates that the incorporation of polymer macrofibres improves the rutting behaviour of WMA. In the first place, the W mixture presents an important accumulation of permanent deformations with respect to H, see Figure 4. It can be seen that temperature reduction

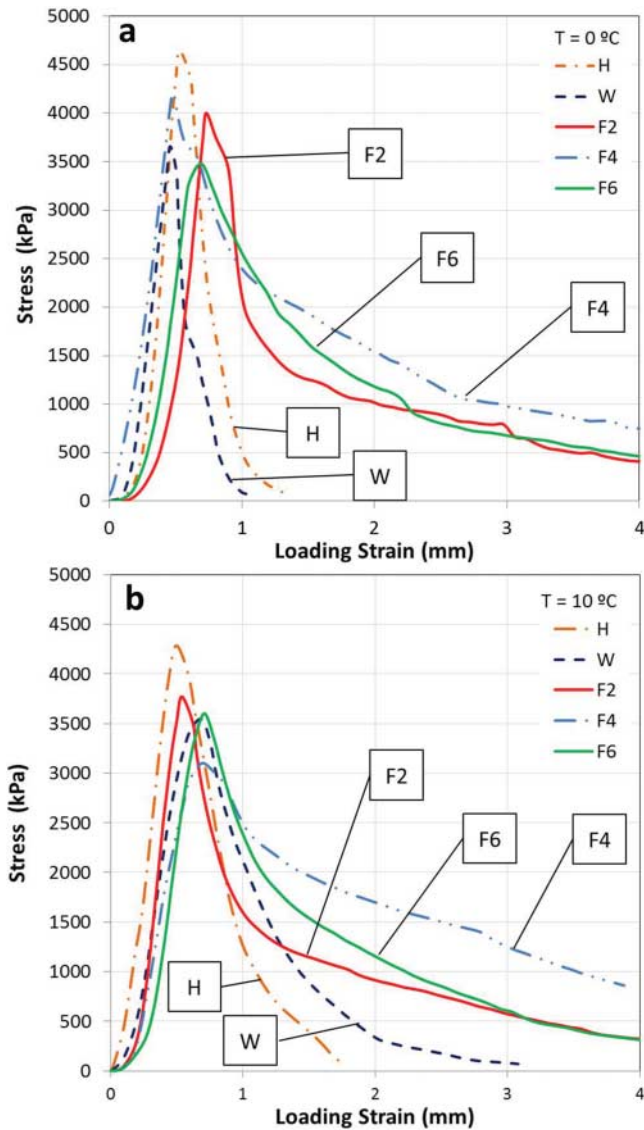


Figure 6. Stress versus loading strain curves of some tested samples in ITS test. (a) $T = 0^{\circ}\text{C}$, (b) $T = 10^{\circ}\text{C}$.

affects the rutting behaviour with an increase of 70% in the WTS of W with respect to the WTS of H. The incorporation of macrofibres improves the rutting behaviour of WMA, reducing the WTS, even with low fibre contents. It can be observed in Table 4 how the WTS of F2 is lower than the WTS of H and nearly half of the WTS of W. It can be mentioned that while fibre content was increased, a higher shear resistance was obtained (lower WTS) and permanent deformations (D_{10000}) were reduced. Up to a 40 and 50% rut depth reduction was reached in the case of F6 with respect to H and W, respectively. It is important to mention the relationship between the densities and rutting resistance of the FRWMAs. While densities decrease (higher air voids) for higher dosages of fibres, the rutting resistance increases. This could be considered contradictory, however the fibres act in the mastic increasing the mastic viscosity due to their length and also



Figure 7. FRWMA sample after indirect tensile strength test.

provide an interlocking mechanism between the aggregates giving more shear resistance, and as a consequence, better rutting resistance is reached despite the higher air voids.

In addition, when comparing WTS and PRD results (see Table 4) with the limits indicated in the Argentinian specification (2017), Table 6, the H mixture meets the criteria for the traffic levels T3 and T4 (if used as the base or surface mixture, respectively), while W doesn't meet any of the criterion. However, incorporating macrofibres improves the rutting response, enabling the use of these asphalt concretes where very high traffics volumes are expected, as in T1 or T2. Then, FRWMA provide a better rutting performance than HMA while maintaining the other benefits of WMA.

Table 6. Wheel tracking limits for asphalt mixtures in Argentinian specification.

Argentinian specification for wheel tracking test (EN 12697-22 – B procedure)*

Position in pavement	Traffic level			
	T1	T2	T3	T4
Surface	WTS \leq 0.08	WTS \leq 0.10	WTS \leq 0.12	WTS \leq 0.15
	PRD \leq 5%	PRD \leq 8%	PRD \leq 7%	PRD \leq 10%
Base	WTS \leq 0.10	WTS \leq 0.12	WTS \leq 0.15	WTS \leq 0.15
	PRD \leq 8%	PRD \leq 10%	PRD \leq 10%	PRD \leq 10%

T1 \geq 1500, T2: 800–1499, T3: 200–799, T4 \leq 199 (vehicles/day).

*Taken from Argentinian Asphalt Concretes Specification (2017).

Regarding fracture behaviour at low temperatures, the incorporation of a minimum amount of macrofibres increases the residual stress capacity of asphalt concrete in both bending and indirect tensile strength tests. Figures 8 and 9 show the calculated toughness at 0°C and 10°C for bending and indirect tensile strength tests, respectively. The advantage of incorporating fibres can be clearly observed in the increase in toughness reached at low temperatures, most evidently at 0°C. At this temperature, the concrete asphalt has a more brittle behaviour, and therefore, the fibres develop a more important role sewing the cracks and transferring the stresses. In addition, it can be observed that while fibre dosage increases, greater improvements of toughness appear. However, there seems to be a minimum fibre content required to improve the fracture behaviour, according to the bending test results shown in Figure 8. While F4 and F6 almost duplicated the toughness at 0°C of H and W, F2 does not show any changes. Another important observation obtained from indirect tensile strength test results (Figure 9) is there appears to be an optimum

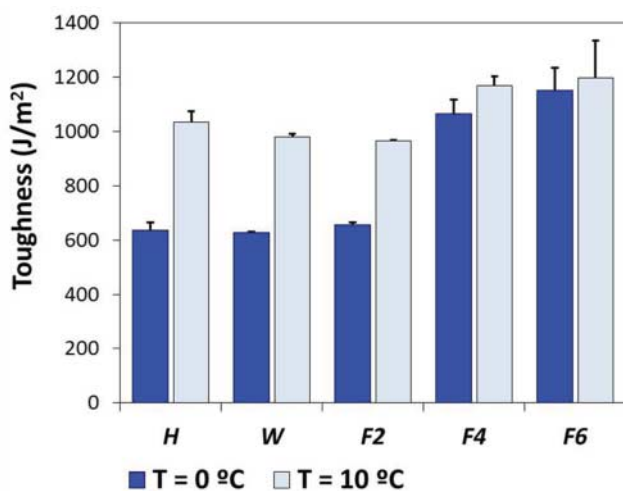


Figure 8. Toughness of asphalt concretes obtained from bending tests.

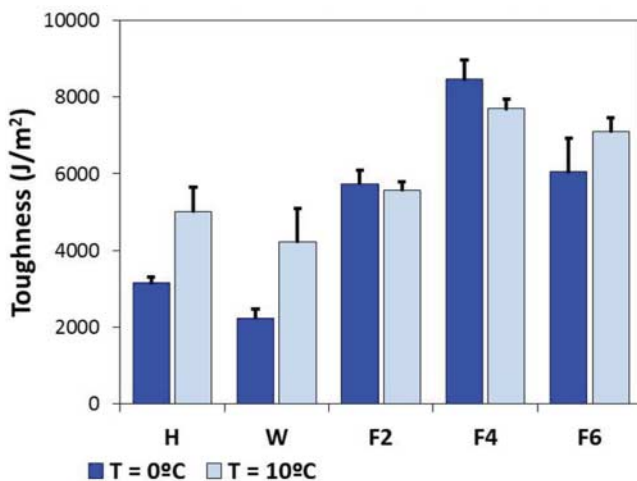


Figure 9. Toughness calculated from indirect tensile strength test.

fibre content to obtain the highest toughness. This is related to optimising the design of these types of asphalt concretes and represents an opportunity for further studies.

FRWMAs present lower densities and higher air voids than control HMA, and as a consequence, lower stabilities were found in these mixtures. These reductions were expected because the design asphalt content of H mixture (4.8%) was kept constant in the FRWMAs. The long shape of the fibres affect the compactability capacity of the mixtures while the asphalt content was not adjusted. Besides, the Marshall compaction method (with blows) affects the compactability of samples at temperatures lower than those of hot mix asphalts. This explains the lower density of W compared to H. Thus, it is evident that for FRWMAs, their asphalt content design should be optimised in accordance with the fibre dosage; however, this was not the main objective of this work. In this way, the results found here for FRWMAs could be considered conservative. Even with these disadvantages, it is proved that the synergic contribution of synthetic macrofibres and WMA technologies represents a promising alternative that must be explored in depth. It is important to mention that a design method for fibre asphalt mixtures is not defined at the moment and remains as a future challenge. It is expected that as fibre asphalt mixtures design is optimised, better improvement in mechanical behaviour will appear.

5. Conclusions

This work explores the possible improvements in asphalt concrete performance due to the incorporation of synthetic macrofibres in Warm Mix Asphalt (WMA). The main conclusions are as follows.

Fibre Reinforced Warm Mix Asphalt (FRWMA) could provide a better performance than Hot Mix Asphalt (HMA) and offers all the benefits of WMA.

The incorporation of macrofibres clearly improves the rutting performance. Wheel tracking test parameters showed improved results for FRWMAs when compared to WMAs without fibres and were even better than those measured on HMAs.

The benefits of synthetic macrofibres also appear when the fracture resistance at low temperatures (0°C) is considered, providing residual post peak load capacity and increasing the toughness.

It must be noted that in this study, the densities of fibre asphalt mixtures were lower than in HMAs since the asphalt content was based on HMA without fibres design and not modified for FRWMAs. The behaviour can be even better after mixture design optimisation; in this sense a proper design method for mixtures would be useful.

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