FIBRA

Fostering the implementation of fibre-reinforced asphalt mixtures by ensuring its safe, optimized and cost-efficient use

Deliverable 3.2

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Executive Summary

This document presents part of the experimental results obtained during the WP3 of the FIBRA project. In particular, those relatated to the thermal and chemical anaylisis corresponding to the different fibres studied as well as the assessment of the rheological properties of the bituminous binders recovered from the asphat mixtures prepared and tested during the WP3. This deliveralble 3.2 summarizes the most important mechanisms governing the microstructural properties of the asphalt matrix concerning the different fibre dispersions.

This report is divided into four parts:

- CHAPTER 1 provides an introduction to the framework of the study;
- CHAPTER 2 specify the type of fibres and bituminous binders as well as the techinques used for their characterizations;
- CHAPTER 3 shows the main experimental results related to the effect of the fibres on the performeance of the asphat mixtures;
- CHAPTER 4 summarizes the most relevant conclusions

1 Introduction

The objective of the FIBRA project is to overcome the technical barriers for the safe and cost-efficient implementation of fibre-reinforced asphalt mixtures (FRAM) with which an increase in the asphalt pavements durability could be achieved.

Previous analysis of diferent experiences with fibres was used in order to select the most promising ones (deliveralble D2.1), a preliminary study has been carried out in order to evaluate the effect of two types of fibres on conventional dense and porous asphalt mixtures (deliveralble D3.1.). In parallel, to understand the performance of FRAM, aspects such as themal properties of the fibres, fibres distribution within the asphalt matrix or rheological behaviour of the recovered binders were studied. In the following sections, details of the materials and techinques used in this study are specified as well as the most relevant results which help to explain the physical mechanisms consequence of the addition of the fibres.

2 Materials and experimental characterization

2.1 Thermal characterization of the fibres

In WP3, two commercially available fibres were selected to investigate the effect of their incorportation into conventional asphalt mixtures. Type A consists on a combination of aramid (A1) and polyolefins (A2) fibres of 19 mm length. The ratio of this blend of fibres was approx. 1:7 (aramid :polyolefins). The other type is polyacrylonitrile fibres (type P) of 4 mm length and nominal diamterer ca. 10 um. Images of both types of tibres are showed in Figure 1.

Figure 1. Images of the different fibres used for the WP3. Type P (left) and type A (right).

In order to investigate the thermal transitions of these three types of fibres, a differential scanning calorimeter (DSC) from PerkinElmer was used. A sample of 5 ± 1 mg was prepared for the DSC analysis. The heating ramps were conducted at a rate of 20 °C/min. Furthermore, the thermal stability of the fibres was analyzed by thermal gravimetric analysis (TGA) under a nitrogen atmosphere using TGA 209 from NETZSCH.

2.2 Evaluation of the fibres distribution by imaging analysis and rheological evaluation of the binders recovered from the FRAM

As part of the experimental plan designed for the WP3, four dense asphalt mixtures (AC 22) were preapared and cylindrical specimens were compacted to evaluate their mechanical properties (Table 1); one control mixture with polymer modified bitumen, one reference mixture with bitumen 50/70 and two FRAMs; one with fibre type A (0.05%wt.) and the other with fibre type P (0.15%wt.). The amount of fibre was suggested by the manufacturers of the two products. More details about the mixing process can be found in the deliverable D3.1.

Table 1. Dense asphalt mixtures (AC 22) prepared for the WP3.

Name	Bitumen (4.2%)	Fibre
ACB22H - Control	PmB 45-80/65	-
ACB22H - Reference	50/70	-
ACB22H – P (0.15%)	50/70	P
ACB22H – A (0.05%)	50/70	A

To understand the chemical effect, if any of the different types of fibres on the behaviour of the experimental mixtures, samples of bitumen were extracted with toluene. Then, the bitumens were recovered by rotatory evaporator (EN 12697-3). The rheological responses and softeining properties of these samples were evaluated. In this study master curves obtained from rheological measurements conducted with a DSR (Physica MCR 301 DSR, Anton Paar) were analysed. The parallel plate configuration with diameters of 8 mm and 25 mm corresponding to test sample thicknesses of 2 mm and 1 mm respectively was used. A constant strain amplitude of 1% for high-temperature range (40 °C – 80 °C) and 0.1% for low temperature range (40 °C–0 °C) with testing frequencies between 0.1 and 20 Hz at each temperature were used. Peltier Systems H-PTD200 and PPTD200 were used for cooling.

In parallel, samples of the FRAMs were evaluated using the Environmental Scanning Electron Microscope (ESEM). For this purpose, specimens with dimension 28×47×10 mm^3 were cut from the centre of a cyclindrical specimens. The samples were impregnated with resin and polished using sand paper and water as lubricant (Figure 2). The ESEM experiments were performed with an FEI Quanta 650 by Thermo Fisher. This study was conducted in the low vacuum mode in order to avoid any specimen perturbations. Specifically, lower inert gas purge (IGP) of 1.9×10^{-7} mbar and upper IGP equal to 1.2×10^{-9} mbar and current emission of 309 mA. Moreover, the ESEM analysis was performed with EDX (Energy Dispersive X-Ray Spectroscopy) option for chemical analysis.

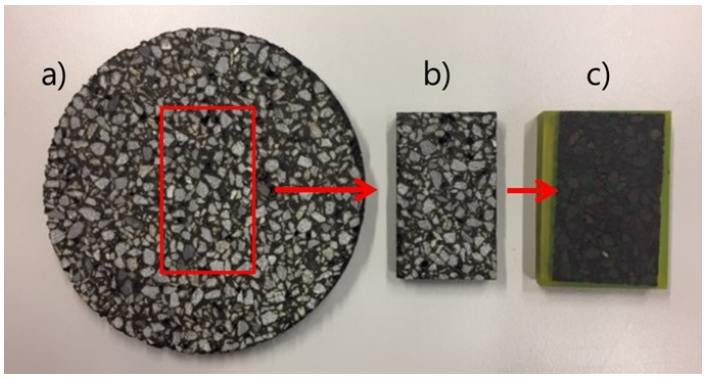

Figure 2. ESEM specimen preparation steps. (a) Marshall sample cylinder (102 mm x 10 mm); (b) Centre cut (28 mm x 47 mm x 10 mm); (c) impregnated and polished ESEM sample.

3 Experimental results

3.1 Fibres characterization

In an asphalt plant, the blending conditions of the asphalt binder components depend on the type of asphalt mixing adopted. Hot mixing of asphalt at temperatures from 130 °C to 160 °C, depending on the binder viscosity, is the common technology for preparing asphalt mixtures. The required mixing temperature should provide sufficiently low viscosity of asphalt binder to ensure full coating of all aggregates. Therefore, the fibres must be able to survive high temperatures and mechanically tough mixing conditions. In order to investigate such conditions, the fibres type A (aramid and polyolefins) and type P (polyacrilonitrile) were subjected to heating ramps using TGA. The measured weight loss of the fibres versus temperature is reported in Figure 3.

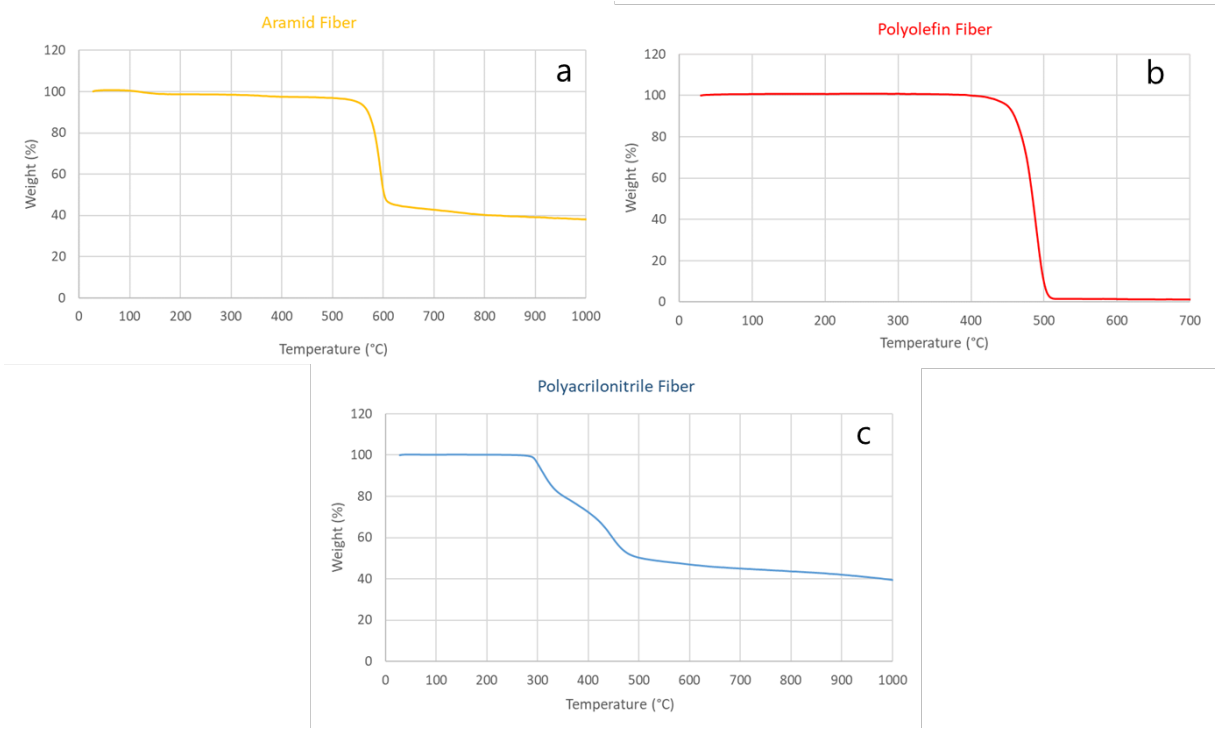

Figure 3. Weight loss of fibres versus temperature during TGA heating ramps. Aramid fibre (a), polyolefin fibre (b) and polyacrilonitrile fibre (c).

It can be observed that for both fibres forming type A (aramid and poyolefins), the weight of the sample is kept constant until temperatures above 450°C. This means that, at conventional temperatures for hot asphalt mixes (ca. 160°C), the fibres will not suffer any thermal degradation. Regarding the fibre type P (polyacrilonitrile), the weght loss starts at a lower temperature (ca. 280°C) compared to fibres type A. It can be also seen that the curve is smoother revealing different degradation temperatures probably linked to the various chemical composition of the fibre. Nevertheless, these temperatures are still far from the mixing tempatures normally used in standard procedures, so, it can be confirmed that no themal effect is expected for this kind of fibre either.

The fibres were also thermally analyzed for a large temperature range (from 20 °C to 500 °C) in detail by calorimetry in order to study the thermal transitions of the fibres. The heat flow curves for fibres type A and P are given in Figure. 4.

During the heating, the fibres could melt and hypothetically react chemically with the bitumen and modify it. This could then change the binder properties affecting the performance of the asphalt mixtures. In Figure 4, it can be observed that aramid fibres (type A1) above 400 °C, so no chemical modification would occur and any effect on the response of the FRAM will be due to a physical interaction. On the other hand, the polyolefin fibres (type A2) showed a melting temperature of 124.5 °C which is lower than the mixing temperature used for the experimental FRAMs (i.e. 160°C). Although the concentration of this type of fibres was low (lower than 0.05%wt.), the addition of the polyolefin fibres was recommended to be done directly into the hot binder. This fact could lead to a modification of the bitumen influencing the behaviour of the corresponding FRAM (ACB22H-A). For that reason, it was decided to extract and recover the bitumen from samples of the different dense mixes prepared during WP3 (control, reference and FRAMs) in order to study this potential bitumen modification. Results from this analysis are shown in the next section (3.2.2).

Concerning the polyacrilonitrile fibres (type P), the DSC measurement (Figure 4c) shows a sharp exothermic peak close to 300°C. This could meand the fibre already starting decomposig at this temperature. This behaviour coindides with the result observed from TGA. In this sense, the provider has pointed out that the fibre degrades before reaching its melting temperature (ca. 350 °C). As a consequence, energy freed from this initial degradation could locally raise the temperature.

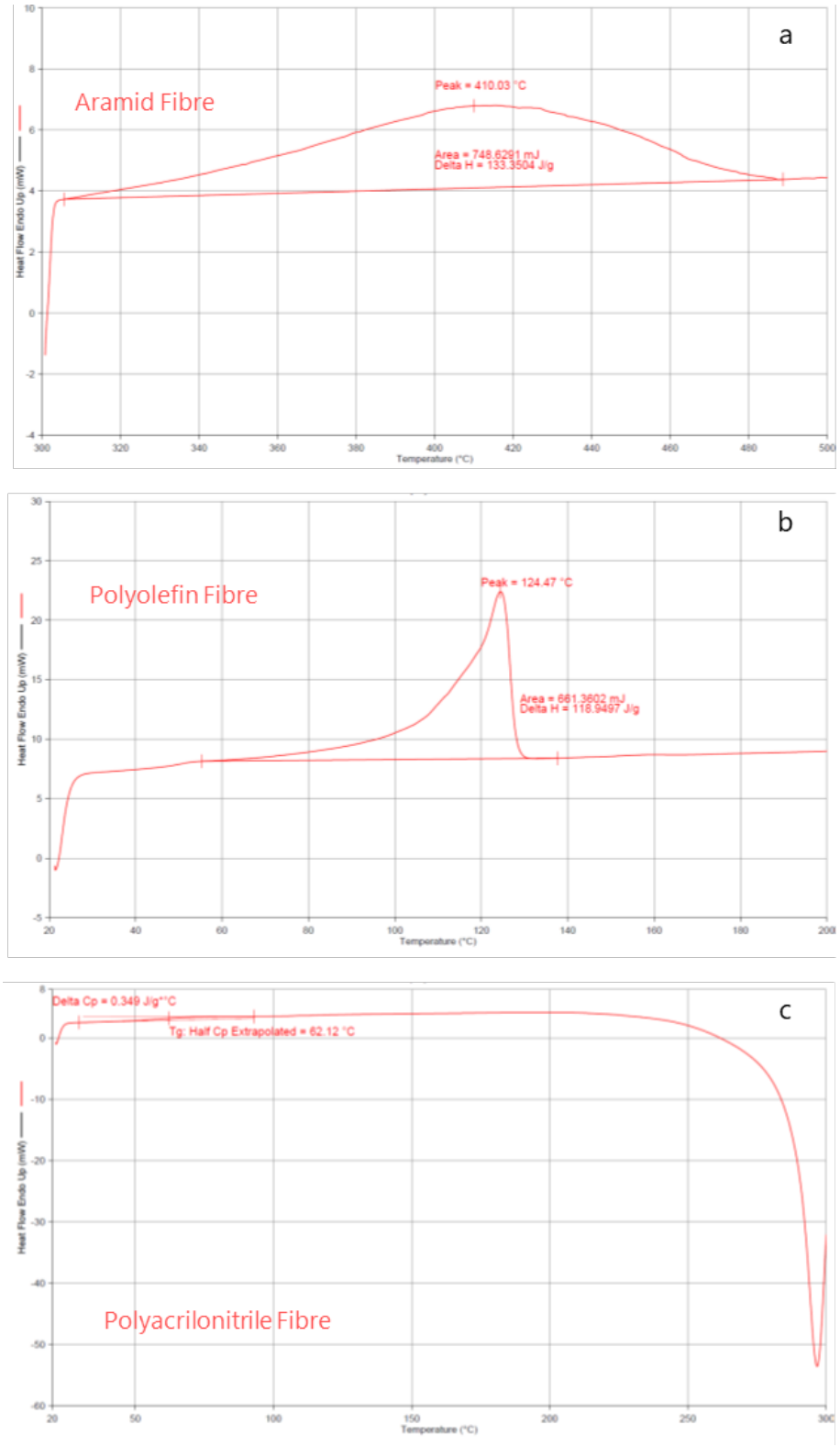

Figure 4. Heat flow curves versus temperature for the different fibres studied. Aramid fibre (a), polyolefin fibre (b) and polyacrilonitrile fibre (c).

3.2 Imaging analysis

The aim of this part of the study is to visualize the fibres whithin the asphalt matrix in order to understand their role based on this aspect. The use of the EDX technique will help to identify the fibres thanks to the analysis of the chemical elements. First, single analysis of the fibres were done to charaterize their chemical compositions. ESEM image of polyolefin fibres and its elemental analyses of two characteristic points are shown in Figure 5 and Table 2, respectively.

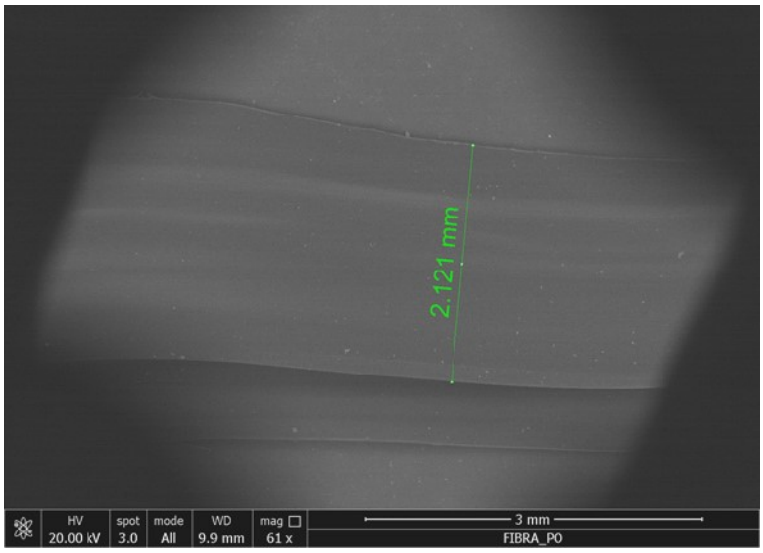

Figure 5. ESEM micrograph of a polyolefin fibre (Type A2).

Table 2. Elemental analysis (atom%) of the polyolefin fibre.

	C
FIBRA PO(1)_pt1	100.00
FIBRA PO(1)_pt2	100.00

The EDX analysis of the polyolefin fibres (type A2) indicates that they contein mainly carbon elements. If these fibres are not melted, as we discussed in the previous section, this fact will make it more difficult to identify them within the asphalt matrix which also contains hydro carbons.

The ESEM image of the aramid fibres (type A1) and its elemental anaylsis are shown in Figure 6 and Table 3. Here, it can be seen that more atoms appear (N, O or S) which reveals the presence of their characteristic amide groups. These particularities will later help to analyse the micro-images from the FRAM modified with fibres type A.

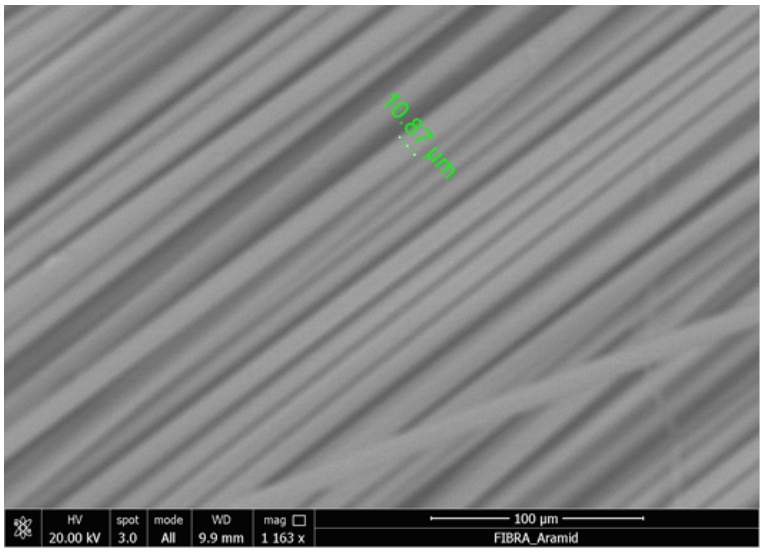

Figure 6. ESEM micrograph of an aramid fibre (Type A2).

Table 3. Elemental analysis (atom%) of the aramid fibre.

	C	N	O	Na	S
FIBRA Aramid(1)_pt1	61.24	21.08	17.28	0.29	0.11

Last, the ESEM image of the fibres type P (polyacrilonitrile) and their elemental composition are shown in Figure 7 and Table 4.

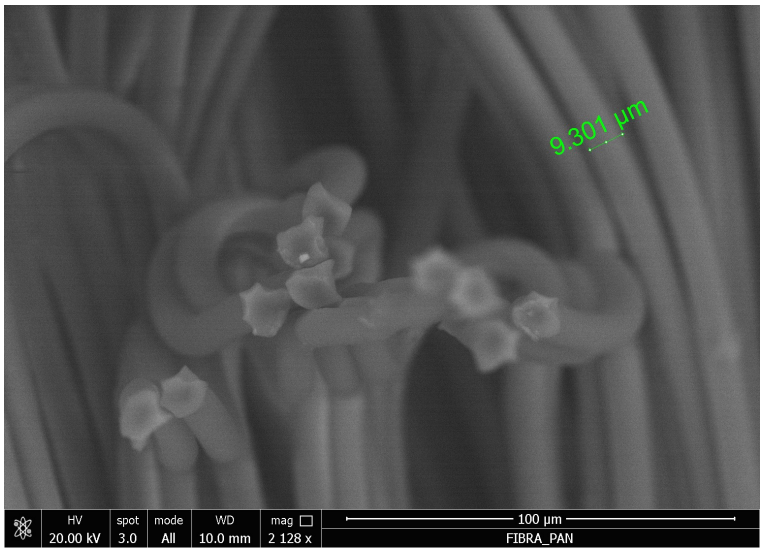

Figure 7. SEM micrograph of a polyacrilonitrile fibre (Type P).

Table 4. Elemental analysis (atom%) of the polyacrilonitrile fibre.

	C	N	O	Al	Si
FIBRA(1)_pt1	54.80	36.97	8.10	0.03	0.09
FIBRA(1)_pt2	52.30	38.73	8.78		0.19

In this case, the EDX analysis of the fibres type P (polyacrilonitrile) indicates that they primarily consist of carbon (C), nitrogen (N) and oxygen (O). This is directly related to the presence of the acrylonitrile groups. Again, the presence of nitrogen will be crucial to identify these fibres mixed with the asphalt and mineral aggregates.

Next, the ESEM image from the sample of the FRAM mixture using fibres type A (aramid+polyolefins) is shown in Figure 8. The presence of some aramid fibres (identified by their unusual shape and chemical analysis) can be clearly seen. Polyolefin fibres could have been melted during the mixing process due to their thermal properties and therefore it can be expected that the fibres in Figure 8 are Aramid fibres.

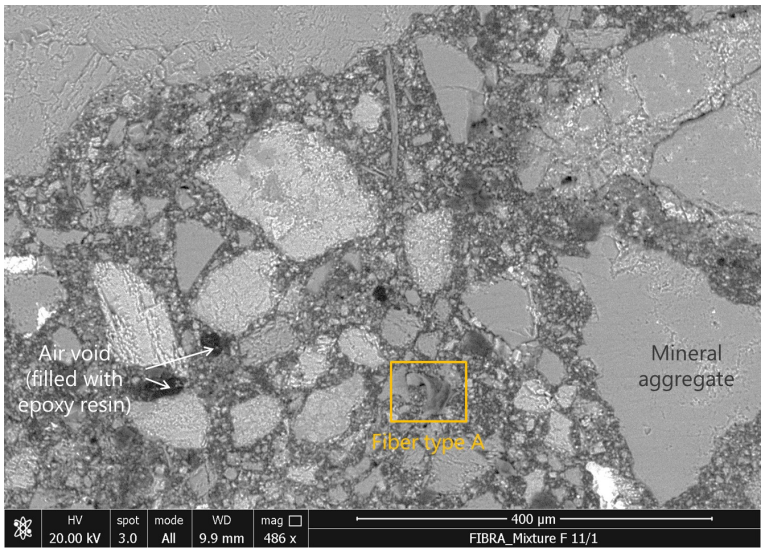

Figure 8. ESEM image of the sample from the FRAM with type A fibres.

An ESEM image of sample obtained from the FRAM modified with the fibres type P (polyacrilonitrile) is shown in Figure 9.

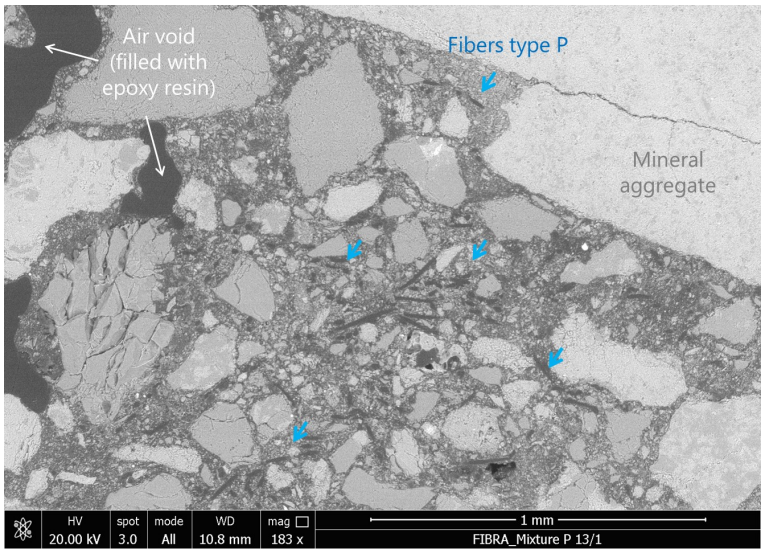

Figure 9. ESEM image of the sample from the FRAM with type P fibres.

In this image, the presence of parts of fibres type P can be clearly observed. These are homogeneously distributed consequence of their higher concentration (0.15%) compared to the FRAM modified with fibres type A (0.05%).

3.3 Evaluation of the bitumen recovered

After the mixing porcess, samples from the different mixes were taken to recover the bitumen in order to study whether the fibres had any chemical effect on the binders. After bitumen extraction, the mix of mineral aggregates and fibres are shown in Figure 10. It can be seen that they suffered a significant change in their morphology (form and structure). Nevertheless, these changes could be a consequence of the solvent (toluene) used during the extraction process.

Figure 10. Mineral aggregates and fibres after bitumen extraction. FRAMs modified with fibres type A (left) and type P (right).

Moreover, details of the fibres can be observed in Figure 11.

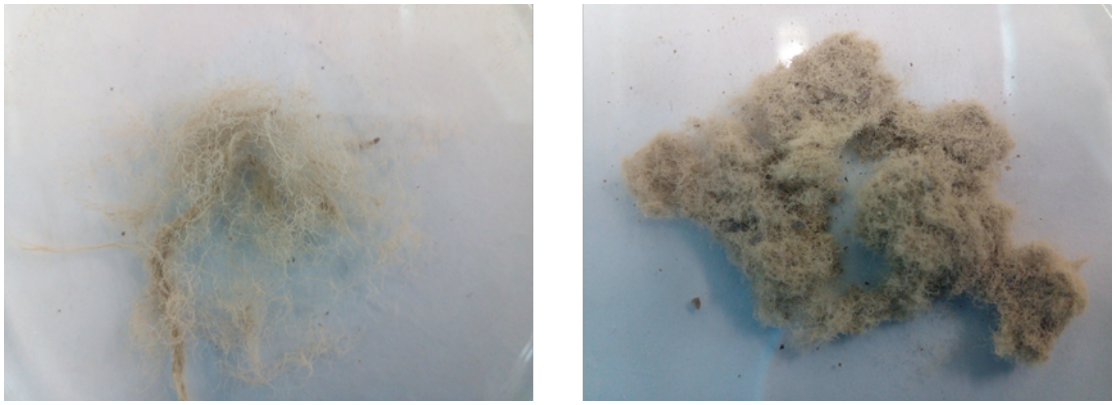

Figure 11. Details of fibres type A (right) and type P (left) after bitumen extraction.

The samples of the different bitumens recovered from the experimental mixtures were first tested to evaluate their softening point. These results are shown in Table 5.

Table 5. Bitumen properties before and after mixing and extraction processes.

	Softening point		°C
Original	Reference	50/70	49.0
	Control	PmB	66.4
After recovery	Reference	50/70	55.0
	Control	PmB	66.8
	ACB22H - P	50/70 + type P	55.5
	ACB22H - A	50/70 + type A	56.1

First, it can be seen that there is a significant ageing effect on the straight bitumen which leads to an increase of the softening point from 49°C to 55°C. This effect was not observed in the polymer modified bitumen used for the control mixture. Regarding the influence of the fibres, at mixing temperature (160 °C), a chemical modification of the bitumen could be expected by the polyolefin fibres (type A2) due to their melting temperature (124°C). Nevertheless, it can be confirmed that there was no chemical modification due to any of the fibres. Values obtained for the bitumens recovered from the FRAMs are quite similar to that obtained for the reference mixture.

This behaviour could be observed in more detail in Figure 12 where the rheological anaylisis is shown. In these graphs, the master curves as well as the black curves obtained from the different bitumen samples indicate that the recovered polymer modified bitumen is the only one showing a different response. On the other hand, the binders from the FRAM present a similar response to the one from the reference mixture (straight bitumen 50/70) confirming that the addition of fibres did not alter the binder .

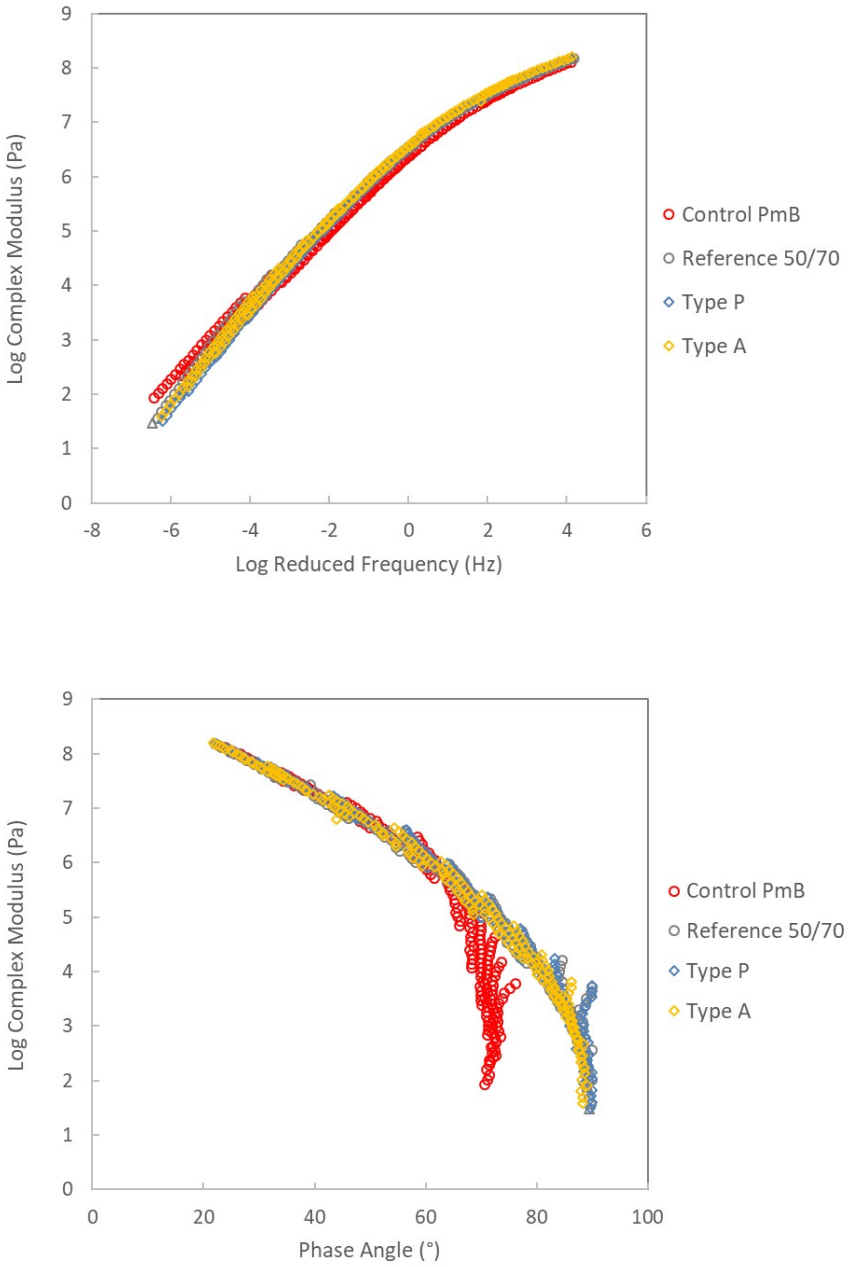

Figure 12. Master curves and Black curves of the different bitumen samples after extraction.

4 Conclusions

In the present document the main results from the thermal characterization of the selected fibres, image anaylsis of samples of the FRAMs and study of the bitumen recovered have been reported. This study aims at understunding the chemo-mechanical mechanims behind the performance obained for the FRAMs in the other experimental phase within this WP3. The following conclusions can be drawn from the anaylis of the two different types of fibres (type A and type P):

- The thermal analysis of the fibres indicate that no degradation will happen during the mixing process due to the temperature. Besides, since their melting temperartures were found higher than the mixing temperatures, polyacrilonitrile (type P) fibres and aramid fibres (type A1) will remain in solid state. However, the melting temperature for the polyolefin fibres (type A2) was found to be lower, thus, they could melt and modify the bitumen. This effect could be increased for the addition of these type of fibres directly to the hot bitumen.

- The imaging analysis with ESEM plus EDX method has confirmed the presence of polyacrilonitrile (type P) fibres and aramid fibres (type A1) within the asphalt matrix. This confirms the improvement of the mechanical properties of the FRAMs is directly related to a physical influence of the fibres.

- After extraction and recovery of the bitumens from the different asphalt mixtures, no significant modification due to the addition of the fibres was observed. The possible melting of the polyolefin fibres (type A2) does not affect the bitumen properties. Unfortunately, the chemical composition of these fibres (mainly carbon) made it impossible to distinguish them within the asphalt matrix in the ESEM analysis. So, it is not clear if these fibres "survive" the mixing process (small mixing times) and play a reinforcement role like the other fibres evaluated in this study.