



HAL
open science

Influence of warm mix additives on the low-temperature behavior of bitumen using the Bending Beam Rheometer (BBR)

Anda Ligia Belc, Ion Octavian Pop, Florin Belc, Ciprian Costescu, Fateh Fakhari Tehrani

► To cite this version:

Anda Ligia Belc, Ion Octavian Pop, Florin Belc, Ciprian Costescu, Fateh Fakhari Tehrani. Influence of warm mix additives on the low-temperature behavior of bitumen using the Bending Beam Rheometer (BBR). *Construction and Building Materials*, 2021, 273, pp.121682. 10.1016/j.conbuildmat.2020.121682 . hal-03705701

HAL Id: hal-03705701

<https://cnam.hal.science/hal-03705701>

Submitted on 22 Mar 2023

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial 4.0 International License

Influence of warm mix additives on the low-temperature behavior of bitumen using the Bending Beam Rheometer (BBR)

Anda Ligia BELC¹, Ion Octavian POP², Florin BELC¹, Ciprian COSTESCU¹, Fateh FAKHARI TEHRANI^{2,3}

¹Universitatea Politehnica Timisoara, Facultatea de Constructii, Timisoara, Roumania

²Univ. Limoges, GC2D EA3178, F-19300, Egletons, France

³Conservatoire National des Arts et Métiers, Paris, France

Abstract

The mechanical and rheological characteristics of bitumen are crucial for the behavior and durability of the bituminous road layers. A bitumen susceptible to high temperatures can lead to plastic deformation of the bituminous layers, whereas a low temperature susceptible binder can lead to cracking of the bituminous layers. The penetration test at 25°C, softening point - ring and ball method and ductility test were carried out on a 50/70 bitumen and five types of bitumen blends. In order to determine the behavior of bitumen at low temperatures, the Bending Beam Rheometer method was implemented. The presented research aims to determine how different additives that have the purpose to increase the workability of asphalt mixtures (for making warm mix asphalts) influence the behavior of a 50/70 bitumen commonly used in Romanian road technique. In this regard, the results regarding the susceptibility of bitumen at low temperatures are presented, compared to those of the same binder in which various additives were introduced. To further highlight the influence that the additives used have on the low temperature behavior of the bitumen, the research is completed by tests on bituminous mastic for each type of binder. The additives do not lead to significant changes in the rheology of pure bitumen. The wax has the biggest influence on the bitumen's properties. The limit temperature increases with the addition of filler in the blends.

Keywords: Bending Beam Rheometer (BBR), bitumen, bituminous mastic, flexural creep stiffness, the slope of the stiffness versus time curve

1. Introduction

This research is part of a larger study, which aims at finding sustainable methods to improve the rheological characteristics of asphalt mixtures and determining the additive that has the best behavior to be used for the preparation of warm mix asphalts in the specific weather and traffic conditions. It should be noted that in the last period of time the road specialists are more and more concerned about by finding viable technologies to reduce the fuel consumption and greenhouse gas emissions. Thus appeared the problem of reducing the temperatures of preparation and laying the asphalt mixtures by a few tens of degrees, without compromising the performance achieved for the road layers [1,2]. In this context, an important part of the research is assigned to the characteristics of the bitumen and their variation in relation to the additives usable for the preparation of warm mix asphalts. The literature shows that the application of these technologies can lead to the reduction of gas emissions by 10 - 50%, respectively the reduction of fuel consumption by 11 - 35% [1]. In this context, the behavior of bitumen at low temperatures is monitored and how it is influenced or not in terms of rheological characteristics by the additives used to reduce the temperature of preparation and laying of asphalt mixtures.

Generally, the classic (hot) asphalt mixes are prepared by heating the component materials (aggregates and bitumen) at temperatures of 160-180°C, which leads to significant fuel consumption and gas emissions in the atmosphere throughout the manufacture, transport and laying, but also at the risk of accidents at work. Thus, the warm asphalt mixes, which have significantly lower preparation and laying temperatures than in the case of hot mix asphalts, have been designed worldwide (preparation temperatures of about 120-140°C and laying temperatures of 100-120°C) [1–5]. In order to increase the workability required to put them into operation in good conditions special additives (chemical additives and organic additives) or zeolites are used [1–4,6].

Recent study reveals, a nonlinear rheological behavior of the bituminous mastic was observed and it was concluded that the high temperature performance of the mastic is influenced more than the low temperature performance by the amount of filler used [7]. Another study on the performance of bituminous mastic at low temperatures shows that the chemical composition and the shape of the filler do not seem to have an effect on the flexural creep stiffness of the mastic at

low temperatures. The only thing that affects this performance being the amount of filler added in the mastic [8].

The bitumen is a hydrocarbon binder used in road technology in several countries around the world that, due to its easy use and its main properties (especially related to: adhesiveness, plasticity, ductility, insolubility in water and inertia to numerous chemical agents), has undergone an important development in the road and industrial fields [9–12]. It has a large monolithic capacity because it adheres to most of the usual materials: natural aggregates, cement concrete, wood, metal or glass. Unfortunately, its rheological behavior is different depending on the conditions of load and temperature [13–17]. It deforms differently for normal ambient temperatures and for loads from extremely varied traffic in size and frequency of application [6,18–20]. The asphalt mixtures that deform easily are prone to rutting, and those that are too rigid are susceptible to fatigue and cracking. Understanding bitumen flow and deformation is important for evaluating the performance of asphalt mixtures. The main characteristics by which the quality of a road bitumen is currently analyzed are related to consistency (viscosity, standard penetration and penetration index), plasticity (softening point ring and ball, breaking point Fraass, ductility) and adhesiveness, to which are added characteristics related to aging (loss of mass and determination of thin layer stability RTFOT - Rolling Thin-Film Oven Test or TFOT - Thin Film Oven Test, [21]).

To highlight the rheological character of the bitumen, additional laboratory tests are required. For these tests, high precision equipment in the variation of the temperatures and the loads applied to the samples is required during the test. Among these common tests, the following ones are reminded:

- The Bending Beam Rheometer test was developed in the US as part of the Strategic Highway Research Program (SHRP) to facilitate the determination of the rheological characteristics of bitumen at low temperatures. These characteristics serve to estimate the possibility of thermal cracks occurring in the bituminous layers [22,23];
- The DSR (Dynamic Shear Rheometer) is used to characterize the viscous and elastic behavior of bitumens at medium and high temperatures. With the help of this test the rutting and fatigue cracking behavior of the asphalt mixes can be evaluated [24,25];
- Direct Tension Test (DTT) helps measuring the low temperature stiffness and the

relaxation properties of bitumen. These parameters provide guidance on the ability of a bitumen to withstand cracking at low temperatures. DTT is used in combination with BBR to determine the low temperatures performance grade (PG) of bitumen [26].

The effect of various percentages of organic additive added to the bitumen was previously analyzed and it was determined that adding a high percentage of this warm mix additive can lead to a lower cracking performance [27].

A type of organic additive, synthetic wax, is found to have a positive effect on the high temperature performance if used in percentages up to 2% by the weight of the bitumen, but it has a negative effect on the low temperature performance [28]. A research [29] concluded that the addition of a warm mix additive (wax) in a polymer modified bitumen can lead to a lower resistance on low temperature cracking, but other studies state that the increase in the cracking temperature is very small so this type of additive can be used [30]. Another research shows that adding a warm mix additive (commercial wax) can be benefic in terms of increasing the degree of reversible aging in the bitumen and also increasing the high temperature performance grade. [31]

Recent studies [32,33] show that the BBR device has been modified and used to determine the strength of asphalt mixtures. Different methods of sample preparation and loading were analyzed, as well as the factors that may affect the strength of the mixture: the size of the test beams and the cooling environment.

Also, for a better correlation of the results on the six types of binders (bitumen, respectively bitumen with additives), the behavior of the bituminous mastic (filler/bitumen ratio of 1.46) was evaluated at reduced temperatures (same as in the case of non-filler binders).

The two sets of results allowed highlighting the differences and similarities between the behavior of binders and bituminous mastic, with the formulation of conclusions regarding the behavior at low temperatures of asphalt mixtures prepared with such binders.

2. Materials and methods

The binder considered in this study is a bitumen 50/70 from the Mol Refinery, frequently used in the Romanian road technique. In the research, the respective bitumen was blended with various additive substances (i.e. classical synthetic wax, softer synthetic wax, chemical additive and synthetic zeolite), that are used in the road technique to reduce the temperatures of preparation and laying of asphalt mixtures, that is to obtain the warm mix asphalts.

There are three available techniques to produce WMA: chemical additive, organic additive and foaming techniques. [1,2] Regarding the foaming techniques there are two available methods for foaming: direct method (using injection foaming nozzles) and indirect method (using minerals). In the present study the indirect method of foaming (using synthetic zeolite) was studied besides the chemical and organic additives techniques.

The evaluated bitumen blends are: pure bitumen, bitumen with 3% classical synthetic wax by weight of the binder (B+3%W1), bitumen with 1.5% classical synthetic wax by weight of the binder (B+1.5%W1), bitumen with 1.5% softer synthetic wax by weight of the binder (B+1.5%W2), bitumen with 0.5% chemical additive by weight of the binder (B+0.5%C), bitumen with 5.5% synthetic zeolite by weight of the binder (B+5.5%Z), respectively. The percentages of additives/synthetic zeolite were a recommendation from suppliers. The blend between the pure bitumen and the modifier was made with a countertop electric laboratory stirrer (the stirrer length is 64.5 cm and stir blade diameter, fully extended, is 5.5 cm). The stirrer can reach up to 3000 revolutions per minute.

In order to study the morphology and elemental composition of filler and zeolite and to determine if the chemical additive reacts with the filler, these materials (filler, zeolite and filler blended with the chemical additive) were characterized by scanning electron microscopy (SEM: Quanta FEG 250, FEI, The Netherlands) using back scattered electron detector (BSD) and by energy dispersive X-ray spectroscopy (EDX using an Apollo SSD detector, EDAX Inc. US). The microstructure and EDX analysis were performed at about 10 mm working distance (WD) in low vacuum mode in order to avoid surface charging and damage of the analyzed material. The results are presented in Figure 1.

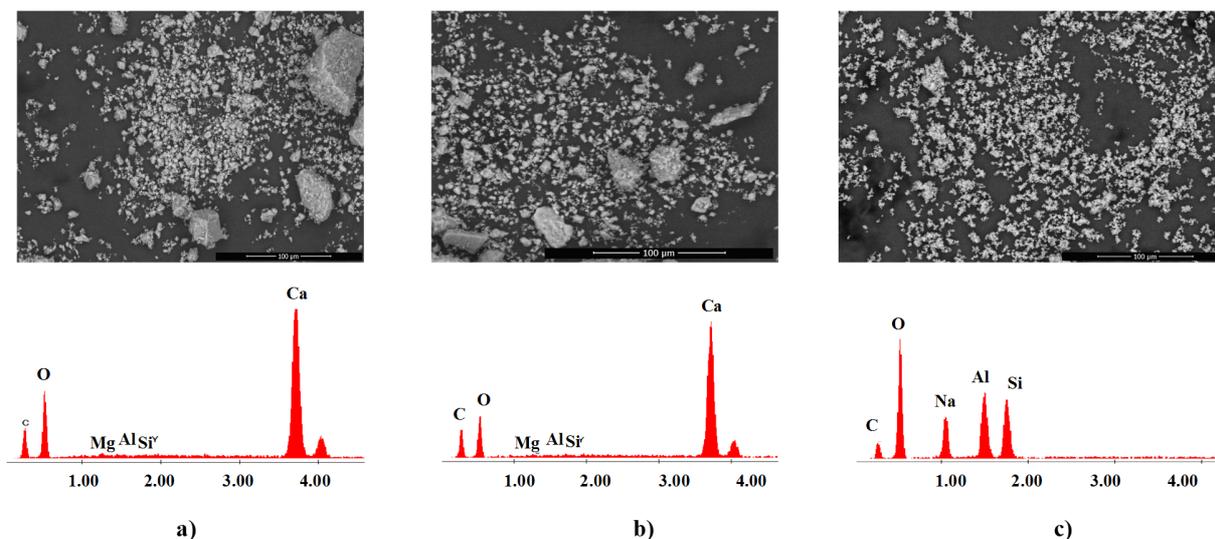


Figure 1. The morphology and elemental composition of: a) Filler; b) Filler blended with chemical additive; c) Synthetic zeolite

As it can be observed from the figures and the elemental composition, the filler does not react with the chemical additive, it keeps the same shape and the elements are not changed. It was concluded that this additive does not react with the filler. On the other hand, there is a clear difference between the filler and the synthetic zeolite which can be noticed in the shape of the particles, as well as in the elemental composition. This is the reason why the zeolite could not be considered as filler.

In addition, tests were performed on bituminous mastic, in a filler / bitumen ratio of 1.46, for both pure bitumen and pure bitumen homogenized in the same percentages with the abovementioned substances. According to [7] a filler / bitumen ratio between 0.9-1.4 is advantageous for both low and high temperature behavior. The selection of the respective ratio took into account the dosage of the asphalt mixture, namely an asphalt concrete for the surface layer with the maximum size of aggregates 16 mm (54% aggregates 4/16, 33.2% sands 0/4, 7.6% filler and 5.2% bitumen by weight of the asphalt mixture). Since the difference between the obtained value based on the reference asphalt mixture and the recommended interval of 0.9-1.4 was almost negligible, it was decided to keep the ratio that resulted from the asphalt mix formulation. It should be noted that, bitumen, bituminous mastic and bitumen, respectively bituminous mastic mixes have been subjected to the same conditions of preparation, pouring and

testing, respectively.

In order to characterize the influence of various additive substances, the penetration test at 25°C, softening point ring and ball method and ductility were carried out. Moreover, the penetration index was determined for all the mentioned cases. The tests were carried out according to the European standards in force [34–36]. The penetration index is determined according to Annex A of [37]. The similarities and differences discovered following the tests were evaluated.

In addition to the classic tests on bitumen, the Bending Beam Rheometer test was conducted on the mentioned bitumen blends in order to analyze the behavior of bitumen at low temperatures. This test was performed according to [38]. The temperatures for which this test was carried out are in the range of -5 to -35°C, the determinations being made with the gradual decrease of the temperature by -5°C. For each temperature considered, three bitumen beam samples were tested, the result considered being the average of the obtained values. Table 1 shows the experimental plan followed in this study.

Table 1. Experimental plan for bitumen specimens

Sample						Temperature [°C]	Replicates	Total Tests
Pure Bitumen	B+3%W1	B+1.5%W1	B+1.5%W2	B+0.5%C	B+5.5%Z			
Additive	synthetic wax	synthetic wax	softer synthetic wax	chemical additive	zeolite			
% of additive	3	1.5	1.5	0.5	5.5			
Tests	Penetration (EN 1426:2015)					25	3	18
	Softening point – ring and ball (EN 1427:2015)					-	2	12
	Ductility (SR 61:1997)					25	3	18
	Bending Beam Rheometer (EN 14771)					-5°C	3	126
						-10°C		
						-15°C		
						-20°C		
-25°C								
Bending Beam Rheometer (EN 14771)					-30°C	3	126	
					-35°C			

2.1 Test method - Bending Beam Rheometer

The Bending Beam Rheometer test principle is the measurement of the deflection in the middle of a bitumen beam placed on two supports (3-point bending) in an ethanol bath, maintained at a constant temperature and under a constant load in the middle of the beam for 240 s (Figure 2). The bitumen beam specimen has the following dimensions: thicknesses: 6.4 ± 0.1 mm, width 12.7 ± 0.1 mm, length 127 ± 5 mm. The distance between the supporting pins is 100 mm. The deflection at that point is measured at 8 s, 15 s, 30 s, 60 s, 120 s and 240 s [38]. The loading is 980 ± 50 mN.

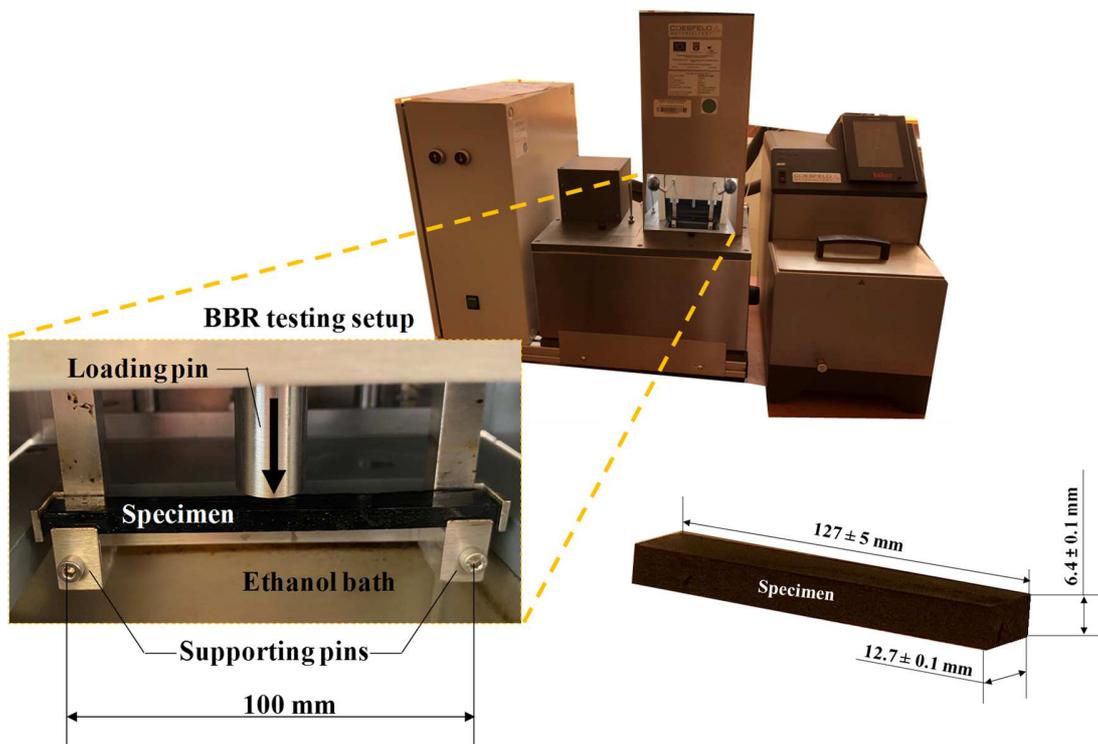


Figure 2. Bending Beam Rheometer test principle

Following this test, two parameters are obtained:

- the flexural creep stiffness, $S(t)$, which indicates the bitumen rigidity at a certain temperature under constant loading;

- the slope of the stiffness curve with respect to time, on a double logarithmic scale, noted below with *m-value* (m), by which one can analyze how the bitumen stiffness changes during the application of a load of constant value.

It can start from the assumption that the flexural creep stiffness of the asphalt mixture after 2 hours of loading can be correlated with the transverse cracks occurring in situ. The extension of this correlation to the bitumen rigidity, and using the principle of time-temperature superposition, leads to the idea that for bitumen, in general, it is valid that the flexural creep stiffness at 2 hours at a minimum temperature T [$^{\circ}\text{C}$] is approximately equal to the flexural creep stiffness at 60 s at a temperature $T' = T + 10^{\circ}\text{C}$ [39]. For this reason, it is recommended to perform the test up to a temperature of 10°C higher than the minimum temperature reached by the bituminous layer in the road structure.

The second parameter, the slope *m-value*, was introduced in the idea that a low value of the slope is unfavorable to the behavior of bitumen because it indicates a lower capacity to release the thermal stresses produced at low temperatures. However, this is debatable, as there are studies that prove that a small slope value can be favorable due to a slower development of thermal stresses [40].

The development of thermal stresses is mainly controlled by the flexural creep stiffness; a harder bitumen leads to higher stresses compared to a soft one, regardless of the slope m . Instead, it was found that for close values of the flexural creep stiffness S , the slope m can greatly affect the development of thermal stresses. Even if in warmer climates where temperatures are not so low a higher slope is desirable, in colder climates this should be treated with caution as thermal stresses can occur faster, leading to cracking [41].

Usually tests on the bending beam rheometer are made between temperatures of -36°C and 0°C , at different temperature intervals (often with a difference of -6°C). Establishing the test parameters is fundamental because, as the loading time increases, the flexural creep stiffness of the bitumen decreases, and the sample deflection increases. In this regard, according to the technical norm [42], the mentioned parameters are recorded at 60 s during the test with the bending beam rheometer, and then they are used to determine:

- the temperature corresponding to a flexural creep stiffness $S(60\text{s}) = 300$ MPa;
- the temperature corresponding to a slope $m(60\text{s}) = 0.30$.

These values (maximum for the flexural creep stiffness, minimum for the slope m) were imposed in order to prevent the cracking at low temperatures and to determine the allowed limit temperature.

3. Results and analysis

3.1 Binder tests

The results of tests performed on the binders considered in the research are presented below. The results of the hardness/softness of bitumen and bitumen with additives are illustrated in Figure 3. Concerning the penetration test, it is widely agreed that if the needle penetration increases, it is considered that the bitumen consistency is reduced and becomes softer.

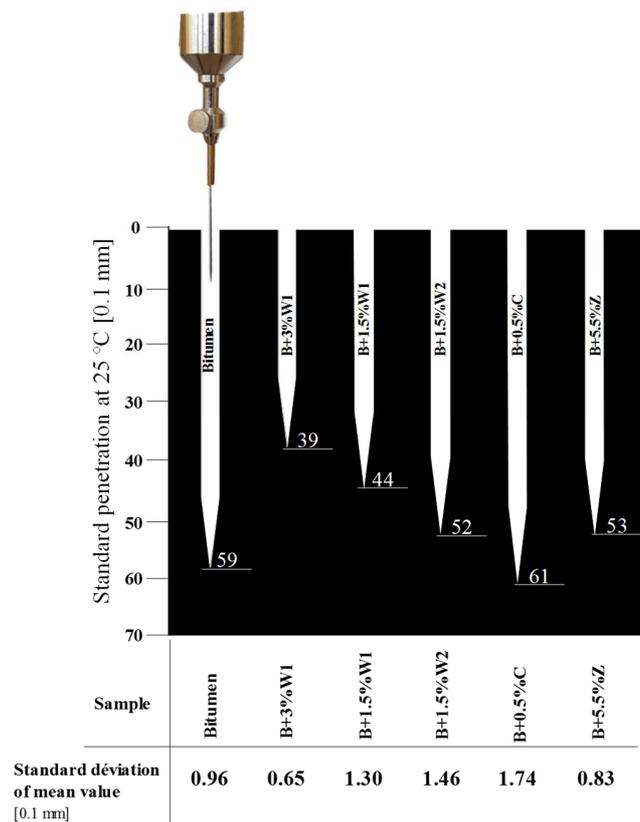


Figure 3. Results of penetration test

As plotted in Fig. 3, in the present case it is found that almost all the additives tested harden the bitumen, but each to a different extent (from a slight decrease in penetration in the case of soft

synthetic waxes, to a considerable decrease in the case of synthetic waxes with a participation rate of 3% by weight of bitumen). The only increase in penetration is recorded when using the chemical additive, but the difference between the pure bitumen and the additive blend is within the precision of the penetration measurement. However, the chemical additive did not change the bitumen grade (50/70). Only the addition of synthetic waxes leads to a change in the bitumen grade, the latter becoming a 35/50.

The softening point results are plotted in Figure 4. This test allows to highlight the consistency of the bitumen. The decrease of the softening point coincides with the decrease of consistency. In the present research an increase of the softening point is observed in the case of almost all the additives compared to the value found in pure bitumen, but the most important increase is also observed in the classical synthetic wax (at 3% by weight of the bitumen). Also, the only additive that does not fit the same pattern is the chemical one.

A hardening of the bitumen with synthetic wax is observed due to the increase of the softening point and the decrease of penetration at 25°C. For bitumen 50/70 according to [37] the softening point must be in the range 46-54°C and the penetration at 25°C in the range 50-70 [0.1 mm].

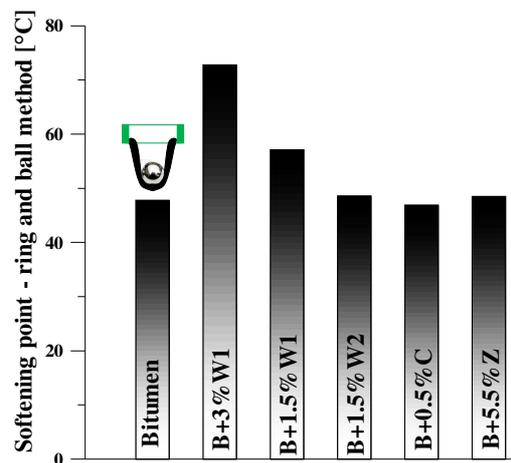


Figure 4. Softening point – ring and ball method

Figure 5 shows the results of the ductility tests. Ductility assesses the flexibility of the bitumen

and its tensile strength. The results of tests reveal that globally the ductility decreases with the addition of additives compared to the case of pure bitumen, which indicates a decrease of the bitumen flexibility when using additives.

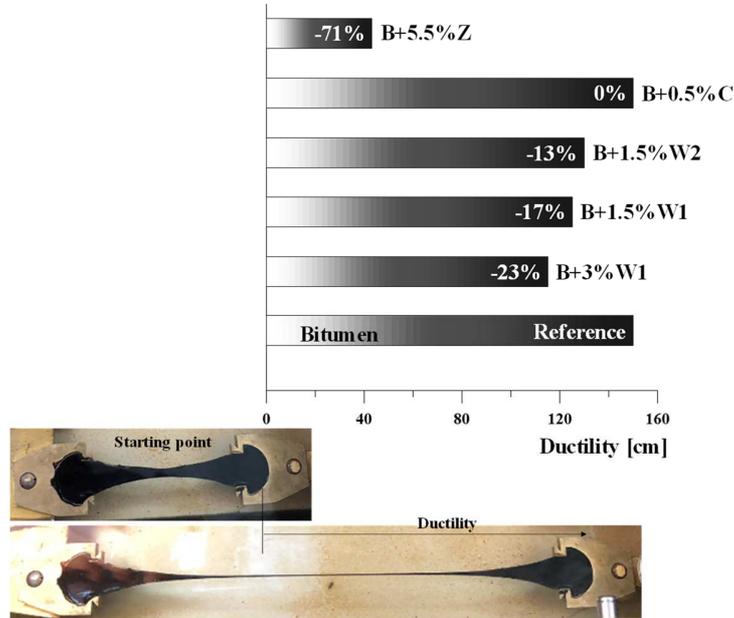


Figure 5. Ductility

A decrease in ductility can be noticed. It can vary between 71% for zeolite and 13% for wax, the exceptions being the samples with chemical additive, where the same ductility as for the pure bitumen can be observed.

The penetration index plotted in Figure 6, indicates the bitumen's temperature sensitivity and is calculated with the relation 1 [37]. In the studied case, an important increase is observed when the organic additive is used - the classical synthetic wax in percentage of 3% by weight of bitumen $1.5 < PI < 0.7$.

$$PI = \frac{(20 \cdot SP + 500 \cdot \log(P)) - 1952}{SP - 50 \cdot \log(P) + 120} \quad (\text{Eq. 1})$$

Where: PI represents the penetration index [-], SP - softening point [°C]; and Log(P) - decimal logarithm of penetration value at 25°C [0.1 mm].

It is also known that a lower value of the penetration index indicates a higher temperature

sensitivity, the lower value being found when using the chemical additive.

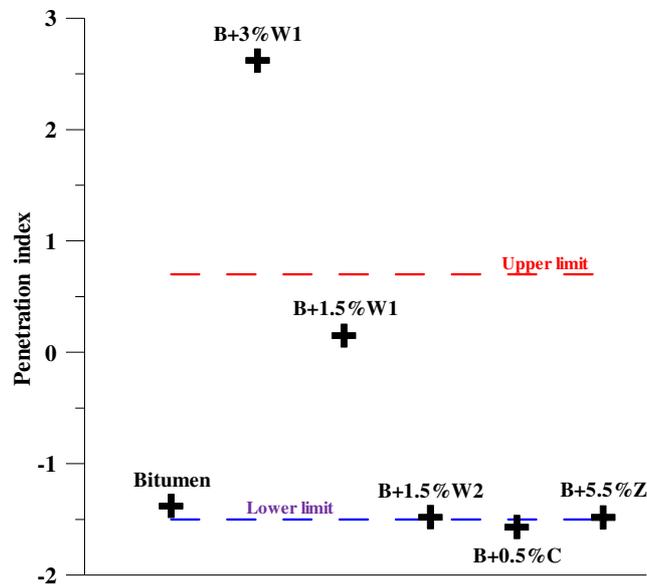


Figure 6. Penetration index for the six bitumen blends

According to the previous results, it is found that the introduction of additives into the bitumen with the purpose of reducing the homogenization temperature of the asphalt mixtures has a certain influence on the modification of the basic characteristics of the binder. Thus, classic wax tends to make bitumen less susceptible to temperature, but with other additives there is almost no difference according to the precision of the tests. The classic wax introduced in a high percentage (3%) leads to the most aggressive modification of the characteristics of pure bitumen, while the other additives do not significantly change these characteristics, they remain within the recommended ranges for 50/70 bitumen.

3.2 Bending Beam Rheometer test results

3.2.1 Bitumen

For the six types of bitumen blends considered, the flexural creep stiffness was determined by means of the bending beam rheometer, in accordance with [38].

Figure 7 shows the results obtained from the BBR test on pure bitumen at the six time steps mentioned before. Further on, for the determination of the allowed limit temperature the values

corresponding to the evaluation time of 60 s are considered.

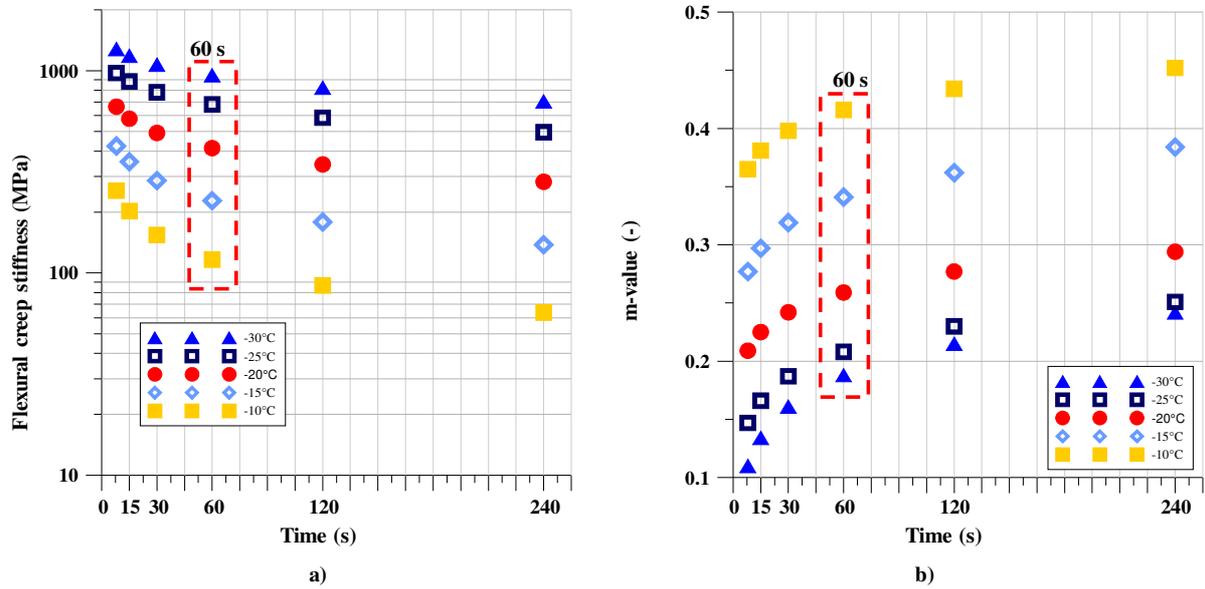


Figure 7. a) Flexural creep stiffness $S(t)$ values for pure bitumen; b) Values of the slope of the stiffness versus time curve, m -value, on pure bitumen

The results obtained for pure bitumen reveal that the flexural creep stiffness increases with decreasing temperature, while the behavior of the slope (m) is inverse, decreasing with decreasing temperature.

The evolution of the temperature-related behavior, for the six bitumen blends, is illustrated in Figure 8. Usually the temperature tests for BBR are selected between -36°C and 0°C , at different intervals. For the present study the following temperatures were chosen: -10°C , -15°C , -20°C , -25°C , -30°C and -35°C .

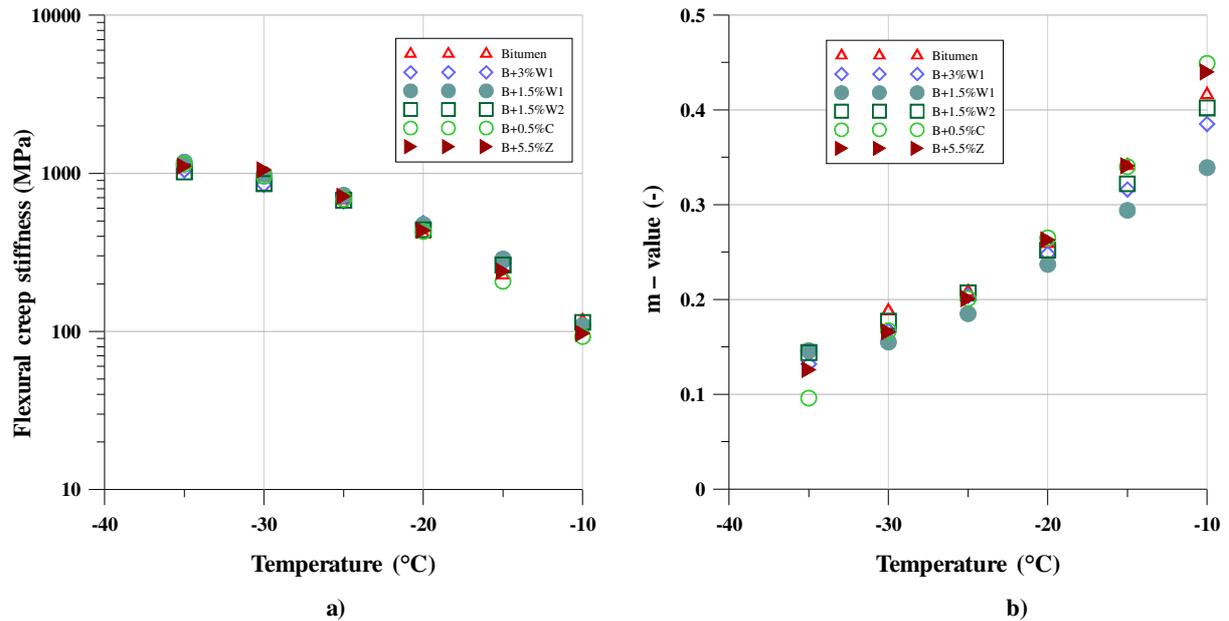


Figure 8. a) Flexural creep stiffness values on bitumen blends; b) Values of the slope of the stiffness versus time curve, *m-value*, on bitumen blends

According to the previous graphs, it can be observed that the tested bitumen blends have relatively close behavior. The following observations can be noted:

- the flexural creep stiffness of the bitumen with additives increases with decreasing temperature, while the behavior of the slope (*m*) is decreasing with decreasing temperature, the same as for the pure bitumen (Figure 8a);
- the tendency of the flexural creep stiffness *S* (*t*) and of the slope *m-value* are almost the same in all six cases (Figure 8b);
- regarding the flexural creep stiffness, at temperatures above -25°C all bitumen blends have values very close to that of pure bitumen, and below this temperature pure bitumen has values higher than blends with 1.5% wax by weight of bitumen, respectively lower than the values of blends with chemical additive, synthetic zeolite and 3 % classic wax by weight of bitumen;
- the samples of pure bitumen have failed at -35°C , which is why no corresponding values are found for this temperature;
- the bitumen blend with synthetic zeolite shows a slightly different flexural creep stiffness behavior at temperatures below -25°C , registering an important increase

up to -30°C compared to the other cases;

- regarding the slope of the stiffness versus time curve, the values closest to those of pure bitumen are found in the case of the bitumen blend with synthetic zeolite, all wax blends have lower values compared to the case of pure bitumen;
- the bitumen blend with chemical additive shows a considerable decrease of the slope of the stiffness versus time curve at -35°C compared to the other blends.

The aforementioned parameters: the flexural creep stiffness and the slope of the stiffness versus time curve are recorded at 60 s during the bending beam rheometer test, and then are used to determine the allowed limit temperature so that cracking at low temperatures is prevented.

For the following values: flexural creep stiffness $S(60\text{s}) = 300\text{ MPa}$, respectively slope $m(60\text{s}) = 0.30$; imposed according to [42], the allowed limit temperature for all bitumen blends was determined and resumed in Table 2. Based on [42], the critical temperature have an accuracy of $\pm 0.1\text{C}$.

Table 2. Allowed limit temperatures after the BBR test on bitumen blends

	Pure Bitumen	B+3%W1	B+1.5%W1	B+1.5%W2	B+0.5%C	B+5.5%Z
Allowed limit temperature for $S(60\text{s}) = 300\text{MPa}$ [$^{\circ}\text{C}$]	-16.9	-15.3	-15.7	-16.1	-17.1	-16.5
Allowed limit temperature for $m(60\text{s}) = 0.3$ [$^{\circ}\text{C}$]	-17.5	-14.3	-16.2	-16.6	-17.7	-17.6
Allowed limit temperature [$^{\circ}\text{C}$]	-16.9	-14.3	-15.7	-16.1	-17.1	-16.5

The allowed limit temperature is set in most cases as given by the stiffness criterion $S(60\text{s})$. The limit temperature corresponding to the bitumen blend with 3% wax is comparable to the results obtained in some laboratories for bitumen 20/30 and the limit temperature corresponding to the other bitumen blends is comparable to the results obtained for bitumen 35/50. [43].

3.2.2 Bituminous mastic

The behavior of the same bitumen blends was evaluated but with the addition of the filler, obtaining 6 types of bituminous mastic (bitumen and filler). The evaluated bituminous mastic mixes are: bituminous mastic without additives (Mastic), bituminous mastic with 3% wax by weight of the binder (Mas+3%W1), bituminous mastic with 1.5% wax by weight of the binder (Mas+1.5%W1), bituminous mastic with 1.5% softer wax by weight of the binder (Mas+1.5%W2), bituminous mastic with 0.5% chemical additive by weight of the binder (Mas+0.5%C), bituminous mastic with 5.5% synthetic zeolite by weight of the binder (Mas+5.5%Z) respectively. In the case of bituminous mastic mixtures, the temperatures for performing the Bending Beam Rheometer test vary between -5 ...-35°C. For the bituminous mastic, the same as in the case of the bitumen, for the determination of the allowed limit temperature the values corresponding to the evaluation time of 60 s are considered.

The flexural creep stiffness increases with decreasing temperature, while the slope of the stiffness curve with respect to time decreases with decreasing temperature, which is also observed in the case of bitumen blends, which indicates that adding the filler does not change the mixing behavior. Figure 9 shows the graphical representations of the flexural creep stiffness and the slope of the stiffness versus time curve for the six bituminous mastic mixes. Also, in this figure these results are compared with the ones obtained for the bitumen blends.

Following the tests performed on bituminous mastic mixes, the following aspects are noted:

- the tendencies of the flexural creep stiffness $S(t)$ and of the slope m -value for mastic mixes are very similar to those of the mastic without additives;
- regarding the creep stiffness, all mastic mixes have values higher than that of the mastic without additives, the only exception being the mastic with chemical additive having a lower value at -30°C;
- with regard to the slope m , up to -20°C, the values for the mastic mixes are lower than those for the simple bituminous mastic (except for the bituminous mastic with chemical additive having slightly higher values), and below this temperature, bituminous mastics with synthetic zeolite, 3% wax and chemical additive have higher values than the classic mastic.

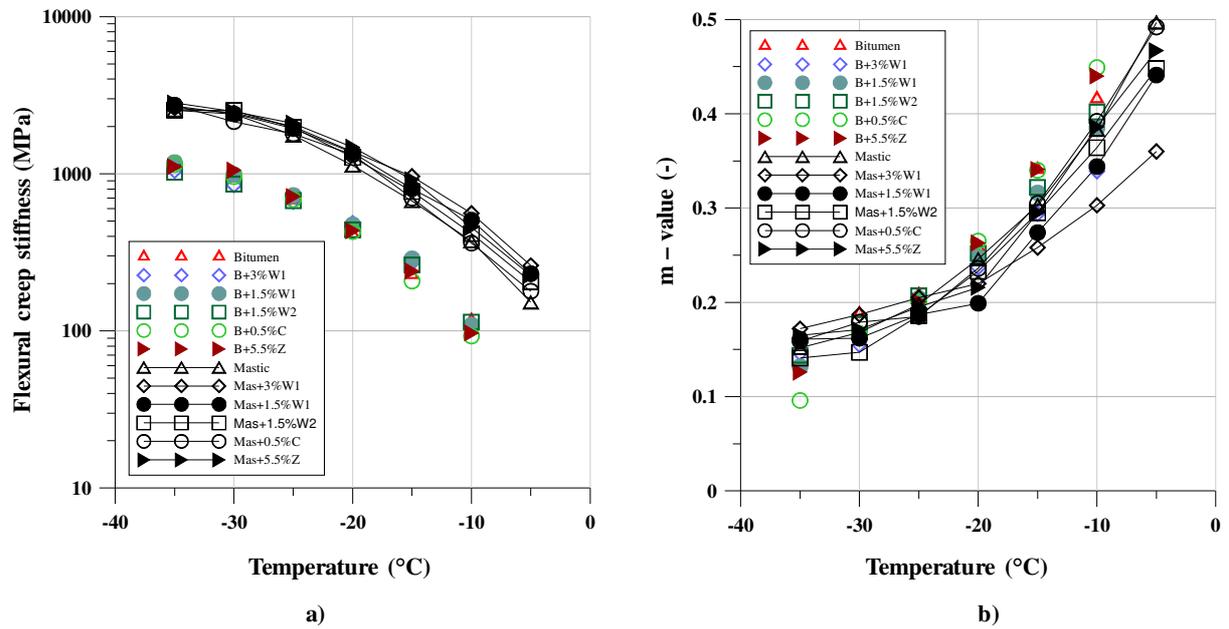


Figure 9. a) Flexural creep stiffness for bitumen vs bituminous mastic with or without additives; b) The value of the slope for bitumen vs bituminous mastic with or without additives

3.2.3 Bitumen vs bituminous mastic comparison

As previously mentioned, Figure 9 presents in parallel the behavior of the six bitumen blends, respectively six bituminous mastic mixes. Comparing the results obtained on bitumen, respectively mastic with additives, the following are noticeable:

- for the pure bitumen, respectively the bituminous mastic without additives, an increase of the flexural creep stiffness is visible, respectively a decrease of the slope m ;
- for the bitumen with 3% classical wax by weight of bitumen and mastic with the same additive we also observe an increase of the flexural creep stiffness, but regarding the slope m an increase is observed in the case of the presence of the filler at temperatures below -20°C ;
- in the case of bitumen with 1.5% classical wax and the mastic with the same additive, we observe, like in the previous case, a considerable increase of the flexural creep stiffness, especially at low temperatures, but regarding the slope m an increase is observed in the presence of the filler at temperatures below -30°C ;

- for bitumen with 1.5% soft wax and mastic with the same additive we observe the same behaviors as in the case of pure bitumen and mastic, with the observation that the flexural creep stiffness is almost constant below the temperature of -30°C;
- for bitumen with chemical additive and mastic with the same additive, there is an increase of the flexural creep stiffness in the case of mastic and a decrease of the slope in the case of mastic with additive with respect to the bitumen with additive over the temperature of -30°C, under this temperature a slight increase is registered;
- the bitumen with synthetic zeolite has a lower flexural creep stiffness compared to the bituminous mastic with the same additive, and with regard to the slope it is lower in the case of synthetic zeolite mastic until the temperature reaches -30°C, following that at lower temperatures it will register a slight increase.

A summary of the results is presented in Table 3.

Table 3. Allowed limit temperatures for bitumen blends and bituminous mastic mixes

Limit temperature [°C]					
Bitumen	B+3%W1	B+1.5%W1	B+1.5%W2	B+0.5%C	B+5.5%Z
-16.9	-14.3	-15.7	-16.1	-17.1	-16.5
Mastic	Mas+3%W1	Mas+1.5%W1	Mas+1.5%W2	Mas+0.5%C	Mas+5.5%Z
-8.4	-5.7	-6.3	-7.3	-8.3	-6.6

The table shows the allowed limit temperatures reached for bitumen blends and mastic mixtures, following the BBR test in all twelve cases. There is a considerable increase in the allowed limit temperature with the addition of the filler in the mixture in all cases.

To highlight the effect of filler on bitumen and bitumen blends, the softening point test has been carried out on all mastic mixes and the results are presented in Table 4.

Table 4. Softening point on bitumen blends vs mastic mixes

Characteristic	Bitumen	B+3%W1	B+1.5%W1	B+1.5%W2	B+0.5%C	B+5.5%Z
Softening point [°C]	47.8	72.8	57.1	48.6	46.9	48.5
Characteristic	Mastic	Mas+3%W1	Mas+1.5%W1	Mas+1.5%W2	Mas+0.5%C	Mas+5.5%Z
Softening point [°C]	56.9	93.6	74.8	59.3	57.5	59.7

The addition of filler to the pure bitumen increased the softening point by 9 °C. The same increase in the limit temperature is observed after the BBR test. The addition of filler to the bitumen blends leads to a higher raise of the softening point, especially in the case of waxes where the temperature increase in softening point is way above the increase of the allowed limit temperature obtained after the BBR test.

4. Summary and conclusions

The laboratory testing with the BBR device of the susceptibility to low temperatures of the binders and bituminous mastics presented above allowed to highlight the fact that the additives considered to be used in the preparation of warm mix asphalts do not lead to significant changes in the rheology of pure bitumen, even if small influences are observed of their presence in mixtures.

The allure and the values obtained for the curves of the flexural creep stiffness and the slope m are close for all the binders tested. It is noticeable that the values of the slope m are very close in the case of bitumen blends, and in the case of pure bitumen, the values are higher. Lower values may indicate that the use of the respective additives leads to a lower capacity of the bitumens obtained to release the accumulated thermal stresses at reduced temperatures.

The allowed limit temperature has resulted for most of the samples studied depending on the temperature at which the limit value of the flexural creep stiffness is reached, and it can be deduced that it mainly controls the development of the thermal stresses. Harder bitumen leads to

higher stresses than softer bitumen, independent of the slope's value. However, for close values of the flexural creep stiffness, the slope m can play an important role in developing stresses at low temperatures.

The allowed limit temperature is slightly higher in the case of bitumen with wax, than in the other cases, the conclusion being that it makes it more susceptible to the temperature. The same is true for bituminous mastic mixes with wax.

In the case of the bituminous mastics tested, the results obtained lead to the same conclusions as for the binders. However, there is an important increase in stiffness, the flexural creep stiffness being 2.5 ... 3.0 times higher in the case of mastics than in the case of binders. Regarding the values of the slope m it is found that they are very close to those obtained on the bitumen blends. Under these conditions, a considerable increase in the allowed temperature limit is observed with the addition of the filler in the mixture in all cases, resulting in a higher susceptibility to the temperature of the bituminous mastic compared to the pure binder used.

The chemical additive is the one that has the lower impact on the bitumen's characteristics, whereas the classic wax is the one that leads to the most important changes. The wax is responsible for a notable increase in softening point and limit temperature, compared to the other studied modifiers.

The addition of filler to bitumen blends leads to a raise of the softening point and of the limit temperature in all studied cases. The same difference can be noticed between the pure bitumen and bituminous mastic in case of the softening point and the limit temperature. This trend can also be noticed in the case of the softer wax, chemical additive and synthetic zeolite. In the case of classical wax, there is a variation of some 10 °C between the differences obtained (bituminous mastic mix - bitumen blend) for the softening point and the limit temperature.

Bibliography

- [1] J. D'Angelo, J. Cowsert, D.D. Newcomb, *Warm-Mix Asphalt: European Practice*, 2008.
- [2] European Asphalt Pavement Association (EAPA), *The Use of Warm Mix Asphalt*, (2014).
- [3] S.D. Capitão, L.G. Picado-Santos, F. Martinho, Pavement engineering materials: Review on the use of warm-mix asphalt, *Constr. Build. Mater.* 36 (2012) 1016–1024. <https://doi.org/10.1016/j.conbuildmat.2012.06.038>.
- [4] M.C. Rubio, G. Martínez, L. Baena, F. Moreno, Warm mix asphalt: an overview, *J. Clean. Prod.* 24 (2012) 76–84. <https://doi.org/10.1016/j.jclepro.2011.11.053>.
- [5] C. Raab, I. Camargo, M.N. Partl, Ageing and performance of warm mix asphalt pavements, *J. Traffic Transp. Eng. Engl. Ed.* 4 (2017) 388–394. <https://doi.org/10.1016/j.jtte.2017.07.002>.
- [6] C. Romanescu, C. Răcănel, *Reologia lianților bituminoși și a mixturilor asfaltice*, Romania, 2003.
- [7] T. Yi-qiu, Z.-H. Li, X.-Y. Zhang, Z.-J. Dong, Research on High and Low-Temperature Properties of Asphalt-Mineral Filler Mastic, *J. Mater. Civ. Eng. - J MATER Civ. ENG.* 22 (2010). [https://doi.org/10.1061/\(ASCE\)MT.1943-5533.0000015](https://doi.org/10.1061/(ASCE)MT.1943-5533.0000015).
- [8] R. Lackner, M. Spiegl, R. Blab, J. Eberhardsteiner, Is Low-Temperature Creep of Asphalt Mastic Independent of Filler Shape and Mineralogy?—Arguments from Multiscale Analysis, *J. Mater. Civ. Eng. - J MATER Civ. ENG.* 17 (2005). [https://doi.org/10.1061/\(ASCE\)0899-1561\(2005\)17:5\(485\)](https://doi.org/10.1061/(ASCE)0899-1561(2005)17:5(485)).
- [9] A. Nikolaidis, *Highway Engineering: Pavements, Materials and Control of Quality*, CRC Press, 2014. <https://books.google.ro/books?id=BiXcBQAAQBAJ>.
- [10] J.-F. Corté, H.D. Benedetto, *Matériaux routiers bitumineux : Tome 1, Description et propriétés des constituants*, Hermes Science Publications, France, 2004.
- [11] G. Lucaci, I. Costescu, F. Belc, *Construcția drumurilor*, Romania, 2000.
- [12] J.C. Petersen, *Binder characterization and evaluation*, Strategic Highway Research Program, National Research Council, Washington, D.C., 1994.
- [13] D. Lesueur, Rhéologie des bitumes: Principes et modifications, *Rhéologie.* 2 (2002) 1–30.
- [14] M. Nivitha, M. Krishnan, Rheological characterisation of unmodified and modified

- bitumen in the 90–200°C temperature regime, *Road Mater. Pavement Des.* (2018) 1–18. <https://doi.org/10.1080/14680629.2018.1552890>.
- [15] M. Marasteanu, T. Clyne, J. McGraw, X. Li, R. Velasquez, High-Temperature Rheological Properties of Asphalt Binders, *Transp. Res. Rec.* 1901 (2005) 52–59. <https://doi.org/10.3141/1901-07>.
- [16] Asphalt, Pavement Interact. (n.d.). <https://pavementinteractive.org/reference-desk/materials/asphalt/> (accessed August 12, 2020).
- [17] D. Lesueur, The colloidal structure of bitumen: Consequences on the rheology and on the mechanisms of bitumen modification., *Adv. Colloid Interface Sci.* 145 (2008) 42–82. <https://doi.org/10.1016/j.cis.2008.08.011>.
- [18] J.G. Speight, *Asphalt Materials Science and Technology*, Butterworth-Heinemann, 2015.
- [19] K. Peterson, B. Bury, C. Duininck, D. Holt, A. Johnson, D. Johnson, R. Kjonaas, M. Marasteanu, M. Marti, J. Quade, G. Skok, D. Van Deusen, R.O. Wolters, *Asphalt Paving design guide*, Minnesota Asphalt Pavement Association (MAPA), USA, 2014.
- [20] T. Mezger, *Applied Rheology – With Joe Flow on Rheology Road*, 1st ed., Anton Paar, Austria, 2015.
- [21] K. Błażejowski, J. Olszacki, H. Peciakowski, *Ghidul bitumurilor*, (2013).
- [22] H. Bahia, D. Anderson, The Development of the Bending Beam Rheometer; Basics and Critical Evaluation of the Rheometer, in: J. Hardin (Ed.), *Phys. Prop. Asph. Cem. Bind.*, ASTM International, West Conshohocken, PA, 1995: pp. 28–50. <https://doi.org/10.1520/STP18187S>.
- [23] Bending Beam Rheometer, Pavement Interact. (n.d.). <https://pavementinteractive.org/reference-desk/testing/binder-tests/bending-beam-rheometer/> (accessed August 12, 2020).
- [24] A. Subhy, Advanced analytical techniques in fatigue and rutting related characterisations of modified bitumen: Literature review, *Constr. Build. Mater.* 156 (2017) 28–45. <https://doi.org/10.1016/j.conbuildmat.2017.08.147>.
- [25] Dynamic Shear Rheometer, Pavement Interact. (n.d.). <https://pavementinteractive.org/reference-desk/testing/binder-tests/dynamic-shear-rheometer/> (accessed August 12, 2020).
- [26] Direct Tension Tester, Pavement Interact. (n.d.). <https://pavementinteractive.org/reference-desk/testing/binder-tests/direct-tension-tester/>

(accessed August 12, 2020).

- [27] Z. You, J. Mills-Beale, E. Fini, S.W. Goh, B. Colbert, Evaluation of Low-Temperature Binder Properties of Warm-Mix Asphalt, Extracted and Recovered RAP and RAS, and Bioasphalt, *J. Mater. Civ. Eng.* 23 (2011) 1569–1574. [https://doi.org/10.1061/\(ASCE\)MT.1943-5533.0000295](https://doi.org/10.1061/(ASCE)MT.1943-5533.0000295).
- [28] K.A. Ghuzlan, M.O.A. Assi, Sasobit-Modified Asphalt Binder Rheology, *J. Mater. Civ. Eng.* 29 (2017) 04017142. [https://doi.org/10.1061/\(ASCE\)MT.1943-5533.0001996](https://doi.org/10.1061/(ASCE)MT.1943-5533.0001996).
- [29] H. Kim, S.-J. Lee, S. Amirkhanian, Effects of warm mix asphalt additives on performance properties of polymer modified asphalt binders, *Can. J. Civ. Eng.* 37 (2010) 17–24. <https://doi.org/10.1139/L09-118>.
- [30] J. Liu, P. Li, Low Temperature Performance of Sasobit-Modified Warm-Mix Asphalt, *J. Mater. Civ. Eng.* 24 (2012) 57–63. [https://doi.org/10.1061/\(ASCE\)MT.1943-5533.0000347](https://doi.org/10.1061/(ASCE)MT.1943-5533.0000347).
- [31] Ding Haibo, Qiu Yanjun, Rahman Ali, Low-Temperature Reversible Aging Properties of Selected Asphalt Binders Based on Thermal Analysis, *J. Mater. Civ. Eng.* 31 (2019) 04018402. [https://doi.org/10.1061/\(ASCE\)MT.1943-5533.0002625](https://doi.org/10.1061/(ASCE)MT.1943-5533.0002625).
- [32] R. Velasquez, A. Zofka, M. Turos, M. Marasteanu, Bending beam rheometer testing of asphalt mixtures, *Int. J. Pavement Eng.* 12 (2011) 461–474. <https://doi.org/10.1080/10298430903289956>.
- [33] A. Cannone Falchetto, M. Marasteanu, S. Balamurugan, I. Negulescu, Investigation of Asphalt Mixture Strength at Low Temperatures with the Bending Beam Rheometer, *Road Mater. Pavement Des.* 15 (2014) 28–44. <https://doi.org/10.1080/14680629.2014.926618>.
- [34] EN 1426:2015, Bitumen and bituminous binders. Determination of needle penetration, (2015).
- [35] EN 1427:2015, Bitumen and bituminous binders. Determination of the softening point. Ring and Ball method, (2015).
- [36] SR 61:1997, Bitumen. Test of ductility, (1997).
- [37] EN 12591:2009, Bitumen and bituminous binders. Specifications for paving grade bitumens, (2009).
- [38] EN 14771:2012, Bitumen and bituminous binders. Determination of the flexural creep stiffness. Bending Beam Rheometer (BBR), (2012).
- [39] D.A. Anderson, T.W. Kennedy, Development of SHRP binder specification, in: *Proc.*

Assoc. Asph. Paving Technol., 1993: pp. 481–507.

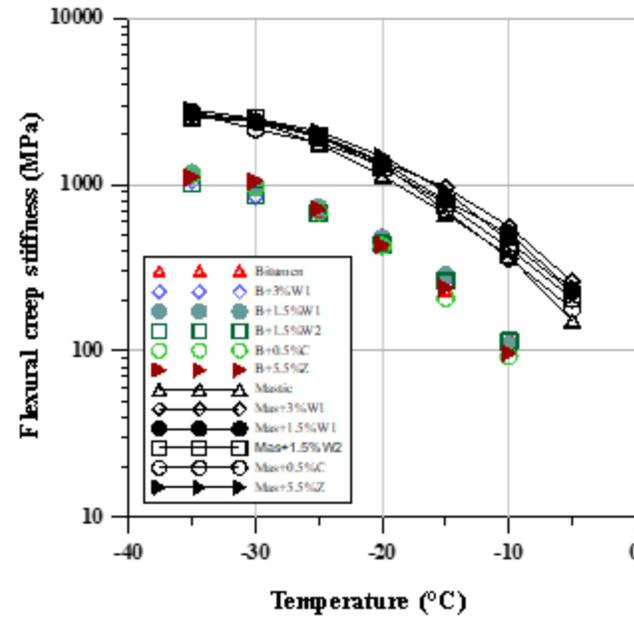
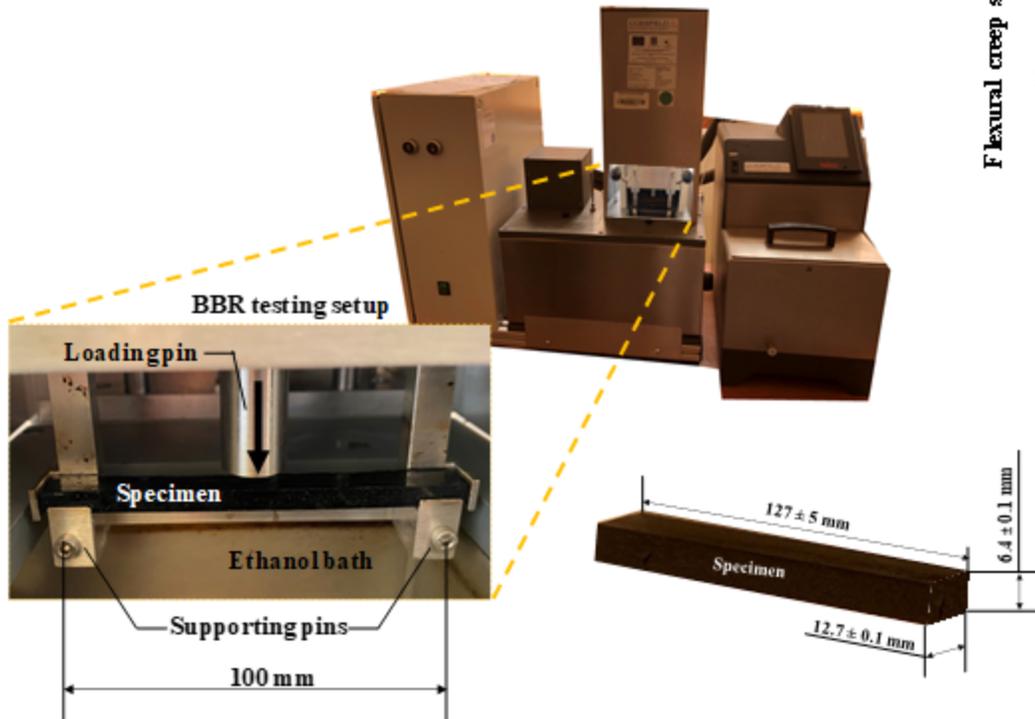
[40] M. Marasteanu, A. Basu, Stiffness m-value and the Low Temperature Relaxation Properties of Asphalt Binders, *Road Mater. Pavement Des.* 5 (2004) 121–131. <https://doi.org/10.1080/14680629.2004.9689966>.

[41] M. Marasteanu, Role of Bending Beam Rheometer Parameters in Thermal Stress Calculations, *Transp. Res. Rec.* 1875 (2004) 9–13. <https://doi.org/10.3141/1875-02>.

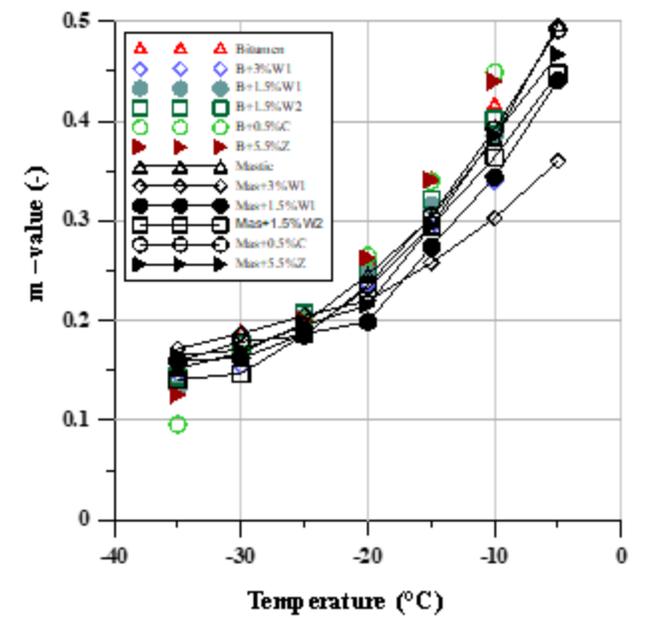
[42] AASHTO T 313, Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR), (2019).

[43] F. Migliori, A.F. Maia, F. Migliori, Essai de fluage par flexion sur éprouvette de bitume à basse température Bending Beam Rheometer Essais inter-laboratoires, (1999) 7.

Influence of warm mix additives on the low-temperature behavior of bitumen using the Bending Beam Rheometer (BBR)



a)



b)

a) Flexural creep stiffness for bitumen vs bituminous mastic with or without additives; b) The value of the slope for bitumen vs bituminous mastic with or without additives