



Evaluation of the potential use of waste sunflower and rapeseed oils-modified natural bitumen as binders for asphalt pavement design

Saannibe Ciryle Somé^{a,*}, Alexandre Pavoine^a, Emmanuel Chailleux^b

^a CEREMA, Laboratoire Eco-Matériaux (LEM), 120 route de Paris, BP 2016, Sourdun 77487 Provins Cedex, France

^b LUNAM université, IFSTAR, route de Bouaye, CS4 44344 Bouguenais Cedex, France

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1. Introduction

Bitumen is the common binder for road building processes. It consists of about 5% of the asphalt concrete mass but provides the major viscoelastic properties needed for the good behavior of the pavement structure. The mechanical performance and the durability, in terms of aging of the asphalt concrete, depend on the choice of the binder and the time evolution of its properties. So, the choice of the binder is of prime interest to guarantee a minimum performance of the pavement structure.

The bitumen consumption is estimated to be about 85 Mt/year in the world. 90% of this consumption is dedicated to the road applications [1]. Bitumen is an oil-refining by-product, so, its production is seen as a consumption of nonrenewable natural resources with a high impact on the CO₂ emissions. Since the last decades, the research on the development of alternative binders' gains interest for several reasons:

- natural nonrenewable resource saving,
- recycling industrial waste products.

This study focuses on the use of vegetable oils and natural bitumen to produce asphalt binder for mixes.

To our knowledge, no study deals with the binder produced with vegetable oils and natural asphalt. Only Yasar

[2] studied the properties of binders produced by mixing thinner oil, gilsonite and soft bitumen.

Traditionally, natural asphalt is used as additive to modify paving grade bitumen or as a part of the filler in the grading curve (in the mineral fractions) [3].

According to Yilmaz et al. [4], the addition of 60% Trinidad lake natural bitumen (TLA) or 10% Iranian' Gilsonite or 9.5% USA' gilsonite into a PG58-34 bitumen provides a new binder. A series of mechanical tests (water sensitivity, resistance to fatigue, rutting, direct tensile strength and stiffness modulus) conducted show an improvement of their performance compared with those obtained with reference bitumen. Ameri et al. [5] highlighted the gradual improvement of the bituminous binder high temperature stiffness due to the addition of 4%, 8% and 12% of gilsonite into bitumen. The conclusions regarding the influence of the gilsonite on the low temperature behavior are contradictory [5,6]. However, no positive impact of gilsonite additive on the low temperature performance has been reported. Recently, Themeli et al. [7] found that Albanian' Selenizza natural bitumen was a good hardener and an aging inhibitor for soft bitumen (with natural asphalt content less than 10%).

Independently to the studies of natural asphalt, recent papers show the capability of the vegetable oils to rejuvenate aged bitumen and to provide more flexibility to the final product [8,9]. The conclusions of these studies are supported by engineering properties (penetration, softening temperature R&B), rheological properties (complex modulus, ductility, creep test) and fatigue resistance measurement. Similar results have been obtained, on site and in laboratory, by Bailey et al. [9] when adding 5% vegetable oil (UVO and

* Corresponding author.

E-mail addresses: ciryle.some@cerema.fr, ciryle.some@gmail.com (S.C. Somé).

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Nomenclature

a_T	shift factor	R	rheological index
$C1, C2$	constant of the Williams–Landel–Ferry’s law	$S(t)$	stiffness modulus, MPa
DT-CY	direct tensile cylindrical load	T	temperature, °C
E^*	tensile complex modulus, MPa	T_g	glassy transition temperature, °C
F	frequency, Hz	T_f	melting temperature, °C
G^*	shear complex modulus, MPa	T_{ref}	reference temperature, °C
G_g	glassy modulus, MPa	<i>Greek symbols</i>	
G_c	modulus at ω_c , MPa	ν	Poisson’s ratio
H	plate thickness, mm	ω_c	cross over frequency, Hz
i/C	water sensitivity	$\sigma(t)$	stress, MPa
MG	multigrade bitumen	$\varepsilon(t)$	strain
m_{ij}	local deformation, mm	τ_β	relaxation time, s
m_{0j}	initial depth measurement, mm	<i>Subscripts</i>	
n	number of values of local deformation	i	plate index
P_i	proportional rut depth, %	j	index of local measurements
$P_{X/Y}$	penetration class of the bitumen, 0.1 mm		
R&B	ring and ball temperature, °C		

Vegetex) into aged bitumen. Moreover, the increase of the percentage of vegetable oils provides sometime better properties than the use of the original bitumen. According to the study of Sun et al. [10], the asphaltene content in vegetable oils is negligible (<1%) compared to that of bitumen. The Aromatics, Resins and Saturates fractions contents in vegetable oils are relatively close to those of the petroleum bitumen. They found a good compatibility between vegetable oils and bitumen under static heated/storage conditions. Moreover, a significant improvement of the stress relaxation of the final binder has been observed. A first chemical and rheological investigation into the role of waste rapeseed and linseed oil into P50/70 bitumen has been addressed by Tachon [11]. She gives the potential chemical reaction between the vegetable oils and the bitumen components.

This study aims to develop new binders by blending Selenizza’ natural bitumen and two waste vegetable oils (rapeseed and sunflower). After the optimization of the composition of the constituent materials of the binders to get similar needle penetration as P35/50 paving grade bitumen, the rheological and the mechanical properties of the binders and the mixes will be characterized.

This paper is organized as follows:

The first part focuses on the binders’ characterization and the second part deals with the mixes’ properties and performances assessment.

Conclusions regarding the conformity of the performances to the requirements defined in the product standard EN 13108-1 (2007) will be drawn.

2. Objectives

The objective of this study is to modify natural asphalt to produce bituminous binders for pavement design. To achieve this, waste vegetable oils have been used to soften

hard natural asphalt. The results in terms of mechanical performances of the produced asphalt mixes are expected to fulfill the requirements of the product standard EN 13108-1 (2007) in terms of water sensitivity, resistance to permanent deformation and stiffness modulus.

3. Materials

The binders consist of natural asphalt (asphaltite) from Albanian’s Selenizza quarry mixed with waste rapeseed or sunflower oil and hard bitumen P15/25. The composition of the blended binders is given in Table 1. This composition has been optimized in the preliminary study to select binders whose penetrations are between 35 and 50 (0.1 mm) as common bitumen used in France. The natural asphalt consists of a blend of hydrocarbon fraction (80%) and mineral fractions (15–18%) [7] which provide hardness to the asphaltite. The penetration of the natural asphalt is nearly zero ($P \approx 0$ mm). Hard bitumen P15/25 has been added to the mixture. The quantity of hard bitumen added is equal to the quantity of mineral filler in the natural asphalt. The use of waste vegetable oils allows softening the final binder. Fig. 1(a) and (b) shows some of the constituents’ materials used to produce the binders.

The binder manufacturing process consists of:

- preheating the natural asphalt into a mixer at 190 °C,
- addition of the waste oil and the hard P15/25 bitumen into the melted natural bitumen,
- mixing these constituents for 30 min’ duration to get homogeneous binder (see Fig. 1c).

A minimum mixing time is required to ensure intimate mixture. However, a long mixing time induces loss of vola-

Table 1
Composition of binders.

Constituent materials	Natural bitumen		Waste vegetable oil	Hard bitumen
	Hydrocarbon	Mineral fraction		
Percentage	60.7%	10.7%	17.9%	10.7%



Fig. 1. Main constituents of binders.

tile compounds and the final binder cannot be used to produce a compactable asphalt mixture.

In this paper, the notation $P_{X/Y}$ means that the needle penetration is between X and Y expressed in 0.1 mm (according to EN 1426).

4. Binders' characterization

4.1. Engineering properties

Two binders have been produced respectively with waste rapeseed and sunflower oil. Their composition complies with Table 1. Their penetration and softening temperature have been measured and represented in Fig. 2(a). The results show that the sunflower oil binder is harder than the rapeseed oil binder. Nevertheless, their penetrations are close to the P35/50 petroleum bitumen commonly used. One can note that their softening temperatures exceed the values obtained from the conventional petroleum bitumen. Therefore, these binders cannot be classified as conventional bitumen. The higher softening temperature of these binders is due, in part, to the mineral filler of the natural bitumen ($R\&B_{\text{asphaltite}} = 119\text{ }^\circ\text{C}$ [7]). The penetration versus softening temperature, represented in Fig. 2(b), shows the position of the produced binders compared to the paving grade bitumen (P35/50 to P160/220), the hard bitumen

(P10/20, P15/25) and the multigrade bitumen (MG 20/30-64/74, MG 35/50-57/67) (defined in the standards EN 12591, EN 13924). It can be observed that the produced binders cannot be classified between the standardized bitumen because of their higher softening temperatures for a given needle penetration.

4.2. Volatility of the constituents of the binders

The volatility of the binders' constituents have been investigated by a thermo-gravimetric analysis (TGA) and represented in Fig. 3. Six samples have been characterized: rapeseed and sunflower oils, asphaltite, hard bitumen P15/25 and the two produced binders. The tests have been carried out from 32 °C to 800 °C at a constant heating rate of 10 K/min. As expected, all the constituents seem non-volatile for temperature $T < 250\text{ }^\circ\text{C}$. These results prove that no significant degradation of the constituents of the binders occurs during the binders' production and during the mixes manufacturing at 190 °C.

4.3. Differential scanning calorimeter analysis

Differential scanning calorimeter (DSC) has been performed to investigate the thermal behavior of the binders. The samples have been heated, from $-80\text{ }^\circ\text{C}$ to $-200\text{ }^\circ\text{C}$ at

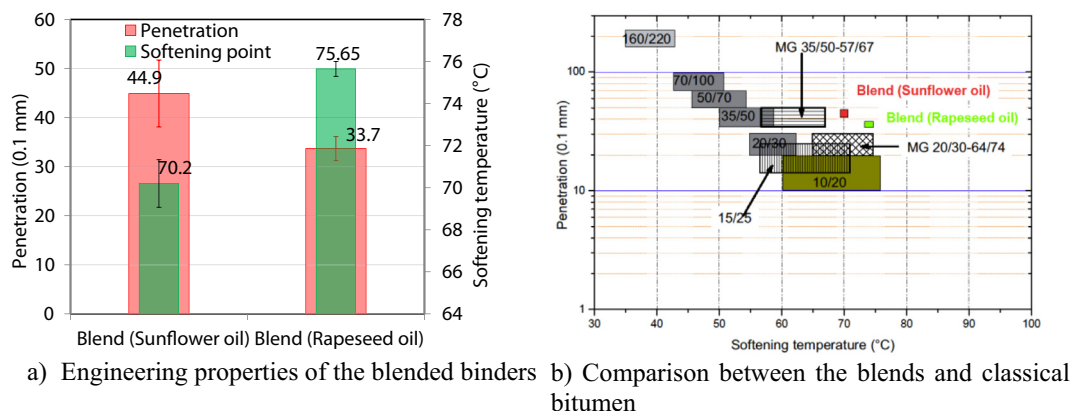


Fig. 2. Classification of the produced binders according to their engineering properties.

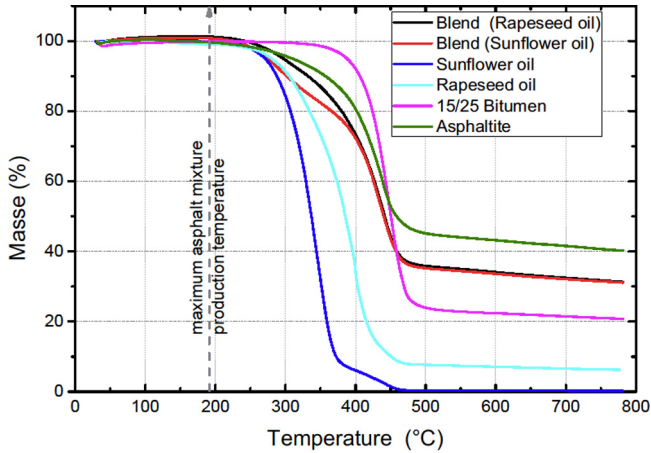


Fig. 3. Evolution of the mass of the constituents of the binders' versus temperature.

a heating rate of 10 K/min. Table 2 gives the glassy transition temperatures T_g and the melting temperatures T_f of the binders constituent materials. Only the glassy transition temperatures for low and ambient temperature condition have been reported. It can be noted that the waste vegetable oil's glassy transition temperatures are lower than those of the bitumen. In addition, the produced binders have lower glassy temperatures than of the original bitumen. This could contribute to increase the lower temperature behavior of the produced binders. However, it was found that P15/25 and P35/50 bitumen have very different T_g , probably due to the difference of their origins.

4.4. Complex modulus

Frequency sweeps have been carried out from 1 to 80 Hz at 10 different frequencies using Metravib apparatus. From $-20\text{ }^\circ\text{C}$ to $20\text{ }^\circ\text{C}$ a cyclic tensile–compression test has been performed on the specimen (see Fig. 4 left) to determine the tensile–compression complex modulus E^* . From $20\text{ }^\circ\text{C}$ to $60\text{ }^\circ\text{C}$, the shear test has been carried out (see Fig. 4 right) to determine the shear complex modulus G^* . E^* and G^* are linked by the following relation: $G^* = E^*/(2(1 + \nu))$. The values of the stiffness modulus E^* for the shear test have been calculated from G^* values by applying an arbitrary Poisson's ratio $\nu = 0.5$, then $E^* = 3G^*$.

Table 2
Glassy transition and melting temperature of the constituent materials.

Material	Glassy transition temperature (T_g)	Melting temperature (T_f)
Waste rapeseed oil	$-30.90\text{ }^\circ\text{C}$	$-2.6\text{ }^\circ\text{C}$
Waste sunflower oil	$-44.3\text{ }^\circ\text{C}$	$-5.65\text{ }^\circ\text{C}$
P15/25 bitumen	$-24.07\text{ }^\circ\text{C}$	–
P35/50 bitumen	$16.4\text{ }^\circ\text{C}$	–
Bend with waste rapeseed oil	$-37.8\text{ }^\circ\text{C}$	–
Bend with waste sunflower oil	$-35.1\text{ }^\circ\text{C}$	–

The results have been represented in Black and Cole–Cole spaces to check the validity of the time–temperature superposition principle (TTSP) (see Fig. 5(a) and (b)). The black space diagram is a convenient way to quantify the changes in the rheological behavior of a bituminous binder due to vegetable oil, additives, aging, etc. The produced binders exhibit a lower phase angle for any given modulus value. This behavior generally characterizes a binder which becomes less viscous or more brittle. However, for the produced binders, this correlation is not proved because their phase angles are higher than reference binder phase angle for high dynamic modulus $|E^*|$ (obtained for high frequencies or low temperatures) as represented in Fig. 5(a). The relatively continuous evolution of the experimental data for each binder in Fig. 5(a) and (b), confirms the validity of the TTSP and allows building the master curves of the complex modulus and the phase angles. Due to the validity of the TTSP, the shift factor a_T values may be approximated by the Williams–Landel–Ferry (WLF) equation [12].

$$\log(a_T) = -\frac{C_1 \cdot (T - T_{ref})}{C_2 + (T - T_{ref})} \quad (1)$$

where C_1 and C_2 are two empirical constants depending of the material and independent to the choice of T_{ref} . Then, the construction of the master curve implies the determination of these constants. The free viscoanalysis[®] software developed at IFSTTAR based on the procedure described in references [13] has been used to build the master curves at a reference temperature of $15\text{ }^\circ\text{C}$. C_1 and C_2 have been found to be respectively 16.27 and 145.24 for the sunflower binder and 14.65 and 131.1 for the rapeseed oil binder.

The binders' complex modulus and phase angle master curves are represented in Fig. 6. It appears that the reference bitumen is stiffer than the produced binders in the temperature range between $-20\text{ }^\circ\text{C}$ and $60\text{ }^\circ\text{C}$. However, the blended binders have relatively similar stiffnesses regarding the accuracy of the data. The evolution of the phase angles represented in Fig. 6, shows that the blended binders have lower phase angles than reference bitumen for the reduced frequency $a_T \times f \leq 2.5\text{ Hz}$ (e.g. $T \geq 20\text{ }^\circ\text{C}$) and higher phase angle for the reduced frequency $a_T \times f \geq 2.5\text{ Hz}$ (e.g. $T \leq 20\text{ }^\circ\text{C}$). Moreover, it can be observed that the produced binders' phase angles are not equal zero, this means that the viscous effects are not negligible compared to reference bitumen. Then, at low temperatures they cannot be assumed to be a purely elastic material. This could be an advantage for low temperature stress relaxation compared to the reference bitumen.

To compare the rheological behavior of the binders, the rheological index (R) and the crossover frequency ($\omega_c = 1/\tau_\beta$) have been assessed and given in Table 3. The crossover frequency (ω_c) is defined as the frequency at which the phase angle is 45° at the reference temperature. It is an indicator of the hardness of the bituminous binder. Lower (ω_c) values indicate a softer binder. τ_β is the

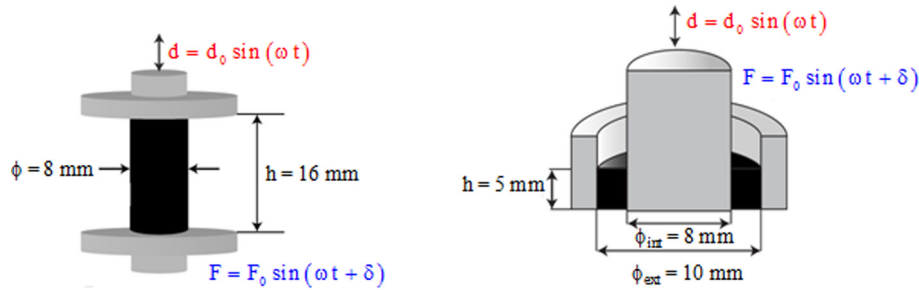


Fig. 4. Principle of the measurement of the binders' complex modulus with Metravis apparatus: tensile-compression test (left) and shear test (right).

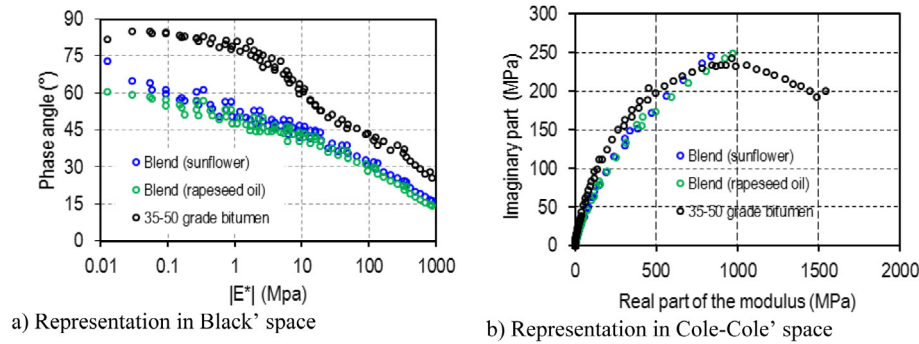


Fig. 5. Evaluation of the applicability of the TTSP based on Black' and Cole-Cole' representations.

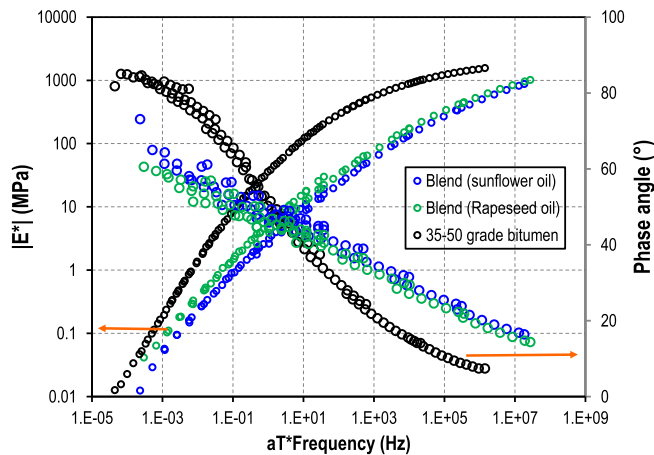


Fig. 6. Binders complex modulus and phase angle master curves at 15 °C.

relaxation time. The rheological index (R) is an indicator of the flatness of the master curves and is given by $R = G_g - G_c$, where G_g is the glassy modulus and G_c the modulus at ω_c [14]. As shown is Table 3, all R values of the produced binders are higher than the R value of reference binder.

5. Mixes characterization

French asphalt concrete BBSG 3, 0/10 (define in EN 13108-1 (2007)) has been manufactured according to the mix composition described in Table 4. The mixture is obtained by mixing the aggregates and the binder during 3 min at 190 °C. According to the laboratory mixing stan-

dard EN 12697-35, which defines the manufacturing temperature, a P35/50 penetration bitumen shall be heated to a maximum temperature of 170 °C. However, it has been found that this standard is not applicable to the produced mixes because the produced binders are still viscous at 170 °C compared to a P35/50 petroleum bitumen. At 190 °C these binders are relatively liquid and allow good coating of the aggregates.

5.1. Resistance to permanent deformation

The resistance of a bituminous material to permanent deformation is grasped by measuring the rut depth induced by the repeated passage of a wheel on a mix plate at a reference temperature of 60 °C according to the standard EN 12697-22 (Large size device). It allows simulating in the laboratory the effect of traffic on a roadway. The plates used have been manufactured and compacted by a roller compactor. For each asphalt concrete, the test has been carried out on two plates. The temperature has been measured during the experiment. The rut depth has been measured with a depth gauge as a function of passes. The first 1000 passes are carried out at room temperature, and the others at 60 °C.

The proportional rut depth, P_i (in percent) of each plate i , has been determined using the following relationship defined in the standard EN 12697-22 for large size device:

$$P_i = \frac{100}{n} \cdot \sum_{j=1}^n \frac{m_{ij} - m_{0j}}{h} \quad (2)$$

Table 3
Rheological parameters of the Christensen's model [14].

Material	Rheological index, R	Crossover frequency ω_c (rad s ⁻¹)
Blend (rapeseed oil)	2.18	1.69
Blend (sunflower oil)	1.91	11.86
35–50 bitumen	1.33	3.9

Table 4
Composition of mixes.

BBSG3, 0/10 according to the EN 13108-1 (2007)	
Granular fractions	Percentage by mass
0/2	26.1%
2/6	23.7%
6/10	42%
Filler (limestone)	1.9%
Binder (asphaltite + waste oil + P15/25 bitumen)	6.3%

P is the measured proportional rut depth in percent (%), h is the plate thickness, n is the number of values of local deformation ($n = 15$ for one specimen), m_{ij} is the local deformation in mm, m_{0j} is the initial measurement at the j location on the surface of the specimen. The average proportional rut depth P is the mean value of the P_i ($i = 1, 2$).

The results are represented in Fig. 7. The rut depths of the mixes manufactured with the produced binders are lower than the control mix rut depth.

According to the standard EN 13108-1 (2007), the percentage of rut depth for a BBSG 3, 0/10 shall be lower than 5% at 60 °C for 30,000 loading cycles. Therefore, the results obtained with the produced binders, represented in Fig. 7, comply with the standard EN 13108-1 (2007). Surprisingly, the rut depths of the produced binders are smaller than the reference mix rut depth. This result seems inconsistent with the evolution of the stiffness modulus. However, we have to clarify that the binders' stiffness modulus test has been carried out from –20 °C to 60 °C and from –10 °C to 40 °C for the mixes (in next section 5.2), while the rut depth has been measured at 60 °C. Then, the rut depth results cannot be compared to the evolution of the stiffness modulus for temperatures lower than 60 °C. At 60 °C (which corresponds to $a_T \times f$ between 10^{-5} and 10^{-3} Hz in Fig. 6, because of the TTSP), the reference binder stiffness is close to the produced binders' stiffness. Further investigations are needed to explain the differences observed in Fig. 7. The better resistances to the permanent deformation obtained with produced binders are probably due to the asphaltite even if the real mechanism that occur is not known yet. This improvement of the resistance to permanent deformation observed is supported by the conclusions of previous studies on the natural asphalts [7,15,16].

5.2. Stiffness modulus

The stiffness moduli have been determined following the standard EN 12697-26 annex E (Direct Tension to

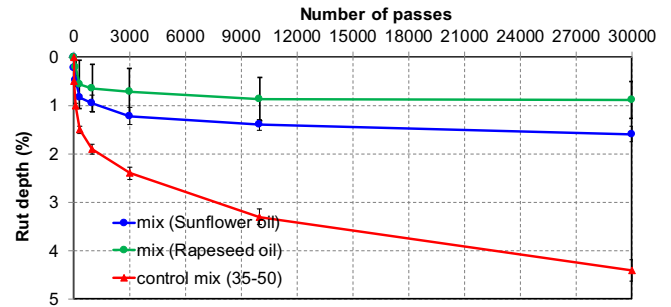


Fig. 7. Evolution of the rut depth.

Cylindrical specimens' DT-CY method). The stiffness modulus test consists of applying uniaxial tensile strain $\varepsilon(t)$ on cylindrical specimens of asphalt concrete (of 200 mm high and 80 mm diameter) and measuring the stress $\sigma(t)$ induced in the specimen. The experiments have been conducted at the following temperatures: –10 °C, 0 °C, 10 °C, 15 °C, 25 °C and 40 °C and for the following loading times: 1 s, 3 s, ..., 300 s. The stiffness modulus $S(t)$ is determined as follows:

$$S(t) = \frac{\sigma(t)}{\varepsilon(t)} \quad (3)$$

The specimens have been cored from plate compacted with roller compactor according to the EN 12697-33.

A preliminary study has been conducted to determine the strain amplitude to be applied during the test to preserve the linear elastic behavior of the samples. According to the standard EN 12697-26, this maximum deformation depends on the temperature as shown in the Table 5. This table gives the maximum strain (50×10^{-6} , 100×10^{-6} , 200×10^{-6} or 300×10^{-6}) to be applied to assess the stiffness modulus in the linear domain according to the temperature and the loading time.

For each temperature and time sweeps, the results have been recorded and used to build the master curves of the stiffness modulus at the reference temperature $T_{ref} = 15$ °C. They have been built by shifting the isothermals in the time domain. They are represented in Fig. 8. It can be seen that the reference mix obtained with the P35/50 bitumen is stiffer than the two others. This is consistent with the evolution of the complex modulus of the binders observed in Fig. 6. However, an inconsistency can be observed due to the difference between the stiffness of the mixes and those of the binders. It can be noted that when the experiment is performed following the recommendation given in Table 5 on specimens which contain rapeseed oil, the linear behavior of the material is not always ensured. Indeed, two specimens containing the rapeseed oil have been broken respectively à 25 °C and 40 °C. More investigations need to be conducted to check the suitability of the recommendations of the standard EN 12697-26 (sum up in Table 5) for this kind of materials.

The estimated values for the stiffness modulus at $T_{ref} = 15$ °C and for a loading time of 0.02 s of the mixes

Table 5
Strain to be applied during a controlled rate test in accordance with the stiffness determined by a preliminary test to 50×10^{-6} (according to EN 12697-26).

Temperature T °C	Stiffness modulus at 10 °C and 3 s		Stiffness modulus at 10 °C and 300 s	
	<7.5 GPa	≥ 7.5 GPa	<1 GPa	≥ 1 GPa
	Amplitude of deformation $\times 10^{-6}$			
$T \leq 10$ °C	100	50	–	–
10 °C $\leq T \leq 20$ °C	–	–	200	100
20 °C $\leq T \leq 40$ °C	–	–	300	200

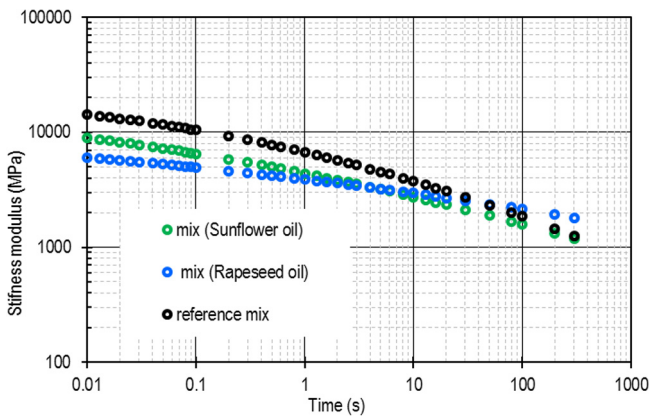


Fig. 8. master curves of the stiffness modulus of the mixes at 15 °C.

($S_{(15^{\circ}\text{C};0.02\text{s})}$) are respectively: 5678 MPa, 8233 MPa and 13171 MPa for the mixes with rapeseed oil, sunflower oil and P35/50 grade bitumen. Then, the stiffness modulus of the rapeseed oil asphalt concrete does not comply with the requirement of EN 13108-1 (2007). Indeed, for a BBSG 3, 0/10, the minimum stiffness modulus should be at least 7000 MPa.

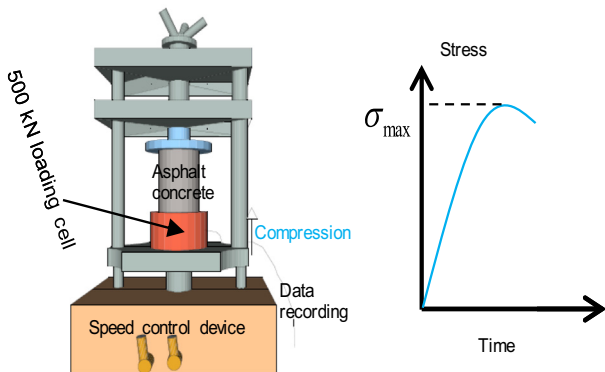
5.3. Water sensitivity

Ten cylindrical samples, with 120 mm diameter, have been manufactured to evaluate the stripping resistance. For each mixture, half of the specimen has been cured in thermoregulated chamber at 18 °C. The other ones have been immersed in water at 18 °C. After seven days' conser-

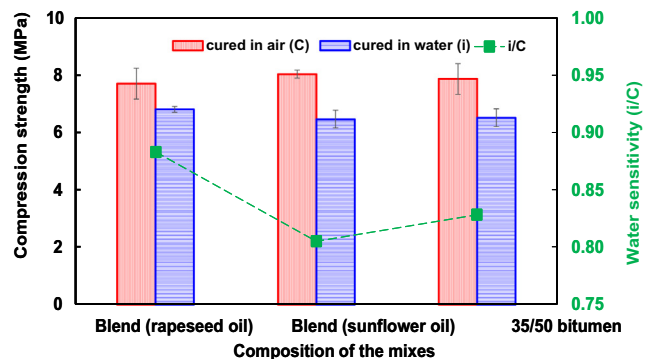
vation, compression test has been carried out on the samples in accordance with the standard EN 12697-12, to measure the failure strength. Fig. 9(a) shows a schematic view of the experimental device used for the compression test [17,18]. The maximum resistances recorded are presented in Fig. 9(b) for samples cured in air (C) and samples cured in water (i). The ratios i/C have been estimated and represented in conjunction with the compression strengths. No significant difference between the vegetable oils mixes and the reference mix is observed regarding the water sensitivity. Since the ratios i/C exceeds 0.7, all the results comply with the standard EN 13108-1 (2007) requirements.

6. Conclusion

The aim of the study presented in this paper was to produce and characterize the properties of new binders. These binders are expected to be used for road building applications. They were produced by mixing 71.4% of natural bitumen, 17.9% of waste vegetables oils and 10.7% of hard petroleum bitumen. This optimal composition was obtained by a preliminary study. The viscoelastic properties of the binders and the mixes were characterized. It was found that the produced binders are softer than the P35/50 petroleum bitumen. In addition, TGA analysis performed indicated a negligible volatility of the vegetable oil compounds during the binders' and the mixes' production. Furthermore, DSC analysis showed a decrease of the final binders' glassy transition temperatures due to the vegetable oils, this could improve low temperature behavior such as thermal cracking.



a) Principle of stripping resistance evaluation [17,18]



b) Compression resistance and water sensitivity of the mixes

Fig. 9. Evaluation of the water sensitivity of the mixes.

Asphalt concretes were manufactured and their stiffness modulus, resistance to permanent deformation and water sensitivity evaluated. The vegetable oil mixes are softer than the reference bitumen mixture. Also, the rutting depths obtained with the reference bitumen is higher than these obtained with the produced vegetable oils. Except the stiffness modulus obtained with the waste rapeseed oil, all the results comply with the standard EN 13108-1 (2007) requirements.

However, this study shall be continued to study the aging, the fatigue and the low temperature properties of these mixes. Further study shall be done to analyze the viability and the economic feasibility of this technology.

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