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Evaluation of different short term aging procedures with neat, warm and modified binders

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HIGHLIGHTS

- Binders modified with polymer and wax were studied.
- The mixing temperature was evaluated through the equiviscous method.
- Short Term Aging procedure was studied with RTFOT at 163 °C and mixing temperature.
- Rheological and chemical properties of unaged and aged bitumens were studied.

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ABSTRACT

This work is based on the characterization of road materials that combine high performance and reduction of the environmental impact during road construction and maintenance. In particular polymer modified bitumens with and without wax additives have been studied. The first increase the elasto-plasticity of the mixture, increasing its durability and fatigue resistance. As for the second the presence of paraffinic additive reduces the bitumen viscosity and consequently the asphalt mixing and compaction temperatures.

A number of studies have shown that the mechanical characteristics of pavement are strongly influenced by the oxidation degree of the organic components of the bitumen during mixing and compaction phases. This phenomenon is known as short-term aging.

Binders modified with polymer and wax were analyzed according to the viscoelasticity theory and different aging conditions were simulated. Tests of advanced rheological characterization were carried out by using the Dynamic Shear Rheometer and the short-term aging was simulated by Rolling Thin Film Oven Test. A new procedure of bitumen aging at the equiviscous temperature or T_{mixing} was proposed. Further rheological tests on aged binders were carried out at this temperature. The results have been supported by FTIR (Fourier Transform Infrared Spectroscopy) analytical chemical tests, analyzing the molecular changes on the aged binders. The standard temperature of 163 °C does not correctly simulate the short term aging of polymer modified and waxes bitumens.

1. Introduction

The binder properties of asphalt mixtures change over time during mixing, compaction and service. In these phases chemical and physical characteristics of bitumen change, in particular oxidation occurs involving the increase of material stiffness. This phenomenon is known as aging and it is one of the key factors affecting the lifetime of an asphalt pavement. Short-term aging is the degradation at hot temperature conditions which occurs during asphalt mix production and field operations. The Rolling Thin Film Oven Test (RTFOT) is the laboratory test method that simulates those conditions. The standard test method is conducted at 163 °C, with the superior limit of temperature necessary to create an optimal adhesion between neat or unmodified bitumen and aggregate during mixing. The Superpave Mix Design and the Marshall mix design requires that specimens are mixed and compacted at equiviscous binder temperatures corresponding to viscosities of 0.17 and 0.28 Pa s, respectively [1]. Those values have been used in the Marshall Mix Design Method (ASTM D 1559) to determine mixing and compaction temperatures. Estimation of proper mixing and compaction temperatures involves developing a temperature viscosity relationship for the binder (ASTM D 2493) [1]. The introduction of waxes and polymers changes the physical properties of

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bitumen; in particular they first reduce the viscosity of the binder, and lower the mixing temperature by 10–30 °C [2]. This technology is classified as warm mix asphalt, and the Fischer–Tropsch waxes with long-chain aliphatic hydrocarbon are characterized by good oxidation and ageing stability [3]. On the contrary the presence of polymers increases the binder viscosity [4]. Simone et al. have evaluated a new RTFOT procedure based on binder viscosity influence that justifies the appropriate laboratory conditions to simulate the effective short term aging during mixing and compaction. The authors have found that the wax binders have a lower value of workability temperatures (T_{work}) than the neat one, and the extracted binder Master Curves are comparable with those obtained from the binders aged at T_{work}, confirming that the RTFOT procedure that follows the EN is not representative of the real short term aging [5]. For this reasons there is a need to define a procedure of RTFOT for modified binders to simulate the real aging at the equiviscous temperature.

The overall objective of this research was to investigate the differences in aging level, in terms of rheological and chemical parameters, between the RTFOT at 163 °C and RTFOT at mixing temperature (T_{mixing}). The research steps were defined as follows:

- a preliminary rheological study of modified bitumen and bitumen with additive was conducted through a dynamic the test like rotational viscometer and Dynamic Shear Rheometer (DSR); the frequency sweep configuration test was adopted to establish the response of binders in term of complex modulus G^* and phase angle δ [6];
- short term aging was simulated with RTFOT at 163 °C;
- short term aging with RTFOT at equiviscous temperature was also assessed;
- dynamic tests were conducted on aged materials to study the variation of rheological parameters after aging. This was necessary to study and validate the new aging method;
- FTIR spectroscopy was used to compare a direct and continued methodological approach of bitumen oxidation process (evaluation of bitumen oxidation).

2. Materials

2.1. Constituent materials

Bitumen 70/100 pen was selected for the analysis and modified as described in Table 1. Warm mix asphalt and polymer asphalt was prepared by adding a paraffinic wax and SBS to the binders. The wax was obtained from coal gasification using the Fisher–Tropsch process.

2.2. Bitumen preparation

The preparation of polymer and wax modified binders includes necessary operations to provide a representative sample of the material. Bitumen is a thermally sensitive material, so the mixing temperature has a great influence on the characteristics of the modification. In order to homogenize the mixture with the polymer, the binder was mixed with SBS at the temperature of 180 °C. The wax (melting point 90 °C) was added at 130 °C. In order to avoid phase separation, the samples were mixed with a Silverson mixer L4R.

2.3. Bitumen characteristics by traditional test

Penetration and Softening Point were conducted in accordance to the European Standards EN 1426-22 and EN 1427-23 (Table 2). SBS and waxes generate a reduction of penetration grade and consequently an increase in softening point. This change in properties is due to crystallization of the introduced wax. The polymer tends to increase the bitumen consistency and this rheological change is even more enhanced with the addition of wax.

Table 1
Materials and tested bitumen code.

Neat bitumen [pen]	Wax [%]	SBS [%]	Bitumen code
Bitumen 70/100	–	–	N
Bitumen 70/100	2	–	W
Bitumen 70/100	–	5	P
Bitumen 70/100	2	5	PW

3. Test methods and procedures

3.1. Rotational Viscometer

A Rotational Viscometer (RV) was used to evaluate the binder viscosity at high temperatures. The RV measures the torque required to rotate a spindle at constant speed while immersed in the simple fluid. Dynamic viscosity is proportional to this measured torque. For all bitumens the dynamic viscosity (η) was calculated in a temperature range between 200 °C and 120 °C. The reading of the viscosity was performed every 5 degrees in the range between 200 °C and 160 °C and every 10° in the range between 160 °C and 120 °C. In order to validate the test, the sample was thermo-regulated 7 min at intervals of 5 °C and 12 min at intervals of 10 °C. Tests were conducted according to EN 13302.

3.2. Dynamic mechanical analysis

Dynamic mechanical analysis, using oscillatory tests, was performed on the four bitumens to determine their rheological properties using a Dynamic Shear Rheometer (DSR). The tests were performed under controlled strain, and the strain amplitude was limited within the linear viscoelastic (LVE) response of the samples. Data were obtained from frequency sweep tests between 0.01 rad/s and 10 rad/s, conducted between 5 °C and 75 °C. The 8 mm measurement system with 2 mm gap was adopted below 35 °C and the 25 mm plate with 1 mm gap at temperatures higher than 35 °C. The rheological parameters obtained from frequency sweep tests were the complex shear modulus G^* and phase angle δ.

3.3. Chemical analysis by FTIR spectroscopy

A PerkinElmer FTIR spectrometer was used to determine the functional characteristics of modified binders before and after aging at the two investigated temperatures and n wavenumbers ranging from 2000 cm^{-1} to 400 cm^{-1}. Binders were dissolved in chloroform $CHCl_3$ with 5 wt.% concentration, then dropped on a sodium chloride NaCl disc for the FTIR analysis.

4. Experimental work

Fig. 1 shows the experimental approach followed for this research.

4.1. T_{mixing} definition by EN 12591 tests

The dynamic viscosity test (EN 13302) was chosen in order to identify the T_{mixing} for the studied binders. The temperature corresponding the viscosity of 0.17 Pa s was assumed as T_{mixing}. Fig. 2 shows the Dynamic Viscosity curve and T_{mixing} evaluation of neat bitumen N, the same procedure was followed for the other binders. Table 3 represents the equviscosity temperature

Table 2
Properties of the bitumens.

Bitumen code	Penetration [dmm] (EN 1426 [22])	Softening point [°C] (EN 1427 [23])
N	87	45
W	66	72
P	52	80
PW	47	88