Test Procedures for Advanced Characterization of Bituminous Binders Employed for Pavement Construction in Public Works Authority Road Projects - State of Qatar

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ABSTRACT

This paper illustrates the approach adopted by the Public Works Authority (Ashghal) of the State of Qatar for the advanced chemical and rheological characterization of bituminous binders (both neat and modified). The ultimate objective of testing is to create a database of employed binders, which may be meaningful for the optimization of the selection of materials and for the consequent enhancement of pavement performance. This paper provides an illustration of employed testing procedure and briefly discusses typical experimental results.

Keywords: Road pavements; Bituminous binders; Test procedures; Chemical characterization; Rheological characterization

1 INTRODUCTION

The road infrastructure network of the State of Qatar has grown significantly in the last few decades, and it has become essential to create a database of the main characteristics and engineering properties of available materials. Such a task has been undertaken by the Quality & Safety Department (QSD) of the Public Works Authority (Ashghal) and by ANAS S.p.A. Qatar Branch as part of the ongoing QA/QC Pavement Consultancy Services contract. Considered materials include those of the "standard" type, which are already fully described in current Qatar Construction Specifications (QCS, 2014), and "alternative" materials which have never been employed in Qatar – or only in experimental trials – and that may be considered for future use.

Since the majority of road pavements constructed in Qatar are of the flexible type, major efforts have been made in order to fully characterize bituminous binders employed for the production of wearing, intermediate and base courses for pavements of all categories. According to the requirements set in (QCS, 2014), these may be either of the "neat" type, or of the "polymer-modified" type (PMBs). However, ongoing developments related to implementation of the so-called "Ashghal Recycling Initiative" (Ashghal, 2018), have recently led to the replacement of PMBs with crumb rubber modified binders (CRMBs) in all projects overseen by the Road Projects Department (RPD) of Ashghal (RPD, 2018).

The State of Qatar does not produce a significant quantity of bitumen from oil refining and therefore has always imported it from foreign Countries. During the entire period of development of the Qatari road infrastructure, bitumen was exclusively imported from a single Country (and a single refinery, which produced it by straight run distillation of a single type of crude). However, since June 2017 bitumen has been supplied from different sources, with the corresponding availability of materials obtained by means of a multitude of crude processing technologies (including air blowing and blending of vacuum tower bottom residues and other petroleum-derived fractions).

As a result of the evolving scenario outlined above, it has become apparent that characterization of bituminous binders employed in the State of Qatar should require the use of advanced testing procedures for the assessment of their intrinsic chemical and rheological properties, which are also affected by ageing. The current characterization approach which is implemented in (QCS, 2014) relies on penetration grading for neat bitumen (as per ASTM D946-15), required to belong to the 60-70 grade, and on the SUPERPAVE system for modified binders (as per AASHTO M 320-17 and AASHTO M 332-14), which have to be classified as PG76-10. While the former system has proven to be scarcely related to actual field performance, the latter one, although conceived with a performance-based character, provides only partial information on the true nature and behavior of bituminous binders, and may therefore fail in capturing relevant differences between products of variable type and origin.

This paper outlines the approach adopted by Ashghal in order to address the issues discussed above, with an illustration of employed testing procedures and typical results obtained in the characterization of different types of bituminous binders, considered in various ageing states. A more comprehensive description of the studies performed on this topic is presented elsewhere (Santagata et al., 2019). Investigations were carried out in the Ashghal Center for Research & Development (ACRD), with the involvement of the Road Materials Laboratory of the Politecnico di Torino, Italy.

2 CHEMICAL CHARACTERIZATION

As part of the advanced approach adopted by the QSD-ANAS team, chemical characterization is carried out, only for neat bitumen, by referring to the classical colloidal scheme proposed by (Nellensteyn, 1928), in which asphaltenes are kept in dispersion in a continuous medium of oils (saturates and aromatics) by means of the peptizing effect of resins which are distributed around asphaltene micelles. The transition of bitumen macromolecules from saturates to aromatics to resins and to asphaltenes occurs with an increase of molecular weight, polarity and aromaticity. The colloidal scheme is a simplification of the true nature of bitumen, which in the reality is formed by a complex and in most part random combination of different macromolecules (Branthaver et al., 1994). Nevertheless, this idealized structure, characterized by given percentages of the four different fractions, can be of support in comparing bituminous binders of different origin, in assessing the effects of ageing, and in explaining some of the specific features of their rheological behavior (Santagata et al., 2009).

Analyses that are consistent with the scheme illustrated above, also known as SARA analyses, are performed by means of an equipment (Iatroscan, model MK-6) which combines thin layer chromatography (TLC) with a flame ionization detection (FID) technique (Leroy, 1989; Ecker, 2001). The adopted testing protocol, validated in the course of previous research work (Santagata et al., 2015), entails the preliminary preparation of solutions of bitumen in dichloromethane with a concentration of 10 mg/ml. A small quantity of these solutions (1 μl) is injected in porous quartz rods covered by a thin film of silica gel. Identification of the four bitumen fractions is thereafter carried out by immerging the rod tips in a set of tanks containing n-hexane, toluene and a mixture of dichloromethane (95%) and methilic alcohol (5%). Finally, percentages of the four fractions, characterized by different polarity, are assessed by means of the FID unit, which operates by measuring the electrical current produced by combustion in hydrogen flame.

Examples of results obtained from SARA analyses are provided in Figure 1, where there are shown in the form of a bar chart (Figure 1a, which contains the relative percentages of all four fractions) and of a triangular chart (Figure 1b, in which saturates and aromatics are considered as a single fraction). Represented data refer to a single neat bitumen (indicated as bitumen A), considered in its three ageing states: virgin (as drawn from plant storage tanks), short-term aged by means of the Rolling Thin Film Oven (RTFO) test as per ASTM D2872-19 and AASHTO T240-13, long-term aged by means of the Pressure Ageing Vessel (PAV) as per ASTM D6521-19 and AASHTO R28-12. Figure 1 also displays other data which have been considered for comparative purposes. These were obtained for two binders which were extracted (as per ASTM D2172-17) from cores taken from an in-service pavement (bitumen B-C) and from reclaimed asphalt pavement (RAP) material retrieved from the stockpile of a local Contractor (bitumen C-R). Results that refer to this last sample are of special interest since in Qatar the use of RAP in asphalt mixes is being strongly encouraged within the "Ashghal Recycling Initiative". Thus, assessing the chemical nature of bitumen contained in RAP is functional for the prediction of its effectiveness as a binder and for the identification of proper rejuvenating treatments (Cong et al., 2016).

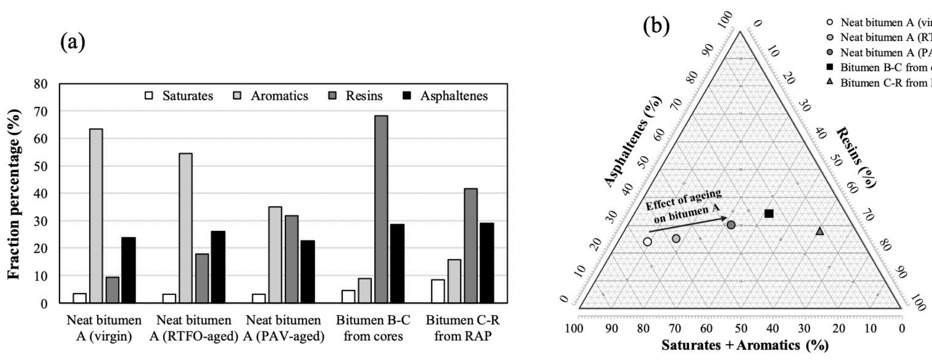

Figure 1: Results of SARA analyses

By considering the results given in Figure 1 it can be observed that SARA analyses clearly describe the evolution of bitumen chemistry as a function of ageing. In particular, as ageing progresses, it causes a gradual reduction of the percentage of dispersing fractions, mainly as a consequence of the transition of part of the aromatics into resins (or asphaltenes) of higher polarity and of the shift of part of the resins to the asphaltene fraction (Eberhardsteiner et al., 2015). Such an evolution is clearly depicted by means of the triangular chart representation, which allows binders coming from different sources and production technologies to be clearly differentiated. Although no information was available on the chemical composition of the bitumen originally contained in the asphalt pavement layer subjected to coring, results obtained on the extracted bitumen are consistent with the observed ageing trends. However, in line with previous findings documented for asphalt mixes employed in Qatar (Sirin et al., 2017), they also suggest that the degree of ageing reached in the field may be superior to that which is mimicked by means of the PAV protocol. Finally, it can be observed that binders contained in RAP, subjected to prolonged ageing, may exhibit a balance between fractions that is completely different from that of all other binders, with an overall reduction of the quantity of oils (saturates and aromatics) and with a significant increase of the percentage of resins. Such an outcome indicates that when using RAP in the production of asphalt mixes it may be necessary to make use of specifically tailored rejuvenating agents, and that for some specific RAP sources the aged binder may be ultimately considered as “black rock” (Chen et al., 2007).

3 RHEOLOGICAL PERFORMANCE-BASED CHARACTERIZATION

According to the approach adopted by the QSD-ANAS team, rheological performance-based tests for the advanced characterization of bituminous binders are carried out by making use of a Dynamic Shear Rheometer (DSR). Employed testing procedures address different aspects of the expected behavior in service, with analyses that are carried out on binders considered in their three characteristic ageing states (virgin, RTFO-aged and PAV-aged).

The following sections contain a description of test protocols and a brief discussion of typical results. Data provided in this paper were obtained by making use of a Physica MCR 302 from Anton Paar Inc., an air-bearing stress-controlled device equipped with a permanent magnet synchronous drive (minimum torque = 0.5 nNm) and an optical incremental encoder for the measurement of angular rotation (resolution = 0.05 μrad).

3.1 Viscoelastic fingerprinting

Viscoelastic fingerprinting of bituminous binders consists in the construction of master curves which provide a full description of their time- temperature- and age-dependent behavior. Such an assessment is of premium importance since parameters obtained from master curve modelling (described in the following) can be related to the synthetic composition parameters derived from SARA analyses (see section 2).

Master curves are obtained by means of frequency sweeps in which the norm and phase angle of the complex modulus is monitored in a wide frequency range (from 1 to 100 rad/s) at temperatures comprised between 4 and 82 °C (with 6 °C increments between each measurement step). Depending upon temperature and frequency, shear strains applied to test specimens are adjusted in order to focus on linear viscoelastic response. A 25 mm parallel plate system is used with a 1 mm gap between the plates at higher test temperatures (34-82 °C), while the 8 mm parallel plate with 2 mm gap is employed at lower temperatures (4-34 °C).

Temperature-dependent shift factors (a_T) are modelled by referring to the following expression, originally proposed by Williams, Landel and Ferry (Ferry, 1980):

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

where C_1 and C_2 are model parameters, T is the generic test temperature, T_{ref} is the reference temperature at which the master curve is evaluated (usually set at 20 °C for all binders).

Measured values of the norm and phase angle of the complex modulus, shifted to the reference temperature, are fitted to the following expression proposed by Christensen and Anderson (1992):

$$G^*(\omega) = G_g[1+(\frac{\omega_c}{\omega})^{\frac{Log2}{R}}]^{-\frac{R}{Log2}} G^*(\omega) = G_g[1+(\frac{\omega_c}{\omega})^{\frac{Log2}{R}}]^{-\frac{R}{Log2}} \tag{2}$$

where $G^*(\omega)$ and $\delta(\omega)$ are the norm and phase angle of the complex modulus at the generic reduced angular frequency ω, G_g is the glassy modulus, ω_c is the crossover frequency, and R is the rheological index.

Examples of obtained results are shown in Figure 2 and in Table 1, which refer to tests carried out on two neat binders of different origin (indicated as D and E) considered in their three ageing states, a PMB (binder F-P), and a binder recovered from RAP (indicated as G-R).

From Figure 2a it can be observed that the two neat binders, although being of the same penetration grade (60-70), exhibit a completely different rheological behavior. In particular, when compared to bitumen D, bitumen E possesses a lower stiffness in all ageing states and is characterized by a lower sensitivity to ageing. By referring to Figure 2b, significant differences can also be found when comparing binders of different type and origin. In particular, binders extracted from RAP may be extremely stiff in the entire range of considered frequencies, while the effects of polymer modification can also be relevant, with a significant change of the master curve shape. These observations can be supported by the critical analysis of the fitting parameters provided in Table 1.

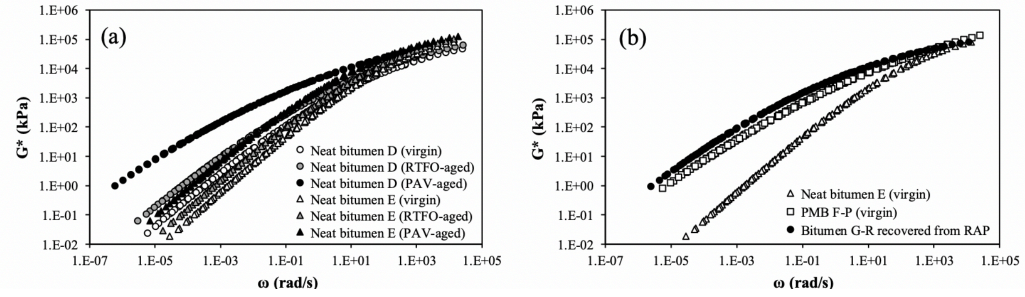

Figure 2: Master curves

Table 1 Master curve parameters

Binder	Type	C_1 (-)	C_2 (°C)	$\log(G_g)$ (kPa)	$\log(\omega_0)$ (rad/s)	R (kPa)
D (virgin)	Neat	15.10	116.87	5.31	1.57	1.65
D (RTFO-aged)	Neat	16.81	126.60	5.53	0.92	1.97
D (PAV-aged)	Neat	31.16	247.87	5.90	-1.55	2.94
E (virgin)	Neat	12.64	110.46	5.42	2.60	1.10
E (RTFO-aged)	Neat	13.98	120.18	5.53	2.28	1.22
E (PAV-aged)	Neat	15.72	127.04	5.60	1.57	1.43
F-P (virgin)	PMB	13.40	89.40	9.93	1.16	6.58
Bitumen G-R	From RAP	23.92	202.49	5.59	-0.61	2.23

3.2 Resistance to permanent deformation

Resistance to permanent deformation is analyzed by performing multiple stress creep recovery (MSCR) tests carried out as per AASHTO T350-19 at temperatures comprised between 52 °C and 76 °C (with 6 °C increments), and two different shear stresses (0.1 and 3.2 kPa). When compared to standard oscillatory tests, MSCR tests are more simulative of in-service loading conditions since they are carried out at higher strain levels, thus allowing a better assessment of rutting potential (D'Angelo, 2009). According to the approach adopted by the QSD-ANAS team, MSCR tests are not simply aimed at identifying temperatures at which AASHTO M 332-14 threshold conditions are met, but rather at giving a full description of the rutting resistance potential in a wide temperature range. Thus, reference is made to the temperature-dependency of percent recovery (R) and non-recoverable shear compliance (J_{nr}), which provide an insight into the degree of elastic response of considered materials.

Examples of obtained results are shown in Figure 3, which refers to tests carried out on two neat binders (indicated as H and I) and two PMBs (J-P and K-P). The two neat binders, of the 60-70 penetration grade and classified as PG64S-22, were of different origin. PMBs, obtained from local Contractors, were reported to contain styrene-butadiene-styrene (SBS) as a modifier and were classified as PG76V-16 and PG76E-22, respectively.

When considering the results obtained at 76 °C (Figure 3a), it can be observed that the two neat bitumens exhibit an almost negligible elastic recovery in the virgin and RTFO-aged state, with no possibility of capturing any significant difference in response. However, in the PAV-aged state they show a different response under repeated loading, the R parameter at 3.2 kPa being equal to 4.25 and 0.76 for bitumen H and I, respectively. Differences between the two PMBs at 76 °C are more noticeable. In particular, it is shown that they are characterized by a different sensitivity to ageing. Additional information on the potential resistance to permanent deformation of the various binders can be extracted from the temperature-J_{nr} plots displayed in Figure 3b. In this case, it is observed that neat bitumens and PMBs can be characterized not only by significantly different values of non-recoverable compliance, but also by a different temperature-susceptibility.

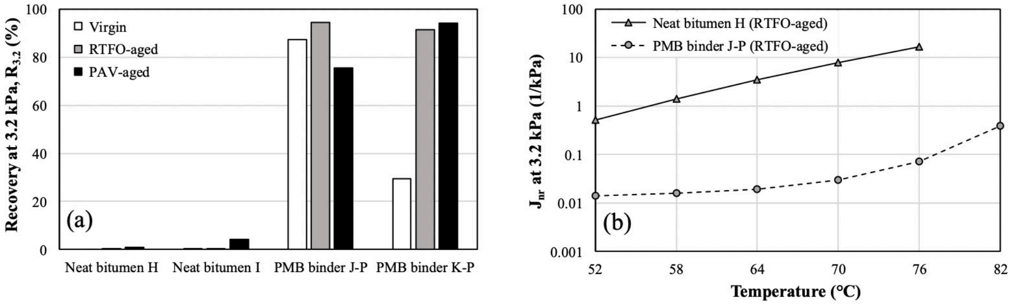

Figure 3: Results of MSCR tests

3.3 Resistance to fatigue damage

Resistance to fatigue damage is investigated by subjecting binder specimens to linear amplitude sweep (LAS) tests as per AASHTO TP 101-14 at 19 °C, with a frequency of 10 Hz and strain amplitudes comprised in the 0.1-30% range. Preliminary characterization in the undamaged state is carried by varying frequency between 0.2 Hz and 30 Hz with an imposed strain of 0.1%.

The final objective of LAS tests is to construct fatigue curves (also known as Wohler curves), which provide a synthetic description of fatigue behavior and allow a straightforward comparison between different materials. These curves are given by the following expression:

$$N_f = \mathrm{A} \cdot \gamma^{-B} \qquad (3)$$

where N_f is the number of loading cycles to failure, γ is initial shear strain, A and B are material-dependent constants derived from fitting of experimental data.

Such an approach to the assessment of resistance to fatigue damage constitutes an improvement with respect to the evaluation of the G*·sinδ parameter referred to in the SUPERPAVE grading system, which shows a poor correlation with field performance as a result of the fact that is obtained in the linear viscoelastic range and after only few cycles of loading (Bahia et al., 2001).

LAS tests have been proposed as an alternative to time sweep tests which have been reported to yield highly variable results (Kim et al., 2006; Botella et al., 2012; Hintz & Bahia, 2013; Santagata et al., 2013). Results recorded during frequency and amplitude sweeps are subjected to Viscoelastic Continuum Damage (VECD) analysis, which eventually leads to the calculation of constants A and B indicated in Equation 3 (Kim et al., 2006; Wen & Bahia, 2009).

Examples of obtained results are shown in Figure 4, in which they are plotted in terms of the N_f/ESALs parameter, given by the ratio between the number of loading to failure (N_f) and the fixed constant (equal to 10^9) provided in AASHTO TP 101-14. Experimental data refer to tests carried out on two neat binders of different origin (indicated as L and M).

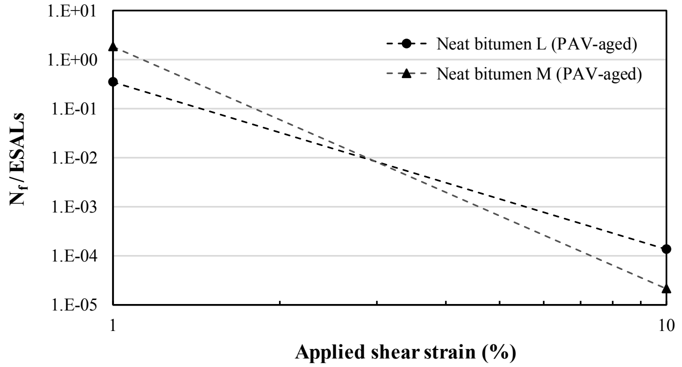

Figure 4: Results of LAS tests

From the analysis of the data presented in Figure 4 it can be observed that the two considered binders display a completely different resistance to fatigue damage, with a relative ranking that depends upon the considered level of strain. Although strain levels typically experienced by bituminous binders in the field are seldom greater than 1% (Masad et al., 2001), such an outcome, which reveals the strong strain-dependency of fatigue resistance, needs to be considered when comparing binders of different origin and type. In particular, it may be of premium interest while analyzing the effects of bitumen modification and of the use of RAP in asphalt mixes.

4 CONCLUSION

Based on the discussion of the results presented in this paper, it can be concluded that the use of advanced chemical and rheological procedures for the characterization of bituminous binders may be of great value in Qatar as a result of the wide variability of available materials. Adopted procedures are capable of highlighting the differences between binders of different origin and type, including those which are modified with polymers or are extracted from pavement layers and RAP samples. The approach adopted by the QSD-ANAS team for binder characterization will be further improved, eventually leading to the fine-tuning of advanced characterization criteria to be included in future specifications. In such a context, the ACRD will have a key role in continuously expanding the chemical and rheological database of bituminous binders employed in Qatar.

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