characterization of asphalt mixture containing bio-extended bituminous binder with spherical indentation test

H. Fadil & D. Jelagin
Department of Civil and Architectural Engineering, KTH Royal Institute of Technology, Stockholm, Sweden

J. Zhu & A. Ahmed
Swedish National Road and Transport Research Institute (VTI), Linköping, Sweden

ABSTRACT: Replacing petroleum-based bitumen with alternative bio-based binders in asphalt mixtures is a promising solution for green transition of road construction industry. The mechanical performance of bio-extended bituminous binders and asphalt mixtures is not yet fully understood, in particular with respect to dynamic mechanical behavior due to traffic and environmental exposures. As bio-binders differ in their chemical compositions, their physical properties and mechanical behavior may vary and differ significantly from bitumen-based materials. This paper aims to contribute to this important topic by evaluating a spherical indentation test as a quasi-nondestructive tool for viscoelastic characterization of the asphalt mixture containing bio-extended bituminous binder. Asphalt indentation tests are more sensitive to the binder phase properties as compared to standard macro-scale asphalt tests and may thus be a potentially useful tool for monitoring binder properties from the measurements performed on asphalt mixtures without extracting the binder.

1 INTRODUCTION

The asphalt pavements constitute a major part of global road network. The petroleum-based bitumen, used as a binder in the asphalt mixtures, has a relatively high environmental impact and carbon footprint. Accordingly, the use of bio-based binders in asphalt mixtures, manufactured from renewable resources such as plant-based biomass, is a promising solution for green transition of road construction industry (He et al., 2023). While a number of promising results have been obtained in this field so far, certain fundamental aspects of material behavior of bio-extended bituminous binders and asphalt mixtures are still not fully understood. In particular, the dynamic mechanical performance is of profound importance as bio-based binders have different chemical compositions compared to bitumen and they may affect mixture durability and performance in a different way as compared to bitumen. This paper aims to contribute to this important topic by evaluating a spherical indentation test as tool to characterize the properties of asphalt mixture containing bio-binder with measurements performed on the asphalt mixture without extracting the binder.

Spherical indentation test for characterization of asphalt mixtures has recently been proposed by Fadil et al. (2021, 2022). The test allows to determine the shear relaxation function $G(t)$ from the measured indentation force $P(t)$ and depth $h(t)$ at arbitrary non-decreasing load history. Indentation measurements allow capturing local mechanical properties of the specimen, as they are primarily influenced by the material properties in the direct vicinity of the indenter-specimen contact point. The spherical indentation test is thus a promising tool for monitoring changes in the binder phase of the material, from measurements performed on asphalt mixtures, as shown by Fadil et al. (2021, 2022) for the conventional mastic asphalts and asphalt concrete mixtures. The indentation test applicability to bio-asphalt, however, has not been evaluated yet.

2 EXPERIMENTAL STUDY

2.1 *Materials*

Two bituminous binders of penetration grade 160/220 were used to prepare dense graded asphalt mixtures: typical Swedish base course mixtures AG16. The mix design procedure was the same and the basic characteristics such as aggregate gradation and volumetric parameters of mixtures were kept as close as possible between the bio-extended variant (BIO) and reference counterpart (REF). The binder content was 4.5% for both mixtures, and the target air void was 4.2%.

Table 1. Binder properties.

Binder	Penetration @25˚C [1/10 mm]	Softening point [˚C]
REF	196	38.8
BIO	193	40.4

2.2 *Rheological characterization*

The binder samples were subjected to laboratory ageing protocols, with the Rolling Thin Film Oven Test (RTFOT) according to EN 12607-1:2024 to simulate the short-term ageing. The dynamic shear moduli, G^* of the binders were measured with the Dynamic Shear Rheometer (DSR), according to EN 14770:2023. For the compacted asphalt mixture specimens, their dynamic moduli, E^*, were measured according to EN 12697-26:2018+A1:2022 (Annex F cyclic indirect tensile test).

2.3 *Spherical indentation tests*

A schematic of the indentation test setup is shown in Figure 1, along with the main test parameters. The photo of the test setup used is shown in Figure 2. In the test, a spherical indenter is pushed into the specimen with a specific indentation depth history, *h(t)*, resulting in a reaction force, *P(t)*. Both *P(t)* and *h(t)* are measured as a function of time for the duration of the test.

The viscoelastic solution for the spherical contact problem, derived by Fadil et al. (2018) is shown in Equation (1):

$$P(t) = \frac{8}{3(1-\nu_o)}\sqrt{R}\int_0^t G(t-\tau)\times\frac{dh^{\frac{3}{2}}(\tau)}{d\tau}\,d\tau \qquad (1)$$

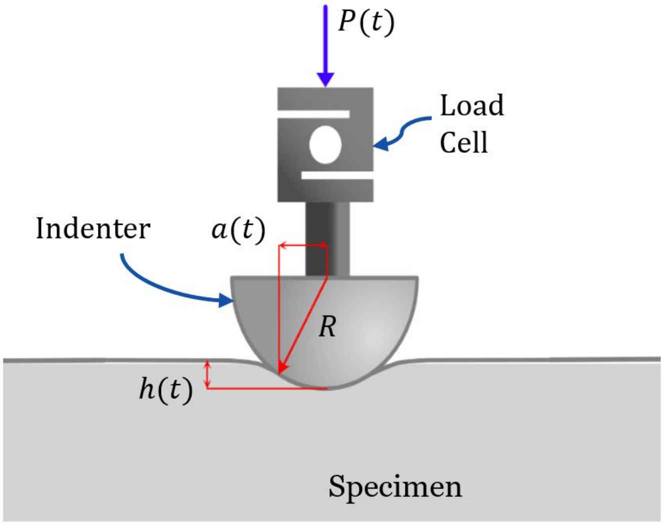

Figure 1. A schematic showing the parameters of the indentation test.

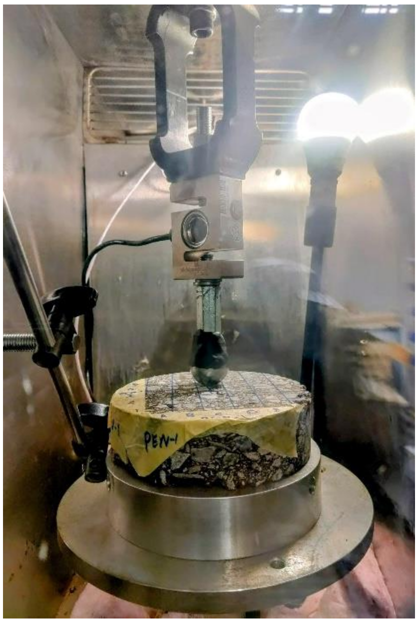

Figure 2. Photo of the indentation test setup.

where *R* is the radius of the indenter (Figure 1), while τ is a dummy integration variable. The validity of Equation (1) is constrained to arbitrary non-decreasing loading. By giving *G(t)* the form of a Prony series as Equation (2), Equation (1) is solved numerically for *G(t)* using linear programming.

$$G(t) = G_\infty + \sum_{i=1}^{N} G_i \cdot e^{-t/\tau_i} \qquad (2)$$

where *N* is the number of branches in the Prony series, G_∞ is the equilibrium shear modulus, τ_i is the relaxation time for branch *i* and G_i is the shear relaxation strength for branch *i*.

As presented in detail in Fadil et al. (2021, 2022), the *G(t)* measured with spherical indentation test is representative for viscoelastic properties of material in the immediate vicinity of the indenter-specimen contact point. The size scale of the region characterized in the test is proportional to the size of the contact area, *a*. As shown in Fadil et al. (2022) for the case of standard asphalt, performing the tests in grid pattern on the specimen surface, results in wide range of measured *G(t)*. This is expected, due to varying distribution of asphalt mixture components in the vicinity of the indentation point, resulting in some measurements being dominated by the mastic phase properties, while others are dominated by the aggregates. Accordingly, a statistical method was proposed by Fadil et al. (2022) to separate the test measurements into two clusters: mastic-dominated and aggregate-dominated. The average of *G(t)* measurements was found to correlate linearly to the asphalt shear relaxation modulus measured with the conventional tests. At the same time, the mastic-dominated results were found to capture the mastic properties.

In this study, the spherical indentation test is applied to characterize REF and BIO asphalts. The tests were performed on laboratory-manufactured asphalt cores of 150 mm diameter. The surfaces were prepared by cutting approximately 5 mm from each side and therefore the specimens had thicknesses of 37-40 mm. Two specimens were used for each mixture. The test setup used a steel spherical indenter with a curvature radius R = 15.875 mm. The test rig was an MTS 810 servo-hydraulic load frame with a 10 kN load cell. The tests were performed in depth controlled mode, applying the maximum indentation depth, h_{max} = 0.3 mm in 0.1 s ramp, and keeping it constant for 200 s. This corresponds to a maximum contact area radius of a_{max}= 2.51 mm. The surfaces of each sample was divided into grid with 25 squares each with side edge size of 2 cm. One indentation test was performed close to the center of each grid square, resulting in 50 tests per specimen in total. That ensures that the distance between each indentation is no less than 10×a. This is to avoid that the effect of indentation in one location interferes with the other measurements. The tests were performed at 0°C.

3 RESULTS AND DISCUSSION

The $G^{*}(\omega)$ measured with the DSR for RTFOT-aged REF and BIO binders are presented in Figure 3. As seen the BIO binder is somewhat stiffer compared to the REF at reference temperature 0°C. The maximum $G^{*}(\omega)$ difference between the materials is approximately 50% and it is diminishing with the frequency to about 30%. It should be pointed out, however, that the measured difference is comparable to the measurement scatter seen in Figure 3.

The dynamic moduli of AG16 REF and AG16 BIO asphalt mixtures at reference temperature 0°C are presented in Figure 4 along with their phase angles. It is observed that, within the frequency range where the binder phase dominates mixture behavior (intermediate to high frequencies), the somewhat stiffer binder in the case of AG16 BIO asphalt results in higher dynamic modulus for the mixture as well. In contrast to the results in Figure 3, however, there is no clear trend in stiffness differences with respect to frequency, which is likely due to the impact of the aggregate phase. The maximum difference in dynamic moduli of the two asphalts reaches approximately 20%. It must be pointed out that, as reported by Zhu et al. (2024), the REF and BIO binders are basically identical in terms of their penetration grade. The difference observed in Figures 3 and 4 are thus attributed to the different dynamic mechanical performance of the two materials. This illustrates importance of controlling dynamic mechanical properties of asphalt materials due to traffic and environmental exposures.

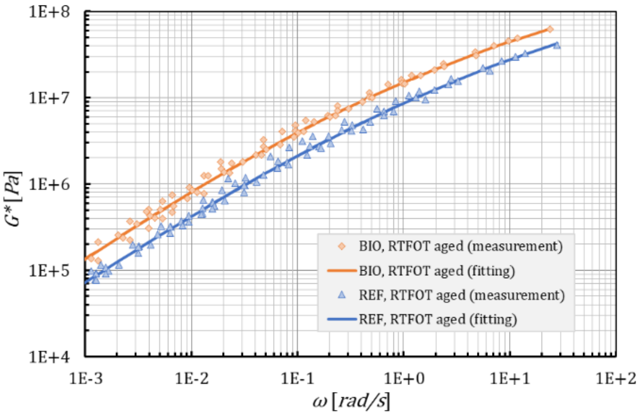

Figure 3. Complex moduli of RTFOT-aged binders, T_{ref} = 0°C.

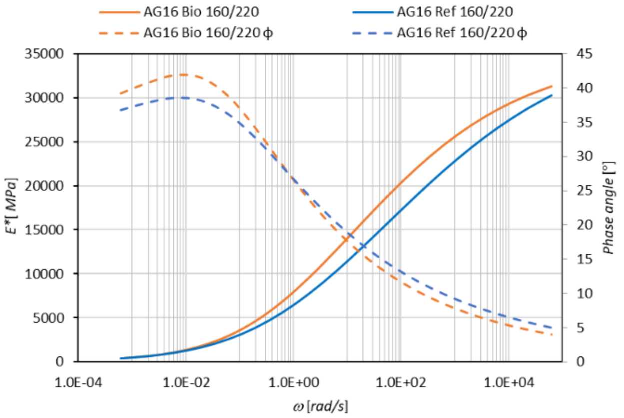

Figure 4. Complex moduli of AG16 BIO and REF mixtures, T_{ref} = 0°C.

The interconversion method proposed by Park & Schapery (1999) is used to convert the $G(t)$ measured on asphalt mixtures with the spherical indentation tests to $G^{*}(\omega)$ to facilitate comparison with the results presented in Figures 3 and 4. In Figure 5 the average mixture $G^{*}(\omega)$ obtained from the indentation test measurements are presented along with their 95% confidence intervals. As may be seen, the average $G^{*}(\omega)$ obtained from the indentation are significantly smaller as compared to the E^{*} values presented in Figure 4. For instance, for ω = 1, the indentation measurements result in $G^{*}(\omega)$ = 0.8 and 1.1 GPa for the REF and BIO asphalts correspondingly. This may be compared to the respective E^{*} values of 6.0 and 8.0 GPa, presented in Figure 4. Even after considering the Poisson's ratio (approximately 0.5 at low temperatures and high frequencies), the shear modulus obtained from indentation test is still relatively low. Similar observation has previously been reported by Fadil et al. (2022) for the tests performed on the conventional asphalt concrete. This discrepancy is attributed to the localized nature of the indentation test, which possibly leads to not capturing fully the effect of the aggregate skeleton on the asphalt mixture properties. At the same time, both in Figures 4 and 5 the BIO asphalt is approximately 30% stiffer as compared to the REF one, which indicates that indentation tests capture the relative differences in the materials and their ranking correctly.

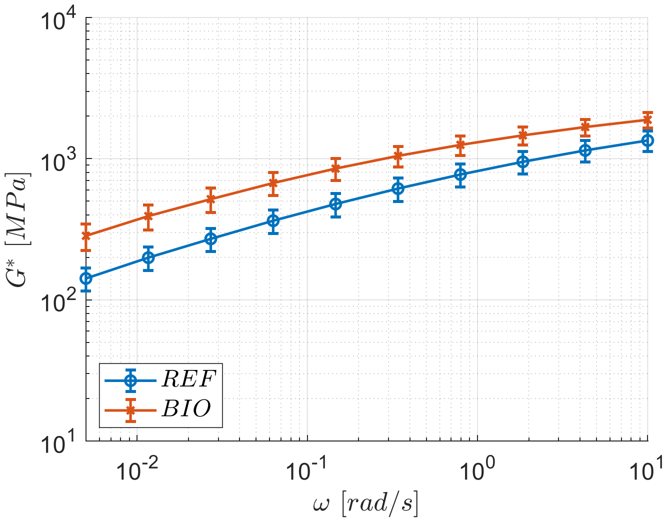

Figure 5. Average mixture $G^*(\omega)$ measured with indentation test on AG16 REF and BIO, T = 0°C.

Focusing on mastic-dominated measurements improves the indentation test sensitivity to the binder phase properties further, as illustrated in Figure 6 where average $G^*(\omega)$ obtained from the mastic-dominated measurements are presented together with the 95% confidence boundaries. $G^*(\omega)$ values in Figure 6 are both lower in magnitude and are accompanied by significantly less scatter as compared to the results presented in Figure 5. In Figure 6, the difference in $G^*(\omega)$ between REF and BIO measurements is within similar range as the DSR results presented in Figure 3, i.e. 40-100%. Accordingly, the results obtained in this study demonstrate feasibility of using indentation test for monitoring evolution of the bio-extended bituminous binders from the measurements performed on asphalt specimens.

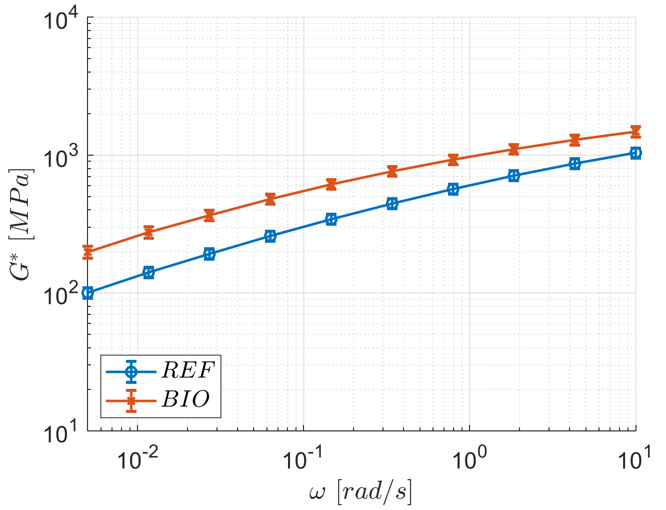

Figure 6. Average mastic-dominated $G^*(\omega)$ measured with indentation test on AG16 REF and BIO, T = 0°C.

4 CONCLUSIONS

Spherical indentation testing has been evaluated in this study as an alternative method for measuring the viscoelastic properties of asphalt mixture with bio-based binder. The increase in the modulus of asphalt mixture resulting from the incorporation of bio-oil, as measured through indentation tests under the specified conditions, showed good agreement with results from the conventional asphalt testing method. Applying a statistical analysis procedure to identify mastic-dominated indentation measurements further enhanced the test's sensitivity to binder properties and significantly reduced measurement variability. The experimental results suggest that the indentation test is a viable alternative for the viscoelastic characterization of bio-asphalts and their binders. Moreover, this method shows great potential for further development to enable in situ monitoring of changes in the viscoelastic properties of the binder phase due to environmental exposures.

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