Dynamic Mechanic Analyzer (DMA) shear test implementation for measurement of G* in fine asphalt mixes

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By

Fabiola Miranda-Argüello (Corresponding Author)
Research
National Laboratory of Materials and Structural Models (LanammeUCR)
University of Costa Rica, P.O.Box 11501-2060, UCR, San José, Costa Rica
Phone: + (506) 2511-2524
Fax: + (506) 207-4442.
E-mail: fabiola.miranda@ucr.ac.cr

Luis Loria-Salazar, Ph.D.
Pavement Infrastructure Program Director
National Laboratory of Materials and Structural Models (LanammeUCR)
University of Costa Rica, P.O.Box 11501-2060, UCR, San José, Costa Rica
Phone: + (506) 2511-4122
Fax: + (506) 207-4442.
E-mail: luis.loriasalazar@ucr.ac.cr

José P. Aguiar-Moya, Ph.D.
Research Program Coordinator
National Laboratory of Materials and Structural Models (LanammeUCR)
University of Costa Rica, P.O.Box 11501-2060, UCR, San José, Costa Rica
Phone: + (506) 2511-2529
Fax: + (506) 207-4442.
E-mail: jose.aguiar@ucr.ac.cr

Paulina Leiva-Padilla
Research
National Laboratory of Materials and Structural Models (LanammeUCR)
University of Costa Rica, P.O.Box 11501-2060, UCR, San José, Costa Rica
Phone: + (506) 2511-2524
Fax: + (506) 207-4442.
E-mail: paulina.leivapadilla@ucr.ac.cr

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ABSTRACT

The following paper corresponds to the characterization of mechanical properties in fine asphalt mixtures by means of a micro scale test. The characterization method involves the design of the fine asphalt matrix, the specimen preparation, the performance of shear tests, and the construction of complex shear modulus master curves based on the obtained results.

The tests were performed by means of the Dynamic Mechanical Analyzer (DMA). The test configuration consists of a temperature and frequency sweep for a given strain level, within the linear viscoelastic range of the material. The test implementation experimental design involved the use of two aggregate sources and three different asphalt types (neat, SBR modified, and ethylene copolymer modified). Based on the results for the different mixes, master curves were calibrated based on the sigmoidal, CA, and CAM general models, using Arrhenius and WLF adjustment factors.

As part of the study, the DMA test based on shear loading mode was successfully implemented and allowed for measurement of a fundamental material property: complex shear modulus (G*). The G* estimation was based on measurement of shear stress, strain, and phase angles. Complex shear moduli in the range of 40 to 170 MPa were obtained, being the fine asphalt mixtures modified with ethylene block copolymer the ones that developed higher stiffness, and the fine asphalt mixtures with neat binder the ones with lower stiffness. Based on the G* results, master curves were developed obtaining the higher fit when using the general sigmoidal formula which indicates the high degree of similitude in behavior between the fine asphalt matrix and the complete HMA asphalt mixtures.

INTRODUCTION

The complexity in behavior of the asphalt binder, due to the its viscoelastic properties (variability associated to temperature, loading time, and age) results in a broad research spectrum for the material. The following study corresponds to a characterization approach associated to one component of the asphalt mixture: the fine asphalt matrix.

With the purpose of performing research associated to the behavior of asphalt mixtures at reduced scales, LanammeUCR acquired the Mechanical Dynamic Analyzer (DMA) which allows for the measurement of the static and dynamic response associated to viscoelastic materials. However, prior to the study, no testing methodology had been proposed for analyzing asphalt mixtures using the equipment.

The importance of the following study is that it allowed for the implementation and standardization of the use of the DMA for characterizing fine asphalt mixtures within the linear viscoelastic range of the material. Three fundamental reasons for the importance of understanding this component of the asphalt mixture are the following: a) microcracking of the asphalt mixture initiates at the fine asphalt matrix and then reflects to the surface of the pavement structure; b) it is the most homogeneous component of the asphalt mixture; and, c) the characterization of the fine matrix allows prediction of the behavior of the asphalt mixture *(Caro, 2013)*.

Furthermore, the study serves as an input for research associated to moisture damage, defined as a loss in adhesivity and durability, due to the presence of water at the asphalt-aggregate interface or within the asphalt matrix *(Lytton et al., 2005)*: a typical phenomena in Costa Rica. The following methodology allows researchers to perform micromechanical analysis associated to moisture damage and generate knowledge that can further improve the understanding of the mechanisms associated to this type of distress.

BACKGROUND

The micromechanical analysis of asphalt mixtures has been researched by the Texas Transportation Institute *(Lytton et al., 2005; Howson et al., 2007)*, where several projects to measure the surface energy of different aggregates and asphalt sources has been performed. However, the previous research also led to the understanding of the moisture damage process, when studying the micromechanisms that affect the adhesive interface between the aggregate and the asphalt, and the cohesiveness and durability of the asphalt matrix. The research was performed in two distinct phases.

The first phase looked at evaluating surface energy and dynamic response of the material *(Lytton et al., 2005)*. The micromechanical tests that were performed are based on the application of a cyclic torsion stress at controlled strain conditions. Testing focused on the measurement of accumulated damage in the asphalt binder and the fine asphalt mixture. The results were used for quantifying the dissipated energy by the material and the energy associated to the viscoelastic deformation *(Lytton et al., 2005)*.

The second phase introduced the concept of energy ratio (ER) that was used to combine the energies associated to the adhesive and cohesive bond, as a parameter to evaluate the compatibility between asphalt binders and aggregates in terms of resistance to moisture damage *(Howson et al., 2007)*. Towards this purpose, a system for the evaluation of moisture damage was developed. The system involves studying the compatibility of an asphalt and aggregate source by means of surface energy and ER, followed by the mechanical dynamic analysis of the fine asphalt mixture. Finally, evaluation of the moisture susceptibility of the asphalt mixture is assessed, in order to evaluate the optimality of the mixture design and volumetric requirements for the HMA.

Because the previous studies demonstrated the efficiency of the micromechanical analysis to assess the resistance of the fine asphalt mixture (FAM) to moisture damage and fatigue cracking, a study to develop a simplified method for the material and a software tool for data analysis (fracture mechanics parameters associated to fatigue cracking and moisture damage related parameters) was performed *(Sousa et al., 2011)*. A project to implement the methodology for DMA specimen preparation was concluded based on different asphalt and aggregate materials. The researchers devised a method to design fine asphalt mixtures based on the actual asphalt content that is used by the fine portion of the gradation, in a given asphalt mixture (material passing the No. 16 sieve).

More recently, research to analyze the linear and non linear viscoelastic behavior of bituminous mortars was performed to characterize the behavior of the asphalt matrix at various temperatures and shear stress levels *(Woldekidan et al., 2013)*. The methodology implemented as part of the study maintains the same approach developed previously by TTI. The research highlights the importance of performing non linear analysis to obtain the true behavior of asphalt mixtures in response to low frequency loading at higher temperatures.

Other studies have also looked at different topics at the micro scale level, such as the anisotropic analysis of stiffness reduction in asphalt mixtures based on micromechanical analysis *(Masad et al., 2013)*. Based on the study, it was found that the asphalt mixture stiffness is 30% higher in the horizontal axis when compared to the vertical axis. The results were possible by studying the properties of the materials at the micro scale level where the homogeneity of the material is higher, as compared to the more heterogenous asphalt mixture.

Additionally, fine mixtures have been implemented in research associated to the use of RAP. In this sense, a project to evaluate the properties of RAP, mixture design, and HMA mixtures with high RAP contents has been performed *(Loría, 2011)*. The author highlights that the characterization of the RAP binder corresponds to one of the fundamental steps in the design of HMA mixtures with higher RAP contents. The material characterization was performed by means of DMA analysis. As previously mentioned, micromechanical analysis of the components of a pavement structure allows the mechanical characterization of the asphalt mixture in a practical, economic and efficient manner.

OBJECTIVE

The project was designed with the objective of implementing a DMA testing method that allowed for the micromechanical characterization of six different fine asphalt mixtures by measuring a fundamental material property: G*. The specific objectives of the study required the definition of a fine asphalt mixture design method based on the experimental design (2 aggregate sources and 3 asphalt types). Based on the previous, an experimental method for measuring the mechanical response of the fine asphalt mixture based on the Dynamic Mechanical Analyzer (DMA) was developed. Finally, based on the experimental results, master curves for G* based on the Sigmoidal, CA, and CAM general formulae using Arrhenius and WLF adjustment factors were generated.

MATERIALS USED IN THE STUDY

As part of the study, 2 aggregate sources of common use in Costa Rica were selected. Based on typical project gradations for these sources and HMA mixture design, the fine portion of the gradation (material passing the No. 50 sieve) was determined. Both mixtures correspond to dense graded mixtures of 12.5 mm and 19.1 mm nominal maximum aggregate size, henceforth referred to as Plant 1 and Plant 2 respectively.

As for the asphalt binders, 3 conditions of one source binder were selected: the binder in its neat condition (PG64-22), and the same binder modified with 1.5% SBR and 1.5% ethylene copolymer (EC). The modifier content was selected based on the workability of the asphalt mixtures and the typical dosage that is used in the Country.

EQUIPMENT DESCRIPTION

The mechanical dynamic analysis is a technique developed to measure the viscoelastic response of materials by means of static or dynamic loading. In this sense, the equipment differs from more traditional testing where only the elastic response is evaluated and hence only an incomplete response of the material is obtained when inelastic and viscous behavior exists. The DMA works mainly in the linear viscoelastic range, and is therefore more sensitive to the true material response.

Dynamic loading tests based on the DMA are the most common and consist of the application of a sinusoidal stress or strain, and the phase lag between the two factors. Based on the DMA Q800 equipment, parameters such as storage modulus, loss modulus, loss/storage compliance, phase angle, complex shear modulus, dynamic or complex viscosity, stress, relaxation modulus, dynamic or static load, temperature, time, frequency, displacement, and stiffness can be measured.

For the current research project, the shear loading mode test was implemented. As part of the test setup, two samples of equal dimensions and material properties are placed between two fixed supports. Between the two samples, a metal plate is used for generating shear (Figure 1). This testing mode is ideal for materials such as gels, adhesives, resins, and other high viscosity compounds *(TA Instruments, 2012)*. Based on the loading mode, the samples to be evaluated can have a maximum dimension of 10 mm in length and width, and 4 mm in thickness.

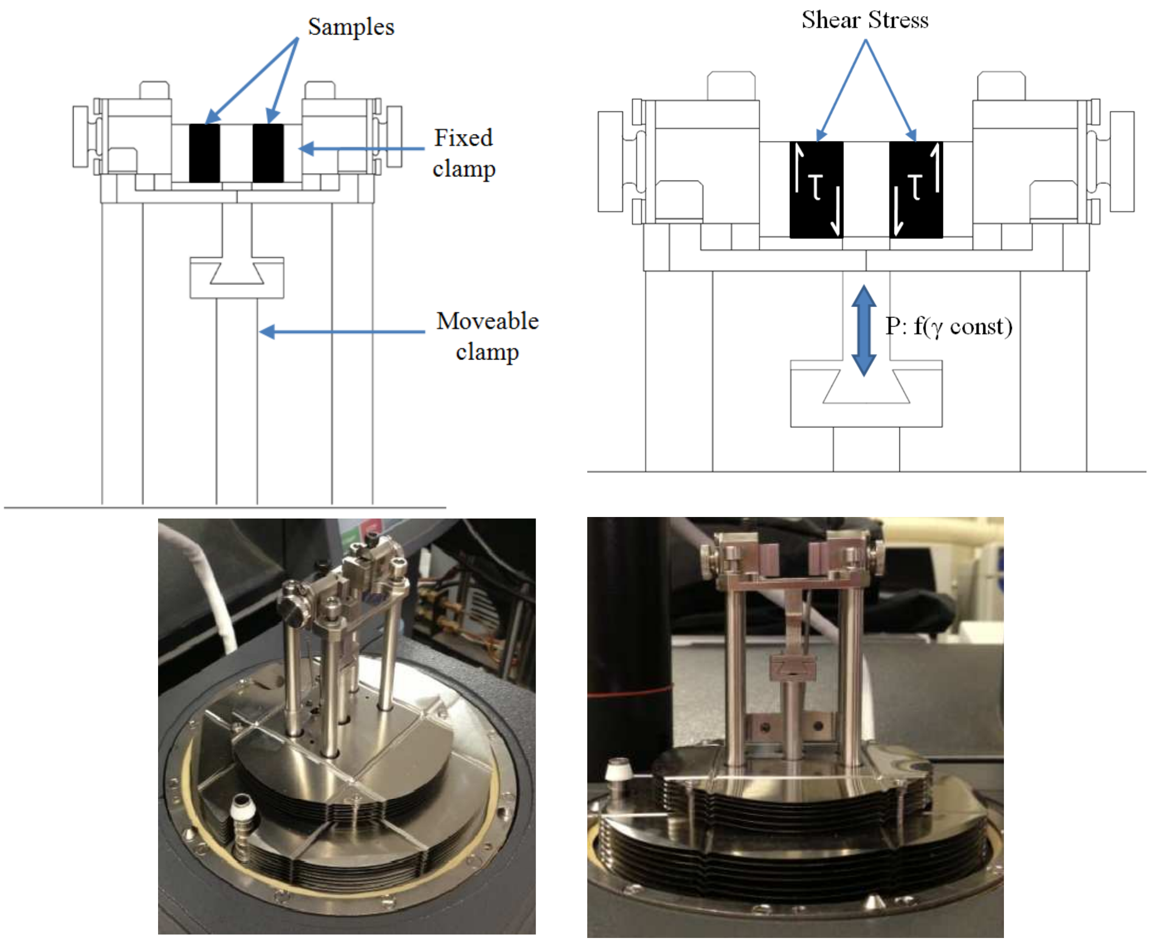

Figure 1. DMA shear test equipment configuration.

FINE ASPHALT MATRIX DESIGN

The estimation of the asphalt content for the fine asphalt matrix required for DMA testing is based on the methodology proposed by Howson *(2007)*. The methodology consists in calculating the thickness of the asphalt film that coats the granular material particles, as a function of gradation, asphalt content of the HMA, and properties of the asphalt binder.

Table 1 shows the asphalt contents that were required by the fine asphalt mixture associated to the six experimental conditions that were tested in the laboratory.

Table 1. Mixture Types Evaluated by Means of DMA

Fine Asphalt Matrix	Aggregate Source	Asphalt Type	Asphalt Content [(1)]
1	Plant 1	Neat binder	17.30 %
2	Plant 2	Neat binder	15.15 %
3	Plant 1	Binder modified with 1.5% SBR	17.30 %
4	Plant 2	Binder modified with 1.5% SBR	15.15 %
5	Plant 1	Binder modified with 1.5% EC	17.30 %
6	Plant 2	Binder modified with 1.5% EC	15.15 %

[(1)] The estimated asphalt content corresponds to the percentage of binder that coats the material passing the No. 50 sieve, with respect to the asphalt content associated to the HMA design.

DESIGN AND PREPARATION OF DMA SAMPLES

For the DMA sample preparation, molds based on the equipment specifications were designed based on the maximum dimensions that can be accommodated by the testing apparatus. The designed molds consist of metallic sections that are clamped together by means of screws, so that the retrieval of the samples is a simple process.

The mixing and molding process considers several concepts introduced by Loría (2011) for characterizing asphalt mortars. The implemented methodology uses a three step procedure, where the first step consists of the material preparation phase: temperature of aggregate and asphalt binder is raised to 150 °C (mixing temperature).

The second phase corresponds to the mixing process. A small metallic container is placed over a Bunsen burner and the materials are placed and mixed until a homogeneous mixture can be observed. It was determined that the maximum mixing time cannot exceed 1 minute.

The third phase corresponds to the specimen molding process. The molds are heated to the mixing temperature to ensure that the fine asphalt matrix temperature does not drop at a high rate, due to a significant temperature differential, resulting in poor compaction. The molds are greased with a Petroleum base grease prior to mixture placement. After the mixture is placed in the molds, uniform pressure is applied on the top face of the specimen by means of a spatula. Finally, the samples are removed from the molds and placed over a rigid layer to avoid any deflection in the material. The samples are placed in a zero humidity chamber at room temperature until ready for testing.

Figure 2 shows the process that was previously described.

Figure 2. Sample preparation

Validation of the sample preparation procedure was performed by means of scanning electron microscopy (SEM) at the sample surface. Figure 3 shows some of the obtained results. The images allowed for monitoring of the surface voids, and verification of the randomness in their distribution. Furthermore, the samples were checked for homogeneity in all sample faces and the possibility of failure planes (none were observed). Based on the previous, the samples were deemed as suitable for testing one discarded.

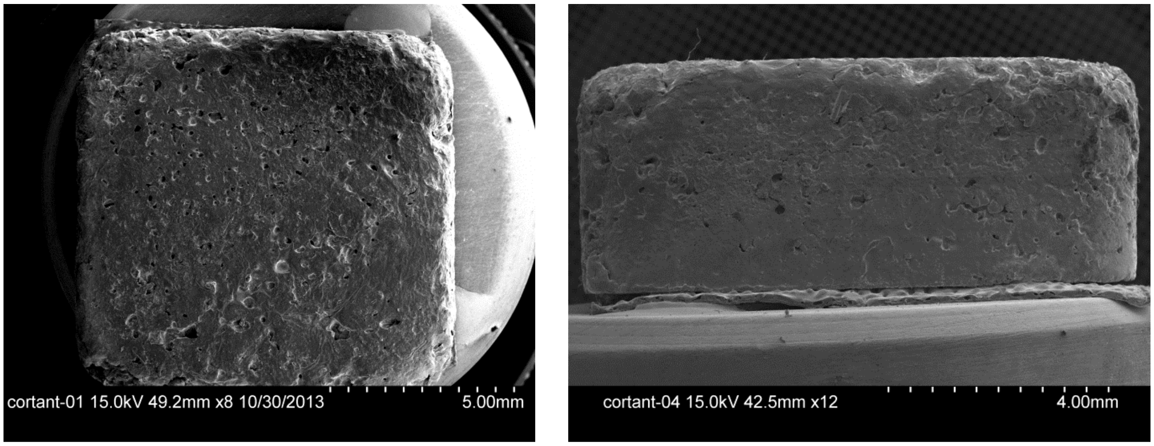

Figure 3. SEM images of DMA sample surface

TEST METHOD DESCRIPTION

The type of test that was used to evaluate the DMA samples in shear mode was a strain controlled multi frequency analysis. The frequencies that were used in the testing procedure correspond to 0.1, 0.5, 1, 5, 10, and 25 Hz, at the following temperatures: -10, 4.4, 21.1, 37.8, and 54.5 °C. The testing parameters are identical to those specified for HMA dynamic modulus testing based on AASHTO TP 62.

The strain level was selected to ensure that the sample remained within the linear viscoelastic. For determining an appropriate strain level, several samples were evaluated at different strain conditions, based on previous experience and equipment capacity. Finally, based on the observed variability and monitoring of the phase angles, a strain level of 0.01% was selected. Figure 4 shows a typical test output at different temperature and frequency conditions. Based on the test setup, the testing time is 112 minutes, accounting for five 12 minute periods required to adjust temperature and stabilize the environmental chamber. The figure shows the expected decrease in modulus associated to temperature increases and frequency reductions.

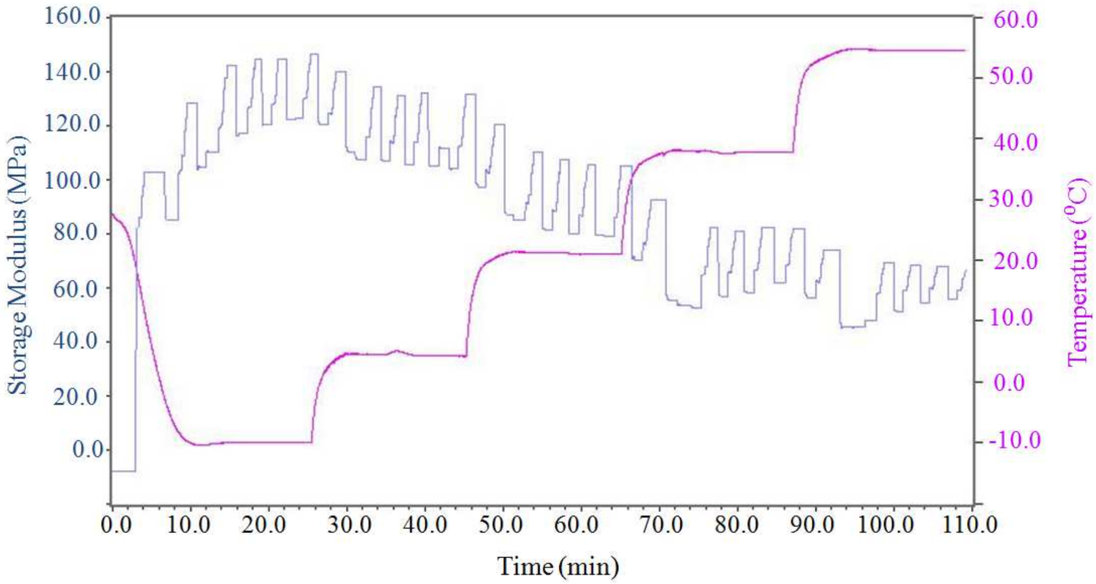

Figure 4. DMA strain controlled multi-frequency shear test output

The selection of the required number of testing replicas was determined by means of a statistical procedure similar to Monte Carlo simulation and Markov chains. The process consisted in recording the variation results based on monitoring of the mean and standard deviations of the measured response, and randomly combining different samples for a given sample sizes (*n*). Consequently, a total of 120 random combinations of 3, 4, 5, 6, and 7 replicas were evaluated. Figure 5 shows the variability associated to standard deviation of the measured properties for different sample sizes.

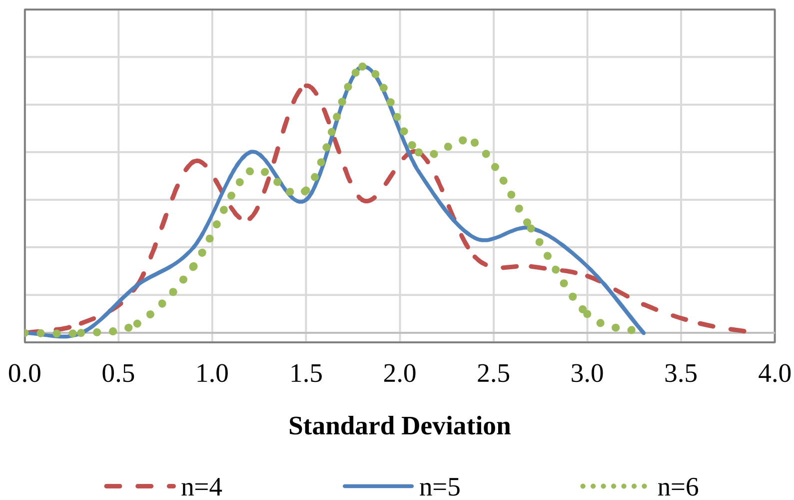

Figure 5. Variability associated to standard deviation of the measured properties for different sample sizes

The least efficient sample size (higher variability) occurred for a sample size of $n = 4$: higher data spread (thicker distribution tails). For a sample size of $n = 5$ and $n = 6$, the spread of the data was similar. Consequently, a sample size of $n = 5$ was considered optimal to ensure repeatability in test results.

Once the required sample size was defined, a sensitivity analysis of the results was performed to account for the error associated to heterogeneity involving specimen preparation. The analysis was based on the Mellin transformation, which applies for functions that are defined for positive numbers only, as is the case of the elastic component of shear modulus. Based on this uncertainty analysis, the most probable variability associated to the modulus was estimated, considering the intrinsic heterogeneity related to the parameters that are involved in the estimation of the modulus: specimen dimensions and sample stiffness.

The Mellin transformation considers the probabilistic distribution of the independent variables associated to the modulus and assigns to each a transformation function. The most likely variability is calculated based on the product of the individual Mellin transformation functions for each independent factor *(Mays, 1999)*. Based on this criteria, the modulus can be expressed as:

$$G' = \frac{3\,Kt}{5\,wh} \qquad \text{[Ec. 1]}$$

$$M_{G'} = (3/5)^{s-1} M_K(s) M_t(s) M_w(2-s) M_h(2-s) \qquad \text{[Ec. 2]}$$

where $M_G, M_K, M_t, M_w,$ and M_h correspond to the Mellin transformations for modulus, stiffness, sample thickness, width, and length. The transformed functions are then evaluated in models associated to the probability distribution of each factor.

Finally, based on the previous analysis an estimated standard deviation of 0.6 MPa for the storage modulus can be expected. Furthermore, because the viscous component for the shear complex modulus as measured by the DMA is in the range of an order of magnitude below that of the elastic component, it is realistic to assume that an uncertainty of 0.6 MPa is adequate for G*.

ANALYSIS AND RESULTS

The individual shear tests were analyzed to verify the variability of the samples. Figure 6 shows the relationship between dynamic modulus and frequency at different temperatures in linear and logarithmic scales, as well as the Black diagram and the Cole-Cole plot for a specific sample. In order to verify the validity of the G* measurements, the data was analyzed for linearity and parallel tendency of the isotherm curves shown in Figure 6 a. and b. The observed behavior reveals adequate material performance, and hence validates the testing procedure

The Black diagram (Figure 6 c.) provides information on the material behavior, and suggests whether the fine asphalt matrix behaves as an asphalt binder or as an asphalt mixture. The hypothesis is based on the fact that for an asphalt binder the phase angle increases as temperature increases, while for asphalt mixes the phase angle increases with temperature up to a given point and then drops *(Pellenin et al., 2002)*. The obtained results indicate an increase in the phase angle from -10 °C up to 21.1 °C, and a decrease from 37.8 °C onward, indicating a response similar to that of an asphalt mixture.

The Cole-Cole plot (Figure 6 d.) allows material analysis at low and intermediate temperatures *(Pellenin et al., 2002)*. Adequate fit in the plot translates to a thermo-rheological simple material, and consequently justifies the application of time-temperature superposition (thermo-rheological simplicity is dependent and strongly correlated to the bitumen composition). It is important to note that higher correlations were observed when comparing neat the binders to the modified asphalt binders.

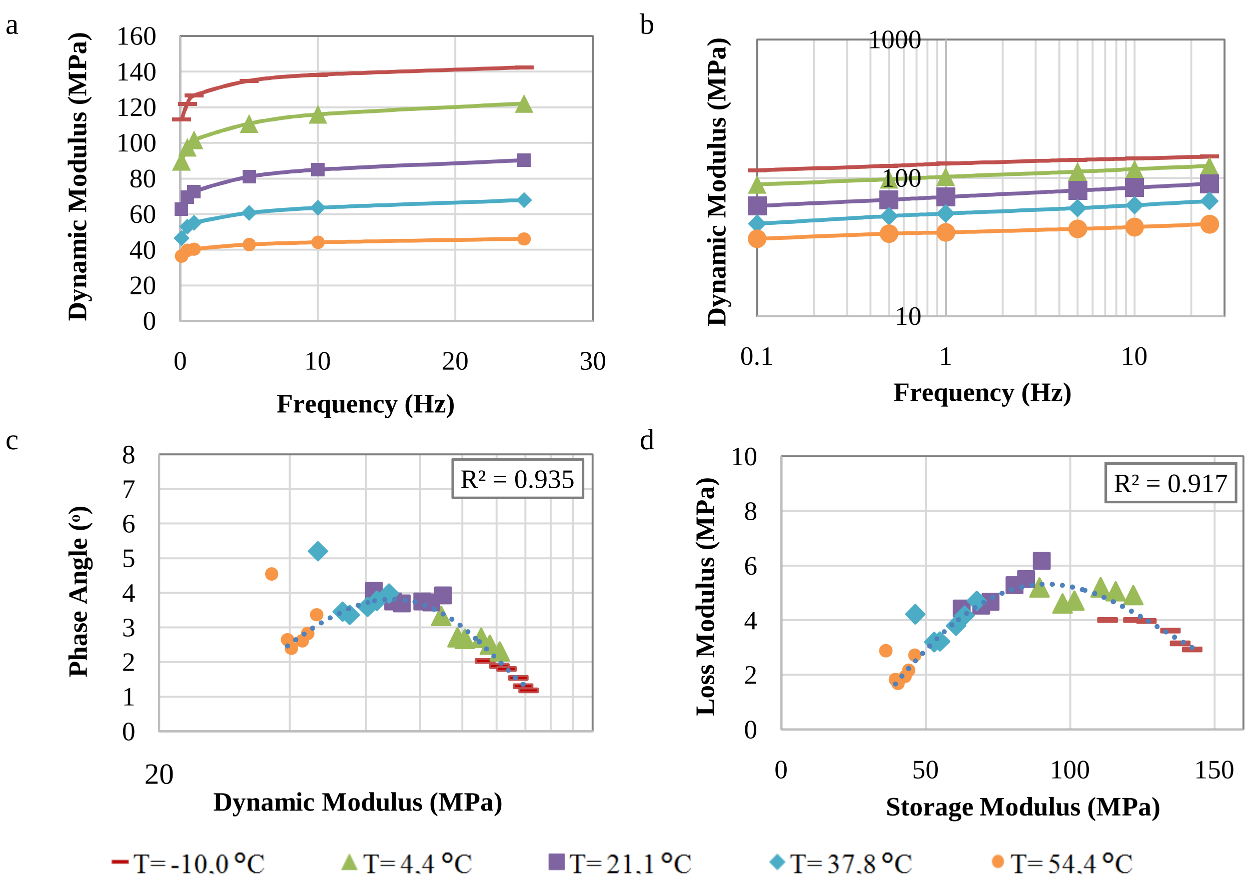

Figure 6. Fine asphalt mixture results.

(a and b) Isotherms (c) Black Space Diagram and (d) Cole-Cole Plot

Coefficients of variation (CV) for all mixtures were obtained and in all cases showed values below 15%. As expected, lower CVs were associated to lower temperatures and high frequencies, while higher CV values to higher temperatures and low frequencies. Consequently, several observations at 0.1 Hz and 54.5 °C had to be discarded because of high variability. Finally, considering a typical variability associated with rheological tests of 20%, the quality of the data was acceptable.

Calibration of the 2S2P1D Model

Based on the DMA shear test results, the parameters for a mechanical model were calibrated in order to evaluate the test results based on fundamental properties of the asphalt binder and the asphalt mixture. The calibrated model represents the 2S2P1D model (abbreviation for 2 springs, 2 parabolic creep type elements, and 1 dashpot model), based on the generalization of the Huet - Sayegh model. Table 2 shows the calibrated model parameters for the six analyzed fine asphalt mixtures. The h, k, G_∞, G_o and η, are parameters associated with: two parabolic creep elements, two springs and a dashpot, respectively (Figure 7).

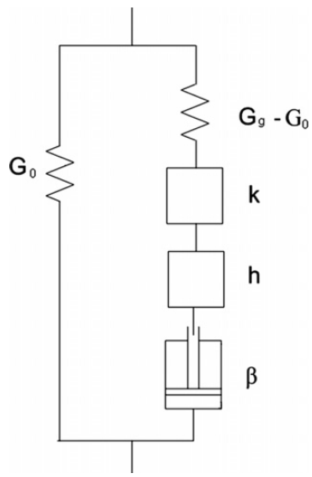

Figure 7. 2S2P1D model

Table 2. 2S2P1D Model Parameters

Fine Asphalt Mixture	G∞ (MPa)	G_o (MPa)	k	h	α	β	τ	Se/Sy	R^2
1	162.953	46.374	0.17	0.48	3.45	798	12.61	0.046	0.998
2	154.709	50.184	0.19	0.43	3.71	729	10.61	0.049	0.998
3	163.428	48.991	0.19	0.51	7.94	202	777.73	0.088	0.994
4	178.923	52.148	0.16	0.50	2.98	222	6.40	0.075	0.996
5	164.414	50.106	0.19	0.68	8.50	202	902.19	0.087	0.995
6	169.861	58.544	0.17	0.62	3.12	209	12.80	0.079	0.996

As can be observed from the table, k values in the range of 0.16 - 0.19 were obtained, while h values ranged between 0.43 and 0.68, indicating an increase in the k value with respect to an increase in mixture stiffness. The fine asphalt matrices containing neat binders, and lower stiffness than their modified counterparts, are associated to the lowest β values. The previous is expected since the parameter is directly related to binder stiffness. Furthermore, due to the thermo-rheological complex behavior, the α parameter serves as an indicator of testing accuracy. In the current study higher α values were associated to the mixtures containing modified asphalt binders.

In general, the goodness of fit indicators associated to the calibrated models are high with R^2 values above 99% and Se/Sy relations below 0.09 for all cases *(Tran, 2005).*

MASTER CURVE ESTIMATION

Master curves for all fine asphalt mixtures using the general Sigmoidal, CA (Christensen-Anderson) and CAM (Christensen-Anderson-Marasteanu) model equations, and applying the Arrhenius and WLF (William-Landel-Ferry) shift factors (Figure 8) were developed. Figure 8 shows a fine asphalt mixture master curve calibrated based on the general Sigmoidal, CA, and CAM models using Arrhenius shift factors. Based on the results, the Sigmoidal function provided the best fit for all evaluated fine asphalt mixtures. Figure 8 a. shows a smooth S curve, with an Se/Sy ratio below 0,05, an R^2 of 0,99 with an associated error of 0,2 %.

In general, all master curves calibrated based on the general Sigmoidal model showed an excellent fit regardless of the selected shift factor (R^2 values between 97% and 99%). The previous observation corroborates the conclusion based on the Black diagram which indicated that the fine asphalt matrices show behavior similar to that of the asphalt mixture.

In the case of the master curves calibrated based on the CA model (Figure 8 b.), high R^2 values were also identified ($R^2 > 92$ % with 1% errors). As for the master curves estimated based on the CAM model, linear trends were observed (Figure 8 c.), and consequently the model is not considered appropriate for the data.

Based on the fit associated to the different models, significant differences were identified in the predicted tails. As observed, the predicted modulus of the fine asphalt matrices is relatively low due to the stiffness associated to the fine aggregate mineral structure in comparison to that of the complete gradation. Furthermore, the high asphalt binder content associated to the fine asphalt matrix results in lower stability and higher flexibility.

The analyzed material showed behavior that is similar to that of conventional asphalt mixtures. However, the previous tend to develop stiffness in the order of 1×10^{10} Pa, while the results measured based on the DMA tests on the fine asphalt mixtures resulted in moduli in the order of 1×10^{8} Pa. Consequently, the fine asphalt mixtures exhibit intermediate modulus values between those observed in asphalt mixtures and those associated to the asphalt binder, presenting a difficulty in the master curve construction.

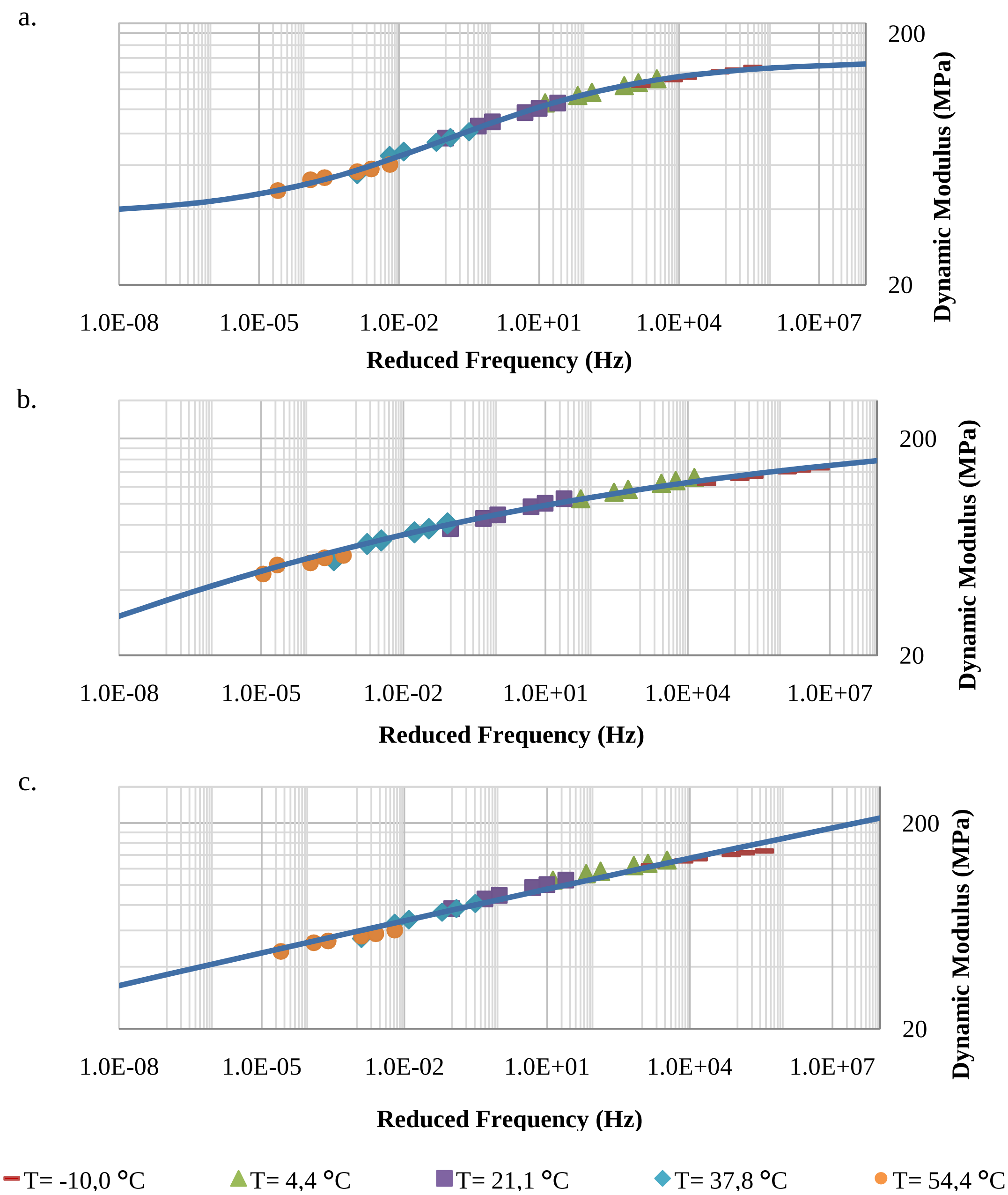

Figure 8. Master curves using Ahrrenius shift factors based on a. Sigmoidal model, b. CA model and c. CAM model

It is important to note that generally, linear viscoelastic behavior occurs between the glassy transition state (in the case of asphalt materials starts at -20°C). In the case of the evaluated fine asphalt mixtures the glassy transition starts at -10 °C (Figure 8 a.). Additionally, the Newtonian liquid state can be expected to occur above 54.4 °C (Figure 8 a.) In general, asphalt binders show this transition at approximately 70 °C). These material properties can be associated with low temperature cracking and rutting respectively.

A comparison between the observed modulus for the six different fine asphalt mixtures was performed. Based on the aggregate source, no significant differences in the material modulus were observed. However, based on the binder type, stiffness differences ranged from 3% at low temperatures to

18% at high temperatures. Figure 9 shows the G* values for the different binders. The higher stiffness values are associated to the fine asphalt mixtures using EC modified asphalt.

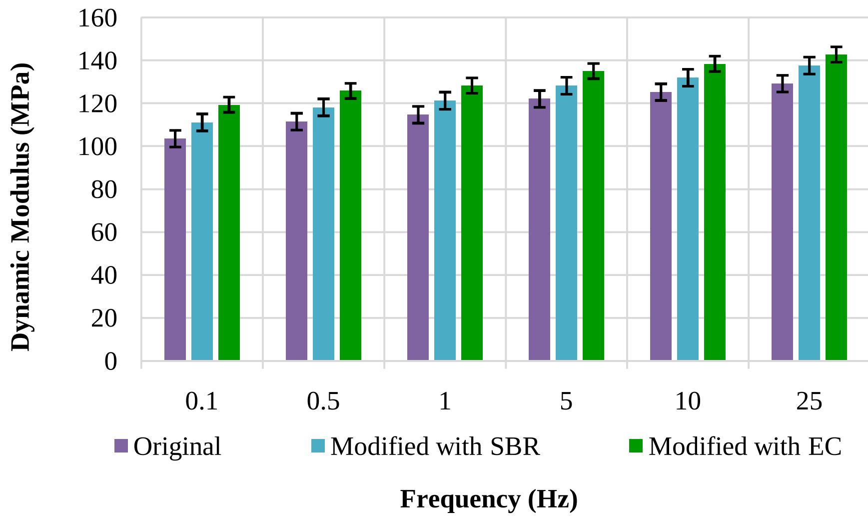

Figure 9. Fine asphalt mixtures G* comparison

SUMMARY AND CONCLUSIONS

The implementation of a DMA test under shear loading mode allowed for laboratory characterization of fine asphalt mixtures based on the material response to dynamic shear loads. The following are the main findings associated to research project:

- DMA complex shear modulus values based on the testing configuration ranged from 40 to 170 MPa, with the higher stiffness associated to modified asphalt binders.
- The material behavior of the fine asphalt mixtures resembles that of the asphalt mixtures, based on the phase angle behavior observed on the Black diagram.
- A sensitivity analysis indicated that the most probable standard deviation associated to the complex modulus based on the uncertainty of testing parameters such as sample dimensions and material heterogeneity is in the order of 0.60 MPa. The previous implies that 97.5 % of all measurements can have an associated error between 0.5% and 3% due to sample preparation and material heterogeneity.
- As the analyzed material mostly behaves as a thermo-rheological complex material, the Sigmoidal general model resulted in the best fit for master curve development, with fine asphalt matrix stiffness values between those associated to an asphalt mixture and those expected of the asphalt binder.
- No significant differences in material response were identified for the fine asphalt mixtures using asphalt binder modified with SBR and EC, at 1.5% concentrations.

Based on the experience developed as part of the project, and the success in implementing the DMA testing methodology, further research is recommended to validate the results obtained based on the proposed method. Finally, the effects associated to changes in the sample geometry, loading ranges and subsequently loading modes need to be further researched. Towards this goal, testing based on compression, tension and bending modes is currently being implemented.

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