Multi-scale Characterization of Asphalt Mastic Rheology

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Abstract. Understanding the influence of the fundamental parameters on asphalt mastic rheology is an important step towards improving the quality of asphalt mixtures. Due to the size of fillers and the sensitivity of the rheological behaviour of mastic, it is not always possible to study the effect of all parameters at one scale. Hence in this study, a theoretical framework is established for calculating the relative viscosity of asphalt mastics as a function of its filler concentration. Furthermore, a new test protocol is introduced for measuring the viscosity of asphalt mastic at higher temperatures and different filler concentrations. To characterize the fillers and their agglomeration and distribution inside solid mastics, X-ray tomography, laser scattering, scanning electron microscopy, BET and Helium Pycnometery were utilized. To characterize the energy dissipation potential of the mastics under cyclic loads, as a function of their fillers, the dynamic mechanical analyzer was utilized. The research shown in this paper further investigated the various dominant parameters related to fillers and bitumen in mastics and relate them to the workability and resulting mechanical properties and developed an overall framework to connect different scales. The developed characterization protocols have the potential to allow the asphalt engineers to design their hot and warm asphalt mixtures on a more fundamental and thus sustainable basis.

1 Introduction

Asphalt mixtures are complex composite materials build up from a mixture of aggregates, bitumen, fillers and additives. As such, the end performance of asphalt mixtures entirely depends on its component's properties, their interaction with to each other and the surrounding environment. Variation in the quantity and quality of the components is, unfortunately, quite normal and has a significant effect on the material's long term performance. Understanding the effect of the individual material components is thus essential for correctly designing asphalt mixtures to meet desirable structural and surface requirements [1-5]. In addition to the base components, there are many other parameters which can be controlled that have a high impact on the ultimate performance of asphalt mixtures.

Being a composite material, also means that the way the mixture is handled and constructed has a large impact on the resulting behavior. In the case of hot mix asphalt (HMA), this means that the material will be heated initially when mixing the components to ensure that the mastic viscosity is reduced sufficiently to coat the aggregates. Then, upon the time of paving, the mixture is heated up again to ensure sufficient workability and compactability. The relationship between the chosen temperatures and the material properties will thus affect the resulting mixture properties. In the case of warm mix asphalts, the use of additives and their interaction with the fillers and bitumen is chosen today, rather arbitrarily and is uncoupled to the short term workability and the long term sustainability demands.

Currently, most mastic parameters are chosen mainly based on experience and measurements of the viscosity of the bitumen only. Yet many parameters have a significant influence, such as aggregate size distribution, filler size distribution, filler agglomeration, mastic viscosity and physicochemical interaction between the aggregates and the asphalt mastic. Studying the influence of all these parameters also means that several scales need to be investigated.

As viscosity of asphalt mixture is a complex phenomenon, for predicting and/or measuring the workability and optimizing the long term field response of asphalt mixtures it is crucial to clearly understand the effect of all the dominant parameters and be able to measure them. To do so, in this research the asphalt mastic phase (i.e. the filler-bitumen mixture) has been identified as an important phase that can be linked to the bitumen level as well as the asphalt mixture level. For the bitumen-mastic connection, the effect of the bitumen viscosity, the filler properties and the filler-bitumen interaction are important aspects to consider. For the mastic-asphalt concrete mixture the mastic properties (from the previous scale), the aggregate properties and the interaction between the two are important. The latter, though part of the overall research project, will not be treated in this paper.

2 Research Aim

In this paper, focus is placed on determining the fundamental parameters which have influence on the rheology of asphalt mastics and evaluating their effect. To do so, first a theoretical framework is established for calculating the relative viscosity of asphalt mastics as a function of its filler concentration. Then, a new test protocol is introduced for measuring the viscosity of asphalt mastic at higher temperatures and different filler concentrations. Subsequently, the results obtained from this viscometry tests are utilized for evaluating the theoretical framework. To be able to understand the underlying mechanism of the measured mastic behavior, the physical properties of the fillers were measured and compared with their corresponding mastic viscosity. To investigate the agglomeration of filler particles after mixing with bitumen, a detailed X-Ray CT analysis is performed. Finally, to

investigate the energy dissipation of the mastic under cyclic loading after cooling down, a new test set-up was developed utilizing the dynamic mechanical analyzer.

3 Theoretical Framework

According to the basic theory, the viscosity of any material can be calculated by dividing the applied shear stress by shear strain rate (equation 1).

$$\eta = \frac{\tau}{\dot{\gamma}} \tag{1}$$

The relationship of shear stress and shear rate for a Newtonian material is linear. For non-Newtonian material this relationship is non-linear, so calculating the viscosity of non-Newtonian material can be a more challenging task. Asphalt mastics at elevated temperatures behave as non-Newtonian suspensions in which bitumen plays the medium role and fillers are act as particles. Suspensions behave differently at varying particle concentration levels, due to a change in the flow profile of the suspension due to presence of particles, causing the non-linear behavior.

In addition to the particle concentration, as mentioned earlier, there are several other parameters that can affect the viscosity of suspensions. Most of the theories which are available for calculating the viscosity of suspension are restricted to certain assumptions and are not necessarily able to model the behavior of mastic at the relevant range of filler concentrations. For this reason a framework was established, based on available theoretical models, to be able to calculate the viscosity of the mastic from dilute to high concentrated suspensions [6].

This framework divides the suspension viscosity behavior into two general regimes: the hydrodynamic and the frictional regime (figure 1) [7]. In the *hydrodynamic* regime, at lower filler concentrations, particles are positioned far from each other, forming thus a so-called dilute suspension. At higher concentration, particles come into a closer position but still do not have any direct filler-filler contact. In the *frictional* regime fillers come into direct content and make a network of filler-filler contacts. In this regime the frictional force is dominant.

As is illustrated in figure 1, in the hydrodynamic regime there are two models that can asymptotically model the mastic relative viscosity, which is the viscosity of the mastic at each concentration divided by viscosity of the neat bitumen. The Einstein formulation (equation 2) and the Frankel formulation (equation3) can be used for lower and higher filler concentration, respectively. Yet there is a gap between the low and high concentrations, which is not covered either by Einstein nor by the Frankel equation. For this zone a transition equation (equation 6) was created to be able to connect the lower filler concentrations to the higher filler concentrations.



Fig. 1 Relative viscosity versus concentration bounded by two asymptotes

$$\eta_r = 1 + k\phi \tag{2}$$

$$\eta_r = C' \left\{ \frac{\left(\frac{\phi}{\phi_{max}}\right)^{\frac{1}{3}}}{1 - \left(\frac{\phi}{\phi_{max}}\right)^{\frac{1}{3}}} \right\}$$
(3)

$$h = 2r \left[\left(\frac{\phi}{\phi_{max}} \right)^{\frac{1}{3}} - 1 \right]$$
(4)

$$h_r = \frac{h}{r} \tag{5}$$

$$\eta_r = C \left(\frac{1}{h_r}\right)^n \tag{6}$$

where η_r is the relative viscosity, k is the Einstein constant, ϕ is the filler concentration, C' is the Frankel constant, ϕ_{max} is theoretical maximum concentration which there is no any free binder in the mastic, r is the is the average weighted radius of the particles, C and n are transition constants and h_r is the average distance between filler grains.

As the fillers come into contact, they form frictional interactions which gets activated upon shearing of the mixtures. So by using the concept of primary and secondary aggregate structures, equation 7 was defined for calculating the relative viscosity of the mastics in this regime.

$$\eta_r = \left(\frac{\delta}{r} - h_r\right) N_c C_1 \tag{7}$$

where δ is the adsorbed asphalt layer thickness, the term of $(\delta/r-h_r)$ shows the strength of contact. N_c is the number of particles and it shows the number of contacts which are producing the friction force and C_1 is the friction coefficient of the whole particles structure [7].

4 New Protocol for Measuring the Viscosity of Asphalt Mastics

After establishing and testing the framework it was necessary to measure the viscosity of asphalt mastic at elevated temperature for a wide range of filler concentrations. Considering that such measurements should be able to clearly identify the effect of various parameters, the robustness, reliability and repeatability of the test should be assured. It was therefor decided to develop a standardized protocol in which the dominant parameters are clearly defined.

The *temperature* is certainly an important parameter, which has a significantly effect on viscosity values. In two stages in the developed test protocol temperature plays an important role: during the filler and bitumen mixing and during the actual measurement. The latter one is more often controlled than the first, but since asphalt mastic is a history dependent material, the entire handling and temperature treatment should be controlled as much as possible. Furthermore, during the mixing process, an optimum temperature is needed to provide suitable bitumen viscosity. So, all mastic samples were mixed at the same temperature (140°C), to reduce the temperature effect of the mixing procedure. This temperature was chosen such that it would be appropriate for mixing the bitumen and fillers at all percentages. Mixing of the bitumen with the fillers was done with a mechanical high shear mixer.

Due to the influence of particle size on the viscosity of mastic, it is important to have a good control on the homogeneity of the filler size. To make sure for all samples that the filler size distribution is the same and segregation does not have any effect on the filler size, sufficient amount of filler for all samples were collected and stirred to ensure a representative filler distribution. Then, the filler was divided into different portions for the mastic samples. At that point filler was placed in the oven for 24 hours to get completely dry and also heated up to the mixing temperature.

Because of the importance of the volumetric characteristics of the fillers and bitumen, the amount of filler content was calculated by volume instead of weight. The weight of volume for a specific concentration was calculated by measuring the density of the fillers and the bitumen at the temperature at which the mastic viscosity will be measured. For calculating the concentration of filler a volumetric calculation was made:

$$\phi = \frac{V_F}{V_b + V_F} \tag{8}$$

Where V_F is the volume of filler and V_b is the volume of bitumen.

Adding filler to the bitumen is an action which seems simple, but can also affect the rate of agglomeration. Agglomerated particles make a bigger artificial particle that can act as coarser filler. For this reason, the adding procedure of the fillers to the bitumen was also standardized as much as possible to avoid introducing additional variables into the mixtures.

Air pockets or enclosed bubbles are an unfortunate reality from the mixing process and can have a significant effect on the viscosity. Air bubbles can be introduced into the mastic during the filler adding into mastic mixing process. Therefore, to prevent adding air bubbles into the mix, the filler was gradually spread in the bitumen during the mixing. The mastic was kept in an oven at 140° C for 2 hours to give the samples time to release any remaining air bubbles. To ensure a homogeneous mixture, the mastic was mixed again at the relevant test temperature before pouring mastic into the cup of the viscometer.

Deciding on the appropriate viscometer cylinder geometry is quite challenging since various geometries are available on the market, but for converting the measured torque and velocity to the correct shear stress and shear rates, some considerations must be made. First of all, some of these geometries are suitable for Newtonian material and others for non-Newtonian. The theoretical equations for calculating shear stress and shear rate usually are based on the assumption that the material moves instantly with the inner cylinder. This means that the system should not have any slipping on inner and outer cylinder wall. The zero shear, or plug, zone is also a non-negligible phenomenon that can occur for some stiffer mastic or for very low shear rate test.

To satisfy all the above conditions, a rotational co-axial viscometer was utilized. This viscometer was found to be able to apply the accurate range of shear stress at different magnitudes for measuring the mastic viscosity with a varying range of viscosity, relevant for mastics. Due to varying flow behaviour of the tested mastics with the different filler concentration, accurate measurement should be possible when mastics display a different type of behaviour such as Bingham flow, shear thinning or thixotropy. For this reason a viscometer was chosen, able to use different geometries and procedures. The geometry and the gap between inner and outer cylinder were chosen very carefully to avoid the influence of the boundaries on the measurements.

The DIN standard (3219:1993(E)) gives the geometry of inner rotor for Newtonian materials (figure 2-c). In this standard, the converting equations are based on the assumptions of dealing with a Newtonian material. As such, the shear stress gradient in the gap between inner and outer cylinder is considered linear, there is no plug zone and no slippage on the wall of inner and outer cylinder. From these assumptions, the limitations of using the cylindrical rotor are considerable.

Bitumen is in principle a Newtonian material. But, when increasing the filler concentration, mastic starts to behave more non-Newtonian. From the particular filler concentration that this behavior is noted, the use of the cylindrical rotor to measure the viscosity is no longer appropriate. This, firstly, because of a boundary bias which starts to occur at that point, and secondly, because the penetration of



Fig. 2 a) The TA Rheometer (viscometer), b) grooved cup, c) cylindrical rotor and d) vane shaped rotor

the inner cylinder or rotor becomes more difficult for stiffer mastics. Lastly, slippage on the wall of the inner cylinder must be prevented, the risk of which becomes quite high for the cylindrical geometry at the higher concentration. For these reasons, a vane shaped rotor (figure 2-d) was chosen in this study for the higher filler concentration. The range of filler concentration for using the two rotors can vary from one type of mastic to the other and it is highly depended on the filler size. From the tested mastics, however, most of the mastics showed that around 20% filler concentration can be a suitable boundary for shifting from cylindrical rotor to a vane shaped rotor.

As mentioned earlier, bitumen is a visco-elastic material and its rheological behaviour is highly depended on the temperature. So, maintaining the accurate temperature during the measurement is crucial. The test temperature can be designed according to the aim of the test. There are, however, some limitations for choosing the test temperature when testing mastics. According to Stokes' law the speed of sedimentation of particles inside a liquid is a function of the weight of the particle and the viscosity of the liquid. Hence, the combination of higher temperature and longer testing time may increase the risk of filler sedimentation. On the other hand, if the testing temperature is too low, the viscosity result may not be representative of or relevant for the viscosity behaviour of bituminous mastic, especially for modified bitumen. To deal with this issue all mastic viscosity measurement were done at 100° C.

5 Material Selection

In this research, for studying the effect of different fillers and filler concentrations, an extensive laboratory program was performed. The fillers were selected to enable the study of a range of parameters, such as filler chemistry, surface area and particle shape on the viscosity of the mastic. Hence, three different types of mineral fillers were selected; two silica based filler (M10 and M600) and fly ash. For

all samples presented in this paper, a standard 70/100 bitumen from Nynas was used. Silica based fillers have a completely different angular shape compared to the rounded fly ash particles. M10 and M600 are both from silica based mineral. M600 is, however, much finer than M10, having thus a much higher surface area and, consequently, behaving very differently in terms of bitumen-filler interaction and agglomeration. The mastic samples were prepared with different filler content ranging from 5% to 50% or more, depending on stiffening effect of the filler on the bitumen.

6 Filler Fundamental Properties

To analyze the characteristics of the selected fillers scanning electron microscopy (SEM), laser scattering, BET (Brunauer, Emmett and Teller theory) and helium absorption were used to determine shape, size distribution, specific surface area and density, respectively.

Figure 3 shows examples of pictures captured by SEM from all fillers. As figure 3-a and 3-b show, M10 and M600 have similar angular shapes, however due to their different size the particle interaction is different, leading to a varying agglomeration. Figure 3-c and 3-d shows this difference. Fly ash filler, figure 3-e and 3-f, has a round shape particle that makes it completely different from the two other fillers in terms of shape.

Laser scattering is one of the possible methods for measuring the size and size distribution of particles with diameter less than 2 mm. In Figure 4 the size distribution of the three fillers measured by laser scattering is shown.

The phenomenon of physical adsorption of gases was taken up the BET to determine the specific surface area of fillers. Measurement of specific surface area of M10, M600 and fly ash by Micromeritics Gemini 2360 Surface Area Analyzer gave 0.93, 4.0 and 1.5 m²/g respectively (Table 1). To determine the density of the fillers, their weight was determined by an accurate scale and their volume by a Helium Pycnometer. According to test results from the Helium Pycnometer the densities 2.79, 2.75 and 2.41 kg/cm³ were obtained for M10, M600 and fly ash respectively (Table 1).

7 Filler Agglomeration in Asphalt Mastics

The filler phase in asphalt mixture gradation is most often noted as the mineral particles which have an average diameter below 63 μ m (or 73 μ m in American standards). From figure 4, however, it can be seen that a wide filler size range was measured, which may affect the mastic rheological behavior. Additionally, it is interesting to know to what extend the fillers form clusters after they are mixed in the bitumen.



Fig. 3 SEM photograph of fillers, a) M10, b) M600, C & d) comparison agglomeration between M10 & M600, e & f) fly ash

To study if the particles agglomerate in the bitumen and how much this agglomeration would affect the actual filler gradation, X-ray computed tomography was used to scan the mastic samples. The 3D X-ray images were then analyzed to measure the volume and size of the filler inside the bitumen mastic and represented in a sieve-size graph to compare to the original filler before mixing. Several samples were prepared with bitumen 70/100 and 30% filler (M600) volumetrically and poured into specially designed silicone molds (figure 5). The sample size is important for the necessary resolution of the scans, so the size of the mold was chosen small enough for the X-rays to be able to capture the fillers as much as possible with good accuracy and big enough to provide suitable data for the statistical analyzing, Figure 6. To avoid erroneous conclusions due to resolution issues, the measured mineral volumes were calculated and compared to be equal to the actual amount as measured from the sample preparation.

The X-Ray image sieve analyses showed a different size of fillers compared to what was measured at the filler's dry stage. As can be seen from the graph, the filler gradation shifted to the right side (coarser side) of the dry filler gradation curve, which indicates the amount of filler agglomeration inside the samples, Figure 6. As can be seen from the graph, the agglomeration is really significant which means that the actual filler concentration will be quite different from what the mastic is originally designed for, had the filler distributed evenly. From the analyses it was also found that the distribution of the fillers within the mastic was not homogeneous and, as can be easily seen with the bear eye in Figure 5, part of the larger filler particles seem to sediment toward the bottom of the sample.



Fig. 4 Filler size distribution for M10, M600 and fly ash

Table 1 BET and Helium Pycnometer results

	M600	M10	Fly Ash
Specific surface area (m ² /g)	4.0	0.93	1.5
Density (kg/cm ³)	2.75	2.79	2.41

8 Viscosity Result Utilizing New Protocol and Framework Validation

To measure the viscosity of the mastics and relate them to the filler properties, several asphalt mastic samples were prepared. In both sample preparation and testing steps of the viscometry, the presented protocol was followed. Figure 7 shows an example of the viscosity result for mastics at different filler concentrations. This figure shows the relationship between shear stress and shear rate for



Fig. 5 Illustration of filler agglomeration, M600, and actual scanned specimen & its silicon mold



Fig. 6 M600 gradation before and after mixing

mastic with filler M600. As can be seen from the graphs, a clear increase of the viscosity of the mastics as a function of filler percentage was measured.

Figure 8 shows the relationship between relative viscosity and filler concentrations at 1.5(1/s) shear rate for three different mastics. The relative viscosity of each filler concentration was calculated by dividing the viscosity of mastic with that particular concentration by the viscosity of the neat bitumen. Figure 8 shows the final viscometry result for all the filler concentrations (from 5% to 50%). It is important to note, however, that for each curve two type of rotor geometry were used. As it discussed earlier for mastics with filler concentration lower than certain concentration (about 20%), the cylindrical inner rotor was used; and for higher than that concentration the vaned rotor was used. In all cases a good connection between the transfer point of these rotors was found in the measurement.



Fig. 7 An example of viscosity results, shear stress vs. shear rate for mastic M600 at different filler concentrations



Fig. 8 Relative viscosity of mastics at 100 C and 1.5 1/s shear rate with combination of cylindrical and vaned rotor (left: complete graphs, right: focused on lower concentrations)

From the measurements the highest relative viscosity for a certain filler concentration was seen with the M600 filler, which is the finest filler with a high surface area and angularity, followed by the mastics with M10 and fly ash fillers. From the first two mastics it can be concluded that with the same mineralogy, but increasing surface area, viscosity will increase. The comparison between the viscosity result of the mastics containing M10 and fly ash shows the effect of shape of filler. The spherical particle of fly ash inside the bitumen under shear stress are rotating and rolling more easily, giving less resistance to flow compare with angular particle of M10.



Fig. 9 Fitting the framework into the experimental results

Figure 9 shows that a good agreement between the experimental result and the developed theoretical framework was found. The fitted framework is able to capture all experimental results quit well; however still makes use of fitting parameters which are presented in table 2. From this it can be noted that a clear distinction can be made between the mastics and that the framework can uniquely describe the behavior of the mastics over a wide range of concentrations. While the fitting factors shows same trend, finding the physical explanation for these factors is the aim of the ongoing project.

Fitting Parameters	M 10	M 600	Fly Ash
$\Phi_{\rm m}$	0.5	0.4	0.6
C'	2.8	3.5	3.6
С	1.5	1.4	1.5
n	1.5	1.7	1.8
Κ	7.8	8.8	5.1

Table 2 Fitting parameters

9 Energy Dissipation of Asphalt Mastic

To investigate the mechanical property of the mastics as a function of the varying filler types and concentrations, the ability of the mastics to dissipate energy is an important property to characterize the mastics. To be able to measure this it is important to vary the train rates in a controlled manner and to create a uniform stress field inside the materials. For this, a new test set-up is being developed that will enable the loading of mastic samples under a uniform stress and strain field and measure their ability to dissipate energy. For this, a DMA Q800 set-up (Figure 10-a) is being utilized and a special sample holder is developed that allows for the required stress and strain fields. In order to increase the accuracy of the sample preparation and reduce the effects of stress concentration on the corners of the rectangular cross section, the standardly available film tension clamp was modified by designing and appending two external parallel ring & plates (Figure 11-a). Consequently the shape of the specimens was changed to a cylindrical shape. Furthermore, in order to mold the specimens with accurate dimensions, a silicon rubber mold and a fixture were designed and built as Figure 11.



Fig. 10 Apparatus for measuring energy dissipation, a) DMA Q800, b) film tension clamp



Fig. 11 a) external parallel ring & plate, b) silicon rubber mold, c) ring & plate in the fixture and d) whole set up

The test is currently still under development, but some preliminary test results are shown in Figure 12. In this graph the displacement versus time is plotted for mastics with different concentrations of fillers. The applied stress field was intended to be a haversine in the tension domain. From the initial analyses it showed that the applied force-field is not yet as needed for the mastic test as is therefore not further shown here. Nevertheless, from the initial results one can see that the test is able to have a first indication of the behavior of mastics, and that the effect of the different filler particles is visible from the test. The development of this test will be further worked upon in the current research project, which will allow for a detailed analyses of the mastic behavior at 100 Celsius. From this the effect of the particle size, distribution and interaction will become clear and will allow for an optimizes mastic design, taking into account the workability of the mastic and the long term performance under the service stress-strain and temperature conditions.



Fig. 12 Comparison of Displacement-Time curves for mastics with different concentration of filler M10, M600 and Fly Ash

10 Conclusion

In this study, theoretical and experimental techniques were utilized for evaluating the effect of fillers on the rheology of asphalt mastics. A theoretical framework was established that is capable of calculating the viscosity of asphalt mastics at different filler concentrations in two hydrodynamic and frictional regimes. The framework was extensively validated and a new protocol to measure the actual viscosity of mastics with different concentrations at higher temperatures was developed. To have a reliable and reputable test the relevant parameters that can affect the viscometry were standardized. To study the effect of the different properties of filler on mastics rheology, three different types of fillers were selected that enable the systematic study of a range of parameters, such as filler chemistry, surface area and particle shape on the rheology of the mastic.

From the measurements it was found that, as M600 is much finer than M10, the M600 has more internal interaction between the particles and showed more agglomeration in the unmixed situation. X-ray tomography also showed that the filler inside the bitumen does not have the same gradation as unmixed. The filler gradation inside the mastic is shifted entirely to the coarser part of graph, which shows the effect of filler agglomeration. The mastic with the finer filler, M600, showed the highest value of viscosity for each concentration, followed by M10 and Fly ash. Fly ash and M10 almost have the same size, however due to the spherical shape of the fly ash particle it showed a lower viscosity compared to M10. The viscometry result also showed that the mastic with higher filler concentration is not Newtonian anymore. So for measuring the viscosity of mastic with higher filler concentration (more than 20%), a vaned rotor was used. Initial measuring of the energy dissipation of asphalt mastics showed that mastics with higher filler concentration have lower visco-elastic energy dissipation potential under dynamic loading. Partly this could be due to the diminished amount of viscoelastic binder (i.e. the bitumen), since the volumes are kept the same. Other reasons for this could be the non-visco-elastic particle-particle interactions at higher concentrations and the increase of bitumen-filler interaction. The mastic energy dissipation test is, however, still under evaluation and not too many conclusions can yet be drawn from its current results. In the continuation of this research, the test will however serve as an important step in linking the production stage behavior of the mastics with the actual long-term field performance.

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