Characterisation and Modelling of Asphalt Mastics and Their Effect on Workability

PhD Thesis

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August 2014
Doctoral dissertation to be defended in Kollegiesalen (the old chapel), Brinellvägen 8, KTH, Stockholm, Sweden, on 12th of September 2014, at 9:00.

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Stockholm, Sweden, 2014

TRITA-TSC-PHD 14-005

Abstract

Sufficient coating, easy paving and good compaction of asphalt mixtures are important parameters to achieve predictable and sufficient long term pavement performance. Being the actual glue between the stones, mastics are thereby an important component in asphalt mixtures. The viscosity of these mastics is affected by several parameters, such as filler concentration, particle shape and size distribution, bitumen type, filler-bitumen interaction and temperature. Having improved control on these parameters can thus lead to better designed mastics, optimized asphalt mixture workability and enhanced long term pavement performance.

In many studies, models have been developed for calculating the viscosity for different types of suspensions at various particle-to-fluid ratios. Unfortunately, none of them are applicable to asphalt mastics, since this material has a much wider range of mineral filler concentration from dilute to very concentrated. Additionally, conventional methods that are currently used for measuring viscosity are designed for bitumen or only suitable at lower or intermediate temperatures. Therefore, a need exists for a laboratory method that is able to investigate the viscosity of mastics at the high temperatures relevant for mixing and compaction of asphalt mixtures.

Considering the importance of diminishing energy usage and greenhouse gas emissions, the asphalt industry has been developing new types of mastics for the past decades (e.g warm mix or cold mix asphalts). These ‘new’ types of materials, also involve new additives and behaviour beyond the ‘normal’ standards. Adding filler into bitumen, is further complicated in the presence of such modifiers, since they may not only change the bitumen properties, but can also change the interaction between the filler and the bitumen matrix. Empirical assumptions thus no longer hold and more sensitive methods and models, based on fundamental understanding, will be needed to bring sufficient control and predictability into mastic and asphalt mix design and production.

This Thesis aimed at developing insight into the effect of different fundamental properties that control the behaviour of asphalt mastics and give tools and methods on how its future mastic and mixtures designs can be optimized. For this, analytical, experimental and computational methods have been used. In the following, a brief overview of the activities and the main findings of the Thesis are given.
In an extensive literature review, the existing viscosity models and their range of applicability to asphalt mastics were evaluated. From this, an analytical framework was developed that removes some of the limitations of the existing theories. Various mastics were placed into this framework and their fitting parameters give a uniform way of comparing their viscosity behaviour over a wide range of filler concentration. As such, the framework will allow researchers and engineers to develop a common understanding in categorizing and choosing various mastic types. It may also assist in further choices for characterization or design. The framework is presented in paper I of the Thesis.

To measure the viscosity of asphalt mastics at elevated temperature and various filler concentrations, a new test protocol was developed. The test protocol is based on a commercially available viscometer for which various rotors and cups are available. In this research, these were investigated over a wide range of filler types and concentrations as well as shear rates. In selecting the fillers, important details were given to the mineralogy and shape of the fillers to enable understanding of these parameters in the resulting mastic behaviour. It was found that a critical filler concentration range exists over which the mastic viscosity behaviour moves away from (linear) Newtonian to (non-linear) shear thinning behaviour. A critical filler concentration was therefore defined, for which the appropriate rotor geometry and resulting interpretation needs to be modified. The effect of a wide range of shear rates was also investigated in detail, from which it became very apparent that limiting rheology investigations of mastics to only a small window of shear rates (as is usually done for bitumen) can pose significant issues and erroneous conclusions. To ensure repeatability of the developed protocol, all steps have been described in detail. The developed test protocol and its considerations are presented in paper II.

To investigate the sensitivity of the developed protocol, environmentally susceptibility was also included in this research by conditioning several samples to ageing and moisture. From this, it was positively concluded that the conditioning was visible in the measured material response. Particle settlement and agglomeration can further modify the resulting mastic behaviour from the intended. To analyze this, X-Ray Computed Tomography was utilized to determine the size and location of the filler particles inside various mastic samples. From the analyses, and comparing with the laser scattering sieve curve, a clear agglomeration of the particles was found. It could be possible that an agglomeration shift factor can be uniquely determined for mastics, depending on the fillers and bitumen types, which could further assist in optimized future design. It also emphasizes the lack of control that we have today on creating homogeneous mastic. These results were presented and discussed in paper III.

In order to investigate the filler-bitumen interaction, the developed protocol was utilized and combined with micro-mechanical Finite Element analyses, in which the shape and size of the filler particles were varied in the bitumen matrix. Combining the experimental and numerical results showed the effect of the interface properties, in which modifiers can affect...
the interface response in such a way that the overall mastic behaviour changes. Furthermore, the posed hypothesis that rounded versus angular particles (thus changing the interlocking effect) and varying size distribution (thus changing the filler surface area) can significantly affect the stresses and deformations inside the mastic matrix was confirmed. These analyses were published in paper IV.

In Sweden, for many years now, a new type of mixing protocol has been applied, in which the order of mixing is changed from the conventional method. Improved workability and diminished mixing and compaction energy needs have been important drivers for this. Considering that it is the mastic phase which is modified by changing the mixing order, it is an interesting case-study to apply the tools and insights developed in this Thesis to further explain the mechanisms of workability in connection with the mastic phase. To do so, the analytical viscosity framework was combined with a mixture morphology framework, to upscale to the mixing level, and tribology principles to explain the interaction between the mastic and the aggregates. From the mastic viscosity protocol it was found that the mixing order significantly affects the resulting mastic viscosity. To analyze the effect of this on the workability and resulting mixture performance, X-Ray Computed Tomography was used to analyze mixtures produced by the two different mixing sequences and mechanical testing was utilized to determine the long-term mechanical performance. In this, mastic viscosity as a function of particle concentration and distribution was directly coupled to improved mixture workability and enhanced long term performance. In Paper V the details and considerations of this investigation were summarized.

**Keywords:** Asphalt, Pavement, Mastics, Bitumen, Viscosity, Rheology, Filler, Filler-Bitumen Interaction, Workability, X-Ray CT.
Acknowledgments

The work presented in this PhD thesis has been carried out at KTH Royal Institute of Technology, at the division of Highway and Railway Engineering.

The Swedish Transport Administration (Trafikverket) and AkzoNobel are greatly appreciated for financing this study.

First and foremost I would like to express my gratitude to my supervisor, Professor Björn Birgisson, who not only offered me an opportunity to join his group as a PhD student, but who supervised me, provided me with solid technical background and gave the tools to further develop myself. I would like to give my sincere gratitude to my co-supervisor, Associate Professor Niki Kringos. I appreciate all her contributions of time and ideas to make my PhD experience productive and stimulating. The joy and enthusiasm she has for the research was contagious and motivational for me, even during tough times in the PhD pursuit.

I am indebted to Dr. Denis Jelagin whose suggestion helped me to develop a framework, which is the first step of this PhD thesis. The assistance accorded by Dr. Alvaro Guarin in scanning and analyzing the samples is gratefully acknowledged. I will also like to acknowledge the discussions and expert advices in regular Friday meeting with Dr. Måns Collin, Dr. Jonas Ekblad, Dr. Per Redelius and other industry members from Trafikverket, Skanska, Nynas, NCC and AkzoNobel.

I would like to acknowledge and thank everybody who has contributed to my pleasant time at KTH, especially my colleagues at the division of Highway and Railway Engineering.

Last but not the least, I wish to thank my parents, Dr. Saeid Hesami and Fariba Pasdar. I owe them everything and wish I could show them just how much I appreciate them.

Ebrahim Hesami
March 2014, Stockholm
I lovingly dedicate this thesis to my precious wife, Anohe Bahramian, who supported me each step of the way.
Publications

Appended Papers

This PhD thesis is based on the work presented in the following appended publications:


Other relevant publications:


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1 Introduction

1.1 Background

In the production phase of asphalt mixtures, their ease of handling and paving (named workability) together with its compacting properties (named compactability) have a great impact on the resulting mechanical performance of asphaltic pavements [1-3]. Though compactability has over the years been studied by many researchers, workability has not been given similar attention. Yet, mixtures with good compactability but bad workability, pose a significant problem for the construction phase as it increases the risks of inhomogeneous pavement properties and enhances the unpredictability. Workability is especially an issue when moving towards modified asphalt mixtures, where additives are used with a specific design purpose in mind. For example, lowering the mixing and compaction temperatures in the case of Warm Mix Asphalt (WMA) or stretching the visco-elastic mix behaviour to lower temperatures, in the case of polymer modification.

Workability of asphalt mixtures is influenced by many different parameters, such as production temperatures, aggregate mineralogy, the aggregate gradation, the size and shapes of the aggregates and the type of bitumen used [4,5]. Though some of these may be more dominant than others, their influence can also vary from case to case, depending on specific mixture characteristics or circumstances. The guidelines and standards that assist pavement engineers in the material design and production phases should therefore be suitable for a wide range of materials and boundary conditions.

Conventionally, viscosity of bitumen has been used to determine the mixing and compaction temperature of the asphalt mixtures [6,7]. Though even for ‘normal’ mixtures, there could be some challenges associated with this; it is certainly no longer possible to directly correlate bitumen behaviour with mixture behaviour when more complex types of materials are used. For example, non-linearity, shear rate dependencies or the interaction of bitumen with different mineral types or additives can show completely different rheological behaviour at higher temperatures. Hence, empirical assumptions no longer hold and more sensitive experimental methods and analytical or numerical models, based on fundamental understanding, will be needed to bring sufficient control and predictability into bitumen and asphalt mix design and production.

There have been some attempts to measure the workability of asphalt mixtures directly from the loose mixture (i.e aggregates coated with the mastic); however for different
technical issues such as device geometry challenges or lack of repeatability, they have not been truly successful [8,9].

Because the mineral filler fraction has a very high surface area in comparison to the coarser aggregates in the mixtures, the physio-chemical interaction between bitumen and fillers may be an important parameter in the mixture performance. The shape of the fillers, their size and size distribution, the nature of their surface texture, their adsorption intensity and the chemical composition of the fillers are all parameters that can potentially have a significant effect on the long-term performance of the entire mixture [5,13,14,15]. Adding filler into bitumen, is further complicated in the presence of modifiers, since they may not only change the bitumen properties, but can also change the interaction between the filler and the bitumen matrix.

So, instead of making the link between bitumen and mixtures, it could be hypothesized that the workability of asphalt mixtures can be directly linked to asphalt mastic behaviour. Having fundamental understanding of mastic behaviour is also relevant beyond the workability properties, as it influences many of the other important properties of asphalt mixtures, such as the overall stability of the mixture, air void distribution, bitumen drain-down during transport [10,11]. Additionally, mastic stiffness and its binding strength is known to influence fracture resistance at low temperature, fatigue resistance at intermediate temperature and permanent deformation resistance at higher temperatures [11].

1.2 Aims and Scope

This Thesis aims at developing insight into the effect of different fundamental properties that control the behaviour of asphalt mastics and develop tools and methods that can be used to optimize the design of mastics. The Thesis also aims to link mastic behaviour to workability of mixtures, taking into account fundamental material properties.

To reach this aim, analytical, experimental and computational methods have been used in which various identified material properties were investigated. In the following, a brief overview of the scope and methods used in the various activities, which are published in the 5 papers, presented in this Thesis.

Having an insight into the methods and models applicable for calculating asphalt mastic viscosity, could lead to a characterization of its behaviour and obtain necessary knowledge needed to interpret mixtures fluidity. To do so, an extensive literature review has been performed. Most of the published approaches were designed for specific material applications, so limitations and boundaries of each model are important to understand. Based on this review, a new analytical framework is developed. As such, the framework will allow researchers and engineers to develop a common understanding in categorizing and choosing various mastic types. It may also assist in further choices for characterization or design.
Measuring the viscosity of asphalt mastics with different filler concentration at elevated temperature is crucial for evaluating the effect of different parameters on the viscosity of asphalt mastics. Conventional methods that are currently used for measuring viscosity of bitumen are designed for bituminous behaviour and are not particularly suitable for mastics. Other methods can be found that are capable of measuring the viscosity of mastics at lower and intermediate temperatures. This, however, does not give enough information for designing the temperature of mixing or compaction of asphalt mixtures, since the flow behaviour of the material will be significantly different at higher temperatures. Hence, to enable control of the mastic viscosity design, a method should be used that is sensitive enough to notice the effect of fundamental parameters on the mastic viscosity and produce reliable and repeatable results.

Therefore, a new test protocol is developed, which is able to measure the viscosity of mastics with a wide range of filler concentrations at elevated temperatures. The test protocol is based on a commercially available viscometer for which various rotors and cups are available. In this research, these are investigated over a wide range of filler types and concentrations as well as shear rates. In selecting the fillers, important details are given to the mineralogy and shape of the fillers to enable understanding of these parameters in the resulting mastic behaviour. To ensure repeatability of the developed protocol, all steps have been described in detail.

In addition to the framework for determining the viscosity of mastics and a protocol for measuring the viscosity of asphalt mastics, focus is placed on the measurement of mastic viscosity as a function of various important parameters such as filler shape, size, size distribution and concentration, which is most relevant to mixing and compaction. Such understanding could lead to improved material component selection, better combination selections and improved predictions of the actual field behavior.

To investigate the sensitivity of the developed protocol, environmentally susceptibility is also included in this research by conditioning several samples to ageing and moisture. Particle settlement and agglomeration can further modify the resulting mastic behaviour from the intended. To analyze this, X-Ray Computed Tomography is utilized to determine the size and location of the filler particles inside various mastic samples.

The differences in viscosity behaviour of mastics partially could be explained from the different shapes and sizes of fillers, but the filler-bitumen interaction also has an important effect on the mastic rheological behaviour. It can be hypothesized that the interaction between the filler and bitumen changes the properties of the bitumen around the filler grains. Fillers influence the properties of this fraction of bitumen, and changing the type of filler can alter its characteristics, such as thickness and stiffness. As such, this influenced layer of bitumen defines the interfacial properties of filler and bitumen, and can have a significant effect on the rheological behaviour of asphalt mastics. Considering the size and variability of this area, investigating the influenced layers directly is rather complex. For this reason, in order to investigate the filler-bitumen interaction, the developed protocol is
utilized and combined with micro-mechanical Finite Element analyses, in which the shape and size of the filler particles are varied in the bitumen matrix.

In Sweden, for many years now, a new type of mixing protocol has been applied, in which the order of mixing is changed from the conventional method. Improved workability and diminished mixing and compaction energy needs have been important drivers for this. However, so far very little fundamental understanding was available to explain the reasons behind these advantages. Considering that it is the mastic phase which is modified by changing the mixing order, it is an interesting case study to apply the tools and insights developed in this Thesis to further explain the mechanisms of workability in connection with the mastic phase.

To do so, the analytical viscosity framework is combined with a mixture morphology framework, to upscale to the mixing level, and tribology principles to explain the interaction between the mastic and the aggregates. From the mastic viscosity protocol it was found that the mixing order significantly affects the resulting mastic viscosity. To analyze the effect of this on the workability and resulting mixture performance, X-Ray Computed Tomography is used to analyze mixtures produced by the two different mixing sequences and mechanical testing was utilized to determine the long-term mechanical performance. In this, mastic viscosity as a function of particle concentration and distribution is directly coupled to improved mixture workability and enhanced long term performance.

1.3 Thesis Outline

In the following an extended summery of five appended papers is presented. The thesis consists of 7 chapters:

Chapter 1: Introduction

The first chapter explains the structure of the research and the relation between the Papers.

Chapter 2: Viscosity framework

Chapter 2 discusses theoretical models which are calculating the relative viscosity of suspension and presents an empirical framework for calculating relative viscosity of asphalt mastic as a suspension and asphalt mixture as a granular paste (Paper I).

Chapter 3: A new protocol for measuring asphalt mastic viscosity

This chapter presents the new protocol for measuring the viscosity of asphalt mastic at elevated temperature and wide range of filler concentration (Paper II).

Chapter 4: Discussion of experimental results
The effect of different filler properties on the viscosity of asphalt mastic is discussed in this chapter, as well as the sensitivity of the viscosity test protocol to the environmental conditioning and different types of fillers. (*Paper III*).

**Chapter 5: Influence of filler-bitumen interface in mastics**

The effect of filler-bitumen interface on the rheology of mastic is presented in this chapter, based on performed experimental and numerical studies (*Paper IV*).

**Chapter 6: Workability of asphalt mixtures and mixing sequence**

Chapter 6 draws a link between the viscosity of mastic and the viscosity framework, and evaluation of the workability of asphalt mixtures. In this chapter sequential and conventional mixing methods are compared in term of the workability, internal structure and mechanical performance (*Paper V*).

**Chapter 7: Conclusions**

The last chapter in the extended summary presents the conclusions that were drawn in this Thesis and recommendations for further research and improvements are made.
2 Viscosity Framework

In this chapter a summary is given of the extensive literature review to evaluate various theories for the calculation of the viscosity of suspensions. Given the limitations of the existing theories, a new analytical viscosity framework was developed that is capable of characterizing the viscosity of suspensions over a wide range of particle concentrations. As such, the framework will allow researchers and engineers to develop a common understanding in categorizing and choosing various mastic types, using five fitting parameters.

2.1 Theoretical Background

Viscosity is defined as a resistance of a material to flow under shear stress. Asphalt mastics are generally made by mixing asphalt binder (i.e. bitumen) with certain percentages of mineral fillers. As such, mastics can be treated as suspensions, in which bitumen is the fluid phase and the fillers are the particles. Over the years, suspension viscosity has received a lot of attention due to its practical importance. Einstein addressed the viscosity solution in his paper in 1906. In his theory, particles are positioned far enough from each other, such that no interaction between the particles will occur [17]. Einstein equation is shown by:

\[ \eta_r = 1 + \eta' \phi \]

where \( \eta_r \) is a relative viscosity of suspension, \( \phi \) is the particle concentration (\( \phi = V_p / (V_l + V_p) \), \( V_l \) = volume of liquid, \( V_p \) = volume of particles) and \( \eta' \) is the intrinsic viscosity which is empirically related to the particle physical characters such as size, shape and rigidity and also particle interaction with the interstitial fluid.

Einstein derived his equation by solving the dissipation of energy for very dilute suspensions and found a value of 2.5 for \( \eta' \) for spherical rigid particles. The Einstein equation is the basis of most theories, which are dealing with the calculation of viscosity of suspensions. Most researchers have tried to extend the Einstein equation for higher percentages of particle concentration. In the paper I a summary is given of several of the most frequently used models. Frankel and Acrivos is one of these models.

Frankel and Acrivos solved the viscous dissipation energy for two neighboring particles with thin hydrodynamic flow between them [18]. From this, they calculated the energy for multiple particles, and suggested the following equation for calculating viscosity.
\begin{equation}
\mu_c = C' \left\{ \frac{(\phi / \phi_c)^{\frac{1}{3}}}{1 - (\phi / \phi_c)^{\frac{1}{3}}} \right\}
\end{equation}

(2.2)

For determining the constant $C'$ they used the Simha cage method [19]. In this method they calculate dissipation energy for a particle surrounded by other particles (six particles) in the cubic configuration plus an influence layer around that particle. They determined $C' = 9/8$ for spherical and $C' = 3\pi/16$ for cubical particles.

### 2.2 New Framework for Calculating the Viscosity of Asphalt Mastics

Some of the previously developed theories are designed for low concentrations, such as the Einstein equation, and some others actually more suited for high concentrations, such as the Frankel equation. These equations could therefore be used as the boundary asymptotes for the viscosity as a function of concentration, Figure 1. Due to Frankel’s definition of maximum concentration, however, at a certain concentration this asymptote tends to infinity. Obviously this can never be an accurate representation of the viscosity of the type of suspensions including granular materials in bitumen at a given temperature. With increased concentration there may be a sharp increase of viscosity, but it would never go to infinity and, as such, would diverge from the Frankel asymptote.

![Figure 1. Relative viscosity versus concentration bounded by two asymptotes.](image)

As discussed earlier, asphalt mastic can be considered as a suspension with different concentration, in which the filler is immersed in the bitumen. In this suspension, the filler consists of particles with different geometries and sizes that are surrounded by a layer of bitumen. This layer around the particle may in fact be divided into two sub-layers: (i) the layer of bitumen around the particle that is partially influenced by the particle and does not have the same flow characteristics as the ‘free’ binder that is farther away from the particle and (ii) the layer of bitumen that has adhered to the particle and as such is acting as though it
Viscosity Framework

has become part of the particle itself. The first layer is here referred to as the “partially influenced bitumen layer (\( \varepsilon \))”, the latter is here referred to as the “adsorbed asphalt layer (\( \delta \))”. Particles with the adsorbed layer will be considered to have a new radius and will be referred to as “effective particles” (Figure 2). The adsorbed bitumen layer (\( \delta \)) could be calculated from [10]:

\[
\delta = \frac{\phi_e - \phi}{\phi G_s A}
\]  (2.3)

where \( \phi_e \) is the effective particle concentration, \( G_s \) is the specific gravity of filler and \( A \) is the surface area of the particles.

![Figure 2](image)

**Figure 2.** Coating layers around the fillers.

The distance between the two effective particles is noted as \( h \). This distance \( h \) is zero if the two effective particles contact each other and infinite for very dilute suspension.

An overall measure of \( h \) in a mixture can be found from [20]:

\[
h = 2r \left[ \left( \frac{\phi}{\phi_m} \right)^\zeta - 1 \right]
\]  (2.4)

where \( \phi_m \) is the maximum concentration and the mixture does not have any free binder [21]. Resistance to the flow comes from particle to particle contacts and makes for a continuum network of friction. The value of the maximum concentration depends on size distribution, shape and type of aggregate and also binder characteristics such as stiffness of binder. Accurately measuring or calculating the maximum concentration according to the above definition is of paramount importance for calculating the viscosity of asphalt mastic.

Under these conditions the resistance to the flow comes from the frictional reaction between particles and the behaviour of material is predictable in the frictional regime.

When the frictional force become dominant, the distance between particles is equal or less than \( 2\delta \). Due to the direct contact of effective particles, most resistance to flow comes from the friction. Hence for calculating the viscosity in this regime the following equation is proposed:
where \( h_r \) is a relative distance between particles and is governed by:

\[
h_r = \frac{h}{r}
\]

(2.6)

where \( r \) is the average weighted radius of the particles.

In equation (2.5), the term of \( (\frac{\delta}{r} - h_r) \) 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.

In each aggregate gradation there is a range of particle size within the entire size distribution that has more influence on the amplitude of the frictional force. This part of structure is here referred to as the “primary structure” or dominant aggregate size range [22]. For bringing the effect of primary structure into the calculation, a term was added to equation (2.4). So the equation changes to:

\[
\eta_r = \left( \frac{\delta}{r} - h_r \right) N_c \cdot C_1 + N_c^p \cdot C_2
\]

(2.7)

where in this equation \( N_c^p \) represents the number of particle in primary structure and \( C_2 \) is a frictional coefficient.

The mastic with concentrations less than maximum concentration behaves in the hydrodynamic regime. In the hydrodynamic regime viscous behaviour of suspensions can be described with two equations, Einstein and Frankel, depending on the percentage of particle concentrations. For mastics with low concentrations the distance between particles is much bigger than the filler radius, so the effect of particles on each other is negligible and the Einstein equation is valid. By increasing the percentage of particles, the distance between particles becomes smaller and the particles start to affect each other. If the distance between particles is \( 2\delta \) up to \( 2\zeta \) (figure 3), the mastic is still in the hydrodynamic regime; however significant interaction of particles increases the viscosity of mastic sharply. In this manner the particles come closer to each other but the friction interaction does not occur. The Frankel equation can explain the viscosity behaviour of mastic very well under these conditions.
Even though the Einstein and Frankel asymptotes can approximate the very low and very high conditions rather well, the transition part between these extreme limits remain without definition. For this reason an equation that can calculate the viscosity of the mastic in the transition part was derived as:

$$\eta_r = C(1/h_r)^n$$

(2.8)

Based on the above discussion, Figure 4 shows the flowchart of the general framework that was described in the above for calculating the viscosity of mastic at different concentrations.

2.3 Summary

Relative viscosity behaviour of a suspension in respect to the particle concentration can be divided into two regimes, hydrodynamic and frictional. Using Einstein and Frankel theories, the relative viscosity of asphalt mastics with lower and higher filler concentrations can be calculated, respectively. The developed transition equation between these two theories can be used for calculating the relative viscosity at the intermediate filler concentration. So based on these three models, the framework can thus cover the viscosity behavior of mastics in the hydrodynamic state for a wide range of filler concentrations. Mastics with very high filler concentration can be considered to behave in the frictional regime in which most of the resistance to flow comes from frictional forces between two particles. For this, a formulation is defined in the framework based on this frictional interaction which can thus be used to calculate the relative viscosity of mastics in the frictional regime.

While having a framework for determining the viscosity over a wide range of particles is a good tool for studying and comparing the viscosity behaviour of asphalt mastics under the influence of various parameters, it does not substitute the need for a reliable and appropriate laboratory method. Nor does can it be used to predict material response, based only on fundamental properties. The latter is addressed in Chapter 4 and the next Chapter is discussing the development of a new test protocol.
Figure 4. General framework to calculate the viscosity of asphalt mast
3 A New Protocol for Measuring Asphalt Mastic Viscosity

Having a reliable and repeatable method for measuring the viscosity of asphalt mastic at appropriate mixing temperature and for a wide range of filler concentrations is important to enable the investigation of the effect of various parameters on mastic behaviour. Due to the high sensitivity of the viscosity to various parameters, such as the size of the filler particles, the mixing condition, the temperature effects and the viscometer geometry; focus has been placed on all these aspects in order to have reliable and repeatable test results. In the following, an overview is given of the extensive laboratory work that has been done in this Thesis towards the development of a new protocol for the measurement of mastic viscosity over a wide filler concentration range.

3.1 The need for a new test

As mentioned in the previous section, fillers that are embedded inside the bitumen can absorb part of the bitumen and form an absorbed layer on the outside of the particle, changing thus locally the flow properties of the bitumen. These new flow conditions in the presence of filler can significantly change the viscosity behaviour of the neat bitumen and should thus be understood. The viscosity of mastics, filler-bitumen mixtures, is a property which is sensitive to the important parameters such as filler size, size distribution, particle shape, particle mineralogy and bitumen rheology.

Due to significant influence of bitumen rheology on asphalt mixtures performance, determining rheological properties of bitumen has received a great deal of interest over the years. A lot of research has been conducted for measuring the viscosity of bitumen using different equipment set-ups and several test standards were established such as, among others: Kinematic Viscosity of Asphalts (bitumen) (ASTM D2170 / D2170M) [23], Viscosity of Asphalts (bitumen) by Vacuum Capillary Viscometer (ASTM D2171 / D2171M) [24], Saybolt Viscosity (T072-10-UL) [25] and Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer (T316-11-UL) [26] (ASTM D4402 / D4402M) [27]. In all these standard test methods, however, bitumen without additive or filler is considered as the testing material.
It would of course be easiest to use an existing viscosity test procedure for mastics, but such a test must be suitable for the rheological behaviour of the mastic particularities and its dependencies. Even for modified bitumen, without the mineral fraction, many different standards have been developed, e.g., Standard Test Method for Apparent Viscosity of Asphalt Emulsion Residues and Non Newtonian Bitumen by Vacuum Capillary Viscometer (ASTM D4957) [28], Standard Test Method for Viscosity of Emulsified Asphalt by Saybolt Furol Viscometer (ASTM D7496) [29] and Standard Test Method for Determining the Viscosity of Emulsified Asphalts Using a Rotational Paddle Viscometer (ASTM D7226) [30].

Despite the fact that there is currently no standardized mastic rheology protocol, some earlier efforts have been made to measure mastic properties. In most of these, focus was placed on low and intermediate temperatures and often a Dynamic Shear Rheometer (DSR) was utilized. Next to the DSR, Di Benedetto and his co-workers [31-33] introduced an Annular Shear Rheometer (ASR) for measuring the complex shear modulus, $G^*$. In this research a new test was developed, which enabled the detailed investigation of filler particles and is focused to linking the mastic level to asphalt mixture workability on the larger scale. In the following the details of the test are further described.

### 3.2 Test Development

Measuring the viscosity of asphalt mastic is an important but also challenging task. As all parameters which were discussed earlier have a significant influence on the measurements, it is essential to understand their effect by having a method that is sensitive enough to note detect the influence of the various important factors and flexible enough to allow for varying them. To have a standardized protocol for such a complex material, it is important that the entire protocol is extremely well defined since any variation could lead to significantly different results leading to erroneous conclusions as to the effect of other parameters. The repeatability of the test result is therefor an important aspect to which a lot of attention has been given in this research.

The temperature is an important parameter, which can affect the viscosity result dramatically. There are two stages where the temperature plays an important role in the protocol: during the filler and bitumen mixing and during the actual measurement. To be able to compare the viscosity result of mastics and making conclusions regarding their fundamental parameters, the mixing temperature should be kept the same and should thus be suitable for all filler concentrations. 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; sedimentation at higher temperature and changes in the viscosity behaviour of mastic at lower temperature especially for the modified bitumen.

The particle size is another parameter which has an important influence on the viscosity of asphalt mastics. It can be even more important when the mastics are tested at different concentrations for comparing the effect of filler content on the viscosity of mastics. Care
should therefore be taken to take a representative sample of the stored filler when adding it into the bitumen to avoid erroneous conclusions. Adding filler to the bitumen can also affect the rate of agglomeration. Agglomerated particles make a bigger artificial particle that can act as a coarser particle. Air bubbles can also be introduced into the mastic during the filler adding into mastic mixing process. Air pockets or enclosed bubbles have a significant effect on the shear stress and thus, seemingly change the measured viscosity result. Therefore, when modifying the filler adding process this issue must be taken into account.

As the intention of this test is to measure the viscosity of asphalt mastics at higher temperature where the asphalt mastics are mostly liquid, the cylindrical viscometer was chosen. Some of the available viscometers on the market are suitable for Newtonian material and others for non-Newtonian flow characterization. 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 mastics or for very low shear rate tests, so the geometry of the cylinders should be designed to avoid this zero shear zone for all filler concentrations.

Bitumen is a Newtonian material, meaning the viscosity of bitumen under different shear rates is constant, yet it is possible that bituminous mastic does not follow Newtonian behaviour for certain filler concentrations [34]. When applying a shear rate that is quite low, the mastic viscous behaviour may show a linear relation between the shear stress and shear rate. When dealing with a “shear thinning” mastic, the relationship between shear stress and shear rate at lower rates can also initially show a linear relationship, followed by a nonlinear behaviour. Hence, designing a suitable test procedure with appropriate boundary conditions is a main key of achieving accurate and sufficient information about the viscosity behaviour of asphalt mastics.

To validate the viscometer’s results, certified reference standard oil produced by Cannon instrument company® was used to compare the measured with the classified viscosity at different temperatures. Table 1 gives both the measured and the classified viscosity. From the comparison, it can be concluded that the instrument gives reliable results. Additionally, to ensure the repeatability of the test, each test was repeated several times and all results were within the range of the accuracy expected from this test.

Table 1. Classified and measure viscosity of standard oil.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Certified Viscosity (mPa.s)</th>
<th>Measured Viscosity (mPa.s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>9754</td>
<td>9743</td>
</tr>
<tr>
<td>50</td>
<td>1487</td>
<td>1481</td>
</tr>
<tr>
<td>100</td>
<td>114</td>
<td>115</td>
</tr>
</tbody>
</table>
3.3 Standardized Test Protocol

3.3.1 Sample Preparation

According to the above discussions, the sample preparation has enormous effect on the viscosity of the asphalt mastics. Therefore, to reduce errors in measuring the viscosity of the mastics as well as creating the possibility of comparison, the sample preparation should be standardized. 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.

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, the filler was placed in an oven for 24 hours to get completely dry and also heated up to the mixing temperature. To mix the bitumen with the filler, the bitumen was also heated to the mixing temperature. For this 200 g of bitumen was placed in the oven for one hour.

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. During this process attention was placed on creating a homogeneous mastic and avoiding filler agglomeration as much as possible. 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.

3.3.2 Test conditions

As a test temperature, 100˚C was chosen. This temperature was selected as the optimum temperature viscosity test for bituminous mastic after several trials. The main reason to note this as the optimum is that, at this temperature, the filler sedimentation speed is moderate and the mastic has more time to be tested as a homogeneous material and still give a repeatable result. Furthermore, at this temperature, all tested mastics had sufficient fluidity, even at high filler concentrations. Finally, the tested mastics still show behaviour in the hydrodynamic regime at this temperature [14].

The used viscometer must be able to apply a constant shear stress in each measurement since, for a Newtonian material, at each shear stress only one shear rate, and consequently, one viscosity will be determined. So for a correct reading it is essential to apply a constant shear stress when the instrument is collecting data. The range of the applied shear rate was chosen to be appropriate to the mastic’s flow behaviour.
3.3.3 Test set-up

To satisfy all the above conditions, a rotational co-axial viscometer was utilized. Due to varying flow behaviour of the tested mastics with the different filler concentrations, it was considered that accurate measurement should be possible when mastics display a different type of behaviour such as Bingham flow, shear thinning or thixotropy.

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 cylinder (rotor) for Newtonian materials (Figure 5). In this standard, the equations used to convert the raw experimental results to viscosity are based on the assumptions of dealing with a Newtonian material. From these assumptions, the limitations of using the cylindrical rotor are considerable. As long as mastics behave like a Newtonian material, the shear gradient can be considered linear in the gap of the coaxial cylinder. And the radiuses of inner and outer cylinder were chosen such as to create a 1.0 mm gap to reduce the plug zone.

When increasing the filler concentration, mastic starts to behave more non-Newtonian. From the particular filler concentration at which this behaviour is noted, the use of the cylindrical rotor to measure the viscosity is no longer appropriate since a boundary bias starts to occur and due to difficulties for the inner cylinder to be placed inside the stiffer mastics. In addition to these, 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 5-d) was chosen in this study for the higher filler concentrations. The range of filler concentrations 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 by volume can be a suitable boundary for shifting from cylindrical rotor to a vane shaped rotor. This threshold could also become a material parameter which later on could be used for comparison and/or design of mastics.

To avoid the slippage on the wall of outer cylinder as much as possible, an outer cylinder with small grooves on the inner wall of the cup was used (Figure 5-b). Considering the important effect of temperature on the asphalt mastic viscosity, having an accurate control on the temperature is essential for any viscosity measurement. For this reason, the whole system was placed in the Peltier Concentric Cylinder Jacket to provide the required temperature. The Peltier jacket with associate cooler can provide a range of temperature from 0°C to 150°C.
3.3.4 Viscosity measurements

The procedure for running the viscometry test was defined according to the mastic properties, after running some simple tests. With both the cylindrical and vane shaped rotors, tests were performed on shear rate control from $5 \times 10^{-3}$ rad/s to 0.4 rad/s for most tests and were varied occasionally for some samples to optimize the measurement. The continuous-ramp procedure was designed into two steps, with first a continuous increase of the shear rate, followed by a continuous decrease.

As mentioned earlier, at elevated temperatures the particles have a tendency to sediment rather rapidly. For this reason, the testing time was held as short as possible to avoid sedimentation of the fillers in the bitumen. Effectively, each ramp took 120 seconds during which period 180 measurements for the shear rate sweep were made. For each mastic type, at least three tests were done to ensure that a repeatable and reliable result was found. Eventually, the relative viscosity for all samples at any given shear rate were computed by normalizing the viscosity of sample with the viscosity of the neat bitumen.

3.4 Summary

In this chapter, first the need for a test to measure the viscosity of the asphalt mastics at high temperatures was discussed. Secondly, focus was places on identifying the dominant factors that may impact the test results and that need to be controlled in the protocol. In this, the stages of sample production and test procedure should be carefully controlled. Additionally, the geometry of the viscometer must be such that it is consistent with the nature of the asphalt mastics.

According to the developed protocol for the samples preparation, filler samples must first be thoroughly homogenized to avoid inconsistent filler gradations in subsequent tests. Samples should be mixed at a temperature at which i) mastic is totally fluid and ii) it is possible to produce all mastics, specially those with higher filler concentrations. Mixing time
should be long enough to ensure the homogeneity of the mastics (i.e. the distribution of the filler inside the mastic) and kept constant for all samples. Mastics at the higher filler concentration are expected to behave as non-Newtonian materials. Thus choosing the correct geometry of the testing equipment that can capture this is rather important. In the developed protocol, a coaxial concentric cylinder is suggested for measuring the mastic at higher temperature. Based on extensive investigations with a wide variety of mastic types, it was concluded that the rotor used should be adjusted to the filler level. As will be further explained in the next chapter, the results showed that at low filler concentration a cylindrical rotor is appropriate and at higher filler concentration a vane-shaped one should be chosen. The concentration level at which the rotor type should be changed, was identified as the critical concentration and was seen at approximately 20% for all tested mastics (also discussed in detail in the next chapter). In addition, to avoid the zero shear zone (or plug zone), the gap between the inner and outer cylinder should be as small as possible and is suggested at $1\text{mm}$. 
4 Discussion of Experimental Results

To investigate the effect of the fillers on the mastics viscosity, an extensive laboratory program was performed. Filler types were carefully selected to investigate the influence of their specific characteristics (e.g. shape or mineralogy). After the calibration and validation procedure, an extensive set of samples were tested for their viscosity as a function of filler concentration and types. Additionally, X-Ray computed tomography was utilized to investigate agglomeration potential of the fillers inside the mastics, which can lead to effectively different filler size.

4.1 Filler Characterization

In this study three types of filler were used. Two of them are silica based (M10 and M600), which are manufactured by SIBLECO, and one type of filler is Fly ash (FA). These fillers were chosen to have different type of parameters to study their effect on the mastic, given the bitumen is kept as a constant. A standard 70/100 bitumen from Nynas AB was used. M10 and M600 have almost the same type of mineral components, but different physical parameters such as particle size, size distribution, specific surface area and density.

To characterize the fillers, scanning electron microscope (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 6 shows examples of pictures captured by SEM from all fillers, Figure 7 shows the filler size distribution and Table 2 shows results of BET and Helium Pycnometer.

Due to high specific surface area of fillers, the internal forces between the grains of fillers are more significant than with coarser particles. Different fillers have different internal forces, and based on the shape and mineral type of filler, the internal forces can be chemical and/or physical nature. When the particles adhere to each other they could effectively form a larger structure, an agglomerated grain, which changes their distribution through the mastic as well as their local interactions. In order to study this potential agglomeration inside the mastic, the difference between the mineral filler gradation before and after mixing in the bitumen was studied utilizing X-Ray tomography. 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
Discussion of Experimental Results

filler gradation curve, which indicates the amount of filler agglomeration inside the samples, Figure 8.

![Filler M10](image1)
![Filler M600](image2)
![Fly Ash](image3)

**Figure 6.** SEM photograph of fillers, a) M10, b) M600, C) fly ash.

![Filler size distribution](image4)

**Figure 7.** Filler size distribution for M10, M600 and fly ash.

**Table 2.** BET and Helium Pycnometer results.

<table>
<thead>
<tr>
<th></th>
<th>M600</th>
<th>M10</th>
<th>Fly Ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific surface area (m²/g)</td>
<td>4.0</td>
<td>0.93</td>
<td>1.5</td>
</tr>
<tr>
<td>Density (kg/cm³)</td>
<td>2.75</td>
<td>2.79</td>
<td>2.41</td>
</tr>
</tbody>
</table>
Figure 8. M600 gradation before and after mixing.

As mentioned in the previous chapter, the results of viscosity testing depend on many factors. To evaluate the sensitivity of designed viscosity test to the other potential factors such as environmental conditions and bio-based materials, some samples were exposed to different environmental conditions before viscometry and for preparing some asphalt mastic samples three types of bio-based mastic additive materials were used. The first type is an oil based product made from cashew nut shells and supplied by Van Weezenbeek Specialities (Netherlands). The product is named Rheofalt HP-EM and will be referred to as ‘Bio1’. For the purpose of these investigations Bio1 is used as an additive to examine the modification of properties when combined with neat binder. Bio1 is mixed at 5% by weight of virgin 70/100 bitumen.

Bio2 was supplied by Biokol Lilliestrale & Co KB (Sweden) and was supplied in two batches, in which batch 1 was grinded mechanically and is a fine material; batch 2 was grinded by hand, leading to a more varied and coarser particle size. Bio3 was the same base material as Bio2 but washed several times to remove certain chemicals, oils and tars.

4.2 Viscosity Measurement Results

The mastic samples were prepared with all three fillers at different filler content ranging from 5% to 50% or more, depending on stiffening effect of the filler on the bitumen. These mastic samples were used to measure their viscosity at 100°C.

Figure 9-a and b show examples of the relationship between shear stress and shear rate at different filler concentrations, which were measured with the cylindrical and vane-shaped rotor respectively. As can be seen from the graphs, the viscosity of the mastics increases as a function of filler percentage, which would be expected. However, this increment is not always linear for all filler concentrations. A clear effect of the filler on increasing the viscosity can be seen in the plot of relative viscosity versus filler concentration (Figure 10). Due to nonlinearity of shear stress and shear rate relationship for mastics with higher filler concentrations, the mastic viscosity is different from one shear rate to another one. So, to have correct result the viscosity is calculated for specific shear rates, even for lower filler concentration mastics, which do display linear Newtonian behaviour.
From the graphs, it can be seen that the relationship between relative viscosity and concentration is linear until a certain critical filler concentration it is reached and it becomes nonlinear. In general, the relative viscosity of bitumen mastic increases by increasing the filler concentration, but from the critical filler concentration the rate of the increment in relative viscosity increases sharply. This critical filler concentration can be different for each type of filler-bitumen combination. Shape, size and size distribution of filler seem to have a significant influence on this value.

**Figure 9.** Viscosity result of mastic with filler M10 at 100 C with a) cylindrical rotor, b) vane-shaped rotor.

**Figure 10.** Relative viscosity of mastics at 100 C and 1.5 1/s shear rate with a) cylindrical rotor and b) vane-shaped rotor.
As mentioned previously, the test results showed that for all fillers after a certain filler concentration the relation between shear stress and shear rate is not linear any more. This concentration is a boundary limit for using the cylindrical rotor as the mastic is not Newtonian anymore. Hence for mastics with lower filler concentration than this boundary concentration, the relative viscosity result from cylindrical rotor can be used and for higher filler concentration, as the mastic is not Newtonian anymore, the result from vane shaped rotor is more accurate. According to this principle, the previously shown graphs are modified and the results from the cylindrical and vane-shaped rotor are combined together and made new relative viscosity versus filler concentration which is shown in Figure 11.

![Figure 11. Relative viscosity of mastics at 100 C and 1.5 1/s shear rate with combination of cylindrical and vanned rotor, a) Entire curves, b) lower part of curves (for better visualization).](image)

As shown in Figure 11, bitumen mastic with filler M600 shows higher relative viscosity for all filler concentrations in comparison to the other mastics. In this thesis, it is hypothesized that this increase in viscosity of mastics with finer fillers can be contributed to the fact that finer fillers provide a larger surface area to interact with bitumen. This may lead to more bitumen being adsorbed by the outer filler layers. Thus a greater volume of bitumen will form a structured layer, attached to the particles and less free bitumen will be available in the mastic matrix, thus reducing the fluidity.

Additionally, it was observed that the mastic with filler M600 showed a sharp increase of its relative viscosity at lower concentrations than mastic with filler M10. This sharp increase could indicate that the relative viscosity of mastics with finer fillers reaches the nonlinear zone at lower concentrations, compared to mastics with coarser particles (given all other parameters being the same).

Comparing the relative viscosity results of mastics with either M10 or fly ash fillers gives insights into the effect of particle shape on the viscosity. Fly ash, as was shown earlier, has round-shaped particles and M10 filler is rather angular in their shapes. It can therefore be hypothesized that the possibility of particle rolling is easier when subjected to shear gives lower resistance to flow seen in the fly ash mastic samples as opposed to more interlocking of the M10 mastics.
4.3 Environmentally Conditioned and Biomass Filler Mastics

To further investigate the sensitivity and potential of the developed protocol for material changes in the mastic components, several environmentally conditioned samples were prepared for viscosity measurements. The measuring device and its geometry, mastic sample preparation and moisture conditioning and aging procedure are explained in paper III.

In figure 12-a, the results for the unconditioned bio-based binders are shown. From the figure it can be seen that Bio1 reduces the viscosity in comparison to the virgin binder while Bio2 and Bio3 increase the viscosity of the binder. Since Bio2 and Bio3 have been used as fillers at 38.5% (weight) this was expected. The difference in viscosity between BMB2 and BMB3 is 18.94 Pa.s with BMB2 having a viscosity of 14.1 Pa.s and BMB3 recording 33.04 Pa.s. One possible explanation for this could be that due to the washing of the Bio3 sample, which removes oils and tars adhered to the surface of the particles, certain substances from the 70/100 binder could be absorbed by the Bio3 particles reducing the overall liquidity of BMB3 and increasing the viscosity. The control 70/100 sample had a viscosity of 2.56 Pa.s and was reduced to 1.58 Pa.s when combined with 5% Bio1. The results indicate that BMB1 could be capable of reducing the viscosity of certain binders and may be appropriate for reducing the viscosity of BMB’s that incorporate fillers and modifiers that naturally increase binder viscosity.

The aged viscosity results are shown in figure 12-b. The results demonstrate that aged material has a higher viscosity than its virgin counterpart, as expected. All results are comparable to the virgin material with the 70/100 binder increasing in viscosity from 2.56 Pa.s to 4.29 Pa.s (67%), BMB1 increased from 1.58 Pa.s to 2.74 Pa.s (74%), BMB2 increased from 14.10 Pa.s to 26.05 Pa.s (85%) and BMB3 increasing from 33.04 Pa.s to 64.18 Pa.s (94%).

Figure 12-c illustrates the moisture viscosity results for the virgin 70/100/M10 & BMB1/M10. In both cases, the moisture conditioned samples (M) have a higher viscosity than the control samples (C), which were not subjected to any moisture. The 70/100/M10 increased by 16% while BMB1/M10 increased by 27%. The viscosity of the 70/100 samples without M10 is negligible with the control at 2.60 Pa.s and the moisture sample at 2.33 Pa.s which is also very similar to the initial virgin viscosity test for 70/100 which was 2.56 Pa.s indicating that the 30 day control period did not have any significant affect the viscosity of the virgin binder samples.

Figure 12-d shows the results of the viscosity testing of the moisture conditioned samples of BMB2/M10 (batch 1) and BMB3/M10. The viscosity for these two samples is very similar between the moisture conditioned (M) and control (C) samples, notably in BMB3/M10 where the change is less than 1%. In the case of BMB2/M10 (batch 1) the viscosity of the moisture conditioned sample has decreased by approximately 10% compared with the control sample. In the case of the BMB3/M10 the addition of M10 and moisture has caused the viscosity to increase from 33.04 Pa.s to 146.7 Pa.s and increase of 443%, the increase in
viscosity is dramatic, signifying the large effect moisture can have on certain modified binders. The BMB2/M10 batch 1 sample has a viscosity of 29.67 Pa.s for the moisture sample, once again confirming the importance of particle size when combining filler material with binder as discussed previously.

### 4.4 Summary

Keeping the bitumen volume constant, filler is the main factor determining the rheological behaviour of mastics. Therefore, various fillers were chosen in such a way that the impact of certain filler characteristics on the viscosity of mastic could be studied. A variety of tests was used to analyze the filler morphology properties. From this it was confirmed that both M600 and M10 fillers are angular while Fly Ash has very round grains. It was also seen that M600 has much higher surface area than M10 and Fly Ash.

Experimental results showed that for similar filler concentrations, the M600 mastics had the highest viscosity, followed by M10 and then fly ash. Although Fly Ash has higher surface area than M10, it showed a lower viscosity. This could be explained from the round shape grains of Fly Ash. Additionally, the results of the environmental conditioning of the biomass-based mastics indicated that the viscosity test is sensitive enough to pick up the effects caused by the environmental conditioning. It also emphasized the importance of developing proper understanding of the effects of adding new filler and additives to asphalt mixtures, since a significant effect was noticed in the mastic samples after moisture and ageing conditioning. These effects may not have been identified, if only the binder itself had been studied, as was also shown in the measurements of the neat binder.

From the X-ray scanning analyses it was shown that an agglomeration parameter could be identified that resulted in an effective shift of the sieve curve of the dry filler material, once the filler was added to the bitumen. This finding has important implications for the resulting behavior of the mastic, since it means a different shape and distribution of the fillers is happening inside the mastic than what the original intention is.
Discussion of Experimental Results

Figure 12. a) Virgin viscosity samples @ 100°C, b) Aged viscosity samples @ 100°C, c) Moisture viscosity - 70/100 & Bio1 @ 100°C and d) Moisture viscosity - Bio2 & Bio3 @ 100°C.
5 Influence of the Filler-Bitumen Interface in mastics

In the previous chapter the influence of physical properties of the filler on the rheology of asphalt mastic was evaluated. The differences in the observed mastic viscosity behaviour were hypothesized to be caused by the different shapes and sizes of fillers and the filler-bitumen interactions. Each filler particle influences its surrounding bitumen layer, and its characteristics, such as thickness and stiffness, can be altered by changing the type of filler. Considering the complexity of investigating interfaces, in this Thesis, combined experimental and numerical techniques are employed.

5.1 The Hydraulic Volume of Filler

In this Thesis, the filler’s surrounding bitumen layer is divided into two parts, adsorbed layer and influenced layer. The size of the rigidly adsorbed layer was previously suggested between 2 - 10 Å [10]. The thickness of the adsorbed layer may, in most cases be negligible, considering the size differences, unless the filler is fine enough that the adsorbed layer becomes significant, for example in the case of lime [35]. In addition to the adsorbed layer the influenced layer, described earlier, is acting around the particles. Combination of the adsorbed layer, influenced layer and the particle itself are the hydraulic volume of the filler (Figure 13).

Figure 13. Scheme of adsorbed layer, influenced layer and hydraulic volume of filler.

Figure 14 illustrates how the mastics with lower filler concentration but higher filler surface activity can have the same filler hydraulic concentration as a mastic with a higher concentration but a lower filler surface activity. Figure 15 shows the same concentration for
nominal filler volume and hydraulic filler volume for the particular filler size. In both cases the concentration are kept the same, however due to the larger size of the hydraulic filler volume, the unit volume of mastic needs fewer but coarser filler grain. In addition to the size effect of the layer around the filler, this layer can also have a smoothening effect. Depending on its properties, it can change the mobility of the filler within the matrix as the interfacial properties between the filler and bitumen change.

Figure 14. Equal filler hydraulic volume concentration for two different circumstances, a) coarser filler with lower surface activity and b) finer filler with higher surface activity.

Figure 15. Equal filler concentration in the unit volume of bitumen, a) only fillers and b) filler hydraulic volume.

The hypothesis as explained above may be dominant in modified mastics, especially for adhesion promoter additives which act at the surface of the filler. A surfactant molecule is typically made of a hydrocarbon chain plus a polar group. If the filler has polar or polarizable components, the polar part of the surfactant molecule will make the polar bond with surface of aggregate, and bitumen will make a Van der Waals forces with the hydrocarbon chain of the surfactant molecule (Figure 16). This effect can increase the thickness of the adsorbed layer.

Figure 16. Adhesion of the surfactant molecules to the solid polar surface.
5.2 Effect of interface layer on the viscosity of mastics

To investigate the effect of the filler-bitumen interface, AkzoNobel Rediset was used for producing the modified mastics. According to the information released by the manufacturer, Rediset is designed to produce warm mix asphalt and it can decrease the mixing and compaction temperature by 30°C. Furthermore, it is suggested that Rediset improves adhesion between the binder and the aggregates by amine surfactants [36]. In this research, 1.5% Rediset (by weight of bitumen) was added to the mastics as the mean value recommended by the manufacturer.

Filler M10, M600 and Fly Ash and bitumen 70/100 were used for producing asphalt mastics. Each type of mastic was produced at two different filler concentrations 20% and 40% by volume. All samples were produced at two Rediset modified and unmodified conditions. All mastics were produced and tested according to the protocol provided in chapter 3.

Figure 17 show the flow curves of modified and unmodified bitumen 70/100, mastic M600, mastic M10 and mastic Fly Ash. Table 3 gives the viscosity of modified and unmodified bitumen and mastics at shear rate 6 (1/s) and 100°C.

![Flow curves of unmodified and modified: a) Bitumen 70/100 at 100°C, b) 20% and 40% of M600 at 100°C, c) 20% and 40% of M10 at 100°C, d) 20% and 40% of Fly Ash at 100°C.](image)
### Table 3. Viscosity of binders and mastics at shear rate 6 (1/s) and 100°C.

<table>
<thead>
<tr>
<th>Filler concentration</th>
<th>Viscosity (Pa.s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Bitumen</td>
</tr>
<tr>
<td></td>
<td>20%</td>
</tr>
<tr>
<td>Unmodified</td>
<td>2.5</td>
</tr>
<tr>
<td>Modified</td>
<td>2.3</td>
</tr>
<tr>
<td>Reduction (%)</td>
<td>10</td>
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</tbody>
</table>

While the unmodified bitumen gives the viscosity of 2.5 Pa.s, modified bitumen gives the viscosity of 2.25 Pa.s which is 10% lower than the viscosity of unmodified bitumen. The ratio of decreases in the viscosity is not the same for all samples. With the same amount of the modifier, the viscosity of mastic with 40% M600 is reduced by 63%. This is the highest reduction of viscosity observed among the modified mastics. The second highest reduction in viscosity is for mastic 40% M10 and then followed by 40% fly ash, 20% M600, 20% M10 and 20% fly ash.

The differences in the reduction in the viscosity of modified bitumen and mastics show that the additive not only changes the viscosity of bitumen, but also change the filler-bitumen interfacial properties. In addition to the surface area, shape of the filler grains also has significant effect on the reduction of viscosity. By comparing the results this can be concluded that, due to the geometry of the angular particles they have more difficulty for movement and/or rotation, this means generally angular particles produce higher viscosity compared to the round shape particles. Hence, softening the structured layer around the angular particles gives more freedom to them for movement and/or rotation, while the round particles have already had this freedom. Therefore the reduction in the viscosity of the mastics with M10 is more than the mastics with fly ash.

### 5.3 Micro-mechanical Finite Element Model

As mentioned previously, the properties of the bitumen in the adsorbed and influenced layers are affected by the filler. These layers define the interfacial properties of filler and bitumen, and can have a significant effect on the rheological behaviour of asphalt mastics. Considering the complexity of studying interfaces, investigating the influenced layers directly is challenging. For this reason, in this research numerical methods are combined with experimental techniques to investigate the influence of filler–bitumen interface in mastics.

The generalized Maxwell model is employed to simulate the viscoelastic behaviour of bitumen [37]. To determine the discrete Maxwell model, the bitumen 70/100 was tested with the dynamic shear Rheometer (DSR) at 25°C, 30°C, 35°C, 40°C and 45°C. Frequency sweeps, from 0.1 to 100 rad/s, were performed at a shear strain value within the linear viscoelastic range. A nonlinear least square regression algorithm has been used to obtain the Prony series
coefficients for viscoelastic model, represented with generalized Maxwell model. A discrete generalized Maxwell model approximates moduli by using n terms as [38]:

\[ G(t) = \sum_{i=1}^{n} G_i e^{\frac{-t}{\lambda_i}} \]  
\[ G'(\omega) = \sum_{i=1}^{n} \frac{G_i \omega^2 \lambda_i^2}{1 + \omega^2 \lambda_i^2} \]  
\[ G''(\omega) = \sum_{i=1}^{n} \frac{G_i \omega \lambda_i}{1 + \omega^2 \lambda_i^2} \]

where \( G(t) \), \( G'(\omega) \) and \( G''(\omega) \) are shear modulus, storage modulus and loss modulus respectively. \( G_i \) and \( \lambda_i \) are shear modulus and relaxation time of each Maxwell branch. The fitted complex modulus is shown in Figure 18.

![Complex modulus fitted by Prony series.](image)

**Figure 18.** Complex modulus fitted by Prony series.

In Figure 19, four different geometries which were used for FE modeling are depicted. To evaluate the effect of filler-bitumen interface with different particle shape and size on the behaviour of mastics, two different particle shapes and sizes were used. The areas of all angular and round, big and small particles are almost the same; this means all samples have almost the same filler concentration. However the surface area (primate) of angular particles is higher than the round particle in both big and small cases.
To define the interface layer between particles and bitumen the Thin Elastic Layer (TEL) was assigned on the boundaries of particles. The TEL contains a spring and a dashpot in series to enable the simulation the simple viscoelastic behaviour. Both spring and dashpot are available in normal and tangential directions (Figure 20).

To study the effect of the properties of the interface, different parameters were assigned for spring constant and dashpot’s viscous property. In Table 4, cases 1 to 4 represent weaker, case 5 and 6 represent same and case 7 and 8 represent stronger interfacial properties than what calculated.

Figure 19. Four different FE model’s geometries with their meshes.

Figure 20. Schematic of Thin Elastic Layer (TEL).
Table 4. Spring constant and viscous force of the interface.

<table>
<thead>
<tr>
<th>Case Number</th>
<th>Thin Elastic Layer (TEL) Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Spring Constant (MN/m)</td>
</tr>
<tr>
<td></td>
<td>1   2  3  4  5  6  7  8</td>
</tr>
<tr>
<td>1</td>
<td>19  19 19e3 19e3 19e9 19e9 19e11 19e11</td>
</tr>
<tr>
<td>2</td>
<td>19  19e3 19e3 19e9 19e9 19e11 19e11</td>
</tr>
<tr>
<td>3</td>
<td>87  87e6 87  87e6 87  87e6 87  87e6</td>
</tr>
<tr>
<td>4</td>
<td>87  87e6 87  87e6 87  87e6 87  87e6</td>
</tr>
</tbody>
</table>

Figure 21 demonstrates examples of the deformed meshes at the end of loading (t=0.095s). From these it can be seen that the levels of debonding are different for varying geometries. For comparing the effect of the interfacial properties on different filler sizes and shapes, overall mesh displacement of a stress controlled test of each geometry was divided by the displacement of the same mesh with rigid bonding (i.e. no interface element).

\[
Overall\ Displacement\ Ratio\ (ODR) = \frac{\text{Displacement with Interface Element, } \mu m}{\text{Displacement without Interface Element, } \mu m}
\]

Figure 21. Example of deformed mesh and colored map for Y component of stress tensor at t=0.095s with scale factor of 50 for deformation.
Figure 22 illustrates the overall displacement ratio of all four geometries and four cases. Results show that the effect of the weak interface (case 1) for angular fillers is higher compared to the round fillers. At weak interfacial properties, the shape of the filler is dominant and can be explained through the high stress concentration at the interface of the angular fillers. Keeping the spring constants the same, but increasing the dashpot constants (case 2), both shape and size of the filler have an influence on the overall displacement. Small particles (angular and round) provide higher surface area, hence show more interface effect upon displacement. Due to higher stress concentration around their interfaces, angular fillers (big and small) show more influence of the interface compared to round fillers.

Increasing the spring constant of the interface, the effect of the surface area becomes dominant. This effect is even more visible when increasing the value of the dashpot constant. Small angular particles showed the highest overall displacement ratio both at the end of unloading and end of recovery, followed by the small round, big angular and big round meshes.

In the case 3 the overall displacement ratio at the end of unloading (t = 0.1s) is affected mostly by the shape of the particles. However at the end of the recovery instead of the shape, surface area of fillers takes over as the dominant effect on the displacement.

For the models without an interface or strong interface properties (Figure 23), the big round particle shows the highest displacement followed by big angular, small round and small angular. From this, it can be concluded that for no interface system or stronger
interfacial condition, bigger filler particles will result in higher overall deformation. Within the bigger filler size the round one gives more displacement compare to the angular one.

Figure 23. Overall displacement ratio in Y direction at the top line without interfacial element.

5.4 Summary

In this Chapter, the impact of the filler-bitumen interface on the rheological behaviour of mastic was evaluated, using experimental and numerical methods. The results of this indicated that the shape and size of the filler have a significant impact on the filler-bitumen interface. This effect is even more pronounced in case of modified mastic. The viscosity results show that the additive is not only changing the viscosity of bitumen but also has significant influence on the surface of the filler. The effect of additives on the finer and angular fillers was found to be larger than the coarser and round filler particles. This effect was more visible for mastics with higher filler concentrations.

According to the results of the numerical modeling, for weak interfacial properties the effect of the shape of the particles was dominant. When representing the interface properties by intermediate spring constants and strong dashpots, the effect of the surface area was dominant at the end of the relaxation period while at the end of the loading the effect of the filler shape seemed more dominant. This emphasizes the importance of creating further understanding to understand all the physical and chemo-mechanical parameters that control filler-bitumen interaction.

In summary, it was shown that interfacial properties have an important effect on the rheological behaviour of asphalt mastics, especially when modified. Having an insight into the effects of filler shape and size on the interfacial properties can lead to better design of mastics in asphalt mixtures.
6 Workability of Asphalt Mixtures and Mixing Sequence

An asphalt mixture can be viewed as a granular paste in which the asphalt mastic plays the media role. Therefore, the rheology of asphalt mastic has a direct impact on the workability of asphalt mixtures. As discussed earlier, there are several parameters which have influence on the rheology of asphalt mastics, such as particle size, shape, size distribution, etc. These parameters still have important impact on the workability of asphalt mixtures, however particles at the mastic level refers to the filler grain but at the mixture level refers to the aggregate grain. If keeping all these variables constant, theoretically the workability of asphalt mixtures should be constant, however some practical issues may be involved which result in different workability. Mixing sequence is one of the parameters for which its effect on the workability of asphalt mixtures can be evaluated by utilizing a viscosity framework.

6.1 Sequential Mixing Method

6.1.1 Instruction of the Sequential Mix

Conventionally, most asphalt mixtures are produced by first mixing all the mineral aggregates together (the ‘dry mixing’), followed by a spraying of the bitumen on this aggregate mixture (the ‘wet mixing’) followed by further mixing [39]. Though the risk for segregation and heterogeneity of the mixture are still noted as issues [40], this type of mixing is still most commonly used.

In Sweden, for many years now, a new type of mixing protocol [41], has been applied, in which the order of mixing is changed from this conventional method:

Step 1: The load carrying aggregates (here named “primary structure”) are mixed with the bitumen.

Step 2: The filler particles are added to the mixture. Filler in this case are the mineral aggregates with size 75\(\mu m\) or smaller.
Step 3: The fine aggregates, which are bigger than filler and smaller than the primary structure (here named “secondary structure”), are mixed in with the entire mixture.

![Sequential Mixing Method Diagram](image)

**Figure 24.** Schematic of conventional Vs. sequential mixing methods.

### 6.1.2 Discussion of the sequential mix benefits

From [41-44] the following four benefits are reported when applying the sequential method:

1) Lower manufacturing temperature
2) More homogeneous mixture
3) Thicker coating around the coarser particles
4) Environmental and economical benefits

The first benefit, the mixing temperature reduction is not only a benefit but also part of the instructions of the sequential mixing. Since at the first step of mixing due to relatively high amount of bitumen against the surface area of the primary structure, there is a risk of the bitumen draining down. To avoid this, the bitumen viscosity should be higher than the viscosity of the bitumen in the conventional mix. Reducing the mixing temperature thus avoids this problem since it increases the viscosity of the bitumen.

Though the total aggregate surface area in this method is the same as in the conventional method, introducing the final fractions at a later stage in the process means that less adhesive forces have to be overcome during the mixing. This also explains why a lowering of the manufacturing temperature is possible.
Due to the high surface area of filler, the filler grains have more interaction with each other and thus have a high risk of forming clusters [44]. These clusters can actually be quite hard to break, once formed, through the mixing process since they will still be relatively small compared to the mechanical mix arms. Additionally, the liquid bridges that bind the particles together, figure 25, arrest part of the bitumen [45]. This portion of the binder is therefore not available as a free binder. This is frequently seen in the normal mixing process through the “dullness” of the loose mixture. In the sequential mixing method, filler is added gradually on the surface of the bitumen that is already partially adhered to the primary structure. As such, the agglomeration problem is significantly reduced in this case. This is also observed through the shininess of this type of mixture.

Conventional mixing procedure all the aggregates are initially mixed dry. Considering that all asphalt mixtures either have a continuous or a gap graded aggregate mixture, there will always be aggregates of different sizes. This means, that in the case of dry mixing of the aggregates, there is a tendency of the smaller particles to move to the top of the mixture. This segregation can thus result in an inhomogeneous mixture upon introduction of the binder. It would take significant mixing effort to reduce this segregation at this stage and “over-mixing” could become a high risk. In the case of the sequential mixing method, the larger aggregate size are first coated with bitumen. Then, upon introduction of the filler there is no chance of segregation since the filler will immediately stick to the binder. This concept is also referred to as cohesive mixing (figure 26) [46]. This thus results in a more homogeneous mixture with less mixing effort.

To assess the above described processes of agglomeration and its viscosity behaviour, viscosity measurement with the shear rheometer were made in which the mixing order of particles and the bitumen was varied:
In the first case (mix I), filler grains were gradually dispersed on top of the bitumen while bitumen was agitated. In the second case (mix II) all mineral aggregates were first placed in the cup and then bitumen was mixed in.

The mixing time for both methods was kept the same (15 min) and done at 100°C. The preparation and test methods follow the protocol for measuring the viscosity of asphalt mastic at higher temperature developed by Hesami et al. [47]. For both samples 100 g bitumen 70/100 were mixed with 150 g fine aggregate (0.5-1 mm) and 40% filler (M600) by volume. In figure 27 the results are shown.

Results show that the mix II has the lower viscosity, which is expected based on the above discussion since the agglomerated minerals formed larger particles in the bitumen matrix thus reducing the overall viscosity [14,47].

![Figure 27. Viscosity result of mastics produced with two different methods at 100°C.](image)

### 6.2 Workability: Theoretical Coating Thickness and Friction Coefficients

To demonstrate the effect of the mixing sequence on the workability of asphalt mixtures, in the following, an example mixing recipe (Swedish ABS 11 [48]) was used. In this mixture, the bitumen content is 6.3% and table 5 shows the aggregate gradation.

<table>
<thead>
<tr>
<th>Sieve size (mm)</th>
<th>16</th>
<th>11.2</th>
<th>8</th>
<th>5.6</th>
<th>4</th>
<th>2</th>
<th>1</th>
<th>0.5</th>
<th>0.063</th>
</tr>
</thead>
<tbody>
<tr>
<td>Passing (%)</td>
<td>100</td>
<td>95</td>
<td>54</td>
<td>34</td>
<td>28</td>
<td>22</td>
<td>19</td>
<td>15</td>
<td>9</td>
</tr>
</tbody>
</table>

To calculate the theoretical workability of the two mixing sequences, the film thickness of the primary structure coating and the frictional coefficients will be calculated. For this, first,
calculations of the size of the course aggregate fractions are made, based on the aggregate gradation shown in Table 5. Combining this with the bitumen percentages, and assuming a simplified packing arrangement, the theoretical film coating thickness of the primary structure can be determined. From this, the aggregate contact points and mixture porosity are determined. These then allow for the determination of the theoretical workability. First, in the following the assumption made and theories utilized are explained.

6.2.1 Categorizing the Aggregates Size

As mentioned previously, the portion of aggregate which has a significant role in carrying the load in the structure of asphalt mixture is called primary structure and aggregates smaller than primary structure and bigger than filler are called secondary structure. In the sequential mixing method it is important to determine the boundary limit between primary and secondary structures. To do so, the weighted average grain size \( D_{w,\text{avg}} \) of the two contiguous sieve sizes should be calculated. Any two contiguous sieve sizes are determined to be in the primary structure range, only if the following equation is fulfilled. The analysis starts from the biggest sieve size in the gradation and progresses down in sieve sizes until the any two of the contiguous sieve sizes stop fulfilling Eqn. (6.1) or when a sieve size of 2.00 and 2.36 mm of European and American sieve size standard is reached, respectively [49].

\[
\frac{1.1 \times D_1 D_2}{\sqrt{D_2^2 + 2.36 \times D_1^2}} \leq d_{w,\text{avg}} \leq \frac{1.1 \times D_1 D_2}{\sqrt{2.36 \times D_2^2 + D_1^2}}
\]

In this equation \( d_{w,\text{avg}} \) is the weighted average void diameter which is equal to \( 0.732 D_{w,\text{avg}} \).

6.2.2 Film Coating Thickness

As explained in Chapter 2, the distance between particles is one of the most important parameter for evaluating viscosity or workability of suspensions and granular paste. Coating thickness at the production phase of asphalt mixture does not only show the quality of asphalt mixture but also gives a rough estimation of distance between aggregates. Once the primary and secondary structure’s range are determined, the film coating thickness can be calculate by using the morphology framework concept [50-52]. In the conventional way of calculating the theoretical film thickness, the contact between the aggregate is neglected, however the morphology framework concept takes into account the film growth imposed by contact between particles based on the aggregate packing arrangement [50]. It is assumed that the aggregates in loose mixture are arranged in the simple cubic packing system (Figure 28). To calculate the film thickness in this method, the volume of unit cell and its porosity must be calculated. A unit cell is a six-side box, which is formed by joining the center of eight spheres. For the simple cubic arrangement, the volume of the unit cell can be calculated by the following equation.
where \( d_p \) is the diameter of a particle and \( \alpha_m \) is a packing factor, which for the simple cubic is equal to 1. Porosity of the unit cell simply can be calculated by subtracting the total volume of the unit cell by the volume of the aggregates and bitumen within the unit cell. In the simple cubic unit cell there is only one spherical aggregate in the unit cell, so by having the average weighted aggregate size for each part of aggregate gradation, the volume of solid in the unit cell can be calculated. Volume of bitumen also can be determined by using the bitumen content in the mix, specific gravity of aggregate and specific gravity of bitumen.

The relationship between the porosity and coating thickness index \( \left( \frac{2L_c}{d_p} \right) \) can be identical from figure 29, which is proposed by Taylor et al. [53].

6.2.3 Calculating Workability of Sequential and Conventional Mixes

According to the developed viscosity framework, the relative viscosity of a suspension, mastic or mixture, can be calculated based on the particle concentration. The relative viscosity of suspension can be calculated in Hydrodynamic or Frictional regimes. As the asphalt mixture in all mixing stages have the high aggregate concentration, asphalt mixtures in the loose stage and during mixing behave in the frictional regime. Hence, the greatest resistance to the shear in loose asphalt mixture comes from the frictional force between the
aggregates. However, aggregates during mixing are not dry, since a thin layer of the bitumen exists between aggregates. This layer of bitumen has a lubrication effect and causes a lower friction coefficient [54].

The Striebeck curve [55] is a theory in Tribology, which explains the changes in friction coefficient due to change in properties of the film lubricant.

Figure 30 shows the Striebeck curve in which the vertical axis is the friction coefficient and the horizontal axis is Sommerfeld number (SN), which is given in equation (6.3).

![Figure 30. Striebeck curve [55].](image)

\[
SN = \frac{Velocity \times Viscosity}{Load} \tag{6.3}
\]

So, changing any of these variables changes the friction coefficient, which may lead to changes in the lubrication regime. According to the Striebeck curve, three different lubrication regimes are distinguishable boundary, mixed and hydrodynamic lubrication. The distance between two surfaces is increased from very narrow thickness in the boundary lubrication to more than 25\(\mu m\) in hydrodynamic regime [56].

Considering that during mixing a relatively high velocity can be expected for two sliding aggregates in the vicinity of each other, as well as a low load magnitude, the hydrodynamic regime is the dominant lubrication regime during the mixing of asphalt mixtures. Therefore, for the same job mix formula and mixing conditions, for a given velocity and load, the higher bitumen viscosity will give the higher frictional force. The friction force of two dry aggregates in contact that have relative lateral motion can be calculated with Coulomb equation (equation (6.4)).

\[
F = \mu \times N \tag{6.4}
\]

where \(F\) is the frictional force, \(\mu\) is the frictional coefficient and \(N\) is the vertical load on the contact point. For any two bitumen coated aggregates, the relative lateral motion follows the viscosity law. With the existence of a layer of a Newtonian fluid between two surfaces, the resistance to the relative lateral motion is calculated by the following equation:
Workability of Asphalt Mixtures and Mixing Sequence

\[
\tau = \eta \dot{\gamma} \\
F = \frac{\eta V A}{h}
\]

(6.5)

where \(\tau\) is the shear stress, \(\eta\) is the viscosity of the liquid, \(\dot{\gamma}\) is a shear rate, \(F\) is the lateral force, \(V\) is the relative velocity, \(A\) is the contact area and \(h\) is the distance between the surfaces of aggregates. Substituting equation (6.4) into equation (6.5) will give the friction coefficient as equation (6.6).

\[
\mu = \frac{\eta V A}{h N}
\]

(6.6)

This equation can be used for a relative comparison of the change of friction coefficient during the mixing for both conventional and the sequential mixing methods. The viscosity of the lubricant in this equation is an important parameter for calculating the frictional force in asphalt mixtures. Therefore, the viscosity of asphalt mixtures as a function of aggregate concentration in each stage of mixing is summarized in table 6.

**Table 6. Summary of viscosity as a function of particle concentration at different stages of mixing.**

<table>
<thead>
<tr>
<th>Conventional Mix</th>
<th>Sequential Mix</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Bitumen + Filler</td>
<td>1. Bitumen + Primary Structure</td>
</tr>
<tr>
<td>(\eta_{Mastic} = f(\phi_{Filler}, \eta_{binder}))</td>
<td>(\eta_{PS} = f(\phi_{PS}, \eta_{binder}))</td>
</tr>
<tr>
<td>2. Mastic + Secondary Structure</td>
<td>2. PS Mix + Filler</td>
</tr>
<tr>
<td>(\eta_{SS} = f(\phi_{SS}, \eta_{mastic}))</td>
<td>(\eta_{PS+F} = f(\phi_{PS}, \eta_{mastic}))</td>
</tr>
<tr>
<td>(\eta_{PS} = f(\phi_{PS}, \eta_{SS}))</td>
<td>(\eta_{PS} = f(\phi_{PS}, \eta_{SS}))</td>
</tr>
</tbody>
</table>

The boundary limit between primary and secondary structures of the example aggregate gradation was calculated as 2 mm. By utilizing the Taylor relationship and the other calculated volumetric parameters, the film coating thickness can be calculated, table 7.

**Table 7. Film Coating Thickness for Sequential and Conventional Mixing Methods.**

<table>
<thead>
<tr>
<th>Sequential</th>
<th>Conventional</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stage 1</td>
<td>Stage 1</td>
</tr>
<tr>
<td>(PS) coating thickness (Bitumen)</td>
<td>(Filler) particles in bitumen (Hydrodynamic Regime)</td>
</tr>
<tr>
<td>(T_{PS1} = 326 \mu m)</td>
<td></td>
</tr>
<tr>
<td>Stage 2</td>
<td>Stage 2</td>
</tr>
<tr>
<td>(PS) coating thickness (mastic)</td>
<td>(SS) coating thickness (mastic)</td>
</tr>
<tr>
<td>(T_{PS2} = 718 \mu m)</td>
<td>(T_{SS2} = 84 \mu m)</td>
</tr>
<tr>
<td>Stage 3</td>
<td>Stage 3</td>
</tr>
<tr>
<td>(PS) coating thickness (mastic + SS)</td>
<td>(PS) coating thickness (Mastic + SS)</td>
</tr>
<tr>
<td>(T_{PS3} = 1044 \mu m)</td>
<td>(T_{PS3} = 1044 \mu m)</td>
</tr>
</tbody>
</table>
Combining the film coating thickness results and friction coefficient equation (equation 8), shows the effect of mixing sequence on the friction coefficient. Table 8 shows friction coefficients at different stages for both methods.

### Table 8. Friction Coefficients at different stages.

<table>
<thead>
<tr>
<th>Stage</th>
<th>Sequential</th>
<th>Conventional</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stage 1</td>
<td>[ \mu_1 = \frac{\eta_B \cdot A_{cPS}}{t \cdot (652 \mu m)} ]</td>
<td>[ \text{Filler particles are not in contact and the resistance to flow comes from the viscosity of mastic.}^* ]</td>
</tr>
<tr>
<td>Stage 2</td>
<td>[ \mu_2 = \frac{\eta_{\text{mastic}} \cdot A_{cPS}}{t \cdot (1436 \mu m)} ]</td>
<td>[ \mu_2 = \frac{\eta_{\text{mastic}} \cdot A_{cSS}}{t \cdot (168 \mu m)} ]</td>
</tr>
<tr>
<td>Stage 3</td>
<td>[ \mu_3 = \frac{\eta_{\text{mastic+SS}} \cdot A_{cPS}}{t \cdot (2088 \mu m)} ]</td>
<td>[ \mu_3 = \frac{\eta_{\text{mastic+SS}} \cdot A_{cPS}}{t \cdot (2088 \mu m)} ]</td>
</tr>
</tbody>
</table>

*Viscosity of mastic \( \eta_{\text{mastic}} \) can be determined in hydrodynamic regime utilizing viscosity framework.

Assuming the same mixing velocity and contact area for both mixing methods, the equations in table 8 illustrate that the friction coefficient during the mixing in the sequential method is lower than conventional mixing. This shows that mixtures prepared through sequential mixing are more workable during mixing compared to mixtures prepared through conventional mixing. However, the friction coefficient at end of mixing is the same for both mixtures. This can only theoretically be correct, in practice due to lower level of workability of conventionally mixed mixtures during mixing; it is difficult to achieve the ideal mixture, typically needing a higher mixing effort.

### 6.3 Effect of mixing Sequence on the Internal Structure of Asphalt Mixtures

Changing the mixing method but keeping the recipe and materials constant, may vary the internal structure of asphalt pavement. Changes in the internal structure of asphalt pavement can lead to changes in behavior of asphalt pavement. So, knowledge of the internal structure of the asphalt mixtures produced by sequential and conventional mixing methods could help explain the connection between mixing method on performance. Therefore, in this study three-dimensional X-ray digital images were analyzed using image processing software (Avizo).

Two asphalt samples, which were produced with sequential and conventional mixing methods, were used for the analyzes. Both samples are Swedish ABS 11 mixtures in which the biggest aggregate size is 11 mm and 6.3% of weight of samples is bitumen 70/100 [48]. Figure 31 shows the aggregate gradation of the specimen.
Workability of Asphalt Mixtures and Mixing Sequence

One of the artifacts of X-ray scanning is beam hardening, which refers to the losing of resolution in the center of the scanned images. In this study, the beam hardening is corrected utilizing background and flat field correction feature in Avizo Fire, figure 32-b. After applying some other image processing options, such as equalization for transferring the grayscale histogram from I shape (limited range) to O shape (wither range), filters for noise reduction and edge sharpening for enhancing the edge of objects, the final image (figure 32-c) is obtained for segmentation and image analyzing. The corrected images were utilized for performing the segmentation. Figure 33-a shows the segmented scanned image of asphalt mixtures with three phases: aggregates, mastic and air void. By combining slice the segmented 3D volume is rendered (figure 33-b) which can be used for image analyzing.

Figure 32. a) Raw image, b) Beam hardening and c) corrected image.

Figure 33. a) Mixture segmentation and b) 3D volume rendered.

Figure 34 shows the 2D area of air-voids per slice along the depth. From the figure it can observe that the sequentially mixed asphalt mixture has an even air-void distribution along
the depth, unlike the conventionally mixed asphalt mixture. This indicates a better compactability of sequential asphalt mixture compare to the conventional one.

**Figure 34.** Density distribution of air-voids with depth.

Correspondingly, the mean distance between adjacent aggregates shows the same trend (figure 35) as the sequentially mixed asphalt shows a lower and more even distribution of mean distance after compaction along the depth of sample.

**Figure 35.** Mean aggregate distance distribution with depth.

Figure 36 shows the computed contact area of adjacent aggregates and shows that the sequential mix shows a slightly higher contact area in each slide. Lower and even distribution of mean distance combined by higher contact area implies that the sequential mix has more homogeneity and better internal structure for transferring the load. To further characterize this, mechanical testing was performed, which is described in the following section.
6.4 Effect of Mixing Sequence on Mixture Mechanical Performance

Mixing itself is not the end purpose of the asphalt mixture production. The mechanical performance of the asphalt mixtures under the traffic load is an important parameter in the quality of the asphalt pavement. For this reason, a laboratory investigation was performed to compare the mechanical performance of asphalt mixtures produced with the sequential and conventional mixing methods.

Six samples with ABT-16 (i.e. biggest aggregate size was 16 mm) [48] and bitumen 70/100 mm/mm were produced with the sequential and conventional mixing methods. Figure 37 shows the aggregate gradation and table 9 gives the other mixture specifications.
Table 9. Mixtures Specifications.

<table>
<thead>
<tr>
<th>Mix Type</th>
<th>Aggregate Density (g/cm3)</th>
<th>Flakiness index (%)</th>
<th>Micro-Deval (%) (10-14mm)</th>
<th>Los Angeles (%) (10-14mm)</th>
<th>Bitumen density (Kg/m3)</th>
<th>Bitumen Content (%)</th>
<th>Mixture Bulk Density (Kg/m3)</th>
<th>Mixture Maximum Density (Kg/m3)</th>
<th>Void content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sequential</td>
<td>2.66</td>
<td>15</td>
<td>10</td>
<td>25</td>
<td>1020</td>
<td>6</td>
<td>2358</td>
<td>2430</td>
<td>3</td>
</tr>
<tr>
<td>Conventional</td>
<td>2.66</td>
<td>15</td>
<td>10</td>
<td>25</td>
<td>1020</td>
<td>6.3</td>
<td>2340</td>
<td>2415</td>
<td>3.1</td>
</tr>
</tbody>
</table>

To ensure consistent and more realistic conditions for both types of mixing, the loose asphalt mixtures were taken from the Skanska Rockneby asphalt plant, which is producing asphalt mixtures with both sequential and conventional mixing methods. The Superpave IDT is utilized to determine resilient modulus (Mr), creep compliance, tensile strength (St), fracture energy limit (FE), dissipated creep strain energy limit (DCSE) and energy ratio (ER).

The material parameters obtained from the Superpave IDT test for both sequential and conventional mix methods are summarized in table 10. From the results, it can be observed that producing asphalt mixture with sequential method resulted in stiffer mechanical behaviour. The Resilient modulus represents the material’s capability to recover after releasing loading, so generally higher resilient modulus shows higher elastic recovery. Resilience modulus (Mr) of the sequential mix is 14% higher than the conventional mix. Furthermore, the creep compliance of the sequential mix at 1000s was found to be reduced by 19%, which shows the better performance of sequential mix in terms of resistance to the permanent deformation. Figure 38 compares the creep compliance of both mixtures.

Table 10. Material Parameters from Superpave IDT.

<table>
<thead>
<tr>
<th>Type of Mixture</th>
<th>Mr (GPa)</th>
<th>D0 (1/GPa)</th>
<th>D1 (1/GPa)</th>
<th>m-value</th>
<th>Creep Rate @ 1000s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sequential</td>
<td>13.26</td>
<td>0.075</td>
<td>0.1828</td>
<td>0.710</td>
<td>0.018</td>
</tr>
<tr>
<td>Conventional</td>
<td>11.59</td>
<td>0.086</td>
<td>0.3203</td>
<td>0.659</td>
<td>0.020</td>
</tr>
</tbody>
</table>

Figure 38. Creep Compliance at 10°C.

As shown in Figure 39 the tensile strength at first fracture for the sequential mix is significantly higher than for conventional mix, about 45%. This can be a result of a better
distribution of the aggregates and bitumen in the asphalt mixture produced by the sequential method, providing thus a suitable structure for transferring loads.

Figure 39. Tensile Strength at 10°C.

The summary of the results from Hot Mix Asphalt Fracture Mechanics (HMA Fracture Mechanics) analysis are presented in table 11. Comparing the fracture energy of these two mixtures shows 22% lower Fracture Energy \( (FE) \) for the sequential mix. This indicates that the sequential mixture seems more susceptible to brittle cracking such as thermal induced cracking or fracture due to a single passing of a very heavy vehicle [27]. However, the energy ratios of both specimens are roughly equal which shows that both specimens have almost the same resistance to fatigue cracking.

Table 11. Mixtures Fracture mechanics Parameter from Superpave IDT.

<table>
<thead>
<tr>
<th>Type of Mixture</th>
<th>FE (KJ/m³)</th>
<th>EE (KJ/m³)</th>
<th>DCSE (_f) (KJ/m³)</th>
<th>DCSE (_{min}) (KJ/m³)</th>
<th>Energy Ratio (ER)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sequential</td>
<td>7.79</td>
<td>0.60</td>
<td>7.19</td>
<td>6.53</td>
<td>1.10</td>
</tr>
<tr>
<td>Conventional</td>
<td>10.05</td>
<td>0.49</td>
<td>9.56</td>
<td>8.12</td>
<td>1.18</td>
</tr>
</tbody>
</table>

6.5 Summary

Unlike conventional mixing methods for producing asphalt mixtures, in the sequential mixing method asphalt mixture components are added into the mixer at different mixing stages. Basically, in the sequential mixing method, bitumen is sprayed on the surface of the course aggregate fraction, then the filler is added to the mix and finally the fine aggregate fraction is added. This method of mixing will result in a lower mixing temperature, lower level of agglomeration and also lower segregation.

The viscosity framework, morphology framework [51,52] and concepts from tribology are used to evaluate the effect of mixing sequence on the workability of asphalt mixture during...
mixing. It is shown that asphalt mixtures produced with sequential mixing method are more workable with less segregation. Furthermore, due to the lower specific surface area of the coarser aggregate which is coated at the beginning of the mixing in the sequential mixing method, bitumen is required to have a higher viscosity which allows for a lower mixing temperature.

Despite the same recipe for both sequential and conventional asphalt mixtures, the image analyzing of 3D pictures obtained from X-ray scanner confirmed that the internal structures of the two mixtures prepared by sequential and conventional mixing are somewhat different. The asphalt mixture produced by the sequential mix method showed an even air-void distribution and mean distance along the depth of sample. This implies that the sequential asphalt mixture may compact more easily. In addition, results of Superpave IDT at 10°C indicated that the asphalt mixture which is produced by sequential mixing method has more resistance to the permanent deformation, while it is more susceptible to the brittle cracking. Both asphalt samples show almost the same resistance to fatigue cracking, however the sequential mix is more durable.
7 Conclusion and Recommendation

7.1 Concluding Discussion

Conventionally, the temperature of mixing and compaction of asphalt mixtures is determined based on the viscosity of the asphalt binders. According to the Marshal standard from 1993, the viscosity range of asphalt binders for mixing and compaction should be $0.17\pm0.2\text{ Pa.s}$ and $0.28\pm0.2\text{ Pa.s}$ respectively. In the Superpave mix design, the viscosity of binder is measured at $135^\circ\text{C}$ and $165^\circ\text{C}$ using a rotational Brookfield viscometer. By assuming linear viscosity-temperature relationship when viscosity is plotted on a double log scale and temperature is plotted on a log scale, temperature of mixing and compaction should then be determined.

In addition to the negative effect of high mixing temperature on the quality of the asphalt binder itself as it enhanced ageing, high production energy usage should also be avoided for environmental considerations. Therefore, the use of additives for producing asphalt mixtures at lower temperature is an important point of interest. Unfortunately, generally, most additives change the linear behaviour of the binder to the non-linear regime. This means that the conventional method for determining temperature can no longer be used. In addition, viscosity of the binder alone cannot be a good indicator to show desired workability and compactability as there are several other parameters that have influence on these properties.

Considering the importance of diminishing energy usage and greenhouse gas emissions, the asphalt industry has been developing new types of mastics for the past decades (e.g Warm Mix or Cold Mix Asphalts). These ‘new’ types of materials, also involve new additives and behaviour beyond the ‘normal’ standards. Adding filler into bitumen, is further complicated in the presence of such modifiers, since they may not only change the bitumen properties, but can also change the interaction between the filler and the bitumen matrix. Empirical assumptions thus no longer hold and more sensitive methods and models, based on fundamental understanding, will be needed to bring sufficient control and predictability into mastic and asphalt mix design and production.

Asphalt mastic was defined as an intermediate material that is more close to the asphalt mixture than the binder alone. In addition, the effect of interaction between bitumen and mineral particles in mastics is more visible as filler grains have high surface area. Therefore, in this research the main focus was placed on investigating the effect of various fundamental
parameters on the rheology of asphalt mastic at elevated temperature and its effect on the
workability of asphalt mixture.

Asphalt mastic can be recognized as a suspension in which fillers and bitumen play the
role of particles and media respectively. Based on the current literature and on the
previously developed models for calculating the relative viscosity of suspensions, in this
Thesis a new framework was established which is capable of calculating the viscosity of
mastic asphalt at different filler concentrations, based on three different equations: the
Frankel, Einstein and a transition equation. Due to the commonly used percentage of
concentration of filler in mastic asphalt, the suspension generally flows in the hydrodynamic
regime. As is expected from the framework, the Frankel equation can just cover the viscosity
of mastic with higher concentration and for intermediate concentration it shows higher
relative viscosity than the real viscosity. At low concentration the Frankel shows lower
relative viscosity. For very dilute mastic asphalt both the Frankel and the transition
equations tend to zero, which is obviously not representing the real behavior. The Einstein
equation perfectly fits the viscosity of very dilute mastic asphalts. From comparisons with
actual data, it was shown that the developed framework may serve as a practical tool for
calculating asphalt mastic viscosity as a function of filler concentration.

The developed framework gives insight into the viscosity behaviour of mastic as a
foundation of its filler concentration, however still there is a need for a test which is able to
measure the viscosity of asphalt mastics at higher temperatures and at various filler
concentrations. Such a test would give the possibility of studying the effect of different
parameters on the viscosity behaviour of asphalt mastics and also give needed parameters to
place the mastic within the mastic viscosity framework. Measuring the viscosity of asphalt
mastic is, however, a challenging task as every steps of the test has an influence on the result.
Hence, in this study, a new protocol for measuring asphalt mastic viscosity was created
which is able to determine the viscosity of bituminous mastics, as a function of filler
concentrations reliably. Having such a test protocol available will open up the possibility of
further investigating mastics for their fundamental properties and will allow for the
development of specific guidelines regarding the effect of particle shape, particle type,
temperature effects and additional modifications.

Experimental results obtained from the viscometry test indicated that the particle shape,
size distribution, as well as varying chemistry are important parameters and the test allowed
for the direct measurement of their effects on the viscosity. A critical filler concentration was
identified beyond which the viscosity behavior became nonlinear. This parameter seemed
unique to filler-bitumen combinations and could become a useful parameter to use in the
characterization of mastics. The rounded filler particles (e.g., fly ash) showed a significantly
lower viscosity build-up, as did the Bio1-based mastics.

The test results also showed that the test is sensitive enough to notice the effect of
moisture and aging on the viscosity of the mastic, which is important as these affect the long-
term response of the asphalt concrete and are normally difficult to predict beforehand.
Moisture and aging effects cannot be identified, if only the binder itself is studied, as shown
in the measurements of the neat binder. This again shows the importance of the developed mastic viscometry test.

From an X-ray scanning analyses it was shown that an agglomeration parameter could be identified that shifted the sieve curve of the dry filler material effectively, once the filler was added to the bitumen. This finding has important implications for the resulting behavior of the mastic and should be studied further.

The differences in viscosity behaviour of mastics could be partially explained from the different shapes and sizes of fillers, but it was concluded that the filler–bitumen interaction also has an important effect on the rheological behavior of the mastics. Therefore, in this Thesis the influence of filler–bitumen interaction on the rheology of asphalt mastic was investigated, placing special focus on shape and size of filler. For this, both experimental and numerical methods were utilized. Measuring the viscosity of different asphalt mastics at 100°C showed that the effect of the additive is more visible in mastics with higher filler concentrations. The additive is not only changing the viscosity of bitumen but also has significant influence on the surface of the filler.

From both the experimental and the numerical analyses, it was found that interface influence changes with different filler shapes, sizes and filler concentrations. Mastics with angular fillers are affected more than mastics with round filler. Mastics with smaller filler size particles showed a significant higher influence compared to the coarser ones. Mastic with higher filler concentrations showed the highest level of influence by the changing the interfacial properties.

The micro-mechanical finite element model showed the influence of varying interfacial properties on the resulting behaviour. For weak interfacial properties the effect of the shape of the particles was dominant. For intermediate spring constant and a strong dashpot viscosity the effect of the surface area is dominant at the end of relaxation period while at the end of the loading the effect of the filler shape is more dominant.

Finally, the workability of asphalt mixtures in relation to the viscosity developed framework and the viscosity of mastics was studied. By using the viscosity framework and other relevant knowledge obtained in this research, workability of two types of asphalt mixtures which are produced by a sequential and a conventional mixing methods were evaluated. The reasons for differences in the rheological properties of sequential and conventional mixing methods are discussed. Some theoretical frameworks such as the viscosity framework, morphology framework and tribology concept are utilized to evaluate the effect of mixing sequence on the workability of asphalt mixture. It is shown that the asphalt mixtures produced with the sequential mixing method is more workable with less segregation. Due to lower specific surface area of the coarser aggregate which is coated at the beginning of the mixing in the sequential mixing method, bitumen is required to have a lower viscosity which can lead to lower mixing temperature.

Despite the same recipe for both, image analyzing of 3D pictures obtained from X-ray scanner showed that the sequential and conventional asphalt field cores did not have similar
Conclusion and Recommendation

internal structures. Asphalt mixture produced by sequential mix method showed an even air-void distribution and mean distance along the depth of sample. This shows that the studied sequential asphalt mixture core had been easier to compact. Furthermore, experimental results showed that the asphalt mixture, which was produced by sequential mixing method, had more resistance to permanent deformation, while appeared more susceptible to the brittle cracking. Both asphalt samples showed almost the same resistance to fatigue cracking, however the sequential mix appeared to be more durable. This experimental result further confirmed the importance of the mastic phase and highlighted the impact that the mixing phase can have on the resulting asphalt mixture performance.

7.2 Recommendations and Further Work

This PhD Thesis has explored the rheology of asphalt mastics and its effect on the workability of asphalt mixtures. Due to the complexity and multidisciplinary of this research topic, the results of this thesis are not all conclusive in themselves. However, some important advances have been made, valuable tools and frameworks have been developed and a solid base for future exploration and research has been further crystalized. In the previous section the main findings of the research were summarized. In the following the recommendations with regard to the continuation of this research are given:

- The viscosity of asphalt mastics is a function of various parameters. The viscosity framework presented in chapter 2 calculating the viscosity of asphalt mastic as a function of filler concentration, and other effective parameters are appeared as the fitting parameters. Finding the physical meaning of each one of these fitting parameters can lead to the more reliable prediction of the viscosity of asphalt mastics.

- Measuring the workability of the asphalt mixtures could be another important research in continuation of this PhD thesis. Designing an instrument, which can be capable to measure the viscosity of asphalt mixture, gives the possibility of evaluating the effect of different parameter on the viscosity of asphalt mixtures directly. This laboratory device should be coupled with a fluid dynamic model to be able to explain the behaviour of the material. Measuring the workability of the asphalt mixture only with simple rotational shaft in a bucket of material is already available which only stay in the comparative stage. Using the computational fluid dynamic (CFD) beside a suitable laboratory device may lead to a comprehensive framework of effect of material and geometry on the workability of asphalt mixtures. It would be important to link this also to the findings of the mastic phase, as found in this Thesis.

- Efficiency in the production of asphalt mixtures is not the only goal in the asphalt mixture industry, but is also needed to ensure that the asphalt mixtures have a desirable and predictable performance. Coupling the workability results with the
mechanical test results of asphalt mixtures may draw a linkage between workability and performance of asphalt mixtures.
Conclusion and Recommendation
References


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