NTNU – Trondheim
Norwegian University of
Science and Technology

Determining the Rheological Properties of Neat and Rubber Modified Soft Bitumen

Andreas Meling Kjosavik

Civil and Environmental Engineering
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Supervisor: Helge Mork, BAT
Co-supervisor: Carl Thodesen, SINTEF Byggforsk

Norwegian University of Science and Technology
Department of Civil and Transport Engineering

NORWEGIAN UNIVERSITY OF SCIENCE AND TECHNOLOGY
DEPARTMENT OF CIVIL AND TRANSPORT ENGINEERING

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Name: Andreas Meling Kjosavik				
Professor in charge/supervisor: Helge Mork				
Other external professional contacts/supervisors: Carl Thodesen, SINTEF				

Abstract:

The properties of bitumen as an asphalt binder are dependent on temperature. At high temperatures, the bitumen softens enabling permanent deformation of the pavement. At low temperatures, the bitumen becomes stiff and inflexible and can crack as a result of strain and thermal contraction. Adding rubber from ground car tires increases the stiffness at high temperatures while maintaining or improving the flexibility of the binder at low temperatures. This thesis studies the interaction between bitumen and rubber by producing rubber modified binders using different mixing times. Two rubber types, special and normal rubber, and a reference binder are compared to find differences in the reactive properties of the rubber and the impacts on key indicators of binder performance. Laboratory testing was done using the Dynamic Shear Rheometer and Bending Beam Rheometer on binder in its original state and after short or long-term aging.

The results show that the rubber modified binders have improved resistance to permanent deformation at high temperatures compared to the reference binder. The resistance to fatigue and thermal cracking at low temperatures is significantly better for the special rubber than the normal rubber, which is significantly better than the reference binder. Mixing time is only significant for the rubber modified binder in its original state, whereas the properties of the reference binder are permanently altered as a result of the mixing. It was not possible to find a clear difference in the reactive properties of the two rubber types. The performance difference is attributed to differences in the properties of the rubber particles.

Keywords:

1. Rubber modified binder
2. Dynamic Shear Rheometer
3. Bending Beam Rheometer
4. Multiple Stress Creep Recovery

Summary

The properties of bitumen as an asphalt binder are dependent on temperature. At high temperatures, the bitumen softens enabling permanent deformation of the pavement. At low temperatures, the bitumen becomes stiff and inflexible and can crack as a result of strain and thermal contraction. Adding rubber from ground car tires increases the stiffness at high temperatures while maintaining or improving the flexibility of the binder at low temperatures. This thesis studies the interaction between bitumen and rubber by producing rubber modified binders using different mixing times. Two rubber types, special and normal rubber, and a reference binder are compared to find differences in the reactive properties of the rubber and the impacts on key indicators of binder performance. Laboratory testing was done using the Dynamic Shear Rheometer and Bending Beam Rheometer on binder in its original state and after short or long-term aging.

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Sammendrag

Egenskapene til bitumen som bindemiddel i asfalt er temperaturavhengige. Ved høye temperaturer blir bitumen mykt, noe som reduserer motstanden mot permanente deformasjoner og spordannelser. Ved lave temperaturer blir bitumen stivt og oppsprekking kan oppstå som følge av påkjenninger og sammentrekning av materialet. Tilsetning av gummi fra oppmalte bildekk øker stivheten til bindemiddelet ved høye temperaturer uten at dette går ut over fleksibiliteten ved lave temperaturer. Denne avhandlingen undersøker interaksjonen som oppstår mellom bitumen og gummi når man produserer gummimodifisert bindemiddel med forskjellige blandetider. To gummityper, spesiell og normal gummi, og et referansebindemiddel sammenlignes med hensyn på reaktive egenskaper til gummien og innvirkningen dette har på ytelsen til bindemiddelet. Arbeidet bestod av laboratorietesting med bruk av Dynamic Shear Rheometer og Bending Beam Rheometer på originalt, korttidsaldret og langtidsaldret bindemiddel.

Resultatene viser at gummimodifiserte bindemidler har økt motstand mot permanente deformasjoner ved høye temperaturer sammenlignet med referansebindemiddelet. Motstanden mot tretthetsbrudd og oppsprekking ved lave temperaturer er bedre for den spesielle gummien enn for den normale som igjen er bedre enn referansebindemiddelet. For de gummimodifiserte bindemidlene er blandetid kun signifikant før aldring. Det ble påvist permanente forandringer i stivheten til referansebindemiddelet som følge av blandetida. Det var ikke mulig å finne en sammenheng mellom ytelsen og de reaktive egenskapene til de to gummitypene. Forskjellen er et resultat av egenskapene til selve gummipartiklene.

Preface

This master's thesis is the final part of a 5 year study of Civil and Environmental Engineering at the Norwegian University of Science and Technology (NTNU). The project was performed during the spring of 2013 in the road laboratory facilities in the Department of Transport and Civil Engineering, which are shared with SINTEF Road and Railway.

I would like to thank my supervisors Helge Mork and Carl Thodesen. Special thanks to Lisbeth Johansen, Haris Brcic and Sara Anastasio for helping me with the laboratory work. Thanks to all the other employees at NTNU and SINTEF for making a good workplace environment and offering advise in my work with this project. Thanks to the Norwegian Public Roads Administration for offering financial support.

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Andreas M. Kjosavik

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Abbreviations

α	=	Significance level
ANOVA	=	Analysis of Variance
ASTM	=	American Society for Testing and Materials
BBR	=	Bending beam rheometer
CRM	=	Crumb rubber modifier
δ	=	Phase angle
DSR	=	Dynamic shear rheometer
J_{nr}	=	Unrecoverable creep compliance
G^*	=	Complex modulus
m	=	Master curve slope
MSCR	=	Multiple Stress Creep Recovery
PAV	=	Pressure Aging Vessel
PG	=	Performance Grade
R	=	Elastic Recovery
R_p	=	Least Significant Range
r_p	=	Least Significant Studentized Range
rpm	=	Revolutions per minute
RTFO	=	Rolling Thin Film Oven
S	=	Creep stiffness
s^2	=	Variance
sin	=	Sine function
SSE	=	Error Sum of Squares
SST	=	Total Sum of Squares
SSX	=	Treatment Sum of Squares
SHRP	=	Strategic Highway Research Program

Chapter 1

Introduction

This thesis contains an outline of the background and process leading up to the laboratory work, as well as the choice of research problem and method. There is then a short literature study and description of the laboratory work and the results. The results are then analyzed and discussed and a conclusion is presented.

1.1 Background

The resistance to wear and permanent deformation of an asphalt pavement is largely dependent on the properties of the asphalt binder. A stiff binder can withstand deformation at high temperatures but is prone to cracking at low temperatures. To cope with a wider range of climatic conditions and increase pavement lifespan, modified binders are used - such as polymer modified binders. Crumb rubber modifier, or CRM, is an asphalt modifier which improves pavement performance and additionally serves as a way of recycling car tires. In Norway, approximately 50000 metric tons of waste car tires are produced every year, of which 60% is exported. Successful uses of recycled car tires include artificial grass, playground surfaces and noise barriers for roads, but in total this use amounts to about 20% of the total amount of waste [1]. Because road construction uses large volumes of materials, it is considered an efficient way of handling the growing amount of waste car tires. There are two different ways of incorporating rubber modifier in asphalt pavements: the dry mix method and the wet mix method. In the dry mix method the rubber is considered to be part of the aggregate portion of the mixture, while in the wet mix method the rubber is mixed and conditioned to react with the asphalt binder before being added to the asphalt mixture. The wet mix method is the most common method both in terms of practical use and in research.

In the late summer of 2012 a SINTEF project on the use of a special type of rubber modifier was undertaken. Following this, I did a project work on the use of rubber modifier in porous asphalt [2]. These two projects are the basis for this master's thesis. Both projects were focused on asphalt mixtures containing different percentages of two types of rubber and the performance of the mixtures in different tests. The tests generally resulted in better results for one of the rubber types which is denoted as special rubber here. However, it was unclear exactly why it performed better. The major selling point from the producer was that it had undergone a special surface activation process which means that it achieves a much higher surface area and reactive properties than is possible using other rubber grinding processes. In normal wet mix, the rubber and bitumen is placed in a reaction tank at high temperature to ensure that the chemical interaction between the bitumen and rubber happens before the binder is then added to the asphalt mixture [3]. This requires additional equipment, which means additional cost. If the added reactive properties of the rubber means that the rubber can be added directly to the asphalt mixture and still interact with the bitumen similarly to the wet mix method, it would be easier for an asphalt producer to use.

1.2 Scope

This study investigates the properties of rubber modified bitumen. This is done by performing binder testing using the Dynamic Shear Rheometer (DSR) and Bending Beam Rheometer (BBR). The testing is done for different binder conditioning states: original, short-term aged and long-term aged. The tests indicate the binders' potential rutting resistance at high temperatures, fatigue resistance and resistance against low temperature cracking. This results in a temperature interval where the binders' performance is acceptable known as the Performance Grade (PG) rating. The work is aimed at testing the following hypotheses:

1. When rubber is mixed with bitumen, the rubber will swell by binding the lighter volatile oil fractions of the bitumen. This can be observed as an increase in stiffness of the binder.

2. The effects of this reaction are dependent on mixing time and the reactive properties of the rubber.

The research work was done in the following steps:

1. Mixing bitumen and rubber using mixing times of 5, 15, 30 and 60 minutes for two rubber types and a reference binder without rubber.
2. Determining the high temperature properties and rutting resistance of the binder using DSR.
3. Subjecting the binder to artificial aging using Rolling Thin Film Oven (RTFO) and Pressure Aging Vessel (PAV).
4. Determining the high temperature properties and elastic recovery of the RTFO-aged material using the DSR and Multiple Stress Creep Recovery (MSCR) test.
5. Determining the intermediate temperature and fatigue properties of the PAV-aged material using the DSR.
6. Determining the low temperature properties and cracking resistance of the PAV-aged material using the BBR.
7. Analyzing the results using statistical methods.

1.3 Method

The method chosen is an experimental approach using laboratory testing. Because bitumen is a material which is particularly sensitive to temperature, having controlled laboratory conditions is advantageous. The test methods quantify the stiffness and flexibility of the binder material for a variety of temperatures. This gives an understanding of the properties of the binder and its strengths and weaknesses. Using standardized test methods and specifications make the results comparable to other binder and modifier materials. Multiple parallels in each test give insight into the possible statistical variations, as well as uncovering possible errors. The state of the binder material can be manipulated using accelerated aging, which enables quick results for conditions that otherwise would take a long time to reach.

One disadvantage is that by only considering the properties of the binder, it's difficult to predict how it will interact with the other parts of the road structure. There are other studies that show correlation between the properties uncovered through binder testing and field performance, but there is always a degree of interpretation. In experiments with a higher scale such as field testing, results may be directly transferred to practical use. In the field, there are so many variables and parameters that influence the performance of an asphalt pavement, so the significance of a very specific set of parameters is difficult to quantify. However, the interaction effects and climatic conditions observed in the field are highly relevant and difficult to replicate in laboratory conditions. A field study could give significant results, but would require more resources and exploring many different parameters is more difficult than for smaller scale experiments. An alternative to testing the binder alone would be to produce small scale asphalt mixtures in the laboratory. This was done in my project work leading up to this thesis. Because the asphalt mixtures include much more than just the binder, it is difficult to isolate the effects of the binder specifically. Due to the complexity of the aggregate-binder interactions, there is much room for variation. In said project, this manifested itself in that for one of the tests, the difference between the results for two separate binder contents were smaller than the variation within the parallel samples. If the sample preparation and testing methods have a large inherent variance, it means that you need a high number of parallel samples. Leading up to the start of the project, the binder testing methods were considered precise enough to look at the seemingly minor parameter which is mixing time. The hope is that in studying such a specific and limited question, it will be possible to find answers and not just more questions.

Chapter 2

Literature Review

This chapter is a summary of different binder testing methods and other authors' conclusions and findings regarding rubber modified binders.

2.1 Binder test methods

The SUPERPAVE binder specification system is a result of the Strategic Highway Research Program (SHRP) from the United States. It addresses the three main causes of deterioration of a pavement: Rutting, fatigue cracking and thermal cracking. This is the basis for a performance grade (PG) rating, which consists of a high and a low temperature. The high temperature is the highest temperature where the pavement has acceptable rutting resistance. The low temperature is the lowest temperature where the pavement is expected to resist thermal cracking. Fatigue cracking requirements are a function of the two other temperatures. The Dynamic Shear Rheometer or DSR is the test apparatus used to determine rutting and fatigue cracking resistance of the binder material. The test method measures the complex modulus G^* which is a measure of the stiffness of the material and resistance to deformation, and phase angle δ which is a measure of the elastic response of the material. The ratio $G^*/sin(\delta)$ is used to determine the high temperature. Because stiffness increases when the binder is aged, the critical binder state is when it is new. The high temperature tests are performed on original binder and binder which has been short-term aged using a Rolling Thin Film Oven or RTFO, which simulates the state just after plant production of the asphalt mixture. The fatigue cracking resistance is determined by the relation $G^* sin(\delta)$ at intermediate temperatures. The low temperature is determined using the Bending Beam Rheometer or BBR. The test method measures the creep stiffness

S and the slope of the stiffness master curve m, which is a measure of the binders ability to relax stresses. Requirements to both parameters must be met to determine the low temperature. Aging stiffens the binders, making it prone to both fatigue and thermal cracking, which means that the long-term aged is the critical state of the binder. For the intermediate temperature DSR test and BBR test the binder is aged using a Pressure Aging Vessel or PAV. [4]

Modified binders have a more complex behavior than conventional binders. One criticism that has been made towards the SUPERPAVE testing methods is that it only considers the binder properties in the linear viscoelastic region, whereas some effects of polymer modification may only be apparent in the non-linear region. The Multiple Stress Creep Recovery (MSCR) test provides a measure of rutting resistance which more accurately takes into account the special properties of modified binders. This method measures the elastic recovery R and non recoverable creep compliance J_{nr}, which is a measure of the resistance to deformation. This is done at the design high temperature and is used to predict the traffic level the pavement will be able to withstand.[5]

2.2 Bitumen-Rubber interaction

Airey et al. [6] investigate the swelling that results from the bitumen-rubber interaction in a dry mix method perspective using the basket draining method. The light fractions of the bitumen are absorbed by the rubber, resulting in an increase in mass of the rubber particles. The initial speed of this reaction is found to be directly linked to the viscosity of the bitumen and size and surface area of the rubber. The residual bitumen show an increase in stiffness, viscosity and elastic response at high temperatures as a result of this reaction. For dry mix purposes, the rubber is seen as part of the aggregates. The rubber influencing the properties of the bitumen is seen as a disadvantage, as the viscosity increases and the total amount of free bitumen decreases. Due to the dry mix perspective, the rubber particles are relatively large and have a lower surface area than typical wet mix rubber, resulting in reaction times of hours rather than minutes. The swelling as a function of curing time is approximated using a log-function and is considered to reach a saturated level after 48 hours at high temperature.

Research on rubber modified binders is focused primarily on the wet mix method. Many factors are considered regarding the performance of rubber modified binders, such as mixing temperature, rubber grinding method and bitumen source. The effect of the rubber modification is primarily an increase in $G^*/sin(\delta)$ at high temperatures, which is favorable [7]. It is indicated that the rubber may reduce the hardening of the rubber resulting from oxidative aging, which reverses the bitumen-rubber interaction, returning the light fractions to the bitumen making it less stiff. Compared to the improvement at high temperatures, the low temperature improvement is considered marginal [7, 9].

2.3 Rubber properties

Rubber size distribution and grinding method are the main differences between rubber types. There are two main grinding methods: ambient and cryogenic. The ambient method produces rubber with a rough particle surface texture resulting in a high specific surface area, while the cryogenic method produces rubber with smooth surfaces similar to shattered glass [9]. The ambient rubber significantly increases viscosity and $G^{*}sin(\delta)$ at high temperatures and significantly decreases creep stiffness S at low temperatures when compared to cryogenic rubber [9, 11]. The effect of the rubber modification can be split into two parts, the interaction effect resulting from the absorption of light fractions of the bitumen and the particle effect of the rubber which acts as a filler. Larger size rubber gives higher G^{*} as a result of the particle effect [8]. Mturi et al. [10] investigate the influence of DSR gap settings for rubber modifiers with different sizes. An optimized gap setting which produces the most consistent results can be found depending on the rubber particle size. Using a gap setting that is too narrow introduces particle effects which may lead to high variance in the results.

My preliminary project work studied the effects of the two rubber types described in this thesis in porous asphalt mixtures [2]. The general trend is that samples with higher rubber content give better results, but this was not true for all binder contents. The special rubber showed better results for all tests relative to the normal rubber. This was primarily attributed to the reactive properties of the special rubber. It was observed that the binder containing special rubber may be more cohesive and flexible than the normal rubber when examining the test samples after the Prall test.

Chapter 3

Experiment

3.1 Materials

The literature suggests that rubber primarily impacts the high temperature performance of the binder. In Norwegian conditions, low temperatures are often more important, so in order to get the best possible performance at low temperatures, a soft bitumen was chosen. It was believed to have a penetration grade of 160/220, but was later found to have a penetration grade of 100/150, most likely due to being stored for a long time. Two types of rubber are used, denoted as special and normal rubber. Both rubber types originate from synthetic rubber from car tires. The special rubber is produced using a special grinding method which leads to a high specific surface area. The normal rubber is added as a fine powder, while the special rubber is in pellets shown in figure 3.1. Despite the visual appearance, the special rubber is thought to have a smaller individual particle size and larger surface area than the normal rubber.

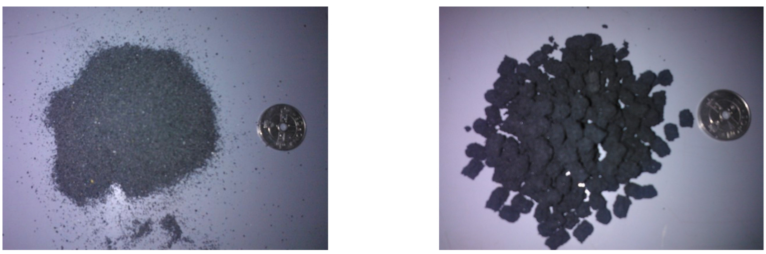

Figure 3.1: Normal Rubber (left), Special Rubber (right)

3.2 Mixing

Mixing is performed to ensure an even distribution of rubber particles in the mixture and sufficient time for the bitumen-rubber reaction to happen. The bitumen was heated in a metal container at a temperature of $150\,^{\circ}\mathrm{C}$ for 1 hour. It was then placed inside a separate oven with an attached shear mixer shown in figure 3.2, also at $150\,^{\circ}\mathrm{C}$. The bitumen was then mixed using the shear mixer at 5000 rpm for 10 minutes. An amount of rubber equal to 10% of the bitumen weight was added and the mixing continued for 5, 15, 30 or 60 minutes. Bitumen with no rubber added was subjected to the same treatment to serve as a reference binder. Approximately 30 grams of binder was poured into a small container intended for DSR testing and both containers were covered and cooled to ambient temperature and stored for at least 18 hours before any re-heating or testing was performed.

Figure 3.2: Shear mixer setup

3.3 Dynamic Shear Rheometer

The DSR is a test apparatus that is used to determine rheological properties of bituminous binders. The test commonly called the DSR-test measures the complex modulus G^* and phase angle δ resulting from oscillating shear stress. The relation $G^*/sin(\delta)$ is found to correlate well with rut resistance at high temperatures, while the relation $G^*sin(\delta)$ is found to correlate well with fatigue resistance at intermediate temperatures. Figure 3.3 shows the DSR apparatus.

Figure 3.3: Dynamic Shear Rheometer

The test was performed in accordance with the NS-EN 14770 standard. The binder was heated to $150\,^{\circ}\mathrm{C}$ for a sufficient amount of time so that it could be poured. Binder was then poured onto a plastic sheet or silicon mould, covered and stored for at least 12 hours for the samples containing rubber and at least 2 hours for the reference samples. The sample was then loaded into the DSR and trimmed using a hot metal spatula. The test is done at set temperatures, ensured by a temperature regulator, at $6\,^{\circ}\mathrm{C}$ intervals. The test continues to the next temperature interval until the threshold value for the given binder conditioning is surpassed. An overview of the different binder aging states and the respective threshold values, spindle sizes and gap settings are shown in table 3.1.

Conditioning	Threshold	Spindle size	Gap setting
Original	$G^{*}/sin(\delta) > 1\,kPa$	25 mm	1 mm
RTFO-aged	$G^{*}/sin(\delta) > 2.2\,kPa$	25 mm	1 mm
PAV-aged	$G^{*}sin(\delta) < 5\,MPa$	8 mm	2 mm

Table 3.1: Overview of DSR test

3.4 Multiple Stress Creep Recovery

The Multiple Stress Creep Recovery or MSCR is an additional test for the DSR. It is designed for modified binders to quantify the elastic response introduced by polymer modification. The test determines the elastic recovery R and non-recoverable creep compliance J_{nr} by subjecting the binder to repeated shear strains and allowing it to recover before reloading. The MSCR test is performed on RTFO-aged material.

The binder sample was left in the DSR after the regular testing. The temperature was set to the highest passing temperature for the DSR test and the test was performed in accordance with the ASTM D7405-10a standard.

3.5 Binder aging

Artificial binder aging is used to simulate different states in the binder life cycle. Rolling thin film oven (RTFO) aging is intended to simulate the aging that happens to the binder during plant production. The RTFO-aging was performed in accordance with the NS-EN 12607 standard. The binder was poured into glass bottles and heated to 163 °C and placed in a rotating stand for 75 minutes. Two of the bottles were used to record the change in mass of the binder over the course of the test. Approximately 30 grams of the binder was poured into a small container intended for DSR testing. The rest of the binder was then subjected to long term aging using the Pressure Aging Vessel (PAV) according to the NS-EN 14769 standard. PAV-aging is intended to simulate oxidative aging by subjecting the binder to high temperatures and air pressure. The PAV aged material was then used for testing using the DSR and BBR. The RTFO is shown in figure 3.5.

Figure 3.4: Rolling Thin Film Oven

3.6 Bending Beam Rheometer

The BBR is used to determine a binder's resistance to low temperature cracking. The test determines the creep stiffness S and master curve slope m at 60 seconds loading time with a constant force of 0.98 N.

The test was performed in accordance with the NS-EN 14771 standard. The PAV-aged binder was heated to $160\,^{\circ}\mathrm{C}$ for a sufficient amount of time so that it could be poured. Metal moulds where lubricated and fitted with plastic sheets to avoid the binder sticking to the moulds. The binder was then poured into the beam moulds and allowed to cool for at least 60 minutes. The top of the samples was then trimmed with a hot spatula and the beams de-moulded and put into the test chamber for temperature conditioning for exactly 60 minutes. The tests are done at set temperatures, ensured by a fluid bath and temperature regulator, at $6\,^{\circ}\mathrm{C}$ intervals. After conditioning, the beams were put onto the supports and subjected to a constant load. New beams were prepared and tested for the next temperature until either of the threshold values $S > 300$ or $m < 0.3$ were surpassed. Figure 3.6 shows a beam and the loading cell inside the BBR test chamber.

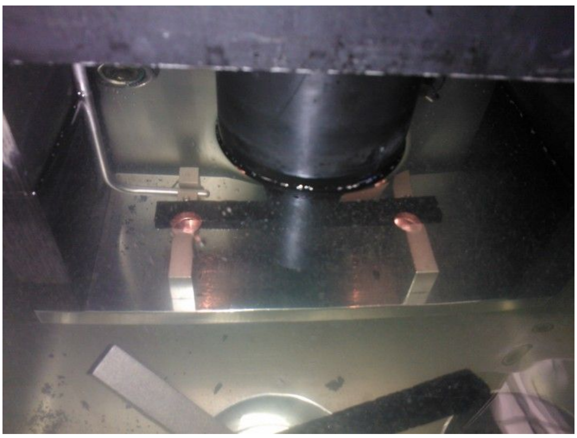

Figure 3.5: Bending Beam Rheometer

Chapter 4

Results

4.1 Overview

Table 4.1 shows the failure temperatures and PG-rating according to ASTM specifications [12] for the different binders and mixing times. Note that the BBR failure temperatures marked (*) are interpolated using results from other mixing times.

Binder	Mixing time	Original	RTFO	PAV	BBR	PG
Reference	5 min	59.2	57.9	15.1	-20.1	52-28
Reference	15 min	62.0	60.0	15.6	-20.0	58-28
Reference	30 min	62.9	61.3	16.0	-19.9	58-28
Reference	60 min	60.7	59	15.3	-20.2	58-28
Normal	5 min	64.4	66.7	14.7	-22.1	64-28
Normal	15 min	65.3	65.8	14.1	-22.5	64-28
Normal	30 min	67.3	66.6	14.2	-23.5	64-28
Normal	60 min	70.8	66.3	14.6	-22.8	64-28
Special	5 min	63.1	65.5	11.6	-24.8*	58-34
Special	15 min	66.1	66.2	11.3	-24.8	64-34
Special	30 min	67.6	65.2	11.3	-25.1	64-34
Special	60 min	68.2	64.1	11.6	-24.7*	64-34

Table 4.1: Failure temperatures and PG-rating

4.2 Dynamic Shear Rheometer

The DSR test gives results for the complex modulus G^* and phase angle δ at all test temperatures. Figures 4.1 through 4.3 show the comparative results of the G^* and $sin(\delta)$ relation for the three binders at a given temperature with respect to mixing time.

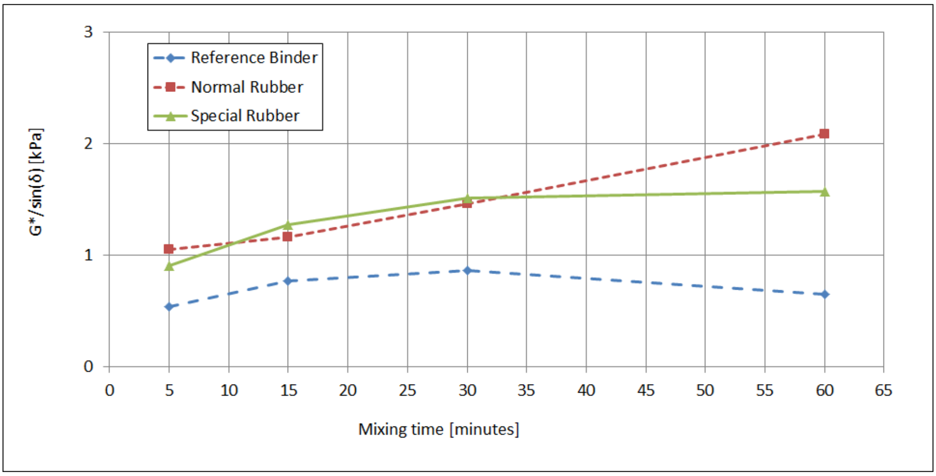

Figure 4.1: DSR Results for original binder at 64 °C

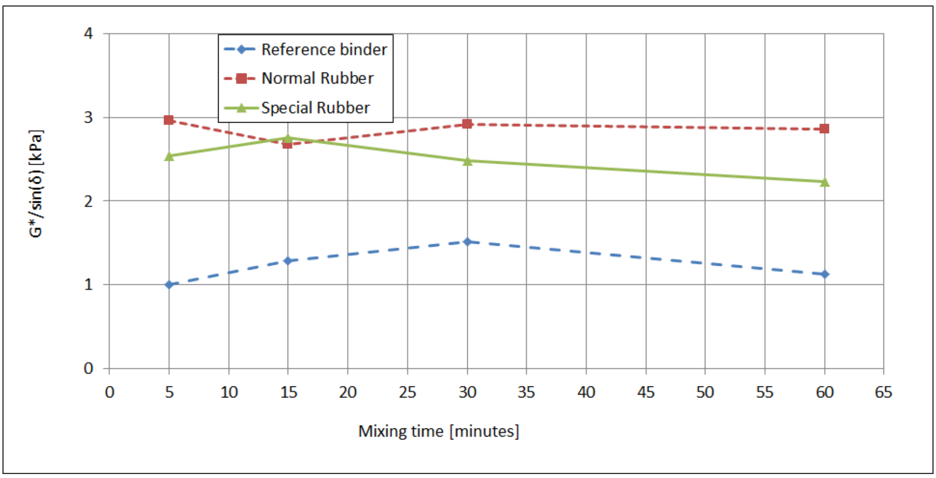

Figure 4.2: DSR Results for RTFO-aged binder at 64 °C

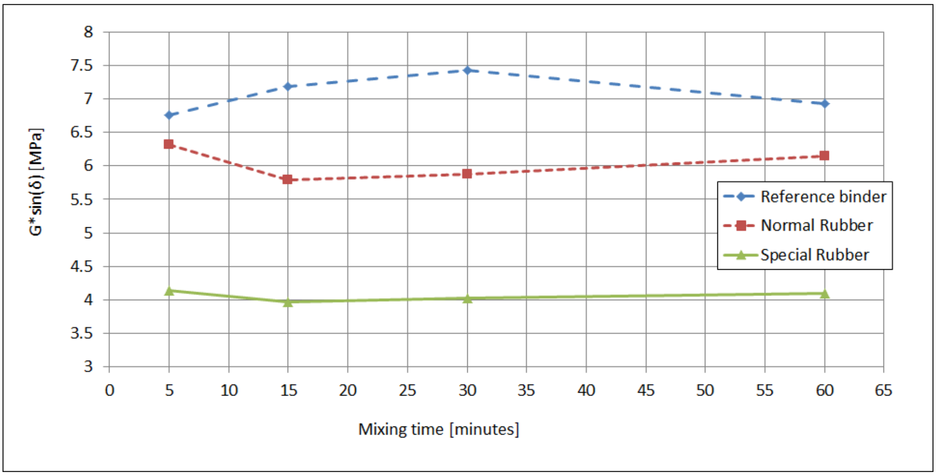

Figure 4.3: DSR Results for PAV-aged binder at 13 °C

4.3 Multiple Stress Creep Recovery

The MSCR test gives results for the non-recoverable creep compliance J_{nr} shown in Figure 4.4 and creep recovery R shown in Figure 4.5 with a shear stress of 3200 Pa. The tests are done at the highest passing temperature from the PG-grading, meaning that the measurements for the reference binder are not at the same temperature as the rubber binders.

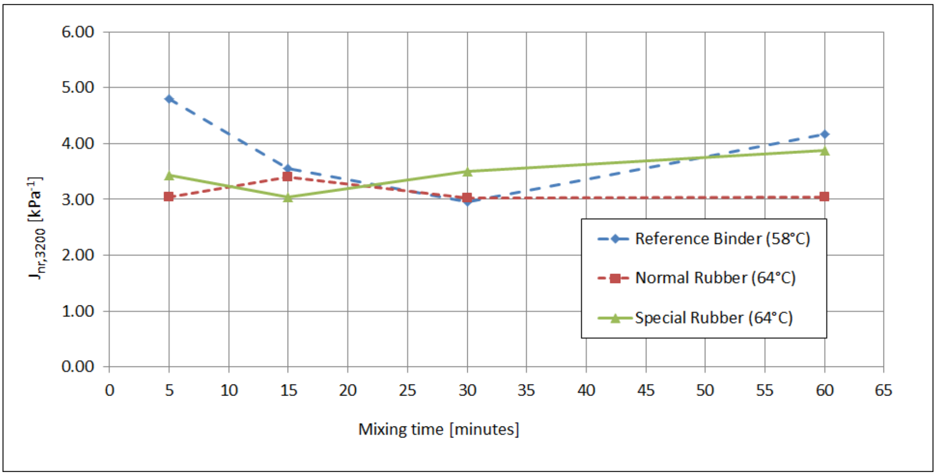

Figure 4.4: MSCR Results for RTFO-aged binder (a)

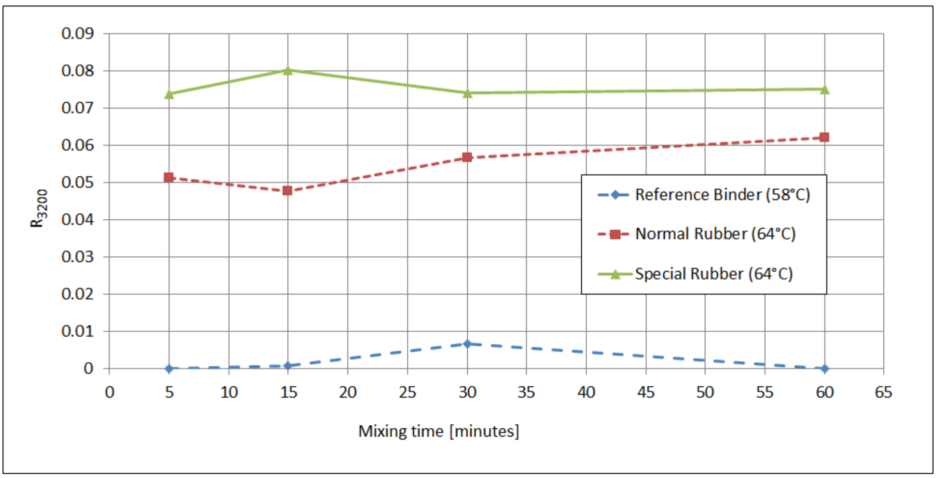

Figure 4.5: MSCR Results for RTFO-aged binder (b)

4.4 Bending Beam Rheometer

The BBR test gives results for creep stiffness at 60 seconds loading time $S(60)$ shown in Figure 4.6 and the slope of the master stiffness curve at 60 seconds $m(60)$ shown in Figure 4.7 at $-24\,^{\circ}\mathrm{C}$ for the three binders and different mixing times.

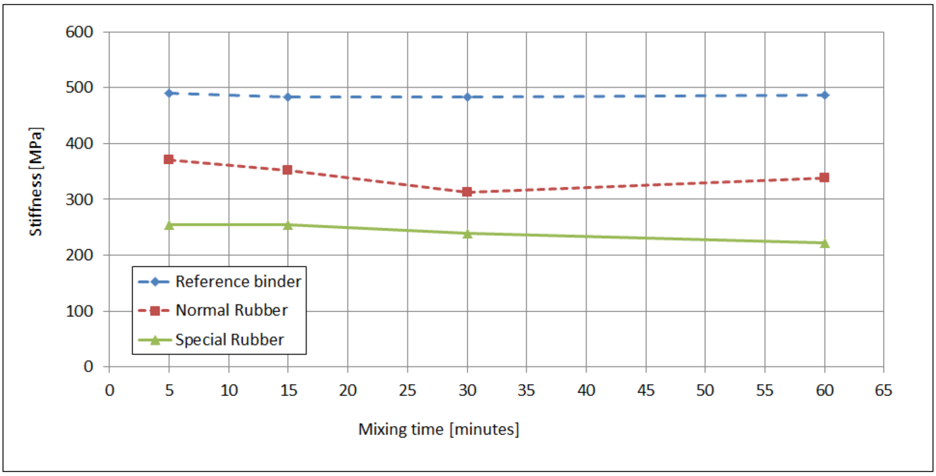

Figure 4.6: BBR Results for PAV-aged binder at $-24\,^{\circ}\mathrm{C}$ (a)

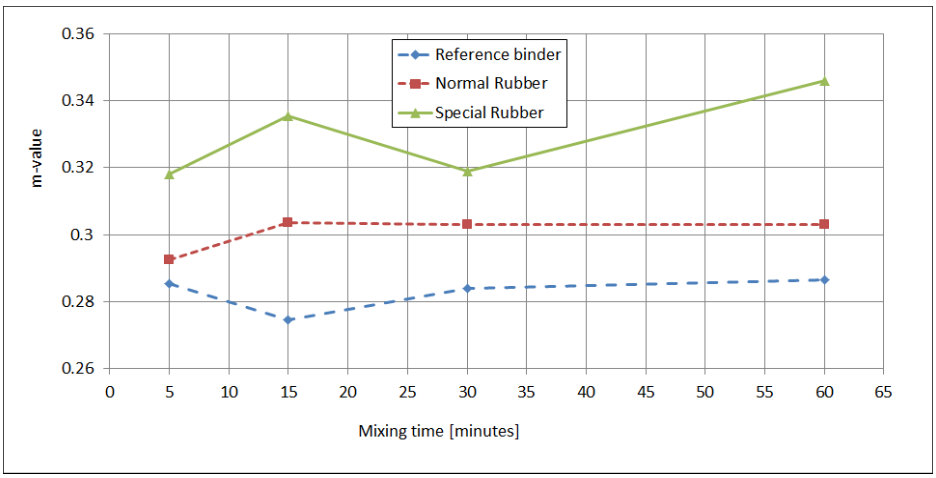

Figure 4.7: BBR Results for PAV-aged binder at $-24\,^{\circ}\mathrm{C}$ (b)

Chapter 5

Analysis

5.1 Statistical Methods

The methods and distributions used here were found in the statistics textbook by Walpole et al.[13]. Analysis of variance, or *ANOVA* is a general technique of using estimated variances from experiments to determine if means are significantly different as a result of different treatments. The zero-hypothesis is that all the means are equal, while the alternative hypothesis is that at least one of the means is significantly different. To test this, an F-test is used with a significance level α of 0.05.

Duncan's test is a more specific method for one-factor experiments that compares all combinations of pairs of measurement means and determines which pairs are significantly different. The method is used in the article by Lee et al.[9], and was chosen based on the similarity in experiment design. The Least Significant Studentized Ranges r_p with a significance level α of 0.05 are used here.

A level of mixing time or rubber type is chosen as the fixed parameter Y, while the other is denoted as X_i with k levels. For each combination of Y and X_i there are $n = 2$ observations, where each observation is denoted as z_{ij}. Each set of z_i is assumed to be normally distributed with a common unknown variance σ^2. It should be noted that the variance is significantly higher for the rubber samples due to limited homogeneity, which would suggest that this assumption is not entirely true. The variance of the results is then split in two parts, the random variance and the variance caused by different levels of X_i, also called treatment variance. An F-test determines whether any of the means are significantly different. If they are, then the differences are compared to the Duncan's Least significant range R_p to see which pairs of measurements are significantly different, where

p is the number of means in the given range. Table 5.1 shows the expressions involved in the calculation steps.

Mean	$\bar{z}_j =$	$\frac{1}{n}\sum_{i=1}^{n} z_{ij}$
Total sum of squares	$SST =$	$\sum_{i=1}^{k}\sum_{j=1}^{n}(z_{ij}-\bar{z})^2$
Treatment sum of squares	$SSX =$	$n\sum_{i=1}^{k}(\bar{z}_i-\bar{z})^2$
Error sum of squares	$SSE =$	$\sum_{i=1}^{k}\sum_{j=1}^{n}(z_{ij}-\bar{z}_j)^2$
Treatment mean square	$s_x^2 =$	$\frac{SSX}{k-1}$
Error mean square	$s^2 =$	$\frac{SSE}{k(n-1)}$
F-ratio	$f =$	$\frac{s_x^2}{s^2}$
F-test	$f >$	$f_{0.05}(k-1, k(n-1))$
Least significant range	$R_p =$	$r_p\sqrt{\frac{s^2}{n}}$
Duncan's test	$(\bar{z}_i-\bar{z}_{i+p}) >$	$R_p\ \forall(i+p\leq k+1)$

Table 5.1: ANOVA expressions

5.2 DSR

The data for the analyses are the $G^*/sin(\delta)$ values at $64\,°\mathrm{C}$ for the Original and RTFO-aged binders and the $G^*sin(\delta)$ values at $13\,°\mathrm{C}$ for the PAV-aged binder. These values were chosen over e.g. failure temperature because they have a higher precision which is important for variance calculation. Tables 5.2 through 5.7 show the Duncan grouping of the means for all the DSR experiments. The letters a to d indicate the relative difference in performance between the means in ascending order. Measurements in the same column that share a letter are not significantly different. Columns containing fs have a negative result of the F-test, meaning that there is no evidence of treatment effect for the respective X_i.

The results have been slightly simplified by only looking at 2 observations for each combination of binder and mixing time. In the case where there were made 3 observations, the latter is ignored due to being considered a biased observation. In the case where one of the parallels failed before the selected temperature the results are extrapolated in a critical manner.

Mixing time	Reference	Normal	Special
5 min	a	a	a
15 min	c	a	b
30 min	d	b	c
60 min	b	c	c

Table 5.2: DSR Original Binder - Mixing time Duncan grouping

Binder	5 min	15 min	30 min	60 min
Reference	a	a	a	a
Normal	b	b	b	c
Special	b	c	b	b

Table 5.3: DSR Original Binder - Binder Duncan grouping

Mixing time	Reference	Normal	Special
5 min	a	f	a,b
15 min	c	f	b
30 min	d	f	a,b
60 min	b	f	a

Table 5.4: DSR RTFO-aged Binder - Mixing time Duncan grouping

Binder	5 min	15 min	30 min	60 min
Reference	a	a	a	a
Normal	c	b	c	c
Special	b	b	b	b

Table 5.5: DSR RTFO-aged Binder - Binder Duncan grouping

Mixing time	Reference	Normal	Special
5 min	f	a	f
15 min	f	b	f
30 min	f	b	f
60 min	f	a	f

Table 5.6: DSR PAV-aged Binder - Mixing time Duncan grouping

Binder	5 min	15 min	30 min	60 min
Reference	a	a	a	a
Normal	a	b	b	b
Special	b	c	c	c

Table 5.7: DSR PAV-aged Binder - Binder Duncan grouping

5.3 MSCR

Tables 5.8 and 5.9 show the Duncan grouping of the mean J_{nr} values for the MSCR experiments. The reference and rubber binder measurements were done at $58\,^{\circ}\mathrm{C}$ and $64\,^{\circ}\mathrm{C}$ respectively, which means that only the rubber samples may be compared in table 5.9. This is because the reference is so significantly different that testing at the same temperature as the rubber would be unwise.

Mixing time	Reference	Normal	Special
5 min	a	f	a,b
15 min	b,c	f	b
30 min	c	f	a
60 min	a,b	f	a

Table 5.8: MSCR - Mixing time Duncan grouping

Binder	5 min	15 min	30 min	60 min
Normal	f	f	f	b
Special	f	f	f	a

Table 5.9: MSCR - Binder Duncan grouping

5.4 BBR

Tables 5.10 and 5.11 show the Duncan grouping of the mean $S(60)$ values for the BBR experiments at $-24\,°\mathrm{C}$. Note that some of the 30 minute mixing time beams were broken before testing, so this mixing time was left out of the calculation.

Mixing time	Reference	Normal	Special
5 min	f	f	f
15 min	f	f	f
30 min	N/A	N/A	N/A
60 min	f	f	f

Table 5.10: BBR - Mixing time Duncan grouping

Binder	5 min	15 min	30 min	60 min
Reference	a	a	N/A	a
Normal	b	b	N/A	b
Special	c	c	N/A	c

Table 5.11: BBR - Binder Duncan grouping

Chapter 6

Discussion

The discussion presents the thought process and possible explanations of the findings in this study. The results and their implications are compared to similar experiments from the literature study.

6.1 Mixing time

Mixing time, temperature and speed are factors that are usually given when rubber modified binders are studied. Examples from the literature study are 1 hour at 160 °C with a high speed mixer [7] and 30 minutes at 177 °C at 700 rpm [8, 9]. Bahia [7] states that too high mixing temperatures can lead to destroying the base bitumen. This is the reasoning behind the mixing temperature of 150 °C due to using a relatively soft base binder. As expected, the rubber modified binders show an increase in stiffness with longer mixing time shown in figure 4.1. This trend is attributed to the rubber absorbing the light fractions of the bitumen, resulting in a stiffer binder [6]. The change between 30 minutes and 60 minutes mixing time for the special rubber is not significant, indicating that the rubber has reached a saturated level after approximately 30 minutes. It also fits well to a log-function similarly to the findings by Airey et al. [6]. The normal rubber follows a linear function all the way to 60 minutes, indicating that the bitumen-rubber reaction takes longer for this rubber. After RTFO-aging the mixing time is essentially insignificant for both rubber modified binders. What happens in the RTFO-aging is not dissimilar from what happens during mixing. The binder is subjected to high temperatures and a rolling movement. This raises the question of whether the binder aging processes are representative for the aging these modified binders would experience in the field. Rubber is thought to reduce the impact of oxidative aging [7], which is what the aging procedures are simulating. The question is if other sources of deterioration may be more relevant for rubber modified binders.

The most surprising findings with respect to mixing time were with the reference binder. The choice to give the reference binder the same treatment as the rubber modified ones is not something that was found in the literature. Up until 30 minutes mixing time, all three binders experience similar increases in stiffness, which leads one to believe that the mechanical work is what induces the effect and not the rubber-bitumen interaction. However, the reference binder has a sharp decrease in stiffness at 60 minutes, which makes it difficult to make that conclusion. A decrease in stiffness indicates that the mixing is more than just an aging process for the reference binder. This is emphasized further by the same relation between stiffness and mixing time being present after RTFO-aging in figure 4.2. The relative difference in stiffness before and after RTFO-aging is about the same as the difference between 5 and 30 minutes mixing time. You can even see remnants of the relation for the PAV-aged material in figure 4.3, however these differences are statistically insignificant. This suggests that the binder is permanently damaged as a result of the mixing. This can be explained as changes in the molecular structure as a result of the mechanical work. For the rubber modified binders, this change can be positive, in that breaking the existing bonds may create new and stronger bonds which incorporate the rubber molecules. The rubber modified binders also have a higher viscosity, which gives a higher resistance to the mixing. There may still be damage done to the rubber modified binder which is interpreted as effects of the rubber. An additional parameter which would be interesting in this case is the mixing temperature to see if the same effect is visible for the rubber modified binders at higher temperatures.

6.2 Rubber properties

The literature suggests that rubber grinding method is significant for the high temperature performance, but only minor differences are found for low temperature performance [9, 11]. The exact nature of the difference between the grinding methods used to produce the normal and special rubber is not investigated in this study. It is assumed that the special rubber has a larger surface area and smaller particles relative to the normal rubber. This should result in higher interaction effects with the bitumen, whereas the particle effects may be smaller [8]. For the original binder DSR testing, the difference between the two rubber types is only significant at a 15 and 60 minutes mixing times, and neither binder is consistently stiffer than the other. For the RTFO-aged binder, the normal binder is significantly stiffer at all but the 15 minutes mixing time using the DSR test, whereas for the MSCR test only the 60 minute mixing time gives significant differences between the two. Overall it must be concluded that high temperature properties of the two rubber types are not significantly different. For the PAV-aged binder, the special rubber has a significantly lower stiffness for both the DSR and BBR tests which indicate intermediate and low temperature performance, respectively. These trends differ from the results found in the literature, where high temperature performance is primarily where the effects of rubber types are found [7, 9, 11].

One explanation for the difference between the rubber types is that the reactive properties of the special rubber means that a larger amount of the light oil fractions of the bitumen is absorbed by the rubber. During aging, the oxidation leads to further reduction of the concentration of these light oil fractions to the point that the absorbed oil starts diffusing from the rubber back to the bitumen. This effectively delays the aging process making the binder softer. If the rubber holds more of these light oil fractions, the effect should be greater. The DSR tests for the original and RTFO-aged binder give no indications that the bitumen-rubber interaction is more prevalent for the special rubber. If this was the case, a higher $G^* sin(\delta)$ would be expected for the special rubber.

Another possible explanation for the difference between the two rubber types is introduced in my preliminary project work [2]: The special rubber showed an overall better performance, and it was observed from the Prall test that the special rubber showed a more elastic and cohesive behavior. This can be linked to the MSCR test and the elastic recovery of the binders, shown in figure 4.5. The normal rubber is stiffer, but the special rubber has a higher elastic recovery. This indicates that the special rubber particles are more flexible, both in terms of being less stiff and more elastic. This is confirmed by the results from the BBR test, where the special rubber ends up being a full PG-rating better at low temperatures. Overall, it seems likely that the particle effect is the differentiating factor between the rubber types, whereas the differences in reactive properties are minimal.

6.3 Sources of error

The primary concern of this study with regards to systematic error is the gap setting for the DSR. Mturi et al. [10] state that gap setting should be set according to the particle size distribution of the rubber. An optimal setting is possible to find by repeating tests and looking at the resulting variation. This was not known prior to starting the testing due to being unfamiliar with the settings of the test apparatus and not having read a sufficient amount of support literature. In retrospect, the best solution would have been to use a 2 mm gap setting for the rubber modified binders as is used for the PAV-aged material. Using a 1 mm gap is likely the reason why the rubber modified binders show relatively high variance. This hypothesis is further emphasized by the reference binder showing a higher variance when the 2 mm gap is used for the PAV-aged binder. This questions the validity of the statistical analyses, as the models assume a common test variance for the different treatments [13]. In other words, the reference binder may not be directly comparable to the rubber modified binders because of different test variances.

Chapter 7

Conclusion and future work

7.1 Conclusion

The following conclusions can be drawn from this study:

- Mixing rubber with bitumen leads to an increase in binder stiffness at high temperatures, which improves rut resistance. A interaction happens between the rubber and bitumen where light fractions of the bitumen are absorbed by the rubber, indicated by an increase in $G^* sin(\delta)$ from the DSR test with longer mixing times. In addition to this interaction, there are particle effects which increase the stiffness and elastic recovery of the binder, shown by the MSCR test.

- Mixing time is also significant for the stiffness of the reference binder. This effect is interpreted as permanent damage to the chemical structure of the bitumen due to it being visible after aging. No significant relation between mixing time and stiffness was found after aging for the rubber modified binders, indicating that no permanent damage was done during mixing. This can have implications for mixing of rubber modified binders, meaning that caution is advised when mixing soft binders at high temperatures and high mixing speeds.

- The two rubber types used in this study did not show significantly different high temperature performance. Little evidence supports the presumed reactive properties of the special rubber. The special rubber has significantly better low and intermediate temperature performance than the normal rubber, indicating better fatigue resistance and resistance to low temperature cracking. This is attributed to differences in the properties of the rubber particles, where the special rubber has a higher elastic recovery and flexibility.

- Based on the tests performed in this study, the PG-ratings of the binders are:
  - Reference PG 58-28
  - Normal rubber PG 64-28
  - Special rubber PG 64-34

7.2 Future work

The experience from working with rubber modified binders has led to the following thoughts concerning future work and possible challenges:

- This study shows that the properties of rubber modified binders are different from the base bitumen. This means that recipes using rubber modified binders should be tailored to the properties of the binder.
- Particle effects are considered a major part of the properties of the different rubber types. Interaction with the filler particles in asphalt mixtures can be studied.
- A binder's cohesive properties and problems with stripping of aggregates when exposed to water is a challenge today. The effects of rubber modification on these properties can be studied.

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Appendix

MASTEROPPGAVE

(TBA4940, Veg, masteroppgave)

VÅREN 2013
for
Andreas Kjosavik

Fastlegging av rheologiske egenskaper for myk bitumen med og uten gummitilsetning

BAKGRUNN

Gamle bildekk utgjør en stor avfallsmengde, og i mange land er dette et stort problem som utgjør store volumer. I Norge blir en god del brukt som brensel i sementproduksjon, noe som også har sine miljømessig uheldige sider, men store deler av de utrangerte dekkene ender som problemavfall, gjerne i andre land enn opphavslandet. En har opp gjennom tiden derfor prøvd andre bruksområder, eksempelvis som underlag for lekeapparater og i løpebaner etc. Dette blir likevel ikke de store volumene, så bruk i forbindelse med vegbygging er det man har satt et visst håp til for å kunne få til effektiv og fornuftig gjenbruk av bildekk. I Norge prøvde man på 1980- og 1990-tallet å produsere «Gummiasfalt», der oppmalte bildekk ble blandet med steinmaterialet, og ideen var at disse dekkene skulle være så fleksible at piggene i piggdekk ble trykt ned i dekkeoverflata uten å medføre slitasje, og dermed kunne fungere som slitesterke dekke. Det viste seg at disse dekkene hadde relativt liten motstand mot mekaniske påkjenninger, og bruk av ordinært vinter-vedlikeholdsutstyr gikk til dels hardt ut over dekket. Forsøksdekkene ble dermed relativt kortlivet.

Med overgang til mer piggfrie vinterdekk og et generelt sterkere miljøfokus, er det andre egenskaper ved asfaltdekker enn slitasjemotstand det fokuseres på, som deformasjonsmotstand, støy- og støvproduksjon. Det har også blitt utviklet andre produksjonsmetoder for gummiasfalt, eksempelvis ved såkalt «våtprosess», som med hell er brukt også i nordisk klima, og det er utviklet andre gummityper som påstås å ha gunstige effekter på asfalten.

Det er utført flere prosjekt- og masteroppgaver ved NTNU de siste årene der en har sett på ulike gummitilsetninger i asfaltdekker, både tette og åpne, sist ved kandidatens prosjektoppgave høsten 2012. Selve bindemidlet er det derimot ikke forsket så mye på. I denne masteroppgaven skal en derfor fokusere på effekten ulike gummitilsetninger har på de rheologiske egenskapene for bindemidlet.

OPPGAVE

Beskrivelse av oppgaven

Denne masteroppgaven omhandler myke bindemidler med gummitilsetning, og tar spesielt sikte på å sammenligne egenskaper for slike bindemidler tilsatt «vanlig gummi» og et russisk spesialprodukt med bindemiddel uten gummi. Videre vil en se på effekten av ulike blandingstider for originalt og kort- og langtidsaldret bindemiddel med og uten gummitilsetning.

Målsetting og hensikt
En viktig målsetting med oppgaven er å fremskaffe kunnskap om deformasjons-, stivhets- og lavtemperaturegenskaper for bindemiddel tilsatt gummi av ulikt opphav, og å se på effekten av ulike blandingstider. En skal sammenligne disse egenskapene med egenskaper for tilsvarende bindemiddel uten gummitilsetning, og på basis av dette komme med anbefalinger om hvordan eventuell gummitilsetning i mykt bindemiddel bør utføres, og også påpeke eventuelle gevinster og ulemper ved slik tilsetning.

Deloppgaver og forskningsspørsmål
Viktige deloppgaver vil dermed være:

- Gjennomføre et litteraturstudium for å finne eventuell internasjonal dokumentasjon av effekter av gummitilsetning i bindemidler. Dette skal også omfatte en gjennomgang av hvilke tester som anbefales gjennomført for å finne disse effektene.
- For myke bindemiddelprøver med 0 og 10 % gummitilsetning både av «vanlig gummi» og den russiske «spesialgummien», bruke Dynamic Shear Rheometer (DSR) for å teste stivhets- og deformasjonsegenskaper, og Bending Beam Rheometer (BBR) for lavtemperaturegenskaper.
- Bruke DSR og BBR til å teste effekten av blandingstid.
- Dersom litteraturstudiet skulle avdekke andre relevante tester, eventuelt vurdere om slike skal gjennomføres.
- Oppsummere og sammenligne laboratorieresultatene for de ulike kombinasjonene, og hvis mulig rangere de ulike variantene.
- Komme med anbefalinger for hvordan eventuell gummitilsetning i myke bindemidler bør porsjoneres og utføres.
- Anslå eventuelle gevinster og ulemper ved slik gummitilsetning.

Resultatene fra disse forsøkene vil kunne være av internasjonal interesse, så det bør vurderes om resultatene skal publiseres, men dette må klareres med veileder(e) og aktuelle produktleverandører. I så fall bør masteroppgave-rapporten skrives på engelsk.

GENERELT
Oppgaveteksten er ment som en ramme for kandidatens arbeid. Justeringer vil kunne skje underveis, når en ser hvordan arbeidet går. Eventuelle justeringer må skje i samråd med faglærer ved instituttet.

Ved bedømmelsen legges det vekt på grundighet i bearbeidingen og selvstendighet i vurderinger og konklusjoner, samt at framstillingen er velredigert, klar, entydig og ryddig uten å være unødig voluminøs.

Besvarelsen skal inneholde
- standard rapportforside (automatisk fra DAIM, http://daim.idi.ntnu.no/)
- tittelside med ekstrakt og stikkord (mal finnes på siden http://www.ntnu.no/bat/skjemabank)
- forord
- sammendrag på norsk og engelsk (studenter som skriver sin masteroppgave på et ikke-skandinavisk språk og som ikke behersker et skandinavisk språk, trenger ikke å skrive sammendrag av masteroppgaven på norsk)
- innholdsfortegnelse inklusive oversikt over figurer, tabeller og vedlegg
- om nødvendig en liste med beskrivelse av viktige betegnelser og forkortelser benyttet
- hovedteksten
- referanser til kildemateriale som ikke er av generell karakter, dette gjelder også for muntlig informasjon og opplysninger.
- oppgaveteksten (denne teksten signert av faglærer) legges ved som Vedlegg 1.
- besvarelsen skal ha komplett paginering (sidenummerering).

Besvarelsen kan evt. utformes som en vitenskapelig artikkel. Arbeidet leveres da også med rapportforside og tittelside og om nødvendig med vedlegg som dokumenterer arbeid utført i prosessen med utforming av artikkelen.

Se forøvrig «Råd og retningslinjer for rapportskriving ved prosjektarbeid og masteroppgave ved Institutt for bygg, anlegg og transport». Finnes på http://www.ntnu.no/bat/skjemabank

Hva skal innleveres?
Rutiner knyttet til innlevering av masteroppgaven er nærmere beskrevet på http://daim.idi.ntnu.no/. Trykking av masteroppgaven bestilles via DAIM direkte til Skipnes Trykkeri som leverer den trykte oppgaven til instituttkontoret 2-4 dager senere. Instituttet betaler for 3 eksemplarer, hvorav instituttet beholder 2 eksemplarer. Ekstra eksemplarer må bekostes av kandidaten/ ekstern samarbeidspartner.

Ved innlevering av oppgaven skal kandidaten levere en CD med besvarelsen i digital form i pdf- og word-versjon med underliggende materiale (for eksempel datainnsamling) i digital form (f. eks. excel). Videre skal kandidaten levere innleveringsskjemaet (fra DAIM) hvor både Ark-Bibl i SBI og Fellestjenester (Byggsikring) i SB II har signert på skjemaet. Innleveringsskjema med de aktuelle signaturene underskrives av instituttkontoret før skjemaet leveres Fakultetskontoret.

Dokumentasjon som med instituttets støtte er samlet inn under arbeidet med oppgaven skal leveres inn sammen med besvarelsen.

Besvarelsen er etter gjeldende reglement NTNUs eiendom. Eventuell benyttelse av materialet kan bare skje etter godkjennelse fra NTNU (og ekstern samarbeidspartner der dette er aktuelt). Instituttet har rett til å bruke resultatene av arbeidet til undervisnings- og forskningsformål som om det var utført av en ansatt. Ved bruk ut over dette, som utgivelse og annen økonomisk utnyttelse, må det inngås særskilt avtale mellom NTNU og kandidaten.

(Evt) Avtaler om ekstern veiledning, gjennomføring utenfor NTNU, økonomisk støtte m.v.
Det er inngått avtale om økonomisk utgiftsdekning fra Statens vegvesen, Vegdirektoratet. Det vises til avtaleteksten for de betingelsene som må være oppfylt for at avtalt beløp utbetales. Se http://www.ntnu.no/bat/skjemabank for avtaleskjema.

Helse, miljø og sikkerhet (HMS):
NTNU legger stor vekt på sikkerheten til den enkelte arbeidstaker og student. Den enkeltes sikkerhet skal komme i første rekke og ingen skal ta unødige sjanser for å få gjennomført arbeidet. Studenten skal derfor ved uttak av masteroppgaven få utdelt brosjyren "Helse, miljø og sikkerhet ved feltarbeid m.m. ved NTNU".

Dersom studenten i arbeidet med masteroppgaven skal delta i feltarbeid, tokt, befaring, feltkurs eller ekskursjoner, skal studenten sette seg inn i "Retningslinje ved feltarbeid m.m.". Dersom studenten i arbeidet med oppgaven skal delta i laboratorie- eller verkstedarbeid skal studenten sette seg inn i og følge reglene i "Laboratorie- og verkstedhåndbok". Disse dokumentene finnes på fakultetets HMS-sider på nettet, se http://www.ntnu.no/ivt/adm/hms/.

Studenter har ikke full forsikringsdekning gjennom sitt forhold til NTNU. Dersom en student ønsker samme forsikringsdekning som tilsatte ved universitetet, anbefales det at han/hun tegner reiseforsikring og personskadeforsikring. Mer om forsikringsordninger for studenter finnes under samme lenke som ovenfor.

Innleveringsfrist:
Arbeidet med oppgaven starter 14. januar 2013

.
Besvarelsen leveres senest ved registrering i DAIM innen 10. juni 2013 kl 1500.

Faglærer ved instituttet: Helge Mork

Veileder(eller kontaktperson) hos ekstern samarbeidspartner: Carl Thodesen, SINTEF

Institutt for bygg, anlegg og transport, NTNU
Dato: 06.02.2013, (revidert: 06.06.2013)

Underskrift

Helge Mork

Faglærer

MT	#	FT	G^*58	G^*64	$\delta58$	$\delta64$	$G^*sin(\delta)58$	$G^*sin(\delta)64$
5 min	1	59.1	1.15	0.537	87.1	88.2	1.15	0.537
5 min	2	59.2	1.16	0.545	87.1	88.2	1.16	0.545
15 min	1	62	1.67	0.771	86	87.4	1.68	0.772
15 min	2	61.9	1.66	0.762	85.9	87.3	1.67	0.763
30 min	1	62.9	1.91	0.867	85.4	86.9	1.91	0.869
30 min	2	62.9	1.9	0.865	85.4	86.9	1.91	0.866
60 min	1	60.7	1.41	0.655	86.6	87.8	1.41	0.657
60 min	2	60.7	1.4	0.651	86.6	87.8	1.41	0.652

DSR Original Binder, Reference

MT	#	FT	G^*58	G^*64	$\delta58$	$\delta64$	$G^*sin(\delta)58$	$G^*sin(\delta)64$
5 min	1	63.6	2.03	0.951	85.2	86.8	2.03	0.952
5 min	2	65.1	2.39	1.13	84.4	86.3	2.48	1.13
5 min	3	64.6	2.28	1.07	84.4	86.4	2.29	1.07
15 min	1	65.5	2.51	1.18	84	86	2.53	1.19
15 min	2	65.1	2.4	1.13	84.2	86	2.41	1.13
30 min	1	67.3	3.06	1.44	81.2	83.6	3.1	1.45
30 min	2	67.3	3.12	1.46	81.6	84.2	3.16	1.47
60 min	1	70.9	4.14	2.07	74.6	77.4	4.29	2.12
60 min	2	70.6	4.01	2	75	77.5	4.15	2.04

DSR Original Binder, Normal

MT	#	FT	G^*58	G^*64	$\delta58$	$\delta64$	$G^*sin(\delta)58$	$G^*sin(\delta)64$
5 min	1	64.1	2.1	1.01	83.8	85.9	2.12	1.01
5 min	2	62.2	1.66	0.798	84.4	86.1	1.67	0.8
5 min	3	63.1	1.86	0.898	84.3	86.1	1.87	0.9
15 min	1	66.2	2.72	1.28	82.5	84.8	2.75	1.28
15 min	2	66	2.66	1.25	82.5	84.9	2.69	1.26
30 min	1	67.1	3	1.42	81	83.7	3.03	1.43
30 min	2	68.1	3.33	1.59	80.3	83.4	3.38	1.6
30 min	3	67.6	3.14	1.51	80.9	83.8	3.18	1.51
60 min	1	68	3.07	1.53	76.9	80.3	3.15	1.55
60 min	2	68.3	3.15	1.57	76.6	80.2	3.24	1.59

DSR Original Binder, Special

MT	#	FT	G^*58	G^*64	$\delta 58$	$\delta 64$	$G^*sin(\delta)58$	$G^*sin(\delta)64$
5 min	1	58	2.2	1.01	85	86.5	2.21	1.01
5 min	2	57.8	2.14	N/A	84.5	N/A	2.14	N/A
15 min	1	59.9	2.83	1.28	83.8	85.6	2.85	1.28
15 min	2	60	2.87	1.29	83.8	85.6	2.88	1.3
30 min	1	61.2	3.35	1.51	83.1	85	3.37	1.51
30 min	2	61.3	3.38	1.52	83	85	3.41	1.52
60 min	1	59	2.49	1.13	84.4	86.1	2.5	1.13
60 min	2	59	2.5	1.12	84.4	86.1	2.51	1.12

DSR RTFO-aged Binder, Reference

MT	#	FT	G^*64	G^*70	$\delta 64$	$\delta 70$	$G^*sin(\delta)64$	$G^*sin(\delta)70$
5 min	1	66.7	2.84	1.49	74	76.6	2.96	1.53
5 min	2	66.7	2.85	1.49	74.2	77	2.96	1.53
15 min	1	65.3	2.46	1.28	75.8	78.2	2.53	1.31
15 min	2	66.2	2.7	1.42	74	76.7	2.81	1.46
15 min	3	65.8	2.59	1.36	74.7	77.2	2.69	1.4
30 min	1	66.5	2.79	1.47	74.1	76.6	2.9	1.51
30 min	2	66.6	2.81	1.48	74	76.2	2.93	1.53
60 min	1	66.5	2.81	1.46	75.1	77.3	2.91	1.5
60 min	2	66.1	2.72	1.39	76	78.2	2.81	1.42

DSR RTFO-aged Binder, Normal

MT	#	FT	G^*64	G^*70	$\delta 64$	$\delta 70$	$G^*sin(\delta)64$	$G^*sin(\delta)70$
5 min	1	65	2.32	1.28	72.5	75.2	2.43	1.33
5 min	2	65.9	2.52	1.39	71.6	74.2	2.66	1.44
15 min	1	65.9	2.56	1.39	72.8	75.1	2.68	1.44
15 min	2	66.4	2.69	1.46	71.8	74.3	2.83	1.52
30 min	1	64.9	2.32	1.24	73.4	75.6	2.42	1.28
30 min	2	65.4	2.43	1.32	72.7	75.1	2.54	1.37
60 min	1	64.3	2.18	1.19	73.3	75.4	2.28	1.23
60 min	2	63.9	2.09	N/A	73.9	N/A	2.18	N/A

DSR RTFO-aged Binder, Special

MT	#	R_{100}	R_{3200}	R_{diff}	$J_{nr,100}$	$J_{nr,3200}$	$J_{nr,diff}$
5 min	1	2.7%	0%	N/A	4.29	4.80	12%
15 min	1	3.4%	0.1%	97.4%	3.21	3.59	11.8%
15 min	2	3.4%	0.1%	97.4%	3.18	3.53	10.9%
30 min	1	4.5%	0.8%	81.6%	2.65	2.96	11.9%
30 min	2	4.2%	0.5%	87.5%	2.63	2.93	11.5%
60 min	1	2.6%	0%	N/A	3.73	4.13	10.8%
60 min	2	2.4%	0%	N/A	3.78	4.19	11%

MSCR, Reference

MT	#	R_{100}	R_{3200}	R_{diff}	$J_{nr,100}$	$J_{nr,3200}$	$J_{nr,diff}$
5 min	1	29%	5.3%	81.8%	1.90	3.04	59.9%
5 min	2	25.6%	5%	80.5%	2.00	3.04	52%
15 min	1	24.2%	3.4%	86%	2.41	3.64	51.2%
15 min	2	28%	5.4%	80.8%	2.02	3.19	57.8%
15 min	3	28.2%	5.5%	80.4%	2.13	3.37	58.3%
30 min	1	29.2%	6.1%	79.3%	1.90	3.01	58.4%
30 min	2	33.7%	5.3%	84.4%	1.76	3.04	72.5%
60 min	1	27.8%	6.9%	75.3%	1.96	2.93	49.3%
60 min	2	24.1%	5.5%	77.1%	2.21	3.15	42.5%

MSCR, Normal

MT	#	R_{100}	R_{3200}	R_{diff}	$J_{nr,100}$	$J_{nr,3200}$	$J_{nr,diff}$
5 min	1	31.6%	7.3%	77.1%	2.14	3.56	66.3%
5 min	2	34.6%	7.5%	78.4%	1.87	3.32	77.4%
15 min	1	32.4%	7.2%	77.6%	1.92	3.16	64.5%
15 min	2	35.9%	8.8%	75.6%	1.72	2.92	70%
30 min	1	33.1%	7.3%	78%	2.16	3.62	67.6%
30 min	2	31.5%	7.5%	76%	2.07	3.37	62.9%
60 min	1	32.9%	7.7%	76.5%	2.28	3.8	66.8%
60 min	2	30.5%	7.3%	76.2%	2.47	3.95	60.1%

MSCR, Special

MT	#	FT	G^*16	G^*13	$\delta 16$	$\delta 13$	$G^*sin(\delta)16$	$G^*sin(\delta)13$
5 min	1	14.8	5190	8480	53.2	50.2	4160	6510
5 min	2	15.3	5590	9120	53.3	50.2	4480	7000
15 min	1	15.4	5820	9370	51.6	48.7	4570	7040
15 min	2	15.7	6070	9750	51.6	48.7	4760	7320
30 min	1	15.8	6270	10000	50.7	47.8	4850	7420
30 min	2	16.1	6510	N/A	50.8	N/A	5040	N/A
60 min	1	15.1	5500	8910	52.5	49.5	4360	6780
60 min	2	15.4	5780	9350	52.3	39.3	4570	7090

DSR PAV-aged Binder, Reference

MT	#	FT	G^*16	G^*13	$\delta 16$	$\delta 13$	$G^*sin(\delta)16$	$G^*sin(\delta)13$
5 min	1	14.7	5540	8660	49.1	46.5	4190	6290
5 min	2	14.7	5560	8710	49.3	46.7	4220	6340
15 min	1	13.9	4790	7600	50.9	48.2	3720	5670
15 min	2	14.2	4990	7920	51	48.3	3880	5920
30 min	1	14.2	5060	7990	50.2	47.6	3890	5900
30 min	2	14.1	5010	7900	50.2	47.6	3850	5840
60 min	1	14.5	5350	8370	49.4	46.8	4060	6100
60 min	2	14.6	5420	8490	49.3	46.8	4110	6190

DSR PAV-aged Binder, Normal

MT	#	FT	G^*13	G^*10	$\delta 13$	$\delta 10$	$G^*sin(\delta)13$	$G^*sin(\delta)10$
5 min	1	11.6	5460	8540	49	46.5	4120	6190
5 min	2	11.6	5480	8590	49.2	46.6	4150	6240
15 min	1	11.1	5140	8010	48.9	46.5	3880	5810
15 min	2	11.5	5410	8430	48.7	46.3	4070	6090
30 min	1	11.7	5630	8760	48.6	46.1	4220	6320
30 min	2	10.8	5070	7520	49.2	47	3840	5500
60 min	1	11.6	5490	8580	49	46.5	4150	6220
60 min	2	11.5	5340	8370	49.4	46.9	4060	6120

DSR PAV-aged Binder, Special

MT	#	$S(60)$ -18	$S(60)$ -24	$m(60)$ -18	$m(60)$ -24
5 min	1	198	489	0.383	0.29
5 min	2	N/A	493	N/A	0.281
15 min	1	212	481	0.374	0.275
15 min	2	210	486	0.375	0.274
30 min	1	212	483	0.369	0.284
60 min	1	208	495	0.374	0.285
60 min	2	176	480	0.372	0.288

BBR, Reference

MT	#	$S(60)$ -18	$S(60)$ -24	$m(60)$ -18	$m(60)$ -24
5 min	1	142	386	0.354	0.292
5 min	2	149	356	0.36	0.293
15 min	1	156	351	0.369	0.29
15 min	2	134	354	0.395	0.317
30 min	1	153	312	0.374	0.303
60 min	1	144	358	0.38	0.305
60 min	2	140	318	0.381	0.301

BBR, Normal

MT	#	$S(60)$ -18	$S(60)$ -24	$S(60)$ -30	$m(60)$ -18	$m(60)$ -24	$m(60)$ -30
5 min	1	92.7	268	N/A	0.381	0.322	N/A
5 min	2	95	243	N/A	0.404	0.314	N/A
15 min	1	96.9	270	553	0.409	0.336	0.254
15 min	2	83.9	239	608	0.398	0.335	0.255
30 min	1	98.1	265	558	0.404	0.335	0.256
30 min	2	90.8	214	N/A	0.394	0.303	N/A
60 min	1	84.8	173	N/A	0.411	0.266	N/A
60 min	2	92.3	223	N/A	0.422	0.346	N/A

BBR, Special

Reference	Normal	Special	
0.537	0.952	1.01	$Avg = 0.829$
0.545	1.13	0.8	
$SSE = 0.038$	$f(2,3) = 9.55$	$r_2 = 4.501$	$r_3 = 4.516$
$SSX = 0.267$	$10.57 > 9.55$	$R_2 = 0.358$	$R_3 = 0.359$
$\bar{z}_3 - \bar{z}_1 = 0.5$	$0.5 > 0.359$		
$\bar{z}_3 - \bar{z}_2 = 0.136$	$0.136 < 0.358$	$\bar{z}_2 - \bar{z}_1 = 0.364$	$0.364 > 0.358$
Duncan:	Reference	Normal	Special
	A	B	B

Example, Statistical Analysis 5 min Original Binder