Effects of Fillers on the Rheological/Mechanical Performance of Mastics/Asphalt Mixes

P.K. Dehdezi*
SINTEF Building and Infrastructure, Department of Road and Railway Engineering, Trondheim, 7465, Norway.
Email: pejman.dehdezi@sintef.no

C.C. Thodesen
SINTEF Building and Infrastructure, Department of Road and Railway Engineering, Trondheim, 7465, Norway

C. Encinas
Department of Civil and Transport Engineering, Norwegian University of Science and Technology, Trondheim, Norway

M. Mengede
Franzefoss Minerals AS, Olav Ingstads vei 5, Postboks 53, 1309 Rud, Oslo, Norway

Corresponding author

ABSTRACT
This paper summarises the effects of four different fillers on the rheological properties of mastics and mechanical properties of asphalt mixtures produced with these four different types of added fillers. The main aim of the study is to evaluate rheological properties of the mastic that control the asphalt performance (i.e. rutting, fatigue cracking and thermal cracking). The second objective of the study is to evaluate some of the mechanical properties of the asphalt mixtures produced with the studied fillers as well as the optimum bitumen content for a certain aggregates, grading curve and filler content.
Therefore, in addition to a preliminary characterization of the fillers based on the evaluation of the Specific Surface Area (SSA) and Rigden voids, the study is divided in two main sections. Firstly four different mastics have been prepared in order to conduct Dynamic Shear Rheometer (DSR) and Bending Beam Rheometer (BBR) tests and therefore obtain information about the rheological behaviour of the lab prepared mastics. In addition, the performance grade (PG) of the mastics with regards to the main distresses above has been performed. Secondly asphalt mixtures have been produced using the studied fillers to conduct the Marshall method of mix design and thus analyse the volumetric of the mixture as well as its mechanical properties (i.e. Marshall stability and flow) in order to determine the effect of fillers on the mechanical performance of the asphalt mixtures.
The Marshall results revealed a relatively strong correlation between the Marshall stability and the SSA of the fillers that might be attributed not only to the different stiffnesses obtained with fillers of different SSA but also to the ease of a crack path to travel through a mastic produced with lower SSA fillers with regards to those produced with fillers with a higher SSA value. As a result of the DSR test on the lab-produced mastics another important correlation was observed between the SSA and the failure temperature at high test temperatures which depends on the rutting parameter, observing that higher failure temperatures are obtained when fillers with higher SSA values are used.

1. BACKGROUND

In this paper the word bitumen refers to the petroleum product and the word asphalt to a mixture of aggregates and bitumen. Asphalt is an essential construction material and the majority of roads are constructed or surfaced with asphalt. As an engineering material, asphalt is typically designed to provide stiffness and bearing capacity, and resist the repeated loading experienced by a pavement under traffic. Road pavements are typically constructed in layers, with each layer of the pavement fulfilling a slightly different function. The surface layer of the road is subjected to the highest stresses in the pavement as it is in direct contact with vehicle tyres, and additionally the surface is exposed to the elements which results in the surface of the pavement reaching the highest and lowest temperatures. The effect of repeated loading manifests itself in two ways, permanent deformation, commonly referred to as “rutting”, and “cracking” through fatigue of the asphalt. The effect of the climate conditions manifests in thermal cracking of the pavement when its temperature drops rapidly. All asphalt mixtures consist of three components aggregates, bitumen (as a binder) and air.

Aggregates

Aggregate is the term used to describe mineral materials such as gravel, sand and crushed rock. Simplistically, aggregates can be considered as the solid particles in an asphalt mixture. Aggregates provide a structural skeleton to asphalt mixtures and it is this structure that provides mechanical strength to the asphalt. Additionally, because the aggregate constitutes the solid surface of the asphalt mixture and this surface largely governs the durability of the mixture in the presence of water, aggregate type is very important when considering the durability of asphalt mixtures. Aggregates commonly used in asphalt include limestone, granites, amphibolites, diorites, basalt and gneiss. Additionally, recycled aggregates such as crushed glass and secondary aggregates, such as slag from iron or steel production, are also commonly used.

Bitumen

Bitumen is defined in the Oxford English Dictionary as “a tar-like mixture of hydrocarbons derived from petroleum naturally or by distillation”. Bitumen acts as a binder in asphalt and binds, or "cements", the aggregate particles together. Bitumen is a complex mixture of components and as a result, bitumen is considered a material with a complex response to stress. The response of bitumen to stress is dependent on both loading time (frequency) and temperature, and it is this behaviour which characterises the mechanical behaviour of asphalt mixtures (Read and Whiteoak 2003).

Asphalt fillers

Fillers modify the properties, increase the performance of, and provide improved durability to composites, polymers, rubbers, adhesives, coatings and construction materials. Fillers are used to lower the cost of materials, change processing characteristics and increase rigidity (Taylor 2007). Fillers in asphalt can be defined as "finely divided mineral matter such as hydrated lime, rock dust, slag dust, hydraulic cement, fly ash or other suitable matter" and typically this definition refers to the size fraction smaller than 63μm (Taylor 2007). Fillers in asphalt are used to obtain increased stiffness or rigidity, reducing creep (permanent deformation), increase density and lower the cost of asphalt mixtures. Too much filler in asphalt mixtures can lead to cracking or fatigue problems as the stiffness is increased. Too little can lead to "bleeding" of bitumen from the mixture (Taylor 2007). The most frequently used filler in asphalt is limestone (calcium carbonate) which is the general term for rocks where calcite, a form of calcium carbonate, is the predominant mineral. Other materials commonly used as fillers in asphalt include Portland cement and hydrated lime, which possesses well documented properties with regard to mixture durability and reduced potential for moisture damage in asphalt. In order to provide satisfactory properties in the finished asphalt, filler should (Kavussi and Hicks 1997):

- Not have adverse chemical reactions with bitumen
- Not possess hydrophilic surfaces to ensure good adhesion
- Not possess high porous particles which may lead to excessive stiffening through selective adsorption
- Contain a dense (well graded) particle size distribution

When bitumen is combined with mineral filler, a "mastic" is formed. This mastic can be viewed as the component of the asphalt mixture that binds the aggregates together and also the component of the asphalt that undergoes deformation when the pavement is stressed under traffic loading. The characteristics of the filler can significantly influence the properties of the mastic, and thus the filler properties can have significant effects on asphalt mixture performance (Osman 2004).

2. AIM

This paper is intended to examine the effects of four different fillers on the rheological/mechanical performance of mastics/asphalts. To achieve this aim, the specific objectives would be as follows:

- To characterise the fillers and bitumen used in the project,
- To devise an experimental programme to determine the rheological properties of mastics produced with four fillers, including the determination of Performance Grading (PG) of mastics, according to the ASTM standard specification for Performance Graded Asphalt Binder (ASTM 2008),
- To determine the optimum bitumen required to manufacture asphalt mixtures using four different fillers,
- To determine the mechanical performance of asphalt mixtures made with different added fillers.

3. METHODOLOGY

The objectives of this paper are linked together, as shown in Figure 1, in order to complete the study.

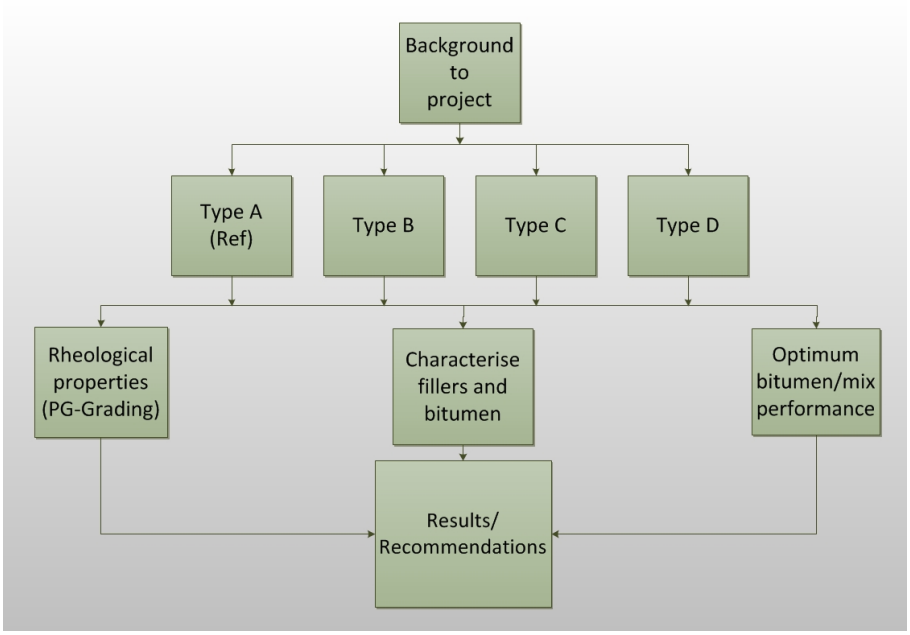


Figure 1: Study flowchart

3.1. Materials characteristics

All types of fillers were tested for their;

- Specific Surface Area (SSA) using BET
- "Rigden Voids" (i.e. voids in the filler in its compacted state) (NS EN 2008)
- Specific gravity

3.2. Mastic preparations

A mortar representative of the actual mastic in the asphalt mixture was prepared assuming that only the added filler will mix with all the bitumen in the asphalt mix to form the mastic component of the asphalt mixture. Based on this assumption and considering a 5% bitumen content by total mass of asphalt, all the percentage of filler in AB11 aggregate grading curve (see Figure 2) were added to 70/100 Pen bitumen which results in ~50/50, bitumen/filler proportions by mass of mastic.

In order to reduce variability in the test results, the mastics were prepared using a procedure suggested by (Osman 2004), as follows:

1) Place a filler sample in an oven at 110±5°C for drying to a constant weight.
2) Place the bitumen into an oven at 160±5°C, until it reaches a uniform temperature of 160°C. Stirring is needed from time to time.
3) After preheating the bitumen and filler samples, remove each from its respective oven.
4) Place the correct quantities of the dried filler sample and the heated bitumen into a sample container and place it in an oven at 160°C and hand mix with a spatula until the air bubbles escape. Care must be taken to prevent loss of fines during mixing. Stirring of the mixture is necessary to produce a homogeneous specimen.
5) When the mastic appears visually homogenous, the mastic will be ready for testing.

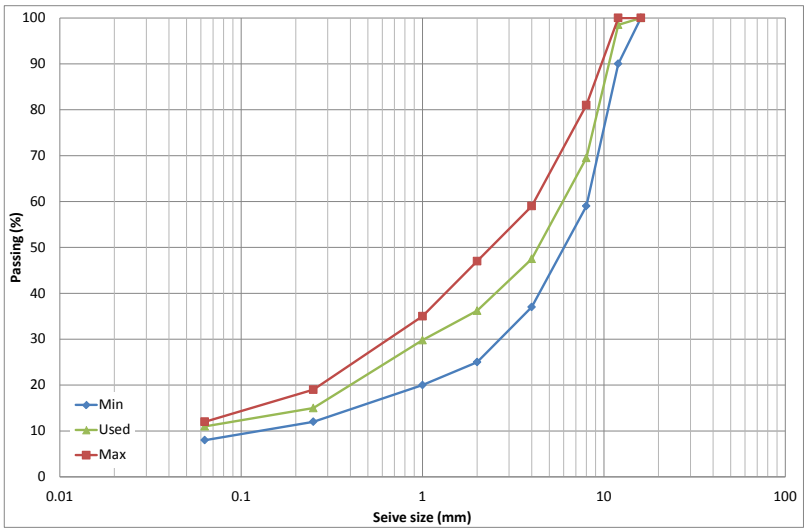


Figure 2: Aggregate grading limits in AB11 mix

3.3. Rheological characteristics of mastics, including Performance Grading (PG)

Dynamic Shear Rheometers (DSRs) and Bending Beam Rheometers (BBRs) (see Figure 3 and 4) are used to measure the rheological characteristics of bitumens. Typically, dynamic tests are performed and the parameters that are determined are the complex modulus and phase angle from which the viscoelastic properties of the bitumen can be evaluated. The DSR has become an accepted test method for determining the dynamic mechanical properties of bitumen in the linear region (Osman 2004; Taylor 2007). The standard DSR test system consists of parallel metal plates, a temperature control chamber, a loading device and a control and data acquisition system. A water chamber controls the temperature of the test specimen. Water is pumped through the test chamber by a separate circulating bath temperature control unit. The water chamber and the temperature control unit can control the temperature of the specimen to an accuracy of ± 0.1°C. Two samples from each mastic were tested as Un-aged, RTFOT (short term aging), and PAV (long term aging) in order to grade the mastic for its performance at high and intermediate temperatures.

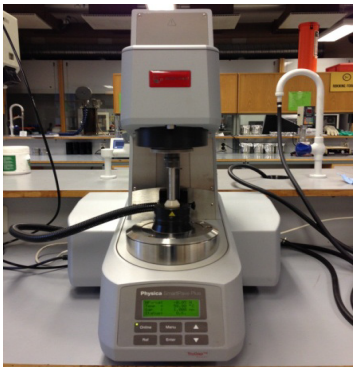


Figure 3: Dynamic Shear Rheometer (DSR) available at SINTEF-NTNU

BBR (see Figure 4) are used to test bitumens at low pavement service temperatures in order to determine its propensity to thermal cracking. The midpoint deflection of a simply supported prismatic and rectangular cross-section beam of bitumen subjected to a constant load applied to its midpoint is measured at different temperatures. From this midpoint deflection, the applied load, the dimensions and the span length of the beam both the maximum bending stress and strain can be calculated and thus the stiffness at different times. The standard BBR test consists of a controlled temperature fluid bath where the beam is placed and loaded with a constant load of 980± 50mN during 240 seconds monitoring the deflection versus time using a computerized data acquisition system. The maximum bending stress at the midpoint is calculated from the dimensions of the specimen, the span length and the load applied for loading times of 8, 15, 30, 60, 120 and 240 seconds. The maximum strain is calculated from the dimensions of the test specimen and the measured deflection at the same loading times. The stiffness of the specimen is then obtained by dividing the maximum bending stress to the maximum strain.

Two PAV-aged samples from each mastic were tested in order to grade the mastic for its performance at low temperatures.



Figure 4: Bending Beam Rheometer (BBR) available at SINTEF-NTNU

3.4. Marshall Mix Design

Marshall method of mix design were used in order to determine the optimum bitumen content required in the asphalt mixes made with different fillers. In this method, the resistance to plastic deformation of

a compacted cylindrical specimen of asphalt is measured when the specimen is loaded diametrically at a deformation rate of 50 mm per minute. There are two major features of the Marshall method of mix design.

1) Density-voids analysis and
2) Stability-flow tests.

The Marshall stability of the mix is defined as the maximum load carried by the specimen at a standard test temperature of 60°C. The flow value is the deformation that the test specimen undergoes during loading up to the maximum load. Flow is measured in 0.25 mm units. In this test, an attempt was made to obtain optimum binder content for the type of filler mix used and the expected traffic intensity. Step of the Marshall designs are as follows (O'Flaherty 2002);

1) Select aggregate grading to be used.
2) Determine the proportion of each aggregate size required to produce the design grading.
3) Determine the specific gravity of the aggregate combination and bitumen.
4) Prepare the specimens with varying bitumen contents.
5) Determine the specific gravity of each compacted specimen.
6) Perform stability tests on the specimens.
7) Calculate the percentage of voids, and percentage of Voids Filled with Bitumen (VFB) in each specimen.
8) Select the optimum binder content from the data obtained.
9) Evaluate the design with the design requirements.

4. RESULTS & ANALYSIS

4.1. Material characteristics

Specific Surface Area

The surface area per unit mass, the Specific Surface Area (SSA), was determined by the BET method. Table 1 shows the SSA of the fillers measured using BET technique. The SSA represents the ratio of a particle's surface area to its mass. From Table 1 can be observed that the SSA of three alternative fillers is significantly higher than the reference filler. The higher SSA means smaller particle size.

Table 1: Fillers Specific Surface Area (m^2/g)

Fillers	**Specific Surface Area (m^2/g)**
Type A (Ref)	0.89
Type B	2.04
Type C	2.25
Type D	2.91

Rigden voids

Voids in the filler in its compacted state, referred to as "Rigden Voids". Table 2 shows the "Rigden voids" of the fillers which were determined according to NS EN (2008). The results show that Type B had the highest Rigden voids followed by Type D, Type C, and Type A filler. Zulkati et al. (2012) observed a set of different fillers under Scanning Electron Microscopy (SEM) and related their particle shapes with the Ridgen voids of fillers. They concluded that particles having larger grain size and smoother faces could result to a lower Ridgen voids. This might be due to the fact that smoother grains have less friction and slide together that could result in a lower void content. By relating Zulkati et al. (2012) finding to this project, it could be concluded that Type A filler have a larger particle size (i.e. having lower SSA) and it may have a smoother faces compared to the other fillers. In addition, (Faheem et al. 2012) reported that, the Rigden Voids has significant influence on two mastic

properties: viscosity and non-recoverable compliance which is related to permanent deformation (i.e. rutting) of asphalt. As the Rigden voids increases, the ability to resist permanent deformation in asphalt mixes as well as mastic viscosity and stiffness will decrease.

Table 2: "Rigden voids" of the fillers

Fillers	**Rigden Voids (%)**
Type A (Ref)	31.6
Type B	46.7
Type C	38.3
Type D	42.0

Figure 5 shows the relationship between the Ridgen voids and the SSA. Although the minimum Ridgen voids occur at the lowest SSA but, there is not any strong linear relationship (with R^2=0.46) between these two parameters.

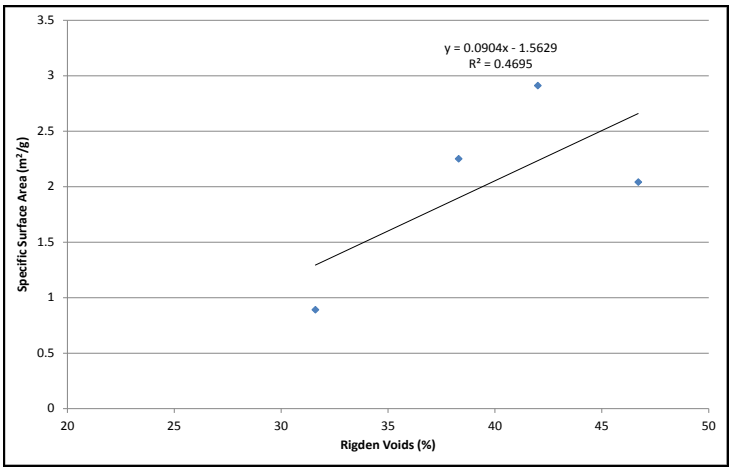


Figure 5: SSA and Ridgen voids relationship for different fillers

4.2. Marshall Tests

For each mix 3 specimens were manufactured at 3 different bitumen contents of 5%, 5.2%, and 5.4%. The maximum theoretical densities of mixes at different bitumen content as well as the compacted density of the specimens were measured prior to the Marshall test. Figure 6 shows that all mixes manufactured with alternative fillers (i.e. Type, B, C, and D) have achieved a higher stability than the reference mix (i.e. Type A). The lower stability of Type A mixes might be attributed to their lower SSA. Figure 7 shows a schematic picture of a reference and alternative mastic. Lower SSA of Type A (i.e. larger particle size) as well as its possible smooth surfaces (i.e. lower Ridgen void) might result in a lower stiffness of mastics. In addition, the crack path travels easier through the Type A mastics compared to the alternative mastics. Therefore, less force is required to crush an asphalt mix composed of Type A, thus, its stability would be lower than alternative fillers.

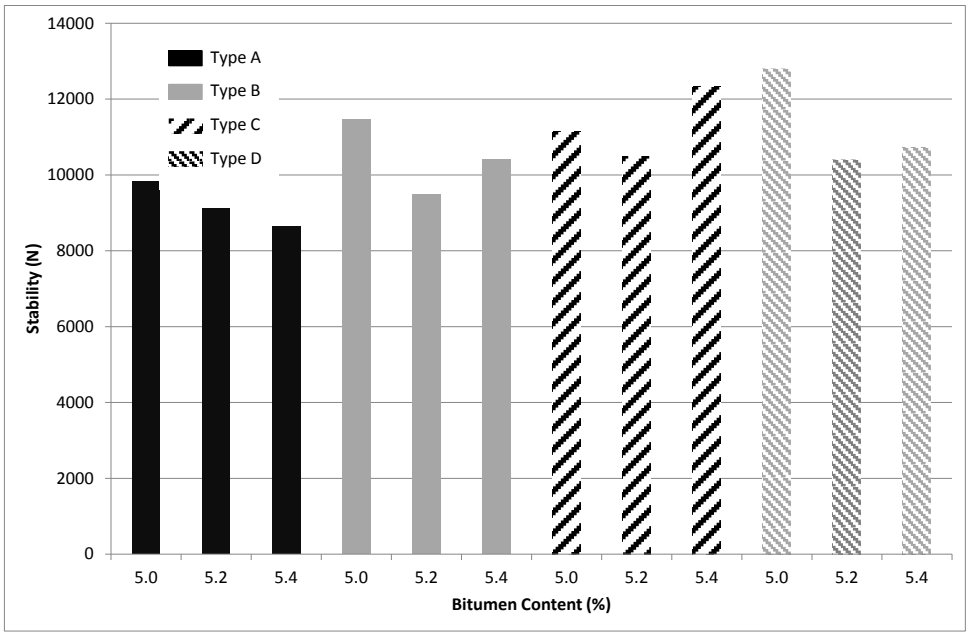


Figure 6: Stability results

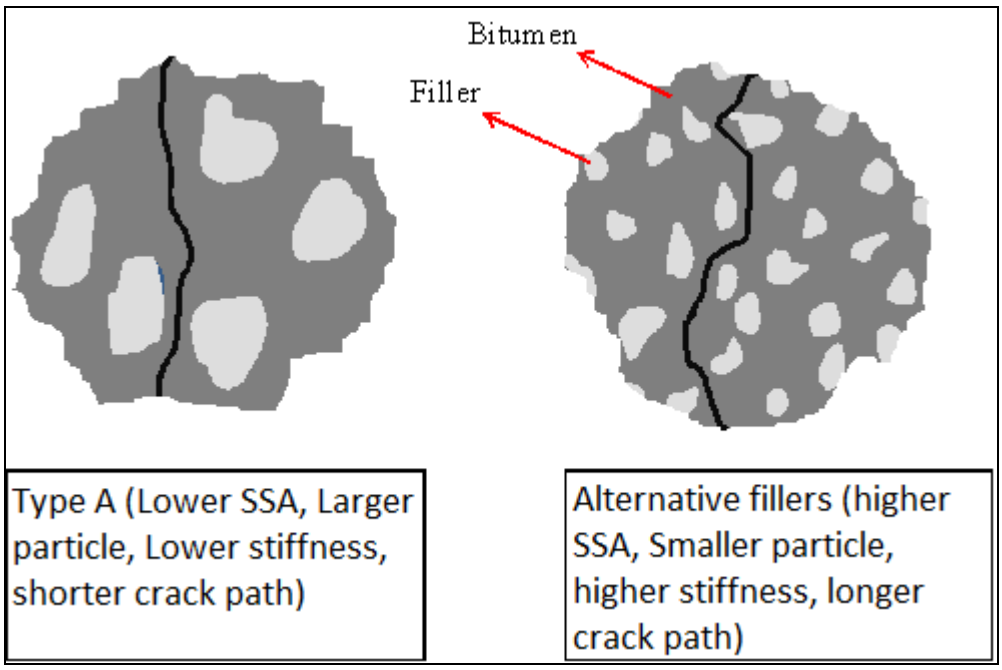


Figure 7: Schematic picture of a reference and alternative mastic

Figure 8 shows the relationship between the average stability of samples and the SSA of fillers. As figure shows, there is a positive and fairly strong relationship between these two parameters.

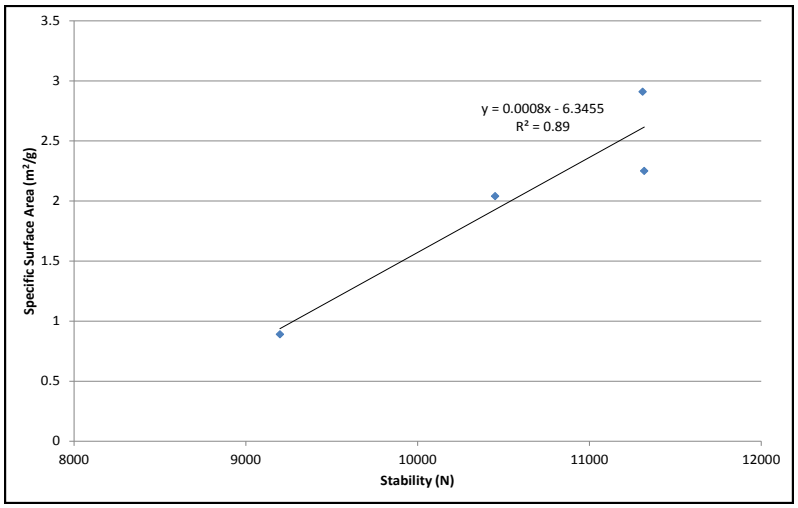


Figure 8: Relationship between average stability and specific surface area of fillers

Table 3 shows the requirements given by Norwegian Public Road Administration (NPRA) for AB11 wearing course (NPRA 2004).

Table 3: Requirements for AB mixes (NPRA 2004)

Requirements for AADT*<5000	**Min (%)**	**Max (%)**
Air Voids	2.0	5.5
Voids Filled Bitumen (VFB)	72	89
*AADT= average annual daily traffic		

Figure 9 shows the air voids values of Marshall samples. From the figure, it can be observed that at least one mix from each alternative fillers can perform in the range recommended by NPRA (i.e. 2%-5.5% in Table 3). None of the mixes composed of reference filler (i.e. Type A) are in the range. Type A filler due to its lower SSA probably requires lower bitumen content than 5%. The mixes that are in agreement with NPRA are as follows:

- Type B with 5.2%.
- Type C with 5.0% and 5.2% binder content.
- Type D with 5.4%.

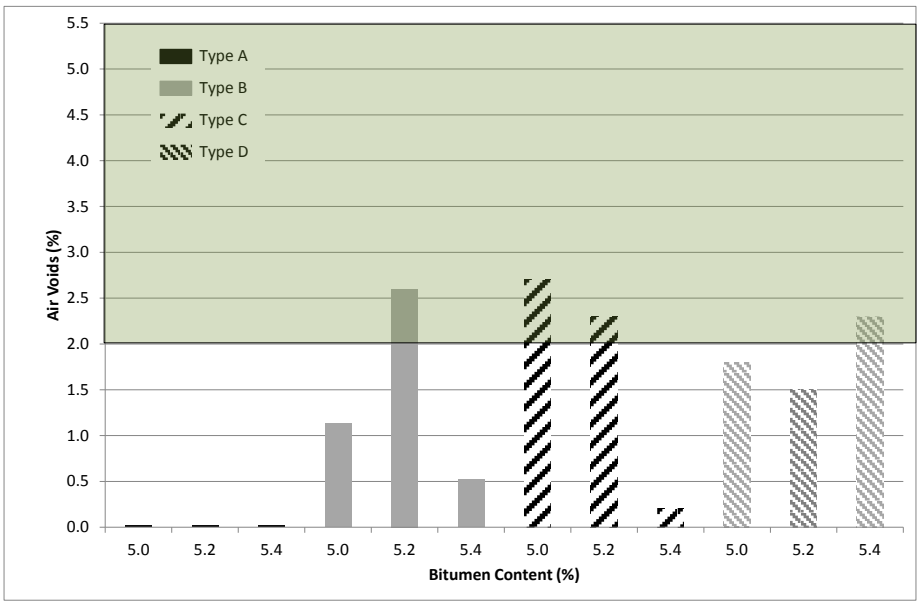


Figure 9: Air void of mixtures

Figure 10 shows the value of VFB for different mixes manufactured with each of the four fillers. Figure shows that mixes manufactured with the reference filler are not comply with the Norwegian standard. The following mixes manufactured from the alternative fillers are in the range of VFB (i.e. 72-89%) recommended by NPRA:

- Type B with 5.2%.
- Type C with 5% and 5.2% binder content.
- Type D with 5% and 5.4%.

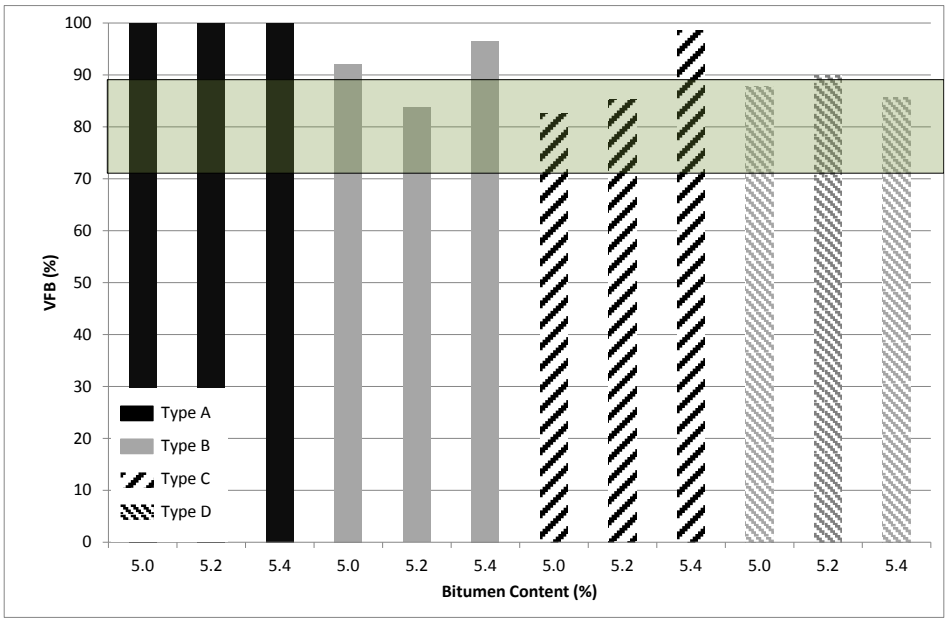


Figure 10: VFB of Marshall samples

In conclusion the following mixes can be used as a wearing course. These mixes are those which have satisfied both the air void and the VFB content according to the Norwegian handbook 018 (NPRA 2004).

- Type B with 5.2%.
- Type D with 5.4%.
- Type C with 5% and 5.2% binder content.

4.3. DSR & Mastic Performance-Grading

Two samples from each mastic were tested in DSR to determine the failure at high temperature (i.e. upper value in PG grading) which depends on the rutting parameter defined as $|G^*|/\sin\delta \geq 1$ kPa and $|G^*|/\sin\delta \geq 2.2$ kPa in Un-aged and RTFOT aged, respectively. Table 4 shows the values at high temperature failure in DSR. It can be observed that, mastic manufactured from the alternative fillers fails at higher temperature compared to that of the reference mastic (i.e. Type A). In addition, mastic composed of Type D shows failure at the highest temperature. These results indicate that, asphalt mixes composed of alternative fillers may have better performance in terms of their rutting resistance.

Table 4: Failure at high temperature in DSR

Fillers	**Un-aged (°C)**	**RTFO (°C)**
Type A (Ref)	72.7	72.8
Type B	74.0	73.3
Type C	74.3	73.2
Type D	76.3	74.7

Another two samples from PAV-aged mastic were prepared and tested in DSR to determine the failure at intermediate service temperature which depends on the fatigue parameter defined as $|G^*| \cdot \sin\delta \leq$ 5000 kPa. Table 5 shows the intermediate failure temperature in DSR. It is observed that Type A shows in this case the lowest failure temperature which means the best resistance to fatigue cracking as in terms of this distress lower temperatures represent more critical service conditions.

Table 5: Failure at intermediate temperature in DSR

Fillers	**PAV (°C)**
Type A (Ref)	30.9
Type B	31.7
Type C	31.8
Type D	33.3

4.4. BBR & Mastic Performance-Grading

Two samples from each mastic were tested in the BBR in order to determine the failure at low temperature (i.e. lower value in PG grading system). The failure temperature in this case depends on two parameters, the s-value ≤ 300 Mpa (creep stiffness s(t)) and the m-value ≥ 0.300 (rate of change m(t) of the creep stiffness). Both s-value and m-value measured at a certain test temperature and at time t=60 seconds are equal, by time temperature superposition, to those s-value and m-value obtained as measured after two hours of loading time at a temperature 10°C lower than the test temperature (Roberts et al. 1996). The failure temperature based on the measurements of s-value and m-value at time t=60 seconds were used to determine the correspondent failure temperature after two hours of loading time, which is referred to as the limiting stiffness temperature and therefore as the lower performance grade in the PG grading system.

Table 6 illustrates the fail temperature values obtained in the BBR according to both requirements in terms of s-value and m-value. The failure temperature is considered to be the highest of the temperatures shown in Table 6 for each mastic.

Table 6: Failure at intermediate temperature in BBR

Fillers	PAV (°C)	
	s-value	m-value
Type A (Ref)	-4.6	-7.7
Type B	-4.8	-9.3
Type C	-4.3	-7.6
Type D	-4.8	-7.1

As shown, filler Type C presented the highest failure temperature and therefore the highest propensity to thermal cracking. However all the tested mastics fell in the same lowest performance grade (-10°C).

It must be noted that although all the mortars fell in the PG 70-10 category, the alternative fillers have a higher temperature failure than the reference one (i.e. Type A) and Type D showed the highest value of 76.3 °C. This might suggest that asphalt mixtures composing of these alternatives fillers could resist more under the rutting, however, more work is needed to verify this. On the other hand the effect of the alternative fillers at low and intermediate service temperatures in comparison with Type A (reference) is not that clear. However, it is interesting to note that Type B presented a reasonably balanced combination of satisfying behaviors at high, intermediate and low service temperatures.

Figure 11 shows the relationship between the SSA of the fillers and their higher temperature failures which is corresponding to their ability to resist permanent deformation (i.e. rutting). Figure 11 suggests that a strong positive relationship exists between the SSA of the fillers and their high temperature failures. As the SSA increases (i.e. filler particles gets smaller), the ability to stand permanent deformation (i.e. high temperature failure) also increases.

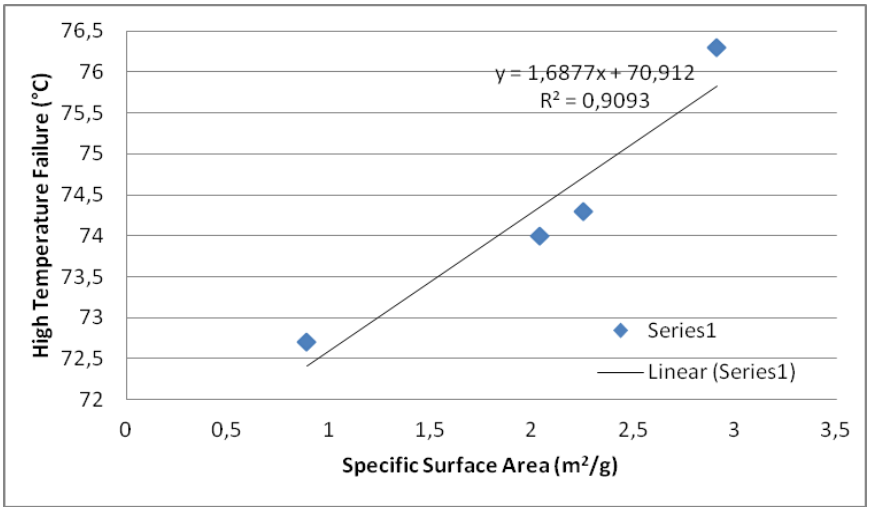


Figure 11: Relationship between specific surface area and high temperature failure

5. CONCLUSIONS AND FUTURE WORK

This study has investigated the effects of four different fillers on the rheological/mechanical performance of mastics/asphalts. Within the limits of aggregate and bitumen type, aggregate grading, and filler content, the following conclusions can be drawn on the basis of the results and analysis presented in this study.

1) All asphalt mixes manufactured with the alternative fillers (i.e. Type B, C, and D) achieved higher stability than the reference mix (i.e. asphalt mix manufactured with Type A). The lower stability of the reference mix might be attributed to the lower Specific Surface Area (SSA) of the Type A fillers that causes a crack to travel easier through the sample under the force.

2) There was a positive and fairly strong relationship (R^2=0.89) between the average stability of asphalt mixtures (determined under Marshall test) with the SSA of the fillers in the mix.

3) The following mixes are those which satisfy the requirements given by the Norwegian Public Road Administrations (NPRA) in handbook 018 (i.e. air void limit and the Voids Filled with Bitumen (VFB) content limit) (NPRA 2004). These filers can be used as AB11 wearing course:
    - Type B with 5.2% bitumen content.
    - Type C with 5.0% and 5.2% bitumen content.
    - Type D with 5.4% bitumen content.
4) A strong correlation (R^2=0.91) was observed between the SSA and the failure temperature at high test temperatures (i.e. higher value in Performance Grading) which depends on the rutting parameter. Higher failure temperatures are obtained when fillers with higher SSA values are used in the mastics.

For the future work it would be interesting to evaluate the resistance of asphalt mixtures, manufactured with mentioned fillers, against fatigue cracking and rutting in order to verify their effects on the mechanical performance of asphalt mixtures. In addition, the effect of filler shape, size and content on the performance of asphalt mixes could be studied.

6. ACKNOLEDGMENTS

The authors would like to gratefully acknowledge Ms. Sara Anastasio (NTNU), Ms. Lisbeth Johansen (SINTEF) and Mr. Andreas Kjosavik (SINTEF) for their valuable help and comments during the Marshall, DSR, and BBR tests.

7. REFERENCES

ASTM (2008). American Society for Testing and Materials, Standard Specification for Performance Graded Asphalt Binder, ASTM D6373-07.

Faheem, A., C. Hintz, H. Bahia and I. Al-Qadi (2012). Influence of filler fractional voids on mastic and mixture performance. In: TRB 2012 Annual Meeting. Washington.

Kavussi, A. and R. G. Hicks (1997). Properties of bituminous mixtures containing different fillers. Proceedings of the Association of Asphalt Paving Technologists. **66:** 153.

NPRA (2004). Norwegian Public Roads Administration, Vegbygging (Materials and Pavement Design Manual), Handbook 018.

NS EN (2008). Tests for mechanical and physical properties of aggregates. Determination of the voids of dry compacted filler, NS EN 1097-4:2008.

O'Flaherty, C. A. (2002). Highways, The location, design, construction and maintenance of road pavements. Oxford, Butterworth-Heinemann.

Osman, S. (2004). The role of bitumen and bitumen/filler mortars in bituminous mixture fatigue, University of Nottingham.

Read, J. and D. Whiteoak (2003). The Shell Bitumen Handbook. London, Thomas Telford Publishing.

Roberts, F. L., Kandhal, P. S. Brown, E. Ray, D. Lee and T. W. Kennedy (1996). Hot Mix Asphalt Materials, Mixture Design and Construction. NAPA Research and Education Foundation, Lanham, MD.

Taylor, R. (2007). Surface interactions between bitumen and mineral fillers and their effects on the rheology of bitumen-filler mastics. Doctor of Philosophy, University of Nottingham.

Zulkati, A., W. Diew and D. Delai (2012). "Effects of Fillers on Properties of Asphalt-Concrete Mixture." Journal of Transportation Engineering **138**(7): 902-910.