DOCTORAL THESIS

Applicability of Limiting Phase Angle Temperatures for Measuring the Low Temperature Performance and Aging of Asphalt Binders

Kristjan Lill

TALLINNA TEHNIKAÜLIKOOL
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Declaration:
Hereby I declare that this doctoral thesis, my original investigation and achievement, submitted for the doctoral degree at Tallinn University of Technology has not been submitted for doctoral or equivalent academic degree.

Kristjan Lill

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Piiravate faasinurga temperatuuride rakendatavus bituumenite madala temperatuuri ja vananemisalase toimivuse mõõtmiseks

KRISTJAN LILL

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List of publications

The thesis has been prepared based on the following peer-reviewed journal (indexed by SCOPUS and WOS) papers:

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III **Lill, K.**, Kontson, K., Aavik, A. (*accepted for publication*, 2024). “Determining Asphalt Binder Aging by Using Limiting Phase Angle Temperature”, The Baltic Journal of Road and Bridge Engineering, 19(4), pp. 28–49, DOI 10.7250/bjrbe.2024-19.647

Author’s contribution to the publications

Contribution to the papers in this thesis are:

I Testing of samples no. 18 to 24, curation and analysis of the data, visualization, drafting and writing the manuscript with all the co-authors.

II Conceptualization, testing and supervision of testing technicians, curation and analysis of the data, visualization, drafting and writing the manuscript with all the co-authors.

III Conceptualization, testing and supervision of testing technicians, curation and analysis of the data, visualization, drafting and writing the manuscript with all the co-authors.

Introduction

Asphalt binder is a complex material that is manufactured from crude oil. Depending on the crude used and the refining process, the properties of the asphalt binder are affected accordingly. Asphalt binder plays a crucial role in the composition of asphalt mixtures, where it acts mainly as an adhesive to hold together the aggregate matrix, but to some extent it also acts as a medium to fill the voids between the aggregate particle to adjust the air void content, which is especially important in northern climatic conditions.

There are many possible pavement defects, two of the most notable are rutting and cracking. Although, the binder does influence the rutting resistance of asphalt, it is clear that this mainly needs to be controlled with a suitable aggregate composition. Cracking on the other hand, especially low-temperature cracking is mainly controlled by the properties of the binder.

Depending on the region, the climatic effects on the binder are different. In the northern part of Europe, binders are subjected to a wide range of temperatures. In the winter months its temperatures can drop below –30 °C, whereas in the summer its temperature can rise much higher than the actual air temperature as the dark asphalt pavement is absorbing the sunlight radiation. For instance, the weather stations from Estonian national roads reveal the pavement temperatures rising up to 60 °C (Kontson et al., 2023, 2024). Their study was conducted on highways, some of which are situated in forest shaded areas, hence it can be assumed that due to heat island effects the pavement temperatures can be even higher in densely built towns.

This yields that the binders must have adequate properties over a broad spectrum of temperatures. The current European specifications in EN 12591 (CEN, 2009) and EN 14023 (CEN, 2010) have been in use for a long time and these versions don't have significant changes comparing to previous editions. This means that the current grading system is decades old. Although the binders supplied to asphalt mixture manufacturers still meet these specifications, there is an understanding that the present binders seem to behave differently as compared to previous decades. The exact reasons behind this are not known, but it cannot be excluded that refineries have enhanced their processes to obtain larger quantities of products with higher profitability, such as different gases and liquid fuels. The refinement of manufacturing processes is considered normal, but it is the task of the specifications to guarantee adequate performance. It should be noted that in the current empirical specifications, there are no restrictions on the chemical composition of the binder. Hence it might be possible that the binders are modified in a way that yield undesirable characteristics.

Updates to the European specifications for paving grade binders presented in EN 12591 (CEN, 2009) and for polymer modified binders in EN 14023 (CEN, 2010) suggested to add more performance-based specifications. Unfortunately, this work has been halted at the moment, as in the current legislative framework there is no possibility for further harmonization. To ensure free movement of products throughout the European Union, the use of harmonized standards is crucial. Fortunately, the legislation is expected to be updated in the near future, allowing thus to implement new specifications for asphalt binders.

Meanwhile new and improved methods for specifying asphalt binders need to be developed. Accordingly, this study focuses on investigating different test methods, found from around the world.

The final focus is on developing a method of using the limiting phase angle temperature for specifying asphalt binder low temperature performance and also for measuring the aging of the binder. Note that the currently commonly used test method for specifying low-temperature performance, the Fraass breaking point, has proven to be unreliable (Baumanis et al., 2021; Besamusca et al., 2010; Bueno et al., 2015; Gražulytė & Vaitkus, 2017), whereas accounting for long-term aging, in general, is lacking in the European specifications. There is only the Rolling Thin Film Oven Test (RTFOT), which is used to simulate short-term aging, but there is no method for predicting the aging characteristics that will occur during the binder's service life within the pavement.

The first part of the research, publication I specified on low temperature performance test methods that are used worldwide. Also, the findings from preceding experimental research by Lill et al. (2019) were included. Resulting from this, it was found that the cumbersome Extended Bending Beam Rheometer (EBBR) test can be the reference method, but the limiting phase angle temperature, measured in the Dynamic Shear Rheometer (DSR), could have the potential to be a quicker alternative. More specifically, the research focused on the temperature where the phase angle equalled 30° (T(30°)).

The second part of the research, publication II focused on the usability of this DSR-based method in the North-Eastern European area and its asphalt binder market. Publication III examined whether the T(30°) parameter could be used for measuring the aging of asphalt binders.

The main novelties of the research are: (i) the T(30°) parameter can be used as a tool for approximating asphalt binder low temperature performance; (ii) the T(30°) parameter is suitable for measuring the aging of asphalt binders; (iii) the T(30°) parameter was capable of discovering a deterioration in asphalt binder quality that remained undetected by conventional methods; (iv) the standard Fraass breaking point should be dismissed as the low temperature specifications in Europe due to its low reproducibility.

Abbreviations

AASHTO	American Association of State Highway and Transportation Officials
ADFT	Accelerated Dynamic Shear Rheometer Fatigue Test
ASTM	American Society for Testing and Materials
BBR	Bending Beam Rheometer
CEN	European Committee for Standardization
DCM	Dichloromethane
DSR	Dynamic Shear Rheometer
DTT	Direct Tension Test
EBBR	Extended Bending Beam Rheometer
FTIR	Fourier-transform infrared spectroscopy
II	Performance grading intermediate temperature grade
LT	Low temperature
PAV	Pressure Aging Vessel
PG	Performance Grading
RAP	Recycled Asphalt Pavement
RTFOT	Rolling Thin Film Oven Test
SARA	Saturates, Aromatics, Resins, Asphaltenes
VET	Viscous to Elastic Transition
XX	Performance grading high temperature grade
YY	Performance grading low temperature grade

1 Background and aims of the study

1.1 Development of early asphalt binder test methods

The use of asphalt binder in paving materials started in the 19th century, when first roads were paved with mixtures containing asphalt binder. Tough, at that time, the binder originated from natural asphalt deposits, such as Trinidad Lake. From the beginning of the 20th century asphalt binder from the crude oil refining process was introduced. Although there have been periods, where other adhesives, like tar and binder derived from shale, have also been used, it is still the crude oil based binder that is being predominantly used in the paving industry for flexible pavements.

As more and more asphalt pavements were constructed, a need for testing the properties of the adhesive arose. The first method of doing this was by chewing. Users of the binder tried to assess the consistency and the adhesive properties of the material with this technique (Rahim et al., 2019). Beside chewing, the oldest binder test is the needle penetration method. In 1888, H.C. Bowen constructed the Bowen Penetration Machine, which is the predecessor of the penetration apparatus used nowadays (Halstead & Welborn, 1974). Another method, that is still used widely, is the softening point determination according to the Ring and Ball method. This was already standardised in 1915 (Halstead & Welborn, 1974) and it is still being used in the current European specifications as the measure for high temperature performance. The low temperature performance is specified in Europe through the Fraass breaking point method, mentioned already in the 1950's (Rigden & Lee, 1953). There are other methods used in specifying asphalt binders in Europe, but these previously mentioned three are the most common. It is noteworthy, that two of the methods are already over 100 and one at least 70 years old. It is questionable if, with developments occurred in the refining industry, the methods are still suitable to assess the performance of binders.

The penetration test is not really a test, which results give insight to a binder's quality or performance. It is an empirical method of determining the consistency of an asphalt binder. In the current European specification, it is used to determine the grade of a binder. For example, the grade 70/100 means that the penetration of this material is within the range of 70 to 100 tenths of a millimetre (0.1 mm). The method is quite reproducible between laboratories, although the allowed deviations in the preparation processes are extensive.

The Ring and Ball softening point is of value, especially together with the change of softening point after short-term aging. Although the softening point does not imply the temperature, where the pavement will fail due to the binder being too soft, it is a good tool to rank different binders. Disregarding the effect of other constituents, a pavement with a higher softening point will experience high temperature defects, like rutting, at higher temperatures, compared to pavements containing a lower softening point binder and vice versa.

The Fraass test, on the other hand, has serious issues. First of all, the procedure states the use of unaged samples as the tested material. As it is a measure of low temperature performance, the use of unaged materials is questionable. It is widely known and many studies (Aliha & Shaker, 2020; Jing et al., 2020; Shaker et al., 2019; Siroma et al., 2022) confirm that cracking becomes an issue, when the binder in the pavement is aged. It is also known that binders age at different rates (Domke et al., 1999). Though the Fraass method is standardized, it has problems with reproducibility and is highly operator dependent, adding to the uncertainty (Baumanis et al., 2021; Besamusca et al., 2010;

Bueno et al., 2015; Gražulytė & Vaitkus, 2017). This is why many previous studies have called for the need to replace the Fraass breaking point, which is a failure test, with a rheology-based method (Lu et al., 2003, 2017; D. Petersen et al., 1997; Ravnikar Turk & Tušar, 2018). To our best knowledge there is no study confirming that there is a good correlation between pavement low temperature cracking and the Fraass breaking point.

1.2 Performance grading

The stakeholders in the Unites States of America came to an agreement that the previously used specifications, together with the testing methods, are not sufficient to specify the performance of asphalt binders. In the 1980's a research program was funded, which resulted in a new specification system called Superpave (Superior Performing Pavements). In terms of asphalt binders, the new Performance Grading (PG) system was introduced (Anderson et al., 1994). Among others, this included the high and intermediate temperature testing with the DSR. Here, using a parallel-plate geometry, the complex modulus and phase angle are measured with oscillatory loading of the sample.

Initially, two low temperature tests were also proposed, the Direct Tension Test (DTT) and the Bending Beam Rheometer (BBR) (Bahia & Anderson, 1995a) test. The former did not receive extensive recognition, but the BBR is still used for low temperature performance testing. In the DTT, binder samples are pulled until failure, which makes it slightly similar to the Fraass test with both being failure tests. The BBR on the other hand is a rheology-based test. Here, binder beams are loaded in a 3-point bending setting and the deflection is measured during the loading.

Another crucial enhancement with the PG system was the addition of the long-term aging of the binders. For both intermediate and low temperature testing, the samples are previously short-term aged with the RTFOT and long-term aged with the Pressure Aging Vessel (PAV). When the method was developed, it was said that it simulates the aging occurring during 8 years of service life (Bahia & Anderson, 1995b). This has been challenged on numerous occasions (Kaveh & Hesp, 2011; Smith et al., 2018, 2019), but nevertheless it is a good procedure to compare the aging rate of different binders.

In the PG system, binder grades are identified by their high- and low-temperature performance grades and the prefix "PG". For example, the grade PG 64-28 should have adequate performance within the pavement temperature of +64 °C to –28 °C. The grading system utilises 6-degree increments, for example high temperature grades next to PG 64 are PG 58 and PG 70. For low temperature grading the increments are the same. It should be noted that the PG grades are given with a confidence level of 95%, which means that there is only a 5% change of the pavement temperature breaching outside of each of the extents.

As with many new things, the implementation of the PG system did not go without problems. As asphalt binder producers had new specifications that they had to meet, many of the previously suitable binders were not on par within the new system. This meant that the producers had to modify their binders to meet the new requirements. Some of the modifications were able to meet the specifications during laboratory testing, but in practise had a very short service life (Rubab et al., 2011). This yielded that many pavements deteriorated prematurely.

1.3 Enhancement of the PG system

When the issues with undesirable modification became evident, researchers based in Kingston, Canada started to work on ways to alter the PG system to eliminate the chances of unsuitable modifications. As the issue was with the low temperature performance, work focused on this field. The research culminated with the development of the Extended Bending Beam Rheometer (EBBR) test, which was first published as a local laboratory standard LS-308 (Ontario Ministry of Transportation, 2007) and later as AASHTO (American Association of State Highway and Transportation Officials) TP 122-16 (AASHTO, 2016). This method is very similar to the regular BBR test, but in addition to testing after 1 hour of low temperature conditioning, samples are also tested after 24 and 72 hours of conditioning.

The prolonged conditioning gave the method the ability to consider reversible aging (Hesp et al., 2007). This phenomenon has been described in literature under many names, such as age hardening (Traxler & Schweyer, 1936), steric hardening (Brown et al., 1957), physical hardening (Blokker & Van Hoorn, 1959; Kriz, 2009), physical aging (Struik, 1978), reversible aging (Hesp et al., 2007) and others. Throughout this study, reversible aging will be used. As the name suggest, this aging can be reversed, and this can be done by reheating the material.

It is of high importance to take this into account, especially in cold climates, where adequate low temperature performance is crucial. Depending on the binder, final equilibrium can be achieved after different times of low temperature conditioning. It is known that binders with high wax content take longer to equilibrate. Coming from this, such binders will be rewarded in the regular BBR test, as the wax would not have set completely after 1 hour of conditioning, when the test is conducted. It is understandable that a shorter testing time is preferable, but in this situation erroneous conclusions could be made. This is why the EBBR test, with its 72-hour total conditioning, is advantageous as it allows for the binder to equilibrate. This is further supported by the fact that binders in service are also not subjected to low temperatures for short periods of time, but rather for days, weeks or even months during winter. Failing to address this critical phenomenon will eventually lead to low temperature cracking (Rigg et al., 2017).

It has been studied that the EBBR test has a good correlation with cracking on the road (Gražulite, 2019; Hesp et al., 2009; Jing et al., 2020; Y. Li & Hesp, 2022; Rigg et al., 2017). Coming from this, it seems that the test is the ideal choice as a low temperature performance specification. As previously mentioned, the advantage of the method is the use of the 72-hour low-temperature conditioning. On the other hand, this is also one of its weaknesses, because the test takes a long period to conduct. Additionally, the amount of binder needed for testing is considerable and it requires significant effort from laboratory technicians. Although the method is used in the province of Ontario in Canada for low temperature binder specifications, it has not spread to many other regions.

1.4 Using phase angle for characterizing asphalt binder

The phase angle can easily be measured with the DSR. While testing, the DSR measures the complex modulus, which can be considered as the total resistance to deformation of the measured sample. The complex modulus composes of two components. The storage modulus, which reflects the elastic component, and the loss modulus, that reflects the viscous component of a binder. Phase angle is the lag between the applied shear stress and the resulting shear strain. The relationships between complex modulus, storage modulus, loss modulus and phase angle are well described by Li and Hesp (2022). These relationships are presented graphically in Figure 1. The DSR was the obvious choice to develop new methods for characterizing asphalt binders, as it was already available in most laboratories due to it being used in the PG specifications high- and intermediate-temperature grading.

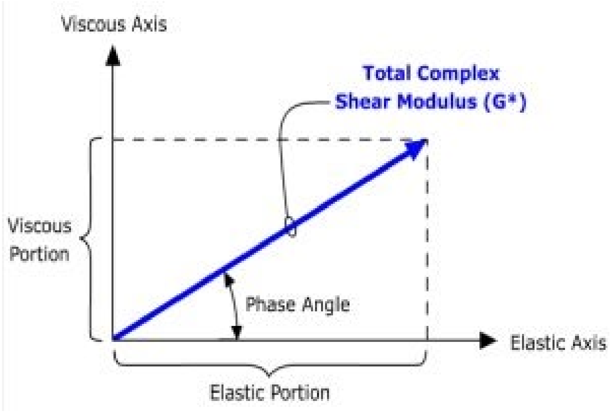

Figure 1. Relationship between complex modulus and phase angle (Abedali, 2017)

Asphalt binders are viscoelastic materials, meaning they have both viscous and elastic properties, they can be characterized by the proportions between these two. A phase angle of 45° is the point where a binder transitions from a more elastic binder to a more viscous binder and vice versa. A lower phase angle means the material is more elastic, with the phase angle of 0° meaning the material is entirely elastic. A higher phase angle means the material is more viscous, with the phase angle of 90° meaning a completely viscous material.

Earlier work in regard to using the phase angle to assess binders was done by Migliori et al. (1993, 1999). A few years later it was Widyatmoko et al. (2005), who followed in the same direction. All of them looked at the temperature, where the phase angle equalled 45°, also known as the Viscous to Elastic Transition (VET) temperature. Migliori et al. (1999) and Soleimani et al. (2009) also looked at the phase angle at a temperature of 0 °C. They concluded that the phase angle at that temperature is around 27° to 28° and that there is an agreement between these results and cracking in the field.

Later, researchers have continued the exploration of the phase angle as a potential binder performance characteristic. Among others, it has been studied, how the phase angle ranking of asphalt binders correlates with other low temperature methods (Angius et al., 2018; Y. Li et al., 2021). Additionally, the important aspect of precision of the phase angle measurements has been proven (Khan et al., 2020). It is also said that due to the nature of the measurement, it is very sensitive to aging, which makes it a great property to measure the aging of asphalt binders (Bird et al., 2020; Kriz et al., 2020; Soleimani et al., 2009).

In relation to the EBBR test, there are clear advantages in favour of the phase angle protocol. Mainly, because the DSR-based test can be run in a matter of hours, not days. Also, the geometry used in the DSR needs only small sample quantities, which is especially helpful when samples are extracted from cores or loose asphalt mixture. These initial findings are presented in (Lill et al., 2019), to be discussed in the context of Publication I.

1.5 Asphalt binder aging

1.5.1 Aging of asphalt binder

It is common knowledge that organic asphalt binders age. This aging causes many different issues for asphalt pavements. This phenomenon has been a research topic for many decades or even longer (Bell, 1989; Richardson, 1905). There is almost an endless amount of literature about asphalt binder aging. Many researchers have compiled comprehensive review papers about asphalt binder aging (Ahmad et al., 2024; Hamzah et al., 2015; Wang et al., 2020; M. Zhang et al., 2020). Others have done more specific studies about asphalt aging (Gamarra & Ossa, 2018; Kleizienė et al., 2019; Liu et al., 1998; Z. Li et al., 2020; Ma et al., 2021; Sreedhar & Coleri, 2022). Numerous papers have been published about how to revert the aging of asphalt binders (Kuang et al., 2019; Mohammadafzali et al., 2017; R. Zhang et al., 2019) and others have investigated the possibility of using anti-aging additives (Camargo et al., 2021; Fu et al., 2024; Gawel et al., 2016; Malinowski et al., 2024).

Aging occurring during the production, transportation and laying of asphalt mixture is known as short-term aging. During this stage the main contributor to aging is the loss of volatile components (Fernandez-Gomez et al., 2013). In addition to short-term aging long-term aging occurs during service life in a pavement. Here the aging can be attributed to oxidation, when the binder reacts with oxygen (Liang et al., 2019). Of course, there is also some loss of volatiles during this aging stage and the effect of ultra-violet radiation (Hunter et al., 2015). In both of the aging stages, the binder becomes stiffer and the viscosity is increased. To some extent, this phenomenon is preferable, as a pavement containing stiffer binder is more resistant to permanent deformations. Though, at some point, the asphalt binder will be aged too extensively, making it brittle. When this stage of binder aging has occurred, the pavement will be more susceptible to both low temperature and fatigue cracking.

Long-term aging is governed by the climatic conditions in which the pavement is located, while short-term aging is affected mostly by the mixing conditions in the asphalt mixture production plant. There are many studies about the effect of mixing temperature on the performance of asphalt binders (Corbett, 1969; Hofko et al., 2017; Liang et al., 2019). Most of these have utilized the RTFOT to simulate the short-term aging in laboratory scale. As already previously mentioned, the PAV is the go-to tool for simulating the long-term aging in the laboratory.

1.5.2 Measurement of aging

From their composition, asphalt binders consist of two main components: asphaltenes and maltenes. The maltenes can additionally be divided into saturates, aromatics and resins (Tauste et al., 2018). Together, these four are called the SARA fractions. A potential method for measuring the aging of asphalt binders is through this chemical composition. The short-term aging affects the binder so, that the fraction of saturates is decreased with the loss of volatiles, while during long-term aging they remain unchanged (Liang et al., 2019). As the binder ages, aromatics are converted into resins, and resins into asphaltenes. Due to this, the asphaltene fraction has the most significant increase (Liang et al., 2019). This method for measuring the aging of asphalt binders was developed already during the 1960’s (Corbett, 1969).

One of the most frequently used methods to measure the aging of asphalt binders, is the investigation of the carbonyl area index via the Fourier-transform infrared spectroscopy (FTIR). This has been used in many studies, but research conducted in Louisiana, USA, found good correlation between the carbonyl area index and both transverse and alligator cracking (Islam et al., 2024).

Similarly to the low-temperature testing, also for aging, the DSR was the go-to tool, as it was already widely available in laboratories. With the increase in stiffness, the binders become more prone to fatigue cracking. This is why the DSR was often used for measuring fatigue properties of binders. Mostly time sweep tests were used, where the studied sample is loaded cyclically for long periods of time until a drop in complex modulus is observed (Hintz & Bahia, 2013). This method, again, having the issue of taking a long time to complete.

To find a quicker method, noteworthy work has been done at the Technische Universität Braunschweig, where Kim et al. (2021) developed the Accelerated Dynamic Shear Rheometer Fatigue Test (ADFT). The same university also worked on the Binder-Fast-Characterization-Test, abbreviated as BTSV, which derives from the German language title “Bitumen-Typisierung-Schnell-Verfahren” (Schrader & Wistuba, 2019). This method has been used to monitor the aging of asphalt binder at high temperatures. To date, this method has been standardized (CEN, 2022).

University of Nottingham is another university to deeply study the effect of aging on asphalt binder. They concluded, that it is important to monitor the aging across the whole temperature spectrum, to which the binder can be subjected to across its service-life (Hu et al., 2023). In regard to fatigue cracking, they refer to the Glover-Rowe parameter (Rowe et al., 2014), which is calculated from DSR frequency sweep data. It is proposed that a limit of 180 kPa is the onset of fatigue failure and 450 kPa as the propagation point of significant failure (Rowe et al., 2014).

1.5.3 Effect of aggregate type on binder aging

Parts of North-Eastern Europe, such as the Baltic states, are situated in an area where there is no native igneous rock available. In Estonia, Latvia and Lithuania the mainly available aggregate types are limestone, gravel and sand. As this geographic location can have harsh conditions during winter, de-icing salt, such as sodium chloride, is used on pavements. Additionally, studded tyres are seasonally allowed on vehicles. The local limestone and also gravel have a relatively low resistance against these impacts. This means that at least the top layer asphalt of a pavement has to be mixed with igneous rock. Local rock is primarily used in binder and base course mixtures. The use of these different rock types has sparked the discussion, whether there is a difference in asphalt binder aging between them.

Literature suggests that binders in mixtures with limestone aggregate age slower compared to igneous rock. Anderson et al. (1994) stated that aggregate with a lower adsorption of highly polar fractions, like granite, shows higher catalytic effect compared to aggregate with higher adsorption, such as limestone. This was later supported by Wu et al. (2014), who concluded that binders, extracted from mixtures containing granite as aggregate, showed higher stiffness when comparing to binders extracted from mixtures with limestone aggregate. Moreover, Petersen et al. (1974) discovered the adsorption of certain polar components onto limestone might be irreversible. As these remain on the aggregate after extraction, the recovered binder will show lower aging indices. Adding to this, Wu et al. (2014) pointed out the possibility of oily fractions being absorbed into the pores of the aggregate and thereby be protected from oxidation.

1.6 Aims of the study

The main goal of this study is to suggest a more optimum test method for the European binder specification to measure the low temperature performance and aging of asphalt binders. For this different test protocols, not common for Europe, were investigated. This aim was chosen to decrease the low temperature and fatigue induced cracking observed on asphalt pavements.

- The first part of the study is dedicated to evaluating new low temperature testing methods, and choosing the most promising protocols for further investigation.
- The methods chosen in the first part of the study are correlated with the Fraass breaking point method, which is the only low temperature specification in the current European binder grading system. This was done to understand if the Fraass method is as unreliable as stated in earlier studies.
- Another aim was to study the feasibility of using limiting phase angle temperatures, measured by the DSR, to specify binder low temperature performance. A correlation with EBBR should be proposed while looking at a broad spectrum of binders from around the world, but the correlation should also be checked for the North-Eastern European binder market.
- The last aim was to investigate, whether the phase angle based parameter can be used for measuring the aging processes of asphalt binders.

2 Experimental

This chapter presents the asphalt binder samples that were tested throughout the process of this study. Additionally, all the methods used in conditioning and testing of the samples are included.

2.1 Materials

A total of 89 samples were studied during the process of this research. 74 samples are included in the publications forming the backbone of this study, but additional 15 binders are included from a related research (Lill et al., 2019). Publication I and Lill et al. (2019) focus on a broader spectrum of samples from around the world, with Lill et al. (2019) focusing on both Northern European and Canadian binders. In addition to Northern European and Canadian binders, Publication I had also samples from Asia, the Middle East and the southern part of Europe. These two publications included 51 of the 89 samples. These samples were used to study enhanced asphalt binder testing methods. This examination determined potential testing methods that could improve the current European binder specifications presented in EN 12591 (CEN, 2009) and EN 14023 (CEN, 2010). The samples tested for Lill et al. (2019) and Publication I are presented in Table 1 and Table 2, respectively. These samples were obtained over a period of around four years from 2016 to 2019.

The research in Publications II and III focused on the Northern Part of Europe, so only binder samples from this region were included. In this part the focus was on this narrow geographical area, as the aim of study is to propose new and improved asphalt binder testing methods for northern European conditions. In this part of the work, 38 binders presented in Table 3 were tested. These were sampled during the years 2020 and 2021, but it should be noted that sample no. 59 was sampled from a storage unit and the binder was produced prior to 2020.

The samples included in Lill et al. (2019) and Publications I and II were all tank samples obtained either straight from refineries or from asphalt mixture or bitumen emulsion plants. Publication III included, in addition to tank samples, also loose asphalt mixture samples and drill cores from which the binders were extracted and recovered. The mixture and core samples were included for the purpose of studying the aging tendencies of the binders.

All of the included binders cover a wide range of grades from both penetration and Superpave grading systems.

Table 1. Samples included in Lill et al. (2019)

Sample No.	Sample No./code in publication	Commercial grade	Origin	Source
1	A	PG 52-34	Canada	Tank
2	B	PG 58-28	Canada	Tank
3	C	PG 64-22	Canada	Tank
4	D	PG 58-28	Canada	Tank
5	E	PG 58-28	Canada	Tank
6	F	PG 58-28	Canada	Tank
7	G	70/100	Lithuania	Tank
8	H	70/100	Russia	Tank
9	I	70/100	Belarus	Tank
10	J	70/100	Poland	Tank
11	K	160/220	Poland	Tank
12	L	70/100	Poland	Tank
13	M	160/220	Poland	Tank
14	N	70/100	Sweden	Tank
15	O	160/220	Sweden	Tank

Table 2. Samples included in Publication I

Sample No.	Publication	Sample No./code in publication	Commercial grade	Origin	Source
16	I	1	n.a.	Canada	Tank
17	I	2	n.a.	Canada	Tank
18	I	3	n.a.	Canada	Tank
19	I	4	n.a	Canada	Tank
20	I	5	n.a.	Canada	Tank
21	I	6	300/400	Canada	Tank
22	I	7	PG 58−28	Canada	Tank
23	I	8	PG 64−22	Canada	Tank
24	I	9	50/70	Kuwait	Tank
25	I	10	160/220	Kuwait	Tank
26	I	11	250/330	Kuwait	Tank
27	I	12	PG 64−22	China	Tank
28	I	13	PG 76−28	China	Tank
29	I	14	PG 64−22	China	Tank
30	I	15	PG 64−22	China	Tank

Table 2. Samples included in Publication I (cont.)

Sample No.	Publication	Sample No./code in publication	Commercial grade	Origin	Source
31	I	16	PG 64−22	South Korea	Tank
32	I	17	50/70	India	Tank
33	I	18	70/100	Poland	Tank
34	I	19	160/220	Poland	Tank
35	I	20	160/220	Poland	Tank
36	I	21	160/220	Belarus	Tank
37	I	22	70/100	Poland	Tank
38	I	23	70/100	Belarus	Tank
39	I	24	70/100	Sweden	Tank
40	I	25	70/100	Spain	Tank
41	I	26	50/70	Spain	Tank
42	I	27	n.a.	Canada	Tank
43	I	28	n.a.	Canada	Tank
44	I	29	n.a.	Canada	Tank
45	I	30	n.a.	Canada	Tank
46	I	31	n.a.	Canada	Tank
47	I	32	n.a.	Canada	Tank
48	I	33	n.a.	Canada	Tank
49	I	34	n.a.	Canada	Tank
50	I	35	n.a.	Canada	Tank
51	I	36	n.a.	Canada	Tank
n.a. – not commercially available					

Table 3. Samples included in Publications II and III

Sample No.	Publication	Sample No./code in publication	Commercial grade	Origin	Source
52	II, III	1 (II) & 1 (III)	100/150	Lithuania	Tank
53	II, III	2 (II) & 3 (III)	70/100	Lithuania	Tank
54	II, III	3 (II) & 6 (III)	160/220	Lithuania	Tank
55	II, III	4 (II) & 9 (III)	70/100	Belarus	Tank
56	II, III	5 (II) & 11 (III)	100/150	Belarus	Tank
57	II, III	6 (II) & 14 (III)	100/150	Belarus	Tank

Table 3. Samples included in Publications II and III (cont.)

Sample No.	Publication	Sample No./code in publication	Commercial grade	Origin	Source
58	II, III	7 (II) & 17 (III)	100/150	Lithuania	Tank
59	II	8	160/220	Sweden	Tank
60	II, III	9 (II) & 19 (III)	100/150	Sweden	Tank
61	II, III	10 (II) & 21 (III)	100/150	Sweden	Tank
62	II	11	100/150	Lithuania	Tank
63	II, III	12 (II) & 23 (III)	100/150	Sweden	Tank
64	II, III	13 (II) & 27 (III)	160/220	Belarus	Tank
65	III	2	100/150	Lithuania	Extracted from mixture
66	III	4	70/100	Lithuania	Extracted from mixture
67	III	5	70/100	Lithuania	Extracted from core sample
68	III	7	160/220	Lithuania	Extracted from mixture
69	III	8	160/220	Lithuania	Extracted from core sample
70	III	10	70/100	Belarus	Extracted from mixture
71	III	12	100/150	Belarus	Extracted from mixture
72	III	13	100/150	Belarus	Extracted from core sample
73	III	15	100/150	Belarus	Extracted from mixture
74	III	16	100/150	Belarus	Extracted from core sample
75	III	18	100/150	Lithuania	Extracted from mixture

Table 3. Samples included in Publications II and III (cont.)

Sample No.	Publication	Sample No./code in publication	Commercial grade	Origin	Source
76	III	20	100/150	Sweden	Extracted from mixture
77	III	22	100/150	Sweden	Extracted from mixture
78	III	24	100/150	Sweden	Extracted from mixture
79	III	26	100/150	Sweden	Extracted from mixture
80	III	28	160/220	Belarus	Extracted from mixture
81	III	29	100/150	unk	Extracted from core sample
82	III	30	70/100	unk	Extracted from core sample
83	III	31	70/100	unk	Extracted from core sample
84	III	32	160/220	unk	Extracted from core sample
85	III	33	70/100	unk	Extracted from core sample
86	III	34	70/100	unk	Extracted from core sample
87	III	35	160/220	unk	Extracted from core sample
88	III	36	160/220	unk	Extracted from core sample
89	III	37	70/100	unk	Extracted from core sample
unk – unknown					

2.2 Methods

The selection of methods includes regular protocols from European specifications (CEN, 2009, 2010) that were mostly used for reference purposes. The other tests were included as they were deemed to have the potential to be proposed as new methods for specifying asphalt binder in Europe.

Depending on the sample, most of the testing was done at Tallinn University of Technology's Laboratory of Roads and Traffic and at Queen's University in Kingston, Canada. Although the main principles are the same, there might be slight differences between methods applied, because in Tallinn University of Technology the European standards from CEN were used and Queen's used the respective AASHTO or ASTM methods.

2.2.1 Laboratory aging procedures

Depending on the test, binders in different conditioning states were needed. Two main laboratory aging methods were used. These were the RTFOT and PAV. The RTFOT simulates the binder aging occurring during the mixing, transportation and paving of asphalt mixtures. This is called short-term aging. In this method, 35 g of binder is poured into a glass tube, the tube is fixed horizontally into a rotating shelf inside an RTFOT oven (Figure 2) that has been heated to 163 °C. For 85 minutes the tubes rotate and during each rotation hot air with a flow of 4 l/min is blown into the cylinder, causing oxidation. This method was conducted according to EN 12607-1 (CEN, 2014) or AASHTO T240 (AASHTO, 2010c).

If needed, the long-term aging procedure with the PAV (Figure 3) was applied on the previously short-term aged sample. In the PAV, 50 g of binder is poured onto a pan that is placed into a vessel, where the sample is conditioned at 100 °C and 2.1 MPa for 20 hours. The PAV method was conducted according to EN 14769 (CEN, 2012) or AASHTO R28 (AASHTO, 2010b).

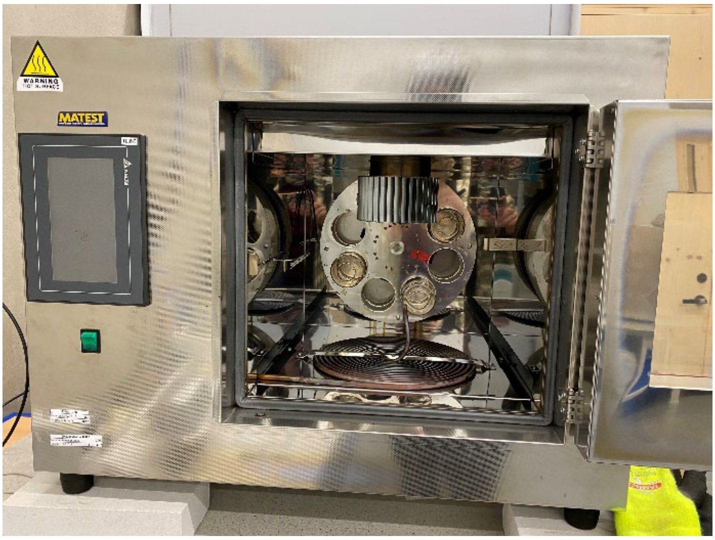

Figure 2. Rolling thin film oven

Figure 3. Pressure aging vessel

2.2.2 Binder extraction and recovery

To obtain binder samples from loose mixture or asphalt core samples, the asphalt samples were loosened for the extraction process. A semi-automatic extraction apparatus (Figure 4) with dichloromethane (DCM) as the solvent was used. The binder, together with the solvent, was separated from the aggregate by the means of EN 12697-1 (CEN, 2020). The solution was introduced into a rotary evaporator (Figure 5) and the DCM was removed with the effect of heating and vacuum. This was done according to EN 12697-3 (CEN, 2018).

Figure 4. Semi-automatic extraction apparatus

Figure 5. Rotary evaporator

2.2.3 Dynamic Shear Rheometer testing

2.2.3.1 Performance grading high temperature

As the DSR (Figure 6) is a very versatile apparatus, it was used extensively in the course of this research. One of the uses was the determination of the high temperature grade in the Performance Grading system. Where applicable, the temperature where the complex modulus divided by the sine of the phase angle (G*/sinδ) equalled 1 kPa or 2.2 kPa was determined. The first for unaged materials and the latter for short-term aged materials, obtained either after the RTFOT or after extraction and recovery. This determination was done according to AASHTO M320 (AASHTO, 2010a). Depending on the use, either the actual PG high temperature grade or the exact true grade was presented.

Figure 6. Dynamic shear rheometer at Tallinn University of Applied Sciences

2.2.3.2 Limiting phase angle temperature measurement

The DSR was additionally used for measuring the low temperature performance of asphalt binders. Mostly using PAV aged binders, but also on samples extracted and recovered from asphalt cores, temperatures were determined, where the phase angle equalled 30°. For this, the binder samples were tested in oscillatory mode with an angular velocity of 10 rad/s at different temperatures and interpolation was used to obtain the temperatures. Extrapolation was used on a limited number of samples, and it was allowed only when the determined temperature was up to 1 °C outside of the datapoints. A parallel plate geometry, illustrated in Figure 7, was used with a 2 mm gap and depending on the measured temperature either plates with a diameter of 8 mm or 4 mm were used.

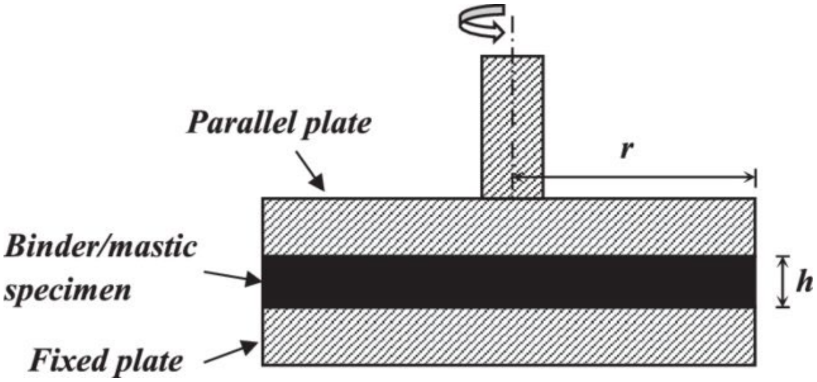

Figure 7. DSR parallel plate geometry (S. Li et al., 2021)

2.2.3.3 Other Dynamic Shear Rheometer tests

In Publication I and Lill et al. (2019), the DSR was also used to determine the PG intermediate grade where the complex modulus multiplied with the sine of the phase angle (G*sinδ) equalled 5.0 MPa.

2.2.4 Bending beam rheometer testing

In the bending beam rheometer test asphalt binder beams are loaded at low temperatures in a 3-point configuration for 4 minutes and the deflection is continuously recorded. This is illustrated in Figure 8. The BBR was tested according to the AASHTO M320 method (AASHTO, 2010a), but additionally the extended version according to AASHTO TP 122-16 (AASHTO, 2016) was used. The main difference between the two is, the first method tests samples only after 1 hour of low temperature conditioning, the latter repeats the testing also after 24 and 72 hours. For this research only the 72-hour tests were done in addition to the regular 1 hour. At each of the conditioning times the temperatures, where the stiffness equalled 300 MPa and m-value equalled 0.300 at a loading time of 60 seconds, were determined. The highest of the two temperatures determines the low temperature grade of the binder. The stiffness is calculated with Equation (1). M-value is the slope of the curve when plotting stiffness versus time as illustrated in Figure 9. The BBR apparatus used in Tallinn University of Technology is shown in Figure 10.

$$S(t) = \frac{PL^3}{4bh^3\Delta(t)} \tag{1}$$

Where
S(t) – creep stiffness at time, in MPa
P – applied constant load, 100 g
L – distance between supports, 102 mm
b – beam width, 12.5 mm
h – beam thickness, 6.25 mm
Δ - deflection at time, in mm

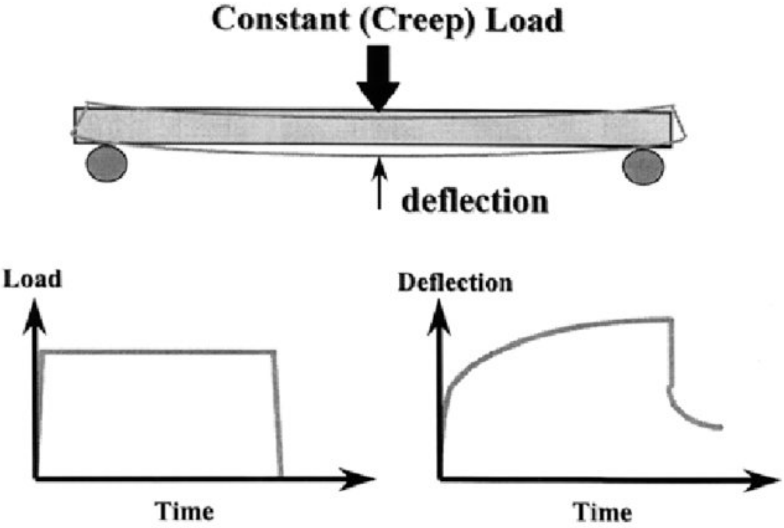

Figure 8. Bending Beam Rheometer test (Rowe et al., 2001)

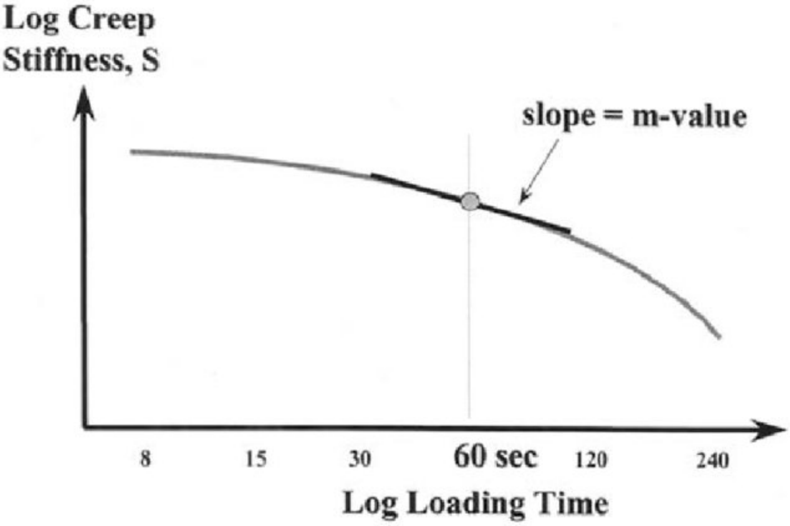

Figure 9. Determination of m-value (Rowe et al., 2001)

Figure 10. BBR apparatus

2.2.5 Needle penetration

The needle penetration is a basic test to characterize asphalt binders. It is a measure of consistency. In the test, the binder sample is conditioned at 25 °C in a water bath and a specific needle with a weight of 100 g is penetrated into the sample for 5 seconds. The penetration depth in tenths of millimetre is considered the needle penetration. This test was done according to the standard EN 1426 (CEN, 2015a). The needle penetration apparatus used for this study is presented in Figure 11.

2.2.6 Softening point

The softening point is used to measure the high temperature performance of asphalt binders in the European specifications. The method is called the Ring and Ball method. In this method, two rings are filled with the sample. The filled rings are conditioned in a beaker filled with 5 °C water. Balls, which are centred with guides, are placed on the filled rings. Starting from the conditioning temperature, the beaker is heated so that the temperature of the water rises uniformly at a rate of 5±1 °C per minute. The average temperature, where the two samples deflect 25 mm due to the weight of the balls, is considered the softening point. Measurement of the softening point is a standardized method covered in EN 1427 (CEN, 2015b). The apparatus used for this study is presented in Figure 12.

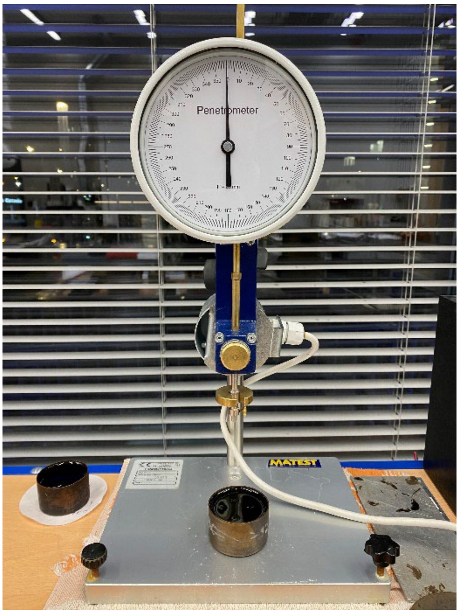

Figure 11. Needle penetration apparatus

Figure 12. Ring and ball apparatus

2.2.7 Fraass breaking point

The Fraass breaking point is used to measure the low temperature performance of asphalt binders in the European specifications. In this method, small metal plates are covered with a thin binder layer. One at the time, the plates are placed into a fixture that can bend the plate. The fixture, together with the plate, is placed in a chamber, which can reduce the temperature at a rate of 1 °C per minute. After each degree the plate is bent and inspected if a crack occurs in the binder film. The average temperature, where the crack occurs, is considered the Fraass breaking point. The test was done according to the standard EN 12593 (CEN, 2015c). Most of the testing was done with an Anton Paar BPA-5 instrument, but some were conducted with a semi-automatic appliance (Figure 13), where the bending is controlled with electronics, but the temperature is lowered manually with the help of dry ice. Although the testing is more complex with the semi-automatic apparatus, confidence in the results was achieved as the Fraass method was accredited at the laboratory of Road Technology Centre in Estonia, where the tests were conducted.

Figure 13. Semi-automatic Fraass apparatus used in this study [Road Technology Centre]

2.2.8 Sum of pavement defects

The Transport Administration of Estonia organises annual inspection of national roads. Among others, the defects on the road, for example the number of transverse cracks, length of longitudinal and joint cracks, area of alligator cracking, the number of potholes and the area of ravelling, are recorded. The guidelines for this inventory method have been published by the Estonian Transport Administration (2021). Using this data, the sum of pavement defects is calculated for sections of 100 meters. The data is held in a publicly accessible database, from where the needed datapoints were extracted. This was used to calculate the average sum of defects per one kilometre for the road sections presented in Publication III.

3 Results and discussion

3.1 Study of different binder testing methods

3.1.1 Superpave grading

Publication I and Lill et al. (2019) focused on studying different binder testing methods that are not commonly used in Europe. This was deliberately done on a wide range of binders sourced from around the world. These samples can be seen in Table 1 and in Table 2.

Throughout the research, to understand the basic properties of the binder, all tank samples were tested for their Superpave grading. The results are presented in Table 4.

As can be seen from the data, the used set of binders is very diverse as the PG high-temperature grades range from a low of +44 °C up to +79 °C. Similarly, the intermediate temperature grades span from +4 °C to +26 °C and low-temperature grades from –24 °C to –41 °C. These results, with the fact that the samples have been obtained from different locations around the world, make the set representative for making widespread conclusions.

Table 4. Superpave grading of Lill et al. (2019) and publication I samples

Sample No.	Commercial grade	XX, °C	II, °C	YY, °C
1	PG 52−34	55	10	-36
2	PG 58−28	60	17	-31
3	PG 64−22	66	23	-28
4	PG 58−28	59	17	-30
5	PG 58−28	60	20	-29
6	PG 58−28	61	19	-30
7	70/100	63	18	-28
8	70/100	62	18	-30
9	70/100	69	13	-31
10	70/100	65	24	-27
11	160/220	51	17	-29
12	70/100	64	19	-28
13	160/220	53	15	-29
14	70/100	63	18	-27
15	160/220	56	13	-32
16	n.a.	53	15	-30
17	n.a.	55	20	-25
18	n.a.	54	15	-32
19	n.a	49	7	-35
20	n.a.	63	20	-26
21	300/400	48	10	-37
22	PG 58−28	60	17	-31

Table 4. Superpave grading of Lill et al. (2019) and publication I samples (cont.)

Sample No.	Commercial grade	XX, °C	II, °C	YY, °C
23	PG 64−22	66	23	-28
24	50/70	67	26	-24
25	160/220	56	15	-27
26	250/330	52	11	-30
27	PG 64−22	66	24	-26
28	PG 76−28	79	20	-29
29	PG 64−22	66	24	-26
30	PG 64−22	64	23	-25
31	PG 64−22	68	23	-25
32	50/70	60	20	-27
33	70/100	65	24	-27
34	160/220	52	17	-29
35	160/220	54	15	-29
36	160/220	53	11	-31
37	70/100	60	18	-28
38	70/100	63	14	-31
39	70/100	65	20	-26
40	70/100	63	21	-27
41	50/70	66	21	-26
42	n.a.	44	4	-41
43	n.a.	51	7	-36
44	n.a.	55	13	-33
45	n.a.	59	15	-32
46	n.a.	62	16	-30
47	n.a.	64	19	-29
48	n.a.	57	13	-31
49	n.a.	58	14	-30
50	n.a.	60	17	-29
51	n.a.	65	21	-27

The range between the PG high temperature grade and low temperature grade is considered the grade span. This is the span of temperatures, where a binder is expected to perform adequately in a pavement and no serious defects are to be expected. It is obvious that a wider grade span is preferred and thus, in terms of cost, usually binders with a wider grade span are more expensive.

The achieved grade spans for the studied binders are presented in Figure 14. The results range from a low of 80 °C to a high of 108 °C with the average being 89.2 °C. The largest grade span was achieved by sample no. 28, which is a polymer modified binder. Another out of the ordinary result was for sample 9, with a result of 100 °C. This sample is believed to be an air-blown binder. A clear tendency can be seen that softer binders have lower grade spans. Going through the results, it can be seen that softer binders have notably lower high temperature grades, but only slightly better performance at low temperatures, resulting in a narrower grade span.

When focusing only on the penetration grades 50/70, 70/100 and 160/220 then the following can be concluded. The two harder grades have similar grade spans with an average of 90 °C for 50/70 and 92 °C for 70/100, their respective standard deviations being 2.6 °C and 3.1 °C. The average for 160/220 is much lower with 83 °C and a standard deviation of 2.6 °C.

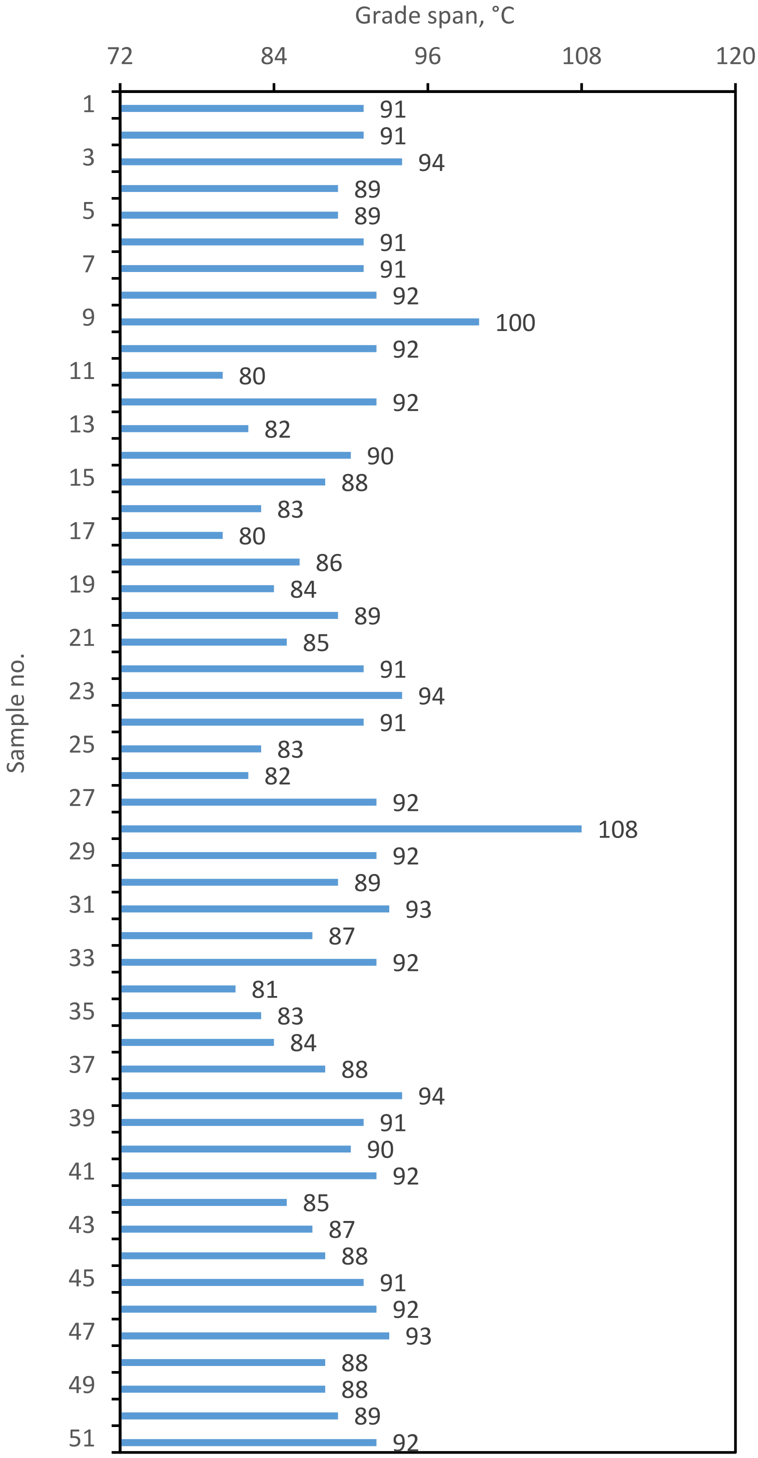

Figure 14. PG grade span (PG HT vs PG LT) (Combination of Lill et al. (2019) and Publication I)

3.1.2 Extended bending beam rheometer testing

All of the samples, included in the research in publication I, were additionally tested for their low temperature performance according to the EBBR protocol (AASHTO, 2016). The same data was available for the samples in Lill et al. (2019). The EBBR test was done to account for the phenomenon of reversible aging. It is known from previous research (Hesp et al., 2009; Rigg et al., 2017) that some binders suffer from a drop in low temperature performance, when subjected to elongated low temperature conditioning.

The low temperature grades according to the EBBR protocol, together with grade losses, are presented in Table 5. The grade loss is the measure of reversible aging. Looking at the results, it is evident that many of the studied samples suffer from this phenomenon. Samples with the worst resistance to reversible aging exhibit grade losses over 6 degrees. This is a huge loss in performance, as it is even higher than a PG grading increment of six degrees. As previously stated, PG grades are assigned with a confidence level of 95% and a drop of more than six degrees can easily reduce this level to around 50%. From the studied 51 binders, a total of 22 lost their initial PG low temperature grade after additional low temperature conditioning.

Although even the smallest grade losses are of concern, Canadian based researchers have proposed a 3.0 °C limit, which still lowers the confidence level noticeably, but removes the worst performers. Depending on the climatic conditions, this much bias can drop the confidence that no low temperature damage occurs to around 70%. The average grade loss for the studied samples is 3.1 °C, meaning that on average less than half of the binders studied would be considered adequate in Canada.

Taking another look at the results, some more conclusions can be made. It seems that the worst performing asphalt binders are from the North-eastern part of the European market. Samples obtained from refineries from Belarus, Lithuania, Poland and Russia (the origin can be seen in Table 1 and Table 2) have all results higher than 3.0 °C and most of them close to or above 6.0 °C.

Exceptions to this observation are the Swedish samples. The least amount of reversible aging can be seen on these samples, showing grade losses around or even below 1.0 °C. This means that these binders almost don’t suffer from additional hardening from elongated periods at low temperatures.

It is similar for many of the Canadian binders, having grade losses around 1.0 °C, though there are some samples having inferior performance with grade losses above 3.0 °C, up to a maximum of 3.8 °C.

The rest of the samples, obtained from Asia, the Middle East and southern part of Europe, fall in between the best and worst performing sources. One noteworthy finding is that from the three Kuwaiti binders, two had also almost non-existent grade losses, but at the same time one binder had a significant grade loss of 5.0 °C. This suggests that these binders have been produced using different crudes or in different ways.

The magnitude of reversible aging seems to be in correlation with the source of the crude oil used to manufacture the asphalt binder. It is known that the samples from the Swedish refinery are from the period, when the Venezuelan crude, which is a naphthenic heavy crude oil, was used. The worst performing binders are from Russian crude. Most likely, the dominant factor causing this is wax content. It is known that Venezuelan crude is low in waxes and the Russian crude, on the other hand, very high in waxes (Hesp et al., 2007).

Table 5. EBBR results

Sample No.	YY_{EBBR}, °C	Grade loss, °C	Sample No.	YY_{EBBR}, °C	Grade loss, °C
1	-35,2	0,9	27	-21,9	4,2
2	-28,4	2,2	28	-24,5	4,1
3	-24,3	3,2	29	-22,0	4,3
4	-26,2	3,5	30	-22,2	2,5
5	-26,7	2,1	31	-19,5	5,9
6	-26,6	3,7	32	-21,3	5,2
7	-21,3	6,6	33	-20,4	6,2
8	-22,9	6,9	34	-25,7	3,3
9	-24,4	6,9	35	-25,1	3,8
10	-20,4	6,2	36	-27,2	3,7
11	-25,7	3,3	37	-21,6	6,7
12	-23,1	5,0	38	-25,8	4,8
13	-23,3	5,2	39	-24,9	1,3
14	-26,9	0,3	40	-22,8	4,4
15	-31,3	0,3	41	-22,1	3,8
16	-28,7	1,6	42	-39,2	1,8
17	-24,6	0,8	43	-34,3	1,4
18	-29,7	1,8	44	-32,3	0,6
19	-34,3	1,1	45	-30,1	1,5
20	-23,9	2,3	46	-28,5	1,9
21	-33,3	3,8	47	-26,6	2,0
22	-28,4	2,2	48	-29,0	1,7
23	-24,3	3,2	49	-28,6	1,4
24	-19,4	5,0	50	-27,5	1,3
25	-26,1	0,6	51	-25,2	1,7
26	-29,2	1,0	-	-	-

3.1.3 Limiting phase angle grading

While the EBBR has proven itself to be a very effective test to measure the low temperature performance on binders, as already stated before, it is very cumbersome and takes four days to conduct without accounting for sample preparation. This is why a quicker method is sought after and the limiting phase angle temperature is studied. The temperature, where the phase angle equals 30°, T(30°), is proposed as a feasible surrogate for the EBBR. As most binders are m-value controlled in the EBBR test, and as the m-value from the more cumbersome test, and the phase angle from the DSR-based method, are closely linked, it is expected that a noticeable correlation exists between the two (Khan et al., 2020; Soleimani et al., 2009).

Previous research suggested that there is a clear correlation between the PG low temperature grade and the T(30°) parameter ($R^2 = 0.71$), but an even better correlation exists between the EBBR-based low temperature grade and T(30°) ($R^2 = 0.88$) (Angius et al., 2018; Ding et al., 2018).

First efforts by the author, in this regard, were done for Lill et al. (2019). To set the scene for the results of Publication I, the correlations found in Lill et al. (2019) are presented and discussed. These correlations, together with the correlations found in previous work from other authors (Angius et al., 2018; Ding et al., 2018) are presented in Figure 15 and Figure 16. Open symbols mark the data points from Angius et al. (2018) and Ding et al. (2018) and the solid symbols the data from Lill et al. (2019). As can be seen from the results on the figures, the newly found correlations were nowhere near those from past research. The coefficient of determination between BBR and T(30°) data added in Lill et al. (2019) was found to be only $R^2 = 0.22$ and between EBBR and T(30°) it was once again better, but still quite low, with $R^2 = 0.33$. The coefficients of determination for data from previous research (Angius et al., 2018; Ding et al., 2018) were $R^2 = 0.71$ and $R^2 = 0.88$, respectively for BBR and EBBR. The reasons for the lower correlations in the newer research are not exactly known, but they definitely are affected by the smaller amount of data points, but there could also be some erroneous results from both the EBBR/BBR or the DSR.

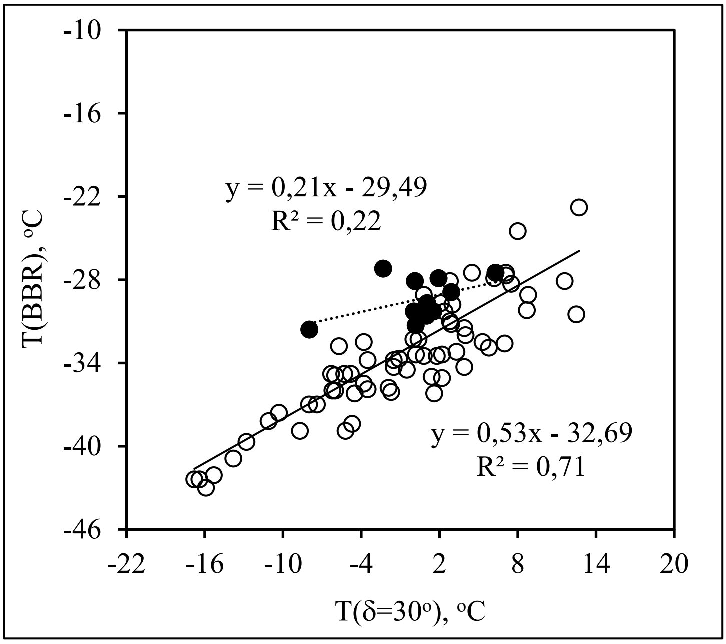

Figure 15. Comparison between BBR grade and limiting phase angle temperature (Lill et al., 2019)

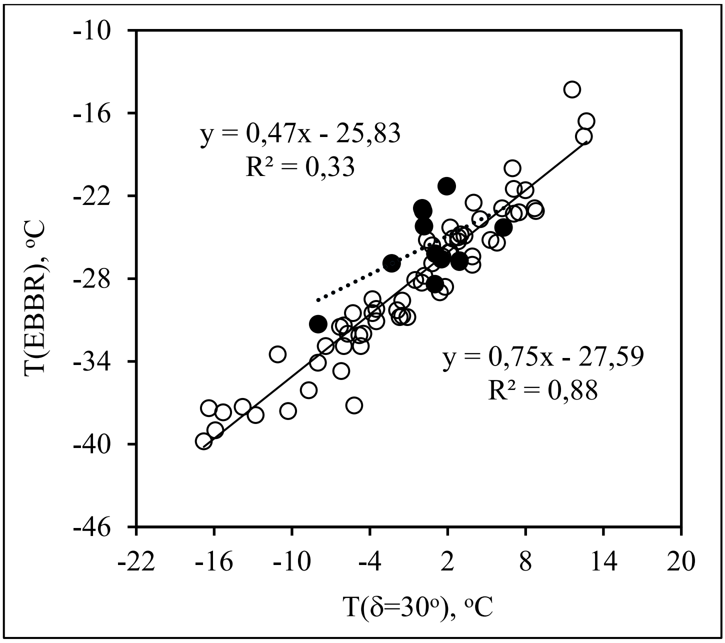

Figure 16. Comparison between EBBR grade and limiting phase angle temperature (Lill et al., 2019)

Due to the fact that the results from Lill et al. (2019) were inconclusive, another effort was made to study these correlations. For Publication I, another 36 binders were tested for this purpose. The correlation between BBR and T(30°) is presented in Figure 17 and between EBBR and T(30°) in Figure 18.

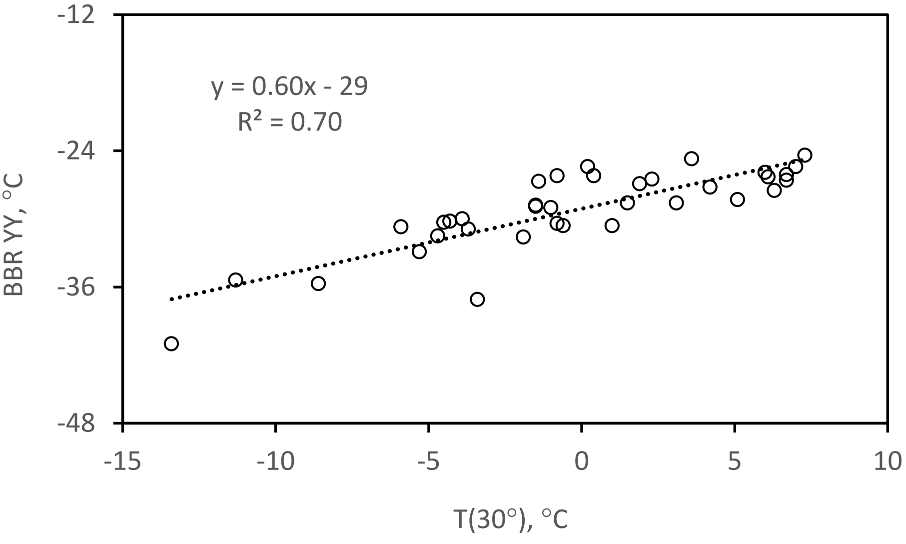

Figure 17. BBR low temperature grade YY and T(30°) correlation (Publication I)

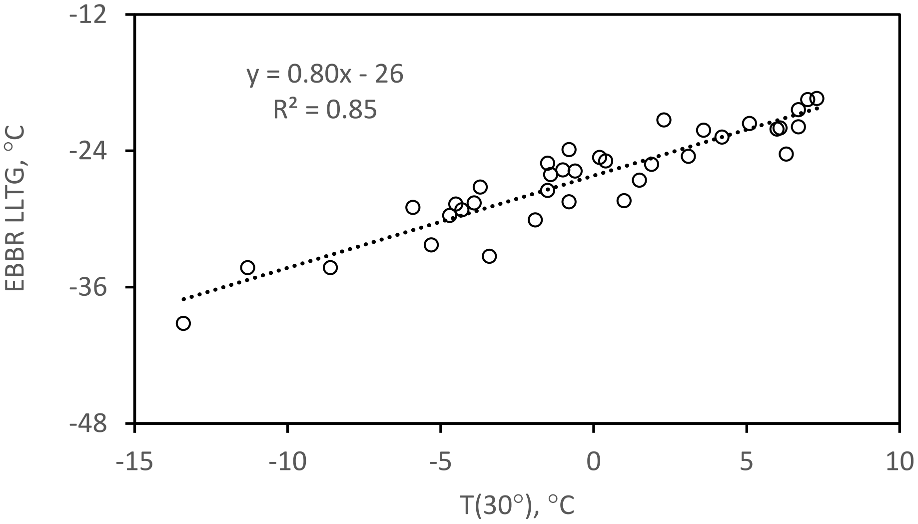

Figure 18. EBBR limiting low temperature grade and T(30°) correlation (Publication I)

As can be seen from the figures, the correlations with both BBR and EBBR have extensively improved and are very similar to the findings from Angius et al. (2018) and Ding et al. (2018). Looking at the BBR and T(30°) correlation, the R^2 equals 0.70 which is a good result, meaning that the conditioning time of 10 minutes in the DSR seems to be able to mimic the cooling for 1 hour in the BBR.

On the other hand, the correlation between the EBBR and T(30°) results is even better with R^2 equalling 0.85. This suggests that during the 10-minute equilibrium time in the DSR, even the reversible aging phenomenon can be considered in some extent.

Coming from this, a linear correlation between EBBR and T(30°) was proposed in Publication I. This correlation can be presented as Equation (2).

$$YY_{EBBR} = 0.8 * YY_{T(30°)} - 26 \tag{2}$$

Where

YY_{EBBR} – calculated extended bending beam rheometer test result, °C

$YY_{(T30°)}$ – measured limiting phase angle temperature

To prove the suitability of Equation (2), the differences between the measured and calculated EBBR low temperature grades were investigated. The mean deviation between the two values was only 1.4 °C. When applying the already previously mentioned (3.1.2) limit of 3.0 °C, then only two samples deviated more than this value. These samples were no. 21 and 23, with deviations of 4.4 °C and 3.2 °C, respectively. Again, the exact reasons for these deviations are not know, but testing errors are not excluded. Hence, the T(30°) parameter and the proposed correlation are promising tools for determining the low temperature performance of asphalt binders.

Another takeaway from these results is the average T(30°) which was found to be –0.3 °C. This once more confirms the findings of Migliori et al. (1999) and Soleimani et al. (2009), who stated that at around a temperature of 0 °C binders used in northern climates would have a phase angle of 27° to 28°.

3.1.4 Summary

In terms of low temperature performance, it is the EBBR protocol that yields the best confidence in terms of expected future behaviour. Due to the extended conditioning time, the phenomenon of reversible aging is considered, meaning that the binder has achieved equilibrium before the measurement. Resulting from this, the EBBR protocol should be considered for the enhancement of the European specifications, especially for initial type testing purposes.

Nevertheless, the method is burdened by the long test duration. This is why the limiting phase angle temperature should also be considered, as it is much quicker to conduct. Although the data obtained from Lill et al. (2019) was inconclusive, previous research suggests otherwise. Additionally, the results from Publication I restored the confidence in this quicker method.

This is why both, the EBBR and DSR-based T(30°), will be included in the second part of the study to identify the most optimum method for specifying low temperature performance.

3.2 Evaluation of the Fraass breaking point method

As one of the aims of this research is to propose new methods for specifying low temperature performance of binders, it is of utmost importance to understand why this needs to be done. As already previously mentioned, the Fraass method has gross reproducibility and uncertainty issues, leading to many researchers advising to substitute the method (Baumanis et al., 2021; Besamusca et al., 2010; Bueno et al., 2015; Gražulytė & Vaitkus, 2017; Lu et al., 2003, 2017; D. Petersen et al., 1997; Ravnikar Turk & Tušar, 2018). To confirm this once more, the Fraass breaking point was included in Lill et al. (2019) and additionally in Publication I and II of this study.

In Lill et al. (2019), the samples were tested exactly as per the European standard EN 12593 (CEN, 2015c). This meaning that no aging protocol was applied prior to testing. These results are presented in Figure 19. In this paper no correlation study was presented, but there seemed to be negligible correlation between the Fraass breaking point and BBR/EBBR and DSR limiting phase angle temperatures.

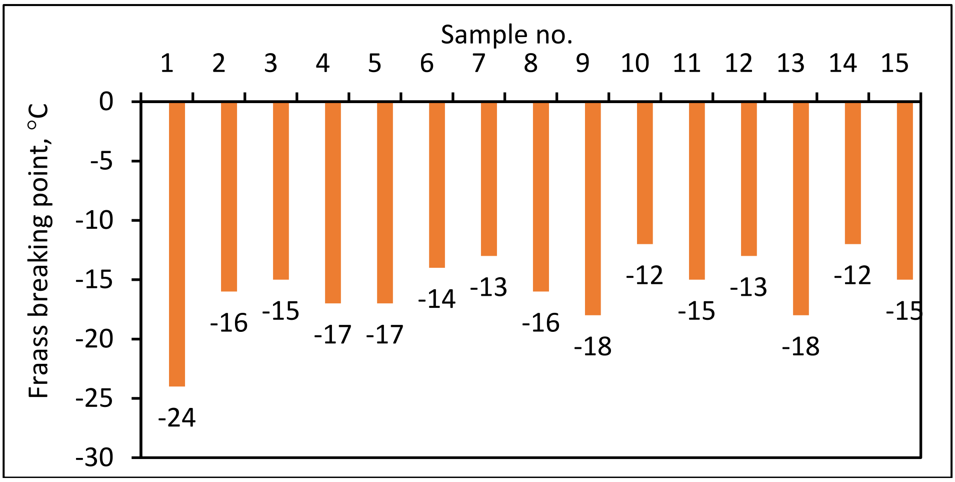

Figure 19. Fraass breaking point results from Lill et al. (2019)

Another look on this topic was given in Publication I. Although this publication included a total of 36 binders, only 26 were tested for their Fraass breaking point, as the other 10 binders were no longer available in the laboratory. The binders studied here covered a range of 11.0 °C in terms of their breaking point. The results ranged from a low of –22.0 °C to a high of –11.0 °C, with an average of –13.5 °C. The Fraass breaking point values for all of the tested materials are presented in Figure 20. Note that the rounding rule of the standard was altered as the results are given with one decimal where applicable.

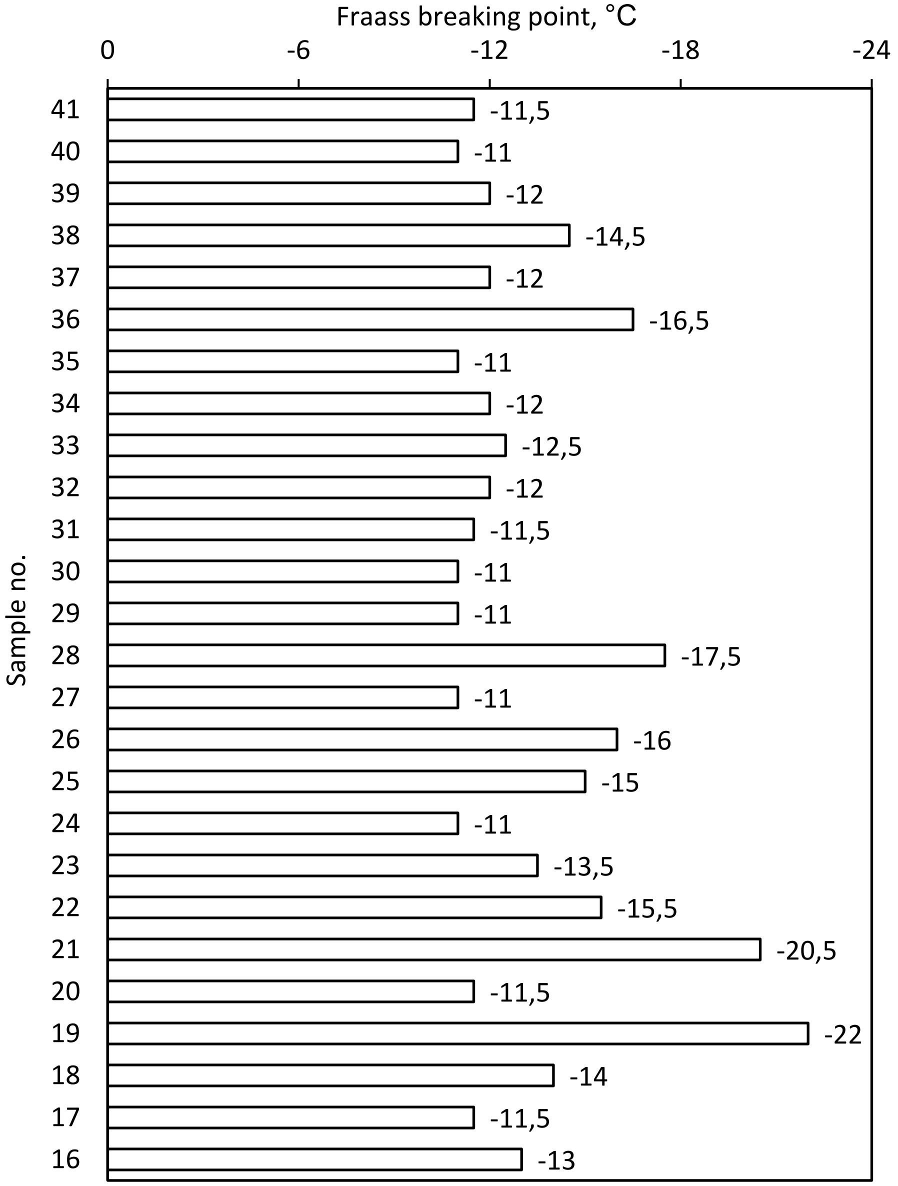

Figure 20. Fraass breaking point results from Publication I

In this study the Fraass breaking point results were correlated with the results from the BBR, EBBR and limiting phase angle tests. These results can be seen in Figure 21, Figure 22 and Figure 23. Somewhat surprisingly, for this set of samples, there was a noticeable correlation with BBR and EBBR results, with coefficients of determination being $R^2 = 0.72$ and $R^2 = 0.67$, respectively. At the same time, the correlation with the limiting phase angle temperature T(30°) had a poor correlation with $R^2 = 0.44$.

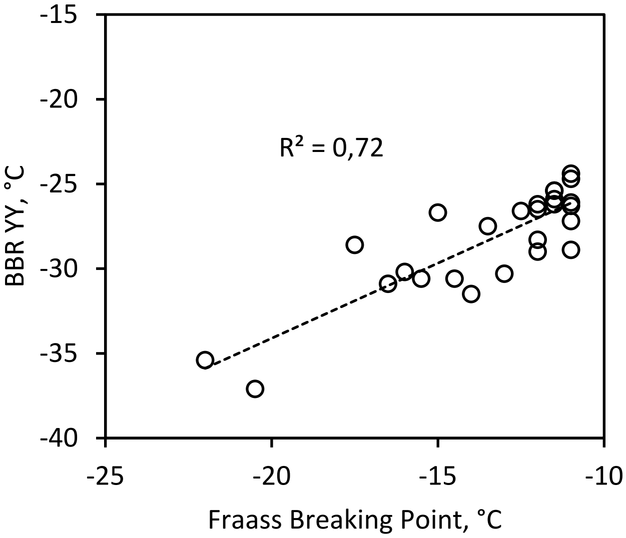

Figure 21. Correlation between BBR and Fraass test (Publication I)

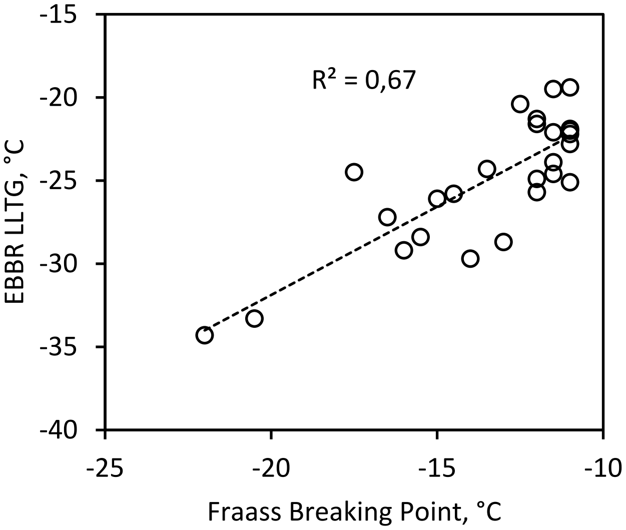

Figure 22. Correlation between EBBR and Fraass test (Publication I)

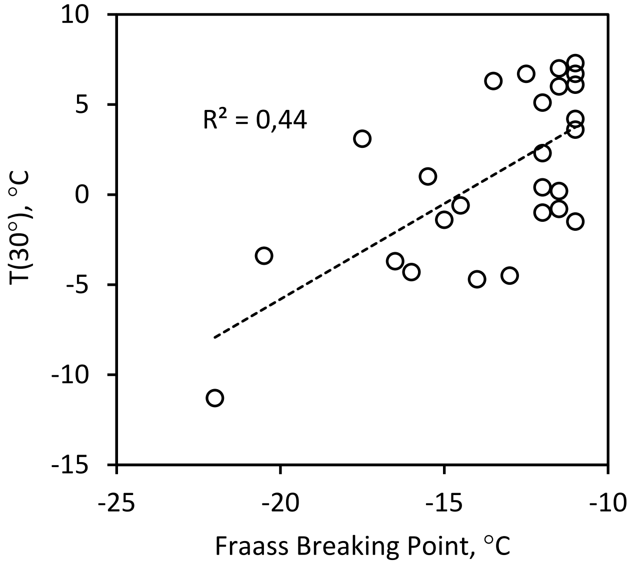

Figure 23. Correlation between T(30°) and Fraass test (Publication I)

One more look into the correlation of Fraass breaking point with other, more advanced methods, was taken in Publication II. It should be noted that in this research the Fraass test was conducted on short-term aged binders. The Fraass results together with the EBBR true grades for the samples included in Publication II are presented in Table 6. The Fraass test was not conducted on sample no. 64, as it was no longer available at the time of the Fraass tests.

The correlation between the EBBR and Fraass test results are presented in Figure 24. Although a low correlation was expected, the correlation in this study was extremely low. The coefficient of determination R^2 equalled only 0.08. One of the reasons could be that the samples were tested at different aging conditions, with the Fraass test being conducted on RTFOT residue in this study and the EBBR on RTFOT and PAV aged materials. This discrepancy was also present in the data of Lill et al. (2019) and Publication II, where the Fraass breaking point was measured on unaged material, but in that case the correlations were better. More likely, it is an issue with the reproducibility of the Fraass method. The Fraass tests for Lill et al. (2019) and Publication I were conducted with an automatic apparatus, while the results for Publication II were obtained from a semi-automatic appliance. Probably the automatic apparatus is capable of more reproducible results and the semi-automatic, where the personnel conducting the test has a larger impact, has worse reproducibility. The semiautomatic and also manual appliances are still widely used throughout European laboratories and this most likely is also the reason why the Fraass test has such a poor reputation. As the automatic Fraass apparatus is in the same price range as the BBR equipment, it is more useful to move towards BBR equipment rather than buying automatic Fraass appliances.

Table 6. Fraass breaking point results (Publication II)

Sample no.	Fraass breaking point (RTFOT residue), °C	EBBR true grade, °C
52	-15	-22.4
53	-17	-23.4
54	-17	-25.7
55	-17	-23.5
56	-17	-24.6
57	-18	-25.0
58	-15	-22.2
59	-18	-32.5
60	-12	-24.4
61	-16	-24.1
62	-15	-24.0
63	-16	-23.9
64	-	-23.9

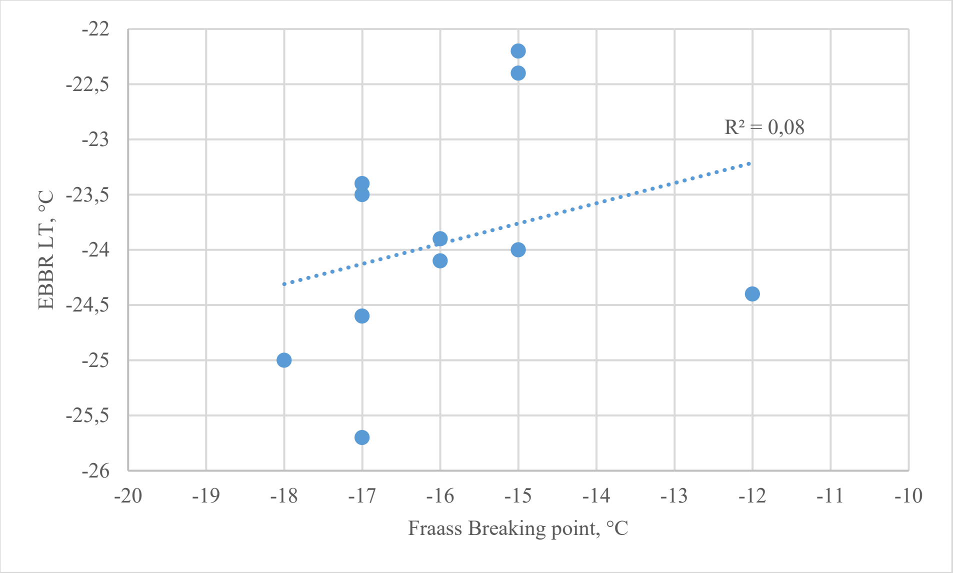

Figure 24. Correlation between EBBR and Fraass test (Publication II)

3.3 Feasibility of limiting phase angle temperatures for specifying asphalt binder low temperature performance in North-Eastern European climatic conditions

The first part of this study with Publications I, together with Lill et al. (2019), focused on a broad spectrum of binders from around the world to evaluate methods for measuring low temperature performance of asphalt binders. The findings were that the EBBR with its ability to account for reversible aging is preferred, but the T(30°) is also valuable, as it seems to lead to similar conclusions with much quicker testing times. A correlation, shown in Equation (2), was proposed to substitute the cumbersome EBBR test with a quick DSR measurement.

This section of the study focuses on the usability of the T(30°) parameter as a surrogate for the EBBR, when focusing on a narrow binder market. In this case the focus is on the North-Eastern European market. A set of 13 binders were sourced from asphalt mixture or emulsion plants in Estonia. As Europe uses penetration grading, the samples covered three grades: 70/100, 100/150 and 160/220. At the time of sampling, the three main countries that supplied Estonia with asphalt binder were Lithuania, Russia and Sweden.

3.3.1 Penetration grading and Superpave grading

The needle penetration and Superpave grade on these 13 samples are presented in Table 7. It should be noted that sample no. 59 is also from a Swedish refinery, but it's from the time when this refinery was still working with Venezuelan crude, which is not available anymore. This sample was included in the study for reference purposes.

According to their needle penetration, all but one sample fall within their respective grade. It is sample no. 59 that falls short by only 1 tenth of a millimetre. The exact reasons for this are not know, but a potential reason could be that the sample was obtained from the emulsion plants warehouse, where it had been stored in a sealed 20-litre container. Although it did not meet its grade, for correlation purposes it was still used throughout the study, where deemed useful. In some cases, the data for sample no. 59 is omitted from the correlations.

The high temperature grades according to Superpave range from 52 °C to 64 °C. As expected, the stiffer 70/100 binders resulted in a PG 64 grade. The binders with the softest penetration grade, 160/220, resulted in two thirds of the occasions with PG 52. It was sample no. 59 that met also the PG 58 criteria. The 100/150 binders resulted in a PG 58 grade, only sample no. 52 missing the grade by a margin of 0.2 °C, meaning that it is a PG 52 binder.

In terms of the low temperature Superpave grades, the binders are divided into two grades with most samples meeting the PG -28 criteria, but samples no. 52 and 58 belong to the PG -22 grade.

Table 7. Needle penetration and Superpave grade (modified from Publication II)

Sample no.	Penetration grade	Needle penetration, 0.1 mm	Superpave performance grade (XX-YY), °C
52	100/150	114	52–22
53	70/100	78	64–28
54	160/220	175	52–28
55	70/100	81	64–28
56	100/150	121	58–28
57	100/150	125	58–28
58	100/150	105	58–22
59	160/220	159	58–28
60	100/150	129	58–28
61	100/150	124	58–28
62	100/150	111	58–28
63	100/150	126	58–28
64	160/220	184	52–28

3.3.2 Low temperature grading

In terms of low temperature performance, the samples were subjected to the regular BBR test, which is part of the Superpave grading. The samples were also tested with the Fraass test and EBBR. These results were already presented in section 3.2, tough missing the grade losses for the EBBR. Additionally, the T(30°) parameter was determined. The T(30°) results, Superpave low temperature true grades and EBBR grade losses are presented in Table 8.

Taking another look at the Superpave low temperature grades, an interesting observation is made. The two previously mentioned samples with low temperature grades PG -22, both miss the PG -28 by the smallest possible margin of 0.1 °C. When looking at the Superpave low temperature true grades, they expand over only a range of 5.8 °C. Despite being from three different penetration grades, they seem to perform very similarly in terms of low temperature performance.

Looking at the grade losses, a sad truth is revealed. A higher grade loss is a disadvantage for binders and as already previously mentioned, Canadian-based researchers have suggested a grade loss limit of 3.0 °C. This means that from the studied binders only sample no. 59 with a grade loss of 1.2 °C would pass this criterion. It should be reminded that this binder was refined from Venezuelan crude oil, which has a low wax content, but sadly the binder is unavailable on the current market. The other twelve binders exhibit very similar performance with grade losses ranging from 4.7 °C to 6.3 °C, meaning that they stem from similar crude sources and refining processes. As these failing binders are being commonly used in flexible pavement construction in the area, an increase in low temperature induced defects is to be expected. Nevertheless, at the moment, the proposed 3.0 °C grade loss limit cannot be implemented in North-Eastern Europe, as this would mean none of the currently available binders would meet the specifications.

As the phase angle defines the viscous and elastic components of a sample, it is expected that it can determine the binder’s performance. It is preferable that a binder preserves as much of its viscous component at lower temperatures. Meaning a lower T(30°) parameter is advantageous. From the results of Publication II, it can be seen that binder no. 59 has the lowest T(30°), with a result of –5.3 °C. Another softer 160/220 grade binder (sample no. 54) came second with a result of –0.5 °C. As expected, the stiffer 70/100 binder samples no. 53 and 55 show the highest T(30°) results with 4.3 °C and 4.6 °C, respectively. All other binders fall in between these extents with one interesting observation. The third 160/220 binder (sample no. 64) has a higher T(30°) than some of the stiffer 100/150 graded binders. It is even more peculiar as this sample had the highest needle penetration, which means it is the softest of all the studied binders and therefore the best low temperature performance would have been expected.

Table 8. T(30°) results, Superpave LT true grades and EBBR grade losses (Publication II)

Sample no.	T(30°)	Superpave low temperature true grade, °C	Grade loss, °C
52	3.7	-27.9	5.5
53	4.3	-28.1	4.7
54	-0.5	-31.4	5.7
55	4.6	-28.5	5.0
56	1.3	-30.0	5.4
57	1.4	-31.0	6.0
58	3.4	-27.9	5.7
59	-5.3	-33.7	1.2
60	1.0	-29.9	5.5
61	2.3	-29.7	5.6
62	3.2	-30.3	6.3
63	2.8	-29.2	5.3
64	1.9	-29.7	5.8

3.3.3 Correlation between low temperature grading methods

As already pointed out many times, the EBBR, with its ability to consider the reversible aging phenomenon, is considered the reference method. The results for the EBBR were already correlated with Fraass results in section 3.2. In this section the correlations with the regular BBR test (Figure 25) and T(30°) (Figure 26) are presented and additionally the correlation between the BBR and T(30°) is included (Figure 27). These correlations are presented without sample no. 59.

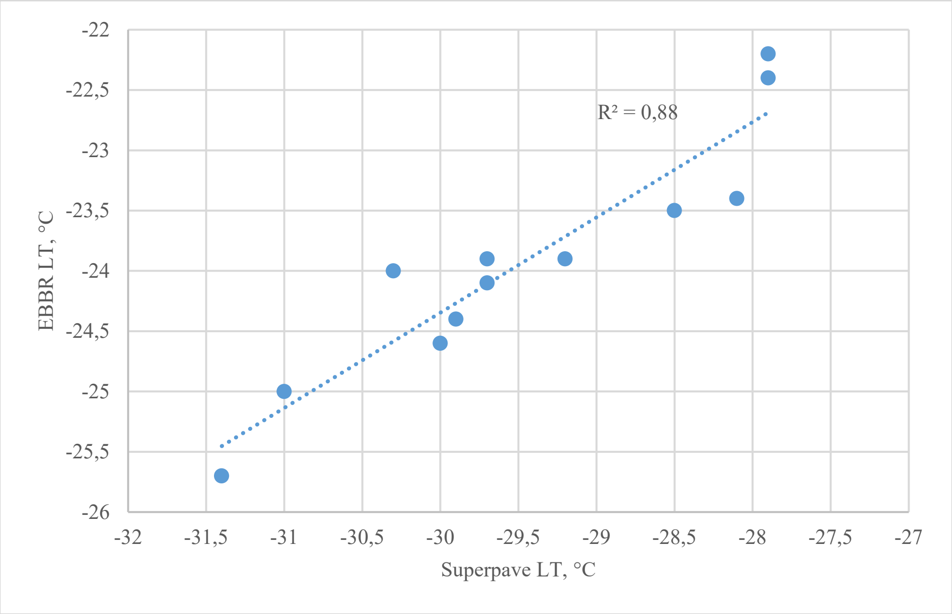

Figure 25. Correlation between EBBR and Superpave low temperature true grades (Publication II)

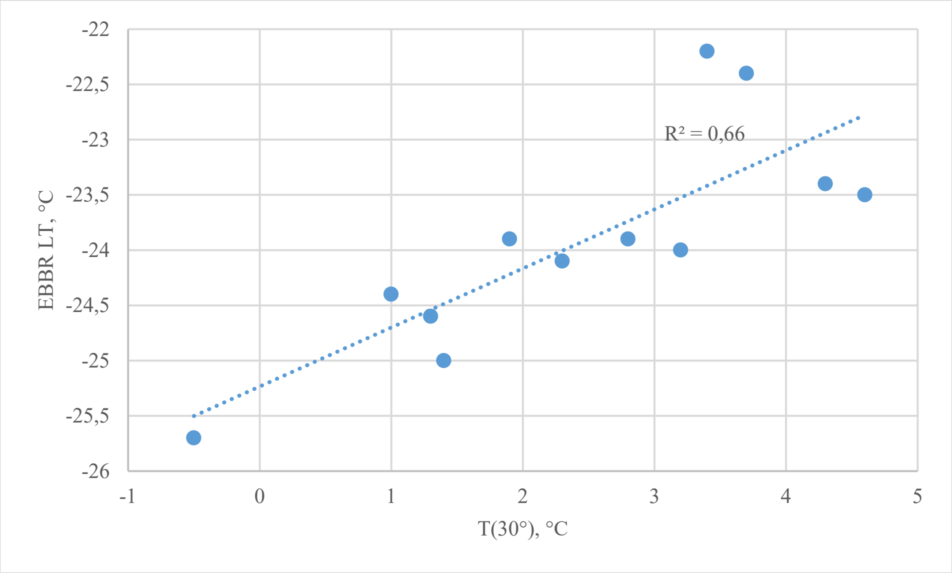

Figure 26. Correlation between EBBR and T(30°) (Publication II)

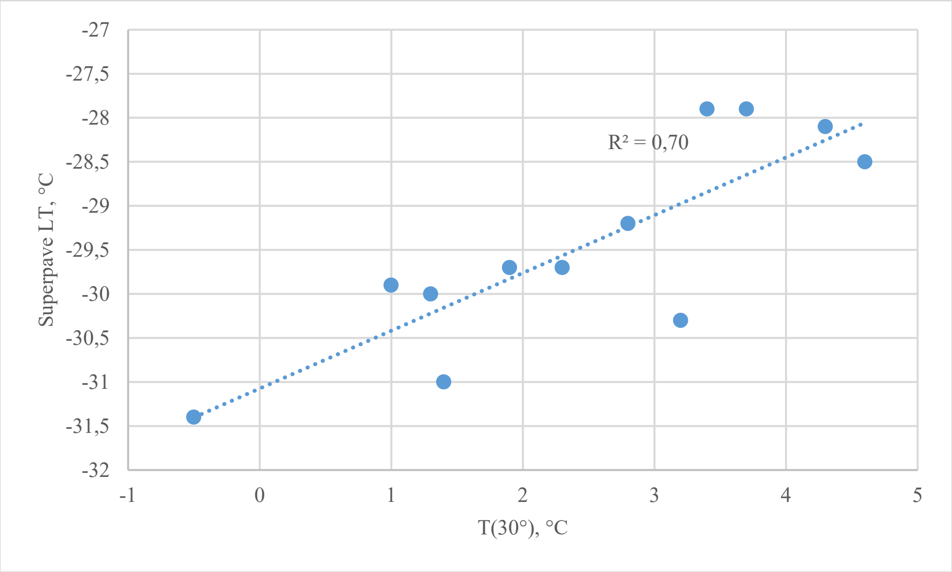

Figure 27. Correlation between Superpave low temperature true grades and T(30°) (Publication II)

Interestingly, the correlation between the BBR and EBBR results is rather high with a coefficient of determination R^2 equalling 0.88. This does not match the results from Lill et al. (2019) and Publication I. The reason for this is most likely the fact that the binder samples are from very similar crude sources and refining processes, thus having all very similar grade losses. Hence in the current market situation in North-Eastern Europe, there is no noticeable advantage in using the tedious EBBR protocol instead of the BBR, but in the long run, with new binders entering the market, the changes in grade loss would go unnoticed.

When investigating the correlations on Figure 26 and Figure 27 another intriguing finding is noticed. In Lill et al. (2019) and Publication I, there was a better correlation between T(30°) and EBBR results, then in this case the opposite is observed. Although, the correlations are quite similar, the R^2 for EBBR is 0.66 and for BBR 0.70. From this it can be concluded that the T(30°) parameter is a satisfactory method for approximating the low temperature performance, but the valuable information of the reversible aging will be lost.

3.3.4 Application of previously found correlation for the conditions of North-Eastern Europe

In Publication I a correlation between EBBR and T(30°) was proposed as Equation (2). This correlation was found on a larger set of binders, which were obtained from around the world.

As the correlation between EBBR and T(30°) were less satisfactory for the sample population in Publication II, a check was conducted to assess whether Equation (2) could be used to predict EBBR results from the measured T(30°) results. The measured EBBR results and the calculated EBBR results with their differences are presented in Table 9.

From the table it can be seen that the differences are rather small, with most being below ±1.0 °C. Only two samples go slightly over this value, with differences of –1.2 °C and 1.1 °C. It is only sample no. 59 that is an exception, having a difference between the measured and calculated EBBR of –2.3 °C. In this specific case there would not be any concern with the binders expected performance in the field, as the predicted value underestimates the performance of the sample, but in certain situations this underestimation could lead to a situation, where the binder cannot be used, although it would perform adequately. On the other hand, the correlation could also overestimate a binder's performance. Due to this, the use of this equation is proposed as a crude tool for approximating the low temperature performance of asphalt binders. Hence, it is always advisable to conduct the actual EBBR test to obtain accurate results.

Table 9. Comparison between the measured EBBR results and the predicted values according to Equation 2 (Publication II)

Sample no.	EBBR low temperature true grade, °C	EBBR low temperature true grade prediction according to Equation 2, °C	Difference, °C
52	-22.4	-23.0	0.6
53	-23.4	-22.6	-0.8
54	-25.7	-26.4	0.7
55	-23.5	-22.3	-1.2
56	-24.6	-25.0	0.4
57	-25.0	-24.9	-0.1
58	-22.2	-23.3	1.1
59	-32.5	-30.2	-2.3
60	-24.4	-25.2	0.8
61	-24.1	-24.2	0.1
62	-24.0	-23.4	-0.6
63	-23.9	-23.8	-0.1
64	-23.9	-24.5	0.6

3.3.5 Summary

This part of the study focused on the feasibility of using a limiting phase angle temperature for the purpose of low temperature specifications for common asphalt binders in the North-Eastern European market. For this purpose the regular BBR, EBBR and T(30°) were measured for a set of 13 binders inherent to this region.

First of all, it was noticed that when measuring with the BBR method, the samples had similar low temperature performance covering only the PG grades -22 and -28 and a temperature range of 5.8 °C. This is surprising as the samples cover three different penetration grades.

The EBBR results uncovered an unfortunate situation in the region, as none of the currently available binders would pass the grade loss limit of 3.0 °C proposed by Canadian-based researchers.

It was the Venezuelan crude oil based binder (sample no. 59) that presented best performance regardless of the method used to determine low temperature performance.

The different low temperature methods were correlated, and some interesting conclusions could be made. The correlation between the BBR and EBBR results was unexpectantly high and the correlation between BBR and T(30°) was better than between EBBR and T(30°).

When studying the correlation presented as Equation (2) with the smaller number of binders from only North-Eastern Europe, it was found that the equation can be used as a crude tool to predict low temperature performance. As the equation might over- or underestimate the actual performance of a sample, it is always advisable to conduct the actual EBBR test for a more precise evaluation.

3.4 Determining asphalt binder aging by using limiting phase angle temperature

The last part of this study focuses on the potential of using a limiting phase angle temperature, like T(30°), for the purpose of measuring the aging of asphalt binders. For this purpose 37 binder samples were obtained, some of which were tank samples and others were extracted and recovered from loose mixture sampled from the auger of the paver and some extracted and recovered from core samples drilled from the pavement. In terms of penetration grading, the binders belong to three different grades: 70/100, 100/150 and 160/220.

Table 10. Basic properties of the tank samples (Publication III)

Sample no.	Penetration, 0.1 mm	Softening point, °C	Superpave performance grade (XX-YY), °C
52	114	41.2	52–22
53	78	48.0	64–28
54	175	39.2	52–28
55	81	47.8	64–28
56	121	43.0	58–28
57	125	42.6	58–28
58	105	43.2	58–22
60	129	41.4	58–28
61	124	41.9	58–28
63	126	41.6	58–28
64	184	40.6	52–28

To understand if the tank samples are in compliance with the current European binder specifications, they were tested for their needle penetration and Ring and Ball softening point. Additionally, for informational purposes, the Superpave grades were determined. These pertinent properties can be found in Table 10. For all of the studied tank samples the needle penetration and softening point fall within the specification requirements.

According to the Superpave grading, the dominating binder grade is PG 58-28, which is suitable for Estonian conditions, although the Estonian PG design temperatures are calculated differently as in the usual Superpave system (Kontson et al., 2023).

As this part of the research focuses on the usability of T(30°) as a parameter to measure the aging of asphalt binders, it was determined for all of the samples. These results are presented in Table 11, but it should be noted that the tank samples were both short-term and long-term aged with the RTFOT and PAV prior to measurement. The samples extracted from loose mixture were additionally aged with the PAV before testing, but the samples extracted from core samples were tested without any additional aging.

Table 11. T(30°) results – numbering as in the publication (Publication III)

Sample no.	T(30°), °C	Sample no.	T(30°), °C
1	3.7	20	3.8
2	3.7	21	2.3
3	4.3	22	3.3
4	4.7	23	2.8
5	0.6	24	5.6
6	-0.5	25	2.8
7	-0.8	26	2.1
8	-4.7	27	1.9
9	4.6	28	1.1
10	4.1	29	-5.7
11	1.3	30	-3.5
12	1.9	31	-2.3
13	-0.7	32	-7.6
14	1.4	33	-3.1
15	1.4	34	-4.0
16	-3.7	35	-8.3
17	3.4	36	-8.8
18	2.8	37	-2.4
19	1.0	-	-

When focusing on tank samples and averaging the T(30°) results for each penetration grade, Figure 28 can be presented. It is evident that stiffer 70/100 binders have a higher T(30°) and the parameter reduces with softer binder grades. These results are as expected, as T(30°) shows the viscous and elastic proportions of materials and stiffer binders achieve the same level of viscous to elastic proportions at higher temperatures when comparing to softer binders. A similar trend is expected with the stiffness increase due to binder aging.

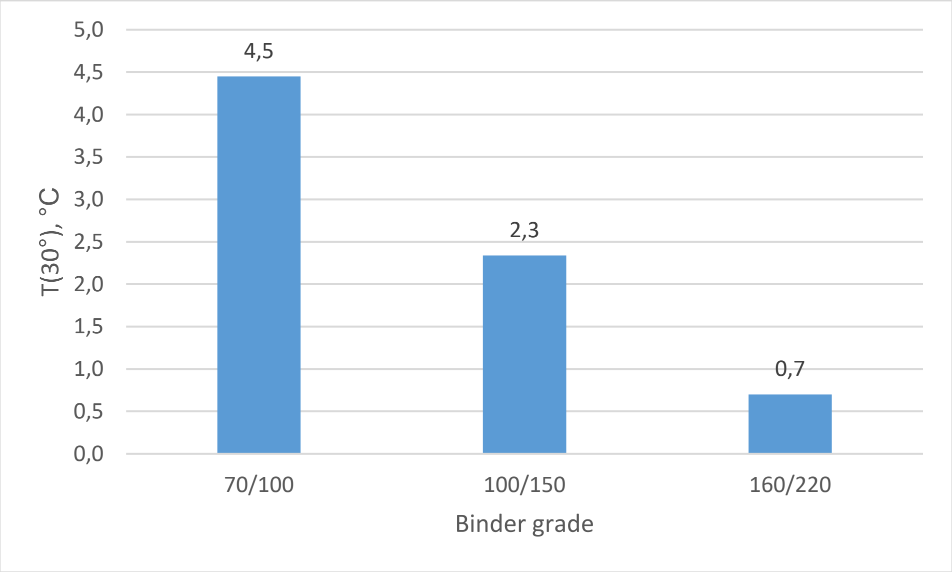

Figure 28. Average T(30°) of different binder grades for tank samples (Publication III)

3.4.1 T(30°) development with pavement age

As previously mentioned, in theory the T(30°) parameter increases with the increase in pavement age. To confirm this, the average T(30°) results for different binder grades and pavement ages are presented in Figure 29, together with linear trendlines for each grade. These results are very controversial, as the data obtained in this study shows the opposite trend across all three grades, then expected. Looking at the data, it seems that binders become more viscous with an increase in pavement age. It is important to note that the data set for this comparison is very limited, but confidence is boosted due to the fact that different binder grades show similar trendline slopes. Additionally, the stiffer 70/100 binders exhibit higher T(30°) and with the increase in penetration grade the T(30°) is decreased.

The most likely reason for this anomaly is the change in binder sources in the North-Eastern part of Europe. As already mentioned multiple times throughout this study, a Swedish refinery used to provide asphalt binder derived from Venezuelan crude oil. This binder had very good low temperature performance and therefore also a low T(30°) value. However, due to geopolitical issues, this binder became unavailable in 2019. The binder samples that were obtained from pavements older than 1 year during coring, are all from the period, where this Venezuelan crude derived binder was still available on the market. To confirm this assumption, it is advisable that future research should measure the aging of binders on specific pavement sections throughout many years, rather than taking samples from different sections with different ages.

It is interesting to note that this fundamental change in binder properties went unnoticed in the European specifications, as all samples have met the requirements presented in EN 12591 (CEN, 2009), but simultaneously there were numerous complaints from the paving industry about binder quality.

Another interesting finding from these results is that the binder present in old pavements is of higher quality than newly refined binder on the market. This is because the binder present in pavements laid prior to 2019 is potentially more viscous at lower temperatures comparing to new binders. In other words, after mixing into the asphalt mixture, newly refined binder is more elastic and thus more prone to cracking due to low temperatures and fatigue, potentially causing pavement defects at an earlier stage in their lifetime. This is something that stakeholders need to acknowledge, as it might be beneficial to start using higher amounts of RAP (recycled asphalt pavement) in new pavements.

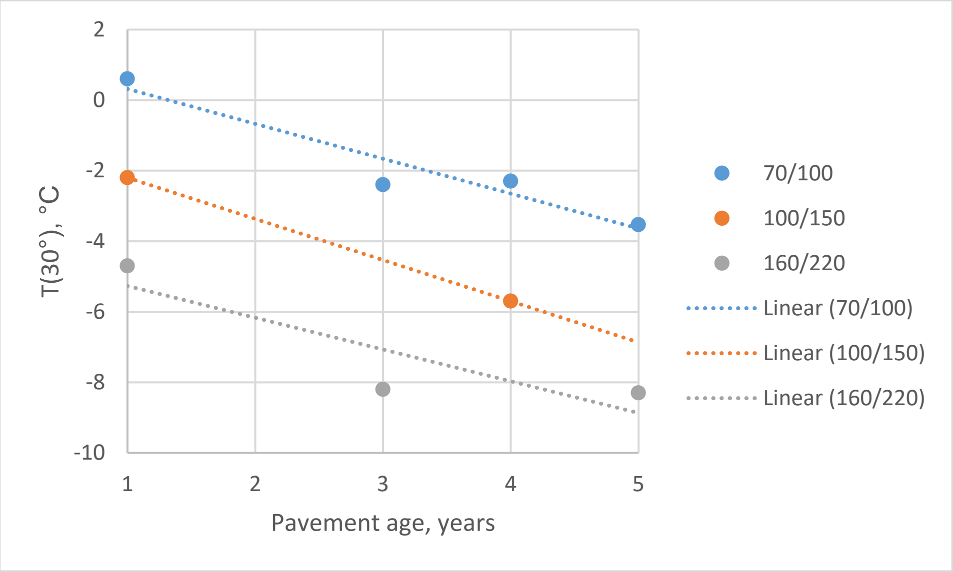

Figure 29. Development of T(30°) with pavement age (Publication III)

3.4.2 T(30°) correlation between tank and mixture samples

Figure 30 presents the correlations between T(30°) for tank samples and mixture samples, noting that the binder used in the mixtures were the same as the tank samples. The correlation includes only sample couples, where both tank and loose mixture samples were available. It can be seen from the figure that the correlation is not great when considering all samples. The coefficient of determination R^2 is 0.51. Taking another look at the data shows that there are two outliers affecting the correlation. These are the tank samples no. 60 and 63 and their respective mixture samples no. 76 and 78. Repeating the correlation without these samples, the R^2 is increased to 0.87, meaning a significant improvement.

The anomaly seems to lie in the results of the mixture samples as they appear to be higher when comparing with the other samples. It is important to understand if the results of the outlying samples are correct or incorrect.

One potential way to clarify this is via high temperature performance grading results. Luckily, this data was available for the studied binders for both the tank samples and samples extracted from loose mixture. These results are presented in Table 12. The data suggests that for almost all sample couples, the RTFOT aged sample has a higher Superpave high temperature grade, compared to the recovered binder sample. As a side note, it can be concluded that the aging caused by the RTFOT tends to be more severe than aging occurring in the asphalt mixing plant.

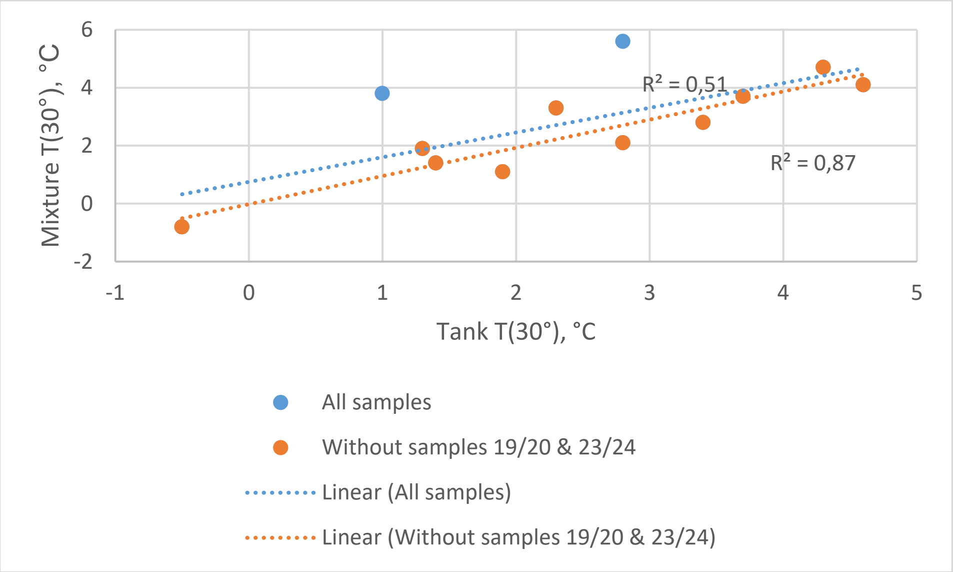

Figure 30. T(30°) correlation between tank and mixture samples (Publication III)

Table 12. Superpave high temperature true grades (Publication III)

Test section	Sample no.	Superpave high temperature true grade for tank sample (after RTFOT), °C	Sample no.	Superpave high temperature true grade for mixture sample (after extraction and recovery), °C
T92 Kanaküla	52	59.4	65	58.5
T65 Veriora (ref)	53	65.8	66	66.9
T65 Veriora (soft)	54	55.0	68	53.1
T92 Rihkama	55	67.1	70	65.3
T52 Metsla (base)	56	60.1	71	58.7
T52 Metsla (surf)	57	59.3	73	57.7
T60 Nätsi	58	60.1	75	58.0
T52 Holstre (base)	60	58.8	76	63.2
T52 Holstre (surf)	61	59.3	77	59.5
T19201 Vahenurme (bin)	63	60.1	78	63.8
T19201 Vahenurme (surf)	63	60.1	79	57.3
T81 Käina	64	54.9	80	53.1

Nevertheless, looking at the data, it is evident that there are some exceptions. For three of the studied samples the trend is the opposite. One of these exceptions is for the site T65 Veriora (ref), where the Superpave high temperature true grade was higher for the recovered binder. The difference was 1.1 °C. For this particular case, the reason for this discrepancy is known, as during sampling for this mixture the asphalt paver broke down and it took around four hours to fix it. The excessive aging was caused by the extended period at elevated temperature, as the mixture was waiting in the truck trailer until paving continued.

The other two exceptions are the already mentioned outlying mixture samples no. 76 and 78. Although the exact reasons for this excessive aging are not known, the most probable reason is that these samples were taken from a mixture with limestone aggregate. Previous research (Anderson et al., 1994; J. C. Petersen et al., 1974; Wu et al., 2014) stated that the aging of the binder in mixtures with limestone is slower than in mixtures with granite aggregate, but in terms of this research the key factor seems to be temperature. Limestone aggregate is much more porous and absorbs more water compared to igneous or metamorphic rock. If the limestone aggregate is wet in the stockpile, and not wanting to compromise the production rate, operators tend to use more heat to dry the aggregate. This results in a higher temperature in the aggregate and mixing this excessively heated aggregate with asphalt binder causes more aging compared to when the aggregate would be less hot. Though, it should be noted that this kind of excessive aging was not observed with sample no. 71, which was also mixed with limestone aggregate. It is not ruled out that at the time of production of this mixture the aggregate was not too wet and no excess heating was necessary.

Jumping back to the discussion about whether the T(30°) results of samples no. 76 and 78 are correct or not. After the previous discussion, it can be stated that the results are correct, and the lower correlation is valid. The findings confirm that the RTFOT is an empirical short-term aging procedure that can be used to assess the aging differences between binder samples, but it cannot always accurately predict the aging occurring in the mixing plant, as there are several factors that affect the aging degree in the plant.

3.4.3 Usability of T(30°) as an aging measuring tool

To study the ability of the T(30°) parameter for measuring asphalt binder aging, data from different aging conditions for a specific binder is needed. In this study, there were three test sections with a total of four asphalt mixtures that had T(30°) data for the tank sample, loose mixture and core sample. The T(30°) results from the core samples and their respective tank samples together with their differences for the three sections are presented in Figure 31. As the pavements were all around 1 year old when coring, similar trends should be observed. This is true for three of the four mixtures.

The AC 16 surface mixture from the section T52 Metsla and the mixtures containing 70/100 and 160/220 binders from T65 Veriora showed similar results with the differences being 3.7 °C to 5.1 °C. However, the difference recorded for T52 Metsla base course mixture was only 2.0 °C. This is an indication that the binder contained in the pavement has aged more severely and its end-of-life could be reached at an earlier stage compared to the other mixtures.

Pinpointing the cause for this excessive aging is difficult, but once again it can be noted that the peculiar result was achieved from a mixture with limestone aggregate. Taking a look at the T(30°) result from loose mixture, it can be marked that no excessive aging occurred at the plant, because the difference between the tank sample and loose mixture was only 0.6 °C. A potential reason for this result is that during the course of a year, lighter fractions of the binders could have absorbed into the pores of the limestone aggregate and were unaffected during the extraction process, meaning the lighter fractions stayed in the pores. Though, this contradicts the finding of Wu et al. (2014). This phenomenon needs to be further investigated in future research, as there in not enough data in the current dataset to draw definitive conclusions.

Still, using this limited amount of data, it is evident that T(30°) has the potential to be used as a parameter to measure the aging of asphalt binders. A key advantage of this parameter is that only a small amount of binder is needed for testing and even one core, drilled from the pavement, provides enough material. If the T(30°) result is known for the tank sample, which has been aged with both the RTFOT and PAV, it can be used as a benchmark. The probability of pavement defects, induced by binder aging, is increased as the T(30°) of the binder in the pavement moves toward or even surpasses the T(30°) result of the tank sample. On another note, this tank sample T(30°) might be useful, when wanting to control the amount and quality of RAP used in new asphalt mixtures.

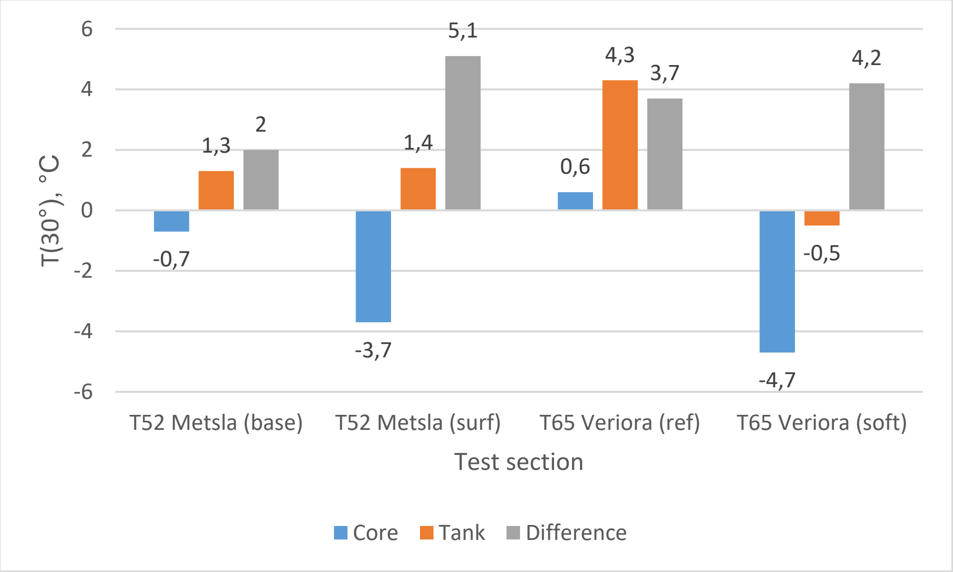

Figure 31. Development of T(30°) (Publication III)

3.4.4 Correlation of T(30°) and pavement defects

The national roads in Estonia are periodically monitored and among others the sum of pavement defects is determined. As this data is publicly available, it was decided to check if the sum of pavement defects per kilometre correlates with T(30°). The data was extracted in the beginning of 2024 and the most recent data for each section was obtained. The section T11152 Kirdalu was last inspected in 2022, but all of the others had data from 2023.

The correlation between sum of defects and T(30°) can be found in Figure 32 and it can be seen that the correlation is found to be negligible ($R^2 = 0.13$). Although a better correlation was hoped for, the result is not unexpected as T(30°) primarily measures the aging degree and low temperature performance of asphalt binders, but not all pavement defects can be attributed to these factors. Additionally, it must be noted that the studied sections vary in Annual Average Daily Traffic and design.

This study was done on relatively new pavements, for which the sum of pavement defects per kilometre were between 0.11% to 4.24%. Currently there are no limits set, but work done in the early 2000s (Kaal, 2003) suggested that a pavement with a sum of defects up to 1% is considered very good and between 2 and 5 good. This means the studied sections all belong either to the good or very good category.

To further explore this topic, it is advisable to include pavements older than 5 years and additionally focus on specific defects that are more closely related to binder aging and low temperature performance, rather than using the aggregated sum of defects data.

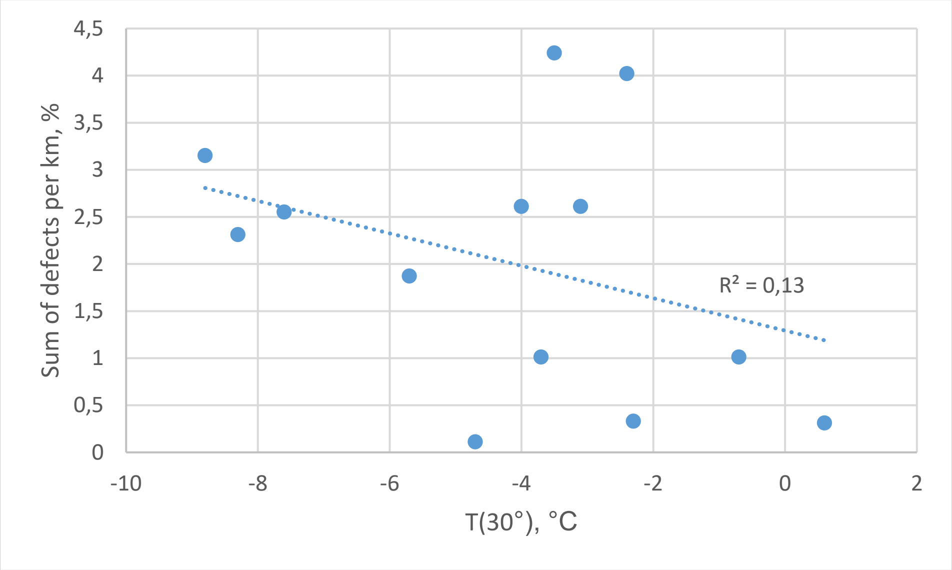

Figure 32. Correlation between sum of defects per km and T(30°) (Publication III)

3.4.5 Summary

This paragraph of the study focused on determining, whether the T(30°) parameter is suitable for measuring asphalt binder aging. For this purpose, 37 binder samples were tested. Among the measured materials, there were tank samples, samples recovered from loose asphalt mixture and samples recovered from core samples.

It was found that the T(30°) parameter can successfully differentiate between softer and stiffer binders with stiffer binders having higher values and vice versa.

A noteworthy finding was that T(30°) was able to detect a drop in binder quality, when regular European specifications could not. Together with this discovery, the revelation came, that pavements constructed before the current decade could contain binder that has better quality than freshly refined binder.

The T(30°) is a suitable measure to monitor the aging of asphalt binders, but a result for the tank sample is necessary for reference purposes. It even has the potential to be used when RAP is added into new mixtures.

Other observations made during this part of the study were that the RTFOT cannot always accurately predict the aging occurring at the asphalt mixture plant as many factors affect the degree of aging in the plant, but the RTFOT has fixed parameters.

On many occasions, the binder in mixtures made with limestone aggregate had aged more than binders in mixtures with igneous rock aggregate. This might be due to higher mixing temperatures used for mixing with limestone, but also the absorption of lighter fractions could be a potential reason.

For the studied road sections, there was almost no correlation between the T(30°) parameter and the sum of pavement defects per kilometre.

4 Conclusions and further studies

The purpose of this study was first and foremost to confirm that the Fraass breaking point is unable to accurately assess binder low temperature performance and to propose new methods for the European specifications. An additional goal was to determine a suitable parameter to measure the aging of asphalt binder.

The first part of the study focused on finding these new low temperature methods. For this purpose, 51 binder samples were tested. The most accurate protocol in this regard was proven to be the EBBR test, which is able to consider the reversible aging phenomenon, but its obvious drawback is the unreasonably long duration of the test. Due to this, an additional parameter, the limiting phase angle temperature T(30°), was introduced. This DSR-based method can be conducted in a matter of hours, rather than days. Both methods were included in further studies and a correlation for these two was estimated.

The correlations between these newly proposed methods and the Fraass test appeared to be scattered. Even though in publication I there was considerable correlation between EBBR results and the Fraass breaking point, but in the other two publications, dealing with the Fraass method, the correlations were negligible. It seemed that results obtained from automatic appliances were of higher quality, but results from semi-automatic appliances deviated extensively. As there is a lot of literature also stating the issues with the Fraass method it was confirmed that this obsolete method needs replacement.

The next part of the study investigated whether the T(30°) parameter is a suitable low temperature performance measure, considering solely the conditions of the North-Eastern European asphalt binder market. For this purpose, 13 local binders were sampled and tested. From these results the following conclusion could be drawn:

- Although being from three different penetration grades, the low temperature performance according to Superpave was very similar, with a true grade range of only 5.8 °C.
- There is a remarkable problem with reversible aging for the binders in the region, as none of the currently available binders passed the grade loss limit of 3.0 °C, proposed earlier by Canadian-based researchers.
- The currently unavailable binder refined from Venezuelan crude oil showed the best low temperature performance, regardless of the studied method.
- An unexpected finding was that BBR and EBBR results correlated very well. This discrepancy is caused by the fact that the studied binders seem to be from very similar crude sources and refining processes.
- The suitability of the EBBR and T(30°) correlation, proposed in the first part of the study, was checked with this limited set of binders. The conclusion was that the proposed correlation can be used as a crude tool for approximating low temperature performance, but it is always advisable to test the EBBR to obtain accurate results.

The last part of the study investigated whether the T(30°) parameter can be used to measure the aging of asphalt binders. This part of the study included 37 different binders, some of which were tank samples, some were recovered binders from loose asphalt mixture and others were recovered from core samples. The following conclusions could be made from the results:

- The T(30°) can successfully differentiate between softer and stiffer binders. Stiffer binders have higher T(30°) results and vice versa.
- The T(30°) parameter was able to distinguish a substantial deterioration in binder quality, which remained undetected by the current EN specifications.
- The T(30°) is suitable to measure the aging of asphalt binders, but a reference result from a tank sample is necessary. Furthermore, the parameter could be beneficial, when trying to control the amount and quality of RAP used in new mixtures.
- Other discovery included that the RTFOT cannot always accurately predict the aging occurring at the asphalt mixing plant, as the test method has fixed parameters, whereas mixing conditions at the plant depend on many factors.
- Another finding from the research was that on many occasions, binders present in mixtures with limestone aggregate, have aged more severely compared to binder from mixtures with igneous rock aggregate. A potential reason for this could be a use of higher mixing temperatures, but the absorption of lighter fractions of the binder into the pores of the aggregate is also a possibility.
- The T(30°) was correlated with the sum of pavement defects, but no significant correlation was observed.

Although the list of findings in the study is extensive, there were still some research questions that were left unanswered. These topics are proposed for future studies. These include the unexpected trend in T(30°) with pavement age presented in Figure 29. It is of interest whether this is caused by the changes in binder sources or not. Additionally, the data was scattered, and no definitive conclusion could be made, whether limestone aggregate causes more or less aging in the asphalt binder. One more suggestion is about the T(30°) and the sum of pavement defects correlation. Although, the correlation was negligible in this study, future research should look at a longer timeline and also consider other defect parameters rather than the sum of defects.

The findings of this study confirm that the EBBR and T(30°) can be proposed as new and improved methods for specifying low temperature performance of asphalt binders. The EBBR should be the reference method, that should be used for at least initial type testing of a binder. The T(30°) parameter can be used as a quick tool to approximate low temperature performance. This should be used, when a fast decision needs to be made, e.g. for continuous quality control. In addition to this, the T(30°) parameter is of value as a parameter to measure the aging process of a binder. When using both the EBBR and T(30°), the issues with low temperature and fatigue cracking should be reduced.

List of figures

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Abstract

Applicability of Limiting Phase Angle Temperatures for Measuring the Low Temperature Performance and Aging of Asphalt Binders

Asphalt binder is a crucial component of asphalt mixtures. Although, the whole mixture composition is important, it is mainly the binder that controls the pavement’s ability to withstand cracking, both low temperature and fatigue induced. During the service-life of a pavement, the binder within, will be subjected to a wide range of temperatures. It has been found that in Estonia the pavement temperature on highways can rise up to 60 °C during summer and can drop below –30 °C in the winter. To ensure adequate performance throughout this range of temperatures, a set of efficient binder specifications is needed. The motivation of this study is to propose new and improved methods for measuring the low temperature and aging performance of asphalt binders.

The current European paving grade binder specification (EN 12591) has only one parameter to control low temperature performance. This is the Fraass breaking point method. This testing protocol has been deemed unsatisfactory by many researchers due to it having reproducibility and uncertainty issues. This outcome is once more confirmed in this study.

Two new methods to substitute the current Fraass breaking point as the low temperature specifications are proposed. These are the Extended Bending Beam Rheometer (EBBR) test and limiting phase angle temperature T(30°). The first of the two is considered more accurate, because it considers the reversible aging phenomenon of asphalt binders, therefore being an excellent method to differentiate between superior binders and poor performers. The issues with this method are that it requires a large sample, it is very labour intensive as it takes four days to conduct the test without sample preparation. This is the reason why the T(30°) parameter is proposed. The test can be finished in a matter of hours on the Dynamic Shear Rheometer (DSR). The conclusion of the study, regarding this topic, was that the EBBR test should be the reference method for specifying low temperature performance, but the T(30°) parameter is a very useful tool if a quick approximation of the low temperature performance of a binder is needed.

Another drawback of the current European specification is that the aging of asphalt binders has very little focus. Currently only short-term aging is included, meaning that long-term aging performance is completely disregarded. The study proposes the T(30°) parameter as a possible tool for measuring the aging of asphalt binders. As the T(30°) parameter utilizes the phase angle, measured in the DSR, it can accurately determine the viscous and elastic proportions of a binder.

The objectives of this research were:

- Evaluating potential new low temperature methods for specifying asphalt binders.
- Confirm the uncertainty issues with the Fraass breaking point method.
- Determine if the T(30°) parameter is able to substitute the EBBR test.
- Study if T(30°) can be used to measure the aging of asphalt binders.

For this purpose, 89 different asphalt binders were tested. In the first part of the study, binders from around the world were used to select potential methods. The second part of the research focused on the narrow North-Eastern European binder market, utilizing

the methods selected in the first part of the study. Most samples tested were tank samples, but for the purpose of measuring asphalt binder aging, also samples recovered from both loose mixture and core samples were included.

Additional to the already mentioned methods, usual binder testing methods from both penetration grading and Superpave grading were included for informational and reference purposes. These include for example the needle penetration, softening point according to Ring and Ball, Superpave high, intermediate and low temperature grades.

The main outcomes of the study are as follows:

1. The T(30°) parameter is a suitable tool for approximating low temperature performance of asphalt binders.
2. The T(30°) parameter can successfully be used to measure the aging of asphalt binders.
3. The T(30°) parameter was able to discover a drop in asphalt binder quality that was missed by current European specifications
4. The Fraass method was confirmed to be unreliable and the proposal for its replacement is justified.

Lühikokkuvõte

Piiravate faasinurga temperatuuride rakendatavus bituumenite madala temperatuuri ja vananemisalase toimivuse mõõtmiseks

Bituumen on asfaltsegude väga oluline komponent. Kuigi kogu segu koostis on oluline, siis just bituumen on see, mis määrab peamiselt asfaltkatte võimekuse hoida ära nii madalast temperatuurist kui ka väsimusest tingitud pragunemine. Asfaltkattes olev bituumen puutub kokku laia temperatuurivahemikuga. Varasemad uuringud on leidnud, et Eesti teekatte temperatuurid võivad maanteedel tõusta suvel kuni 60 °C ja talvel langeda madalamale kui –30 °C. Et tagada nõuetekohane asfaltkatete toimivus kogu selles temperatuurivahemikus, peavad bituumenile kehtestatud nõuded olema piisavad. Käesoleva teadustöö eesmärgiks on välja pakkuda uued ja täiuslikumad meetodid bituumenite madala temperatuuri ja vananemisalase toimivuse määramiseks.

Euroopas kehtivas teebituumeneid reguleerivas standardis EN 12591 on ainult üks parameeter, millega bituumenite madala temperatuuri toimivust määratleda. Selleks meetodiks on Fraassi murdumistäpp. Paljud teadlased on välja toonud selle meetodi puuduseid seoses korratavuse ja määramatusega. Samad probleemid leidsid kinnitust ka käesolevas doktoritöös.

Töös on välja pakutud kaks Fraassi murdumistäpi asendamiseks mõeldud meetodit bituumeni madala temperatuuri omaduste määratlemiseks. Nendeks on pikendatud painduva tala reomeetri (EBBR) katse ja piirav faasinurga temperatuur T(30°). Neist esimene on täpsem, sest see meetod arvestab ka pöörduva vananemisega, millest tulenevalt suudab see katse edukalt eristada heade ja kehvade omadustega bituumeneid. Selle meetodi murekohtadeks on vajamineva bituumeniproovi kogus, suur töömahukus ja katsetamise aeg, mis on neli päeva ilma proovide ettevalmistamiseta. Selle tõttu pakutakse välja ka T(30°) parameeter. Selle katse saab sooritada mõne tunniga dünaamilise nihke reomeetri (DSR) abil. Doktoritöös jõuti järeldusele, et madala temperatuuri toimivuse määratlemisel tuleks kasutada EBBR meetodit referentsina, kuid T(30°) parameetrit saab edukalt kasutada ligikaudse hinnangu andmiseks.

Üks täiendav puudus Euroopas kehtestatud bituumenite nõuete osas on väikene tähelepanu bituumenite vananemisele. Hetkel on nõuetesse kaasatud ainult lühiajaline vananemine ja pikaajalisele vananemisele tähelepanu ei pöörata. Doktoritöös tehakse ettepanek kasutada T(30°) parameetrit, et mõõta bituumenite vananemist. Kuna T(30°) kasutab DSR-iga mõõdetud faasinurka, siis on see meetod edukalt suuteline määrama bituumeni viskoossuse ja elastsuse suhet.

Selle teadustöö peamised eesmärgid on järgmised:

- Hinnata erinevaid uusi meetodeid madala temperatuuri toimivuse määratlemiseks.
- Kinnitada Fraassi murdumistäpi probleeme täpsusega.
- Välja selgitada, kas T(30°) parameeter on suuteline asendama EBBR katset.
- Uurida, kas T(30°) parameetrit on võimalik rakendada bituumenite vananemise mõõtmiseks.

Kõige eelneva uurimiseks katsetati 89 bituumeni proovi. Uuringu esimeses osas olid kaasatud bituumenid üle kogu maailma, et hinnata erinevate meetodite sobivust bituumenite madala temperatuuri toimivuse määramiseks. Uuringu teine pool keskendus kitsalt Kirde-Euroopa bituumeniturule. Enamus katsetatud bituumeniproove oli võetud mahutitest, kuid sideaine vananemise uurimise osas olid kaasatud ka asfaltsegudest ja puurkehadest eraldatud bituumenite proovid.

Lisaks juba eelmainitud meetoditele kasutati ka enamlevinud bituumenite katsemeetodeid nii Euroopa nõuetest (näiteks nõelpenetratsioon ja pehmenemistäpp Kuuli ja Rõnga meetodil) kui ka USA Superpave süsteemist (näiteks kõrge, keskmine ja madal toimivustemperatuur).

Käesoleva doktoritöö peamised leiud on järgmised:

1. T(30°) parameeter on sobilik meetod bituumenite madala temperatuuri toimivuse ligikaudseks määramiseks.
2. T(30°) parameetriga on võimalik edukalt jälgida bituumenite vananemist.
3. T(30°) parameeter suutis avastada bituumenite kvaliteedi languse, samal ajal kui Euroopas hetkel rakendatud metoodikad seda ei suutnud.
4. Fraassi murdumistäpi ebausaldusväärsus leidis kinnitust ning selle väljavahetamine on põhjendatud.

Appendix 1

Publication I

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Comparison of performance-based specification properties for asphalt binders sourced from around the world

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HIGHLIGHTS

- Asphalts rank rather differently according to Fraass, Superpave, and phase angle protocols.
- T(δ = 30°) correlates strongly with the extended BBR limiting grade temperature.
- Athabasca, Cold Lake, and Laguna viscoelastic binders provide superior performance.

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ABSTRACT

This paper documents and discusses performance-based specification properties of 36 asphalt binders. Materials were obtained from commercial sources and purposely distilled from Alberta oil sands crude oils (Athabasca, Cold Lake). Binders were evaluated according to regular Superpave™ specifications, Fraass test, and enhanced protocols based on limiting phase angles and complex moduli. From an overall performance perspective, sol-type binders made from Athabasca and Cold Lake, Alberta and Laguna, Venezuela crudes outperform gel-type ones made from Chinese, Indian, Kuwaiti, Russian, and South Korean sources. Good amounts of asphaltenes together with low wax contents appear to be critical for the production of high quality and durable asphalt binders.

1. Introduction

Flexible pavements are produced from aggregates of different sizes which are held together by asphalt binder (also known as asphalt cement or bitumen). It is generally accepted that the binder is the component of the mixture that has the most important impact on a pavement's resistance to thermal cracking. This is the reason why it is crucial to set proper acceptance specifications for asphalt binders in order to obtain a satisfactory and balanced performance during service life.

In Europe, the most common materials used in road construction are paving grade and polymer modified binders. All member countries of the European Union have to specify these according to European standard EN 12,591 [1] for paving grade binders, and according to EN 14,023 [2] for polymer modified binders. These standards provide a framework comprised of different test methods of which some are compulsory for the specification, but others can be optional depending on the region. The latter contains the only cold temperature test method—the Fraass breaking point temperature. In other words, the Fraass breaking point is currently the only specification European countries are allowed to use to control thermal cracking of asphalt pavements. However, there are recent developments towards more performance based specifications as standards EN 12,591 [1] and EN 14,023 [2] are in the process of being revised. The first of these revisions has made it to a final draft FprEN 12,591 [3] in 2017, but the proposal has been abandoned and apparently no new initiatives have been started to replace the current 2009 version of the standard. The polymer-modified bitumen standard has reached the final draft status prEN 14,023 [4] and is currently under public review.

It is widely known that the historical Fraass test has many shortcomings. One important problem with the method is that the repeatability and reproducibility are poor for unmodified binders and even worse for modified binders [5]. Migliori and coworkers even concluded that the penetration (pen) and ring & ball softening point temperature ($T_{R\&B}$) tests, both meant to control high temperature performance, are more useful for the prediction

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of cracking in comparison to the Fraass breaking point [6]. Another essential flaw from a design specification standpoint is that the test is done on unaged binder. Binders are known to age at different rates [7], so it is rather important to test low temperature properties on appropriately aged material to separate the poor from the good quality materials. These shortcoming were noticed in North America in the 1980s and a research program was funded which resulted in a new specification system that became known by the Superpave™ acronym (which stands for Superior Performing Pavements).

Among others, a low temperature test called the Bending Beam Rheometer (BBR) protocol was developed as part of Superpave [8,9]. This method was further refined through research in Ontario, Canada, to account for the effects of isothermal conditioning. This new approach was eventually published and implemented for acceptance of the asphalt binder around Ontario as laboratory standard LS-308 [10]. The improved protocol was named the Extended Bending Beam Rheometer (EBBR) test. The extended method has a key advantage over the regular BBR as it takes into account the phenomenon of reversible aging [11]. This method has a much improved correlation with cracking onset and severity in the field [12,13], but also has its disadvantages. EBBR grading takes at least three days, a considerable amount of binder, and significant effort by a technician conducting the tests.

The primary objective of the current/ongoing research is to develop a testing protocol that is as good as, or better than, the EBBR test to differentiate good performing binders from poor performers, but one that needs much less binder, time and effort. A more practical test is desirable as it will allow for a more easy adoption by user agencies of asphalt binder. In this respect, the Dynamic Shear Rheometer (DSR)—currently used under Superpave to determine the high and intermediate temperature properties of the binders—is further assessed to control cold temperature cracking. This equipment needs only a small quantity of binder and most tests can be conducted in a matter of hours. Limiting phase angle temperatures are compared with EBBR results. Complex moduli are assessed at the limiting phase angle temperatures, T(δ). A secondary objective of this research is to compare the quality and durability attributes of purposely-distilled Alberta oil sands-derived binders (Athabasca and Cold Lake) with those for materials sourced from around the world. Binders used in this research were obtained from Belarus, Canada, China, India, Kuwait, Poland, South Korea, Spain, and Sweden.

2. Background

The reversible aging term is used here to describe processes such as wax crystallization, free volume collapse, and asphaltene aggregation [11]. It is important to note that reversible aging occurs at cold temperatures and that the effect can be eliminated by reheating the binder. Throughout this paper the phenomenon will be called reversible aging but different researchers have used other terms to describe it. One of the first to write about this in considerable detail was Traxler who called it “age hardening” [14]. Brown and coworkers called it “steric hardening” [15]. But today it is often described as “physical hardening” [16]. Whatever its name, it is an important aspect to be considered when specifying asphalt binders because otherwise the same might happen as what happened with Superpave, where according to the specification all is fine but in service some binders do not perform as expected [12].

The current European low temperature binder specification using the Fraass test also fails to account for reversible aging. This might be one of the reasons why there is almost no correlation between the Fraass breaking point and BBR or extended BBR results [17]. Of course another reason is that while the BBR and extended BBR are low strain rheological tests, the Fraass breaking point is a failure test.

Asphalt binder is a viscoelastic material, but the viscous (sol) and elastic (gel) components change with temperature. The elastic component is described by the storage modulus while the viscous component is reflected in the loss modulus. The parameter that links these two is the phase angle. A 45° phase angle is the point where a binder transitions from a more viscous state to a more elastic state or vice versa. A lower phase angle means the binder has a more elastic response (a phase angle of 0° would be for materials that are entirely elastic such as metals or a rubber spring). A higher phase angle means that the material is more viscous (a phase angle of 90° would be for a 100 % viscous material such as water or oil). Lower phase angle materials are more susceptible to reversible aging.

Early research by Migliori and coworkers found that the temperature where the phase angle equals 45°, i.e. the Viscous to Elastic Transition (VET) temperature, correlated strongly with wheel path cracking observed on several sites in France [6,18]. This approach was further developed by Widyatmoko and coworkers, who added the complex modulus at the VET temperature, G^*_{VET}, to the analysis [19]. In their later work with several case studies, it was discovered that poor performing sites in terms of cracking had binders with higher VET temperatures and lower complex moduli at those temperatures [20,21]. It is thought that more elastic binders are less likely to heal after microcracks form through fatigue loading or cold temperature exposure.

In this study the temperatures where the phase angle reaches 30° and 45° are investigated as improved low temperature ranking criteria. At around the freeze–thaw temperature (0°C) typical binders used in northern climates reach a phase angle of 27° or 28° (which we round to 30). Migliori et al. [18] and Soleimani et al. [22] found there to be a very good correlation between the temperature where the phase angle reaches 27° or 28°, respectively, and the cold temperature cracking severity. Hence, these two temperature should have particular relevance for cracking and, in comparison with the BBR protocol, are burdened somewhat less by the effects of reversible aging [23,24].

3. Experimental

3.1. Materials

In this research, a total of 36 asphalt binders were sourced from around the world. A number of binders were commercially produced in large refineries in Canada, or purposely distilled from Albertan crude oil sands bitumen. Many other binders investigated were commercially produced in different Baltic nations, China, India, Kuwait, and South Korea. Depending on their origin, binders were either Superpave or penetration graded. Pertinent information for all materials is provided in Table 1.

The samples were either obtained straight from refineries or from asphalt and emulsion plant storage tanks. The Canadian binders were produced from Albertan oil sands-derived crude bitumen. The Polish and Belarusian binders were most likely made from straight Russian crude, known to be moderately high in waxes. The Swedish binder was made from Laguna, Venezuelan heavy crude, known to be very low in wax. Kuwait uses predominantly local heavy crude. The sources for the binders from China, India, South Korea, and Spain were unknown but likely blends of heavy and light crudes from around the world. This study expands on earlier work on a comparison of Canadian and Northern European binders [17].

Table 1
Pertinent Binder Information.

Code	Source	Production Technology	Commercial Grade
1	Athabasca	Paraffinic Froth Treatment	n.a.
2	Athabasca	Paraffinic Froth Treatment	n.a.
3	Athabasca	Steam Assisted Gravity Drainage	n.a.
4	Athabasca	Conventional Heavy Oil Production with Sand	n.a
5	Athabasca	Steam Assisted Gravity Drainage	n.a.
6	Cold Lake	Cyclic Steam Stimulation	300–400 pen
7	Cold Lake	Cyclic Steam Stimulation	PG 58–28
8	Cold Lake	Cyclic Steam Stimulation	PG 64–22
9	Kuwait	Conventional Heavy Oil Production	50–70 pen
10	Kuwait	Conventional Heavy Oil Production	160–220 pen
11	Kuwait	Conventional Heavy Oil Production	250–330 pen
12	China	Conventional Oil Production	70–100 pen
13	China	Conventional Oil Production	50 pen, > 90 % ER, >70 T(R&B)
14	China	Conventional Oil Production	70–100 pen
15	China	Conventional Oil Production	70–100 pen
16	South Korea	Conventional Oil Production	70–100 pen
17	India	Conventional Oil Production	50–70 pen
18	Poland	Conventional Oil Production	70–100 pen
19	Poland	Conventional Oil Production	160–220 pen
20	Poland	Conventional Oil Production	160–220 pen
21	Belarus	Conventional Oil Production	160–220 pen
22	Poland	Conventional Oil Production	70–100 pen
23	Belarus	Conventional Oil Production	70–100 pen
24	Sweden	Conventional Heavy Oil Production	70–100 pen
25	Spain	Conventional Oil Production	70–100 pen
26	Spain	Conventional Oil Production	50–70 pen
27	Cold Lake	Steam Assisted Gravity Drainage	n.a.
28	Cold Lake	Steam Assisted Gravity Drainage	n.a.
29	Cold Lake	Steam Assisted Gravity Drainage	n.a.
30	Cold Lake	Steam Assisted Gravity Drainage	n.a.
31	Cold Lake	Steam Assisted Gravity Drainage	n.a.
32	Cold Lake	Steam Assisted Gravity Drainage	n.a.
33	Athabasca	Steam Assisted Gravity Drainage	n.a.
34	Athabasca	Steam Assisted Gravity Drainage	n.a.
35	Athabasca	Steam Assisted Gravity Drainage	n.a.
36	Athabasca	Steam Assisted Gravity Drainage	n.a.

n.a. = not commercially available so purposely distilled for this research project.

3.2. Accelerated laboratory aging

The samples were aged with two methods. First, the Rolling Thin Film Oven Test (RTFOT) was used to age binders for 85 min at 163°C with 35 g of binder in each tube [25]. The RTFOT is supposed to simulate aging that occurs during mixing, transporting, and laying of the asphalt. After RTFOT, binders were aged in the Pressure Aging Vessel (PAV) for 20 h at 100°C under a dry air pressure of 2.08 MPa [26]. The PAV is supposed to simulate the aging of binders during 8–10 years of service in the pavement.

3.3. Dynamic shear rheometer and bending beam rheometer test

Unaged and aged samples were tested with the DSR according to the regular Superpave grading protocol to determine the high temperature grade denoted by XX and the intermediate temperature grade denoted by II. These were determined with DHR rheometers from TA Instruments. The low temperature grade denoted by YY was determined using a Thermo-Electric Bending Beam Rheometer (BBR) made by Cannon Instruments. All grades were determined in accordance with AASHTO standard M 320 [27].

The DSR results obtained on the PAV-aged binders were further analyzed to calculate the temperatures at which the phase angle equals 30° and 45°. Data obtained at various temperatures at 10 rad/s were used to interpolate or, on a few occasions, extrapolate to 30° and 45°. It is preferred to have higher rather than lower phase angles at low temperatures so that the viscous component is as high as possible allowing shrinkage stresses to relax before they cause irreversible damage [28].

The temperature where the complex modulus equals 60 MPa was similarly found by interpolation. Samples that needed extrapolation were excluded as significant deviations from the true value could occur. These temperatures were also obtained for PAV-aged materials tested at different temperatures.

In addition, stiffness at phase angles of 30° and 45° was found by drawing an exponential trend line through the temperature versus complex modulus data. As the temperatures corresponding to phase angles 30° and 45° were already known, the trend line's equation was used to calculate the corresponding stiffness.

3.4. Extended bending beam rheometer test

The Limiting Low Temperature Grade (LLTG) after 72 h of cold conditioning was determined for each binder according to the extended BBR method embodied in LS-308 [10]. In this method 12 binder beams were tested at pass and fail temperatures. Half of the beams were conditioned at 10°C and the other half at 20°C above the pavement design temperature for 1, 24 and 72 h. The warmest of the limiting temperatures, where the creep stiffness at 60 s of loading, S(60), reaches 300 MPa, or the logarithmic creep rate at 60 s, m(60), reaches 0.300, determines the LLTG.

3.5. Fraass breaking point test

The low temperature properties of binders are specified in Europe with the Fraass breaking point test [29]. To determine the Fraass breaking point, a thin metal plate was coated with a film of binder. The plate was placed in a bending apparatus which was surrounded by a chamber that was cooled at a specified rate. The cooling rate used for this test was 1 °C/min. After each minute of cooling, the plate was bent to a certain degree and the temperature at which the first crack appeared is called the Fraass breaking point. It should be noted that the test is conducted on unaged binder. The Fraass breaking point temperatures for the binders in this study were determined with an Anton Paar BPA-5 instrument.

4. Results and discussion

4.1. Superpave grading

All binders were first tested according to the regular Superpave grading protocols to determine their performance grade, PG XX−II−YY. The temperature where the complex modulus divided by the sine of the phase angle ($G^*/\sin\delta$) equals 1.0 kPa was calculated for the unaged binders. For the RTFO aged binders the $G^*/\sin\delta$ limit is 2.2 kPa. The high temperature (XX) of a binder is determined by whichever of the two limiting temperatures is lower. The intermediate temperature (II) is determined by the temperature where the $G^*\sin\delta$ equals 5.0 MPa. This is measured on PAV-aged material. The PAV-aged material is also used to determine the low temperature performance grade (YY). By means of the BBR, the temperatures where the stiffness equals 300 MPa and the m-value equals 0.300 are calculated. The warmer of these two determines the low temperature grade (YY) of the binder. The test results are presented in Table 2.

As can be seen from the findings, these asphalt binders are meant for very different climatic conditions. The high temperatures vary from 79.0°C for what must have been a modified Chinese binder to 44.1°C for a purposely-distilled Alberta binder.

Table 2
Superpave Grades.

Code	XX, °C	II, °C	YY, °C
1	52.7	15.0	−30.3
2	54.9	19.6	−25.4
3	54.2	14.5	−31.5
4	48.5	7.1	−35.4
5	62.7	19.8	−26.2
6	47.9	9.6	−37.1
7	60.0	16.6	−30.6
8	66.0	23.3	−27.5
9	67.3	26.1	−24.4
10	55.8	15.0	−26.7
11	51.5	10.7	−30.2
12	66.0	23.5	−26.1
13	79.0	20.2	−28.6
14	66.0	24.4	−26.3
15	63.5	23.3	−24.7
16	68.1	23.3	−25.4
17	59.6	20.1	−26.5
18	65.0	23.6	−26.6
19	52.0	16.6	−29.0
20	54.1	14.7	−28.9
21	53.2	11.2	−30.9
22	60.0	17.5	−28.3
23	62.6	14.4	−30.6
24	65.4	19.8	−26.2
25	62.5	21.0	−27.2
26	66.0	20.9	−25.9
27	44.1	3.5	−41.0
28	51.0	7.2	−35.7
29	54.9	13.1	−32.9
30	58.5	15.4	−31.6
31	61.9	15.6	−30.4
32	64.3	19.0	−28.6
33	56.5	13.4	−30.7
34	57.7	13.7	−30.0
35	60.4	16.5	−28.8
36	64.8	20.9	−26.9

Intermediate grades ranged from a low of 3.5°C to a high of 26.0°C. Similarly, the low temperatures range from −41.0°C for the previously mentioned Alberta binder to −24.4°C for a somewhat harder binder from Kuwait. Since this is a wide range of binders produced from crudes from around the world, it is expected that the correlations found in this study are likely applicable to other bituminous materials that are not assessed in this project.

Another useful way of comparing binders is by looking at their grade spans, XX−YY. It is thought that a wider grade span is preferable as the binder should perform well over a wider range of temperatures. It is a fact that binders with wider grade spans are sold at higher prices. The grade spans for these binders are presented in Fig. 1. The average grade span according to Superpave is 88.7°C with a standard deviation of 5.1°C.

Binder 13 is an extraordinary one with a grade span of over 107°C, but it should be noted that this is achieved through modification. In this research, those binders with a grade span lower than the average minus one standard deviation are considered less valuable. In this respect, Binders 1, 2, 10, 11, 19, and 20 miss the limit of 83.6°C. At the same time it should be noted that these binders still might fulfill the requirements for certain climatic conditions but they have a narrower temperature range where they perform as required.

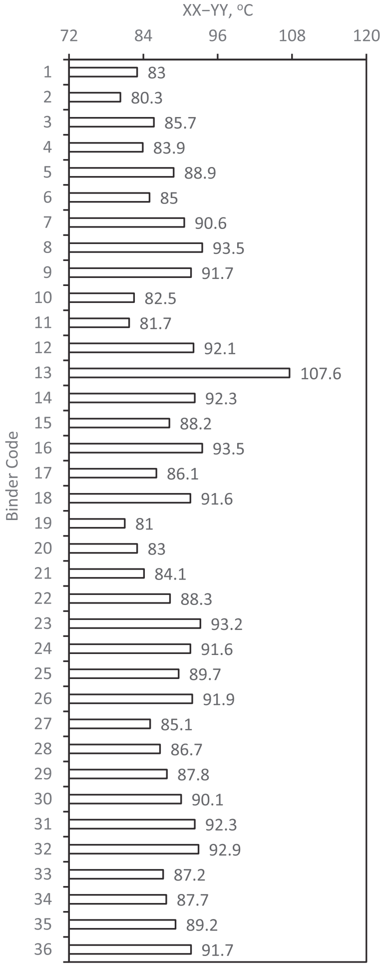

Fig. 1. Superpave grade spans.

4.2. Extended bending beam rheometer testing

The EBBR test was conducted on all the materials to assess the binders' tendency for reversible aging (physical hardening). This test has proven to be able to differentiate superior from inferior performing materials [12,13]. The EBBR low temperature grade together with the grade loss are presented in Table 3. Although all grade losses have a negative effect, in this research a grade loss of more than 3.0°C is considered significant. Depending on the climatic conditions, an error of 3.0°C in low temperature grade can degrade the confidence that no damage occurs in any given year by a significant amount, typically from around 98 % to around 70 %.

By analyzing this set of samples, it is clear that the Alberta oil sands-derived materials (Binders 1–5, 7, 27–36) as well as the Venezuelan heavy oil-derived material (Binder 24) perform very well in this respect, likely due to their naphthenic nature and low wax contents [11]. Only two Canadian binders have a grade loss over the set limit of 3.0°C, Binders 6 and 8. One of the Middle Eastern materials (Binder 9) has a significant grade loss of 5.0°C, while the other two from the same source (Binders 10 and 11) have grade losses of 1°C or less. This would suggest that these binders originate from different crudes or are produced in different ways.

Three of the Chinese binder also fail the limit by having grade losses just over 4°C. The single South Korean binder has a grade loss around 6°C, which is very substantial and typically reduces

Table 3
Extended Bending Beam Rheometer Grades and Grade Losses.

Code	YY_{EBBR}, °C	Loss, °C
1	−28.7	−1.6
2	−24.6	−0.8
3	−29.7	−1.8
4	−34.3	−1.1
5	−23.9	−2.3
6	−33.3	−3.8
7	−28.4	−2.2
8	−24.3	−3.2
9	−19.4	−5.0
10	−26.1	−0.6
11	−29.2	−1.0
12	−21.9	−4.2
13	−24.5	−4.1
14	−22.0	−4.3
15	−22.2	−2.5
16	−19.5	−5.9
17	−21.3	−5.2
18	−20.4	−6.2
19	−25.7	−3.3
20	−25.1	−3.8
21	−27.2	−3.7
22	−21.6	−6.7
23	−25.8	−4.8
24	−24.9	−1.3
25	−22.8	−4.4
26	−22.1	−3.8
27	−39.2	−1.8
28	−34.3	−1.4
29	−32.3	−0.6
30	−30.1	−1.5
31	−28.5	−1.9
32	−26.6	−2.0
33	−29.0	−1.7
34	−28.6	−1.4
35	−27.5	−1.3
36	−25.2	−1.7

the confidence level that no damage occurs in any given year from 98 % to around 50 %. Without exception, none of the Russian binders pass the limit and the same goes for the Indian and Spanish materials.

On the positive side, the binder from Laguna, Venezuela (Binder 24) has an insignificant grade loss of only 1.3°C. It is known that the commercial Laguna asphalt is very low in wax and has moderate amounts of lower molecular weight asphaltenes, which both help it achieve such a desirable result. It is most likely that the Athabasca and Cold Lake binders with low grade losses are similar. These previous results once more prove the need to include a binder specification that verifies reversible aging (physical hardening) effects as the regular BBR test does not take this into account.

4.3. Limiting phase angle grading

While the EBBR is a very effective test method to exclude poor performers, its drawback is the amount of binder needed and the time it takes to complete the test. In the current research an alternative method is evaluated. The low temperature grade is specified by the temperature where the phase angle equals 30°, T(30°). As most binders are m-value controlled in the EBBR test and the m-value and phase angle are closely linked, it is expected that these two correlate well [22,30]. The T(30°) results for this set of binders ranges from a low of −13.4°C for Binder 27 to a high of +7.3°C for Binder 9. It has been previously discussed that typical binders used in northern climates have a phase angle of 27–28° at a temperature near 0°C [30]. This set of materials confirms this once more, as the mean T(30°) for these 36 binders was found to be −0.3°C.

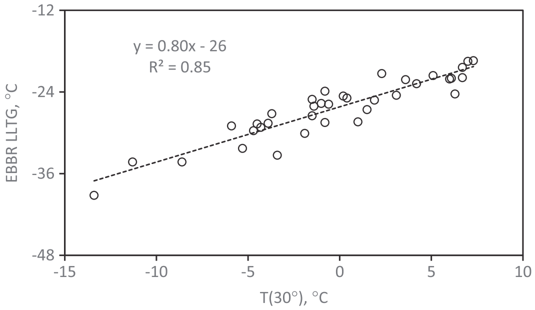

Fig. 2. EBBR limiting low temperature grade and T(30°) correlation.

The linear correlation between the EBBR limiting low temperature grade and T(30°) are presented in Fig. 2. It can be seen that the correlation is strong with a correlation coefficient of R^2 = 0.85. The correlation can be presented as Equation (1). To prove that Equation (1) is suitable, a limit of 3.0°C is set. In this situation we limit the difference between the calculated value and measured value. By analyzing this set of 36 binders, there were only two samples where the calculated and measured temperature deviated from each other by more than 3.0°C. The deviations were 4.4°C and 3.2°C for Binders 6 and 8, respectively. Including these two exceptions, the mean of the deviations is only 1.4°C. The reason(s) for why Binders 6 and 8 deviate more is unclear, but they could relate to testing errors. Hence, T(30°) is a promising parameter to determine a more accurate low temperature grade for asphalt binders.

$$YY_{EBBR} = 0.8 * YY_{T(30^\circ)} - 26 \qquad (1)$$

In the DSR test, which is used to determine T(30°), the samples are equilibrated for only 10 min at each test temperature and this seems to be sufficient to determine a true grade. In the BBR test the samples are conditioned for one full hour yet this does not seem to be sufficient to determine an accurate grade. This can be shown by considering the correlation between BBR low temperature grade YY and T(30°) which is shown in Fig. 3. The correlation is quite a bit lower than for the EBBR with an R^2 = 0.70.

The ability of the T(30°) protocol to predict low temperature field performance is confirmed by two studies. First, the investigations by Migliori and coworkers published in 1999 stated that the cracking severity is highly correlated with the temperature where the phase angle equals 27° [18]. Second, Soleimani et al. [22] found in 2009 that the optimum phase angle cut off was about 28° (tan

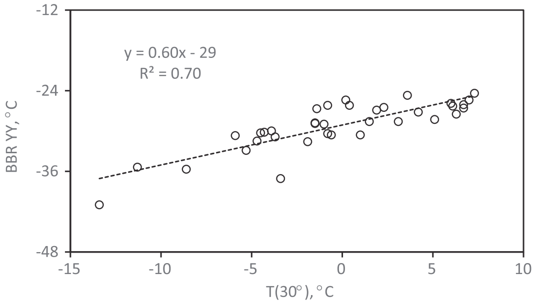

Fig. 3. BBR low temperature grade YY and T(30°) correlation.

(δ) = 0.54). In the current paper the number is rounded to 30. Another reason for why this protocol is suitable is that the correlation between the EBBR LLTG and T(30°) is very high and the EBBR test has been validated earlier [12,13].

4.4. Grade span comparisons and inclusion of complex modulus

Table 4 provides a comparison between the different grade spans as determined by the regular BBR, EBBR, T(30°), and T(45°). The findings show that in this comparison the materials with more elastic behavior (gel and sol–gel type Binders 6, 7, 16, 26, and 31) do relatively better in regular Superpave grading while the materials with viscous behavior (sol-type Binders 1–5, 15, 19, 24, and 33–36) do relatively better in the T(45°) and T(30°) grading.

Another way to reflect this is by calculating the difference between the limiting phase angle temperature and the temperature where the complex shear modulus G* reaches 60 MPa, which we define as ΔT_{cd}, where this stands for the difference between two critical temperatures as measured in the DSR. The ΔT_{cd} is simply a measure of the relative curvature of the rheological master curves (R-value), and thus the viscous (sol) or elastic (gel) type of the binder. Fig. 4 shows these findings for most of the investigated materials (those omitted did not have sufficient data to interpolate accurate T(G* = 60 MPa) values).

The Athabasca materials (Binders 1–5 and 33–36) and the Venezuelan material (Binder 24) do particularly well in this comparison as they are of low rheological index (R-value), low wax content, and moderate asphaltene content. Considering their high temperature grades in Table 2, it shows that these materials will be somewhat softer at high temperatures. Hence, they need to be used in conjunction with good mix designs (angular aggregates), perhaps combined with synthetic fibers or reclaimed asphalt pavement (RAP), to satisfy high temperature rutting performance requirements. Sol-type binders, i.e. those with a positive ΔT_{cd}, are known to perform well in service as shown by earlier research on pavement trials and regular contracts [11,18–21,28,31]. In contrast, gel-type binders are known to suffer more from both thermoreversible and irreversible (oxidative) aging, especially when combined with waxes and RAP [32].

Table 4
Grade Span Comparison.

Sample	XX – BBR YY, °C	XX – EBBR LLTG, °C	XX – T(30°), °C	XX – T(45°), °C
1	83.0	81.4	57.2	44.6
2	80.3	79.5	54.7	45.2
3	85.7	83.9	58.9	43.5
4	83.9	82.8	59.8	45.3
5	88.9	86.6	63.5	51.5
6	85.0	81.2	51.3	30.4
7	90.6	88.4	59.0	40.8
8	93.5	90.3	59.7	42.1
9	91.7	86.7	60.0	46.7
10	82.5	81.9	57.2	43.3
11	81.7	80.7	55.8	41.5
12	92.1	87.9	59.3	43.5
13	107.6	103.5	75.9	59.3
14	92.3	88.0	59.9	45.3
15	88.2	85.7	59.9	46.2
16	93.5	87.6	61.1	45.4
17	86.1	80.9	57.3	45.3
18	91.6	85.4	58.3	42.6
19	81.0	77.7	53.0	29.0
20	83.0	79.2	55.6	42.7
21	84.1	80.4	56.9	41.6
22	88.3	81.6	54.9	38.0
23	93.3	88.4	63.2	45.0
24	91.6	90.3	65.0	51.0
25	89.7	85.3	58.3	42.7
26	91.9	88.1	60.0	43.9
27	85.0	83.3	57.5	40.2
28	86.7	85.3	59.6	42.8
29	87.8	87.2	60.2	43.1
30	90.1	88.6	60.4	43.1
31	92.3	90.4	62.7	45.9
32	92.9	90.9	62.8	45.7
33	87.2	85.5	62.4	47.2
34	87.7	86.3	61.6	46.9
35	89.2	87.9	61.9	46.6
36	91.7	90.0	62.9	48.5

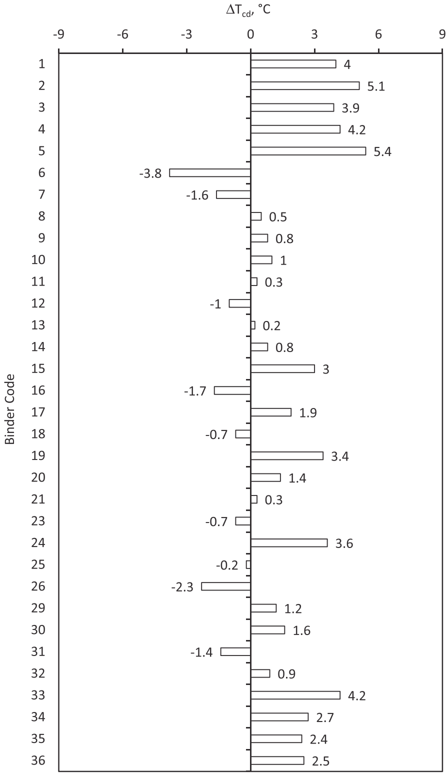

Fig. 4. ΔT_{cd} for temperature where phase angle reaches a critical value of 30° and G* reaches a critical value of 60 MPa.

4.5. Fraass breaking point grading

The Fraass breaking point test results are presented in Figs. 5-8. Only samples 1 to 26 were tested as the other materials were no longer available. The data shows that the Fraass breaking point for these binders covers a range of 11.0°C form a low of −22.0°C to a high of −11.0°C. The average Fraass breaking point was −13.5°C with a standard deviation of 3.0°C, so most binders are very similar in this test. These results were compared to the results of the BBR, EBBR and T(30°). The Fraass breaking point results somewhat correlate with BBR and EBBR low temperature grades with correlation

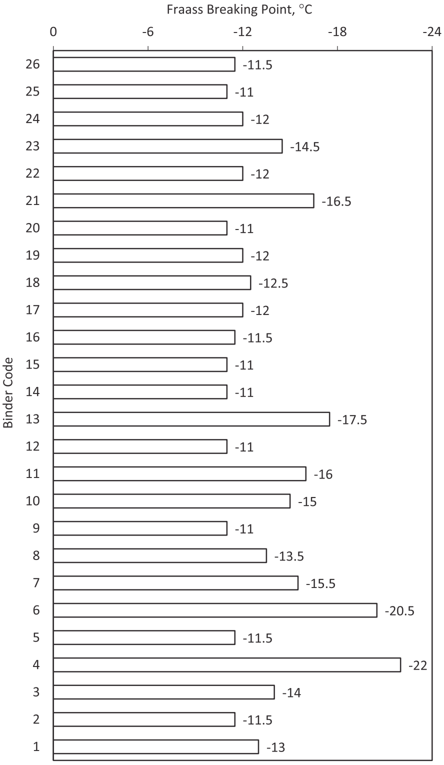

Fig. 5. Fraass breaking point results.

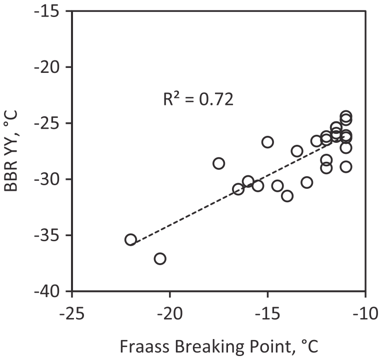

Fig. 6. Comparison of Fraass breaking points with BBR critical temperatures.

coefficients of R^2 = 0.72 and R^2 = 0.67, respectively. At the same time the correlation with T(30°) is very poor with R^2 = 0.44. All this once more confirms the findings from Migliori et al. [6] and others that the Fraass breaking point test is simply a purchase specification with little if any relevance to pavement performance as it has not been proven to correlate with cold temperature distress [30]. In contrast, T(30°) appears to be a preferred specification parameter with a high correlation with pavement performance [6,18,22].

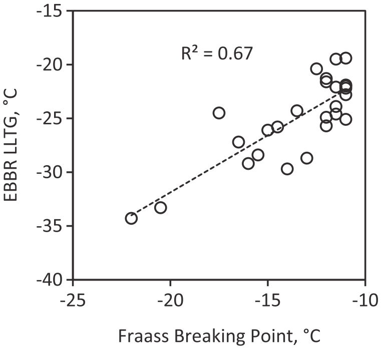

Fig. 7. Comparison of Fraass breaking points (FBP) with EBBR critical temperatures.

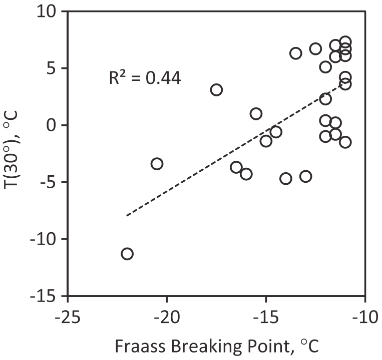

Fig. 8. Comparison of Fraass breaking points (FBP) with limiting phase angle temperature, T(30°).

5. Summary and conclusions

Considering the results and discussion presented, the following summary and conclusions are offered:

- The reversible aging effect can be a significant factor in the accurate performance ranking of asphalt binders for cold temperature cracking.
- The temperature at which the phase angle reaches 30, T(30°), as measured around the freeze–thaw regime after minutes of temperature equilibration, is highly correlated with the extended BBR limiting low temperature grade (EBBR LLTG) after three days of cold conditioning.
- Grade losses over three days of cold conditioning due to reversible aging phenomena range from negligible for highly durable binders that are low in waxes (Athabaca, Cold Lake, Venezuela), to as high as a full grade of 6°C or more for binders made from lesser quality crude oils (Chinese, Indian, Kuwaiti, Russian, South Korean).
- The Fraass breaking point does not correlate well with any of the BBR or DSR properties measured, and hence its relevance for cold temperature pavement cracking remains uncertain.

Given the level of cold temperature pavement cracking problems in North America, Northern Europe and Northern Asia, it is up to government agencies to take the necessary steps to implement improved asphalt cement specification methods that take the above knowledge into account.

CRediT authorship contribution statement

Kristjan Lill: Conceptualization, Data Curation, Analysis, Writing - Original Draft, Review and Editing of Final Manuscript. **Ahmad Khan:** Data Collection, Analysis. Writing - Review of Final Manuscript. **Karli Kontson:** Conceptualization, Data Analysis. Writing - Review of Final Manuscript. **Simon A.M. Hesp:** Conceptualization, Supervision, Data Curation, Writing - Review and Editing of Final Manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix 2

Publication II

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THE APPLICABILITY OF LIMITING PHASE ANGLE TEMPERATURES FOR SPECIFYING ASPHALT BINDER LOW TEMPERATURE PERFORMANCE

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Abstract. The paper discusses the applicability of using limiting phase angle temperatures, measured in the Dynamic Shear Rheometer, for low temperature ranking of performance, in comparison with limiting low temperature grades in accordance with AASHTO TP 122-16 extended Bending Beam Rheometer method. During this study, also other low-temperature test methods were compared to each other. For this purpose, 13 asphalt binders were sourced from around North-Eastern Europe, twelve of which are currently used throughout Estonia as well as the neighbouring countries. The thirteenth was a high-quality Laguna Venezuela binder that is no longer commercially available in the region but was deemed suitable for comparison. Samples were tested to measure their needle penetration, Superpave Grades, Fraass breaking points, AASHTO TP 122-16 limiting low temperature grades and limiting 30° phase angle temperatures. Additionally, a correlation found in previous work was applied to the set of samples studied in this paper. Of the binders tested, the low temperature behaviour of the Venezuelan binder stands out with better performance. The analysis suggests that the twelve commercially available binders are from a similar source which was observed through their tendency

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to undergo thermo-reversible aging. The study shows that the phase angle approach provides a reasonable surrogate for the AASHTO TP 122-16 limiting low temperature grade. However, the latter should currently remain the preferred approach.

Keywords: asphalt binder, Dynamic Shear Rheometer, Extended Bending Beam Rheometer, Fraass breaking point, low temperature performance, phase angle.

Introduction

In Northern Europe, the roads are mostly paved with asphalt mixtures that incorporate asphalt binder as an adhesive to hold the aggregate matrix together. It is known that the aggregate composition is the most important factor to avoid rutting, one of the most frequent pavement distresses that occurs at elevated temperatures. At low temperatures, it is the asphalt binder properties that control the ability of the pavement to avoid low temperature distress, manifesting mostly as transverse cracking.

Paving grade asphalt binders, produced according to EN 12591:2009 (European Committee for Standardization, 2009), are the most common binders used in asphalt mixtures. When an improvement of performance is needed, the paving grade binder can be substituted with polymer-modified binder that is produced according to EN 14023:2010 (European Committee for Standardization, 2010). Both of these standards establish requirements for asphalt binder production, which is required to conform to the CE certification required for distribution within the European market.

Although various regions of Europe have cold winters, there is only one parameter to specify the low-temperature performance of asphalt binders published within the current standards. This parameter is called the Fraass breaking point. Numerous studies (Lill et al., 2019, 2020; Lu et al., 2003, 2017; Lu & Isacsson, 1997; Turk & Tušar, 2018) have suggested that the Fraass breaking point, which is a failure test, should be substituted with a rheology-based test. One of the issues with the Fraass breaking point approach is that the test is conducted on a sample that is unaged. However, the issues related to the low-temperature performance arise after the asphalt binder has been subject to several operational climatic cycles and is subsequently aged (Shaker et al., 2019; Siroma et al., 2021; Aliha & Shaker, 2020; Jing et al., 2020). Despite the test being standardised, reproducibility is also an issue and is highly operator dependent which in itself yields issues related to gross uncertainty (Baumanis et al., 2021; Bueno et al., 2014; Gražulyte & Vaitkus, 2017; Besamusca et al., 2011). A considerable advantage of the Fraass test is the cost of the apparatus, which is relatively inexpensive when using manual or semi-automatic devices.

Working groups within the European Committee for Standardization (CEN) have undertaken extensive work to develop new and improved standards aimed at the asphalt binder producers. The paving grade asphalt binder standard had a revised draft version until 2017 when the work was discontinued, the polymer-modified asphalt binder standard also had a similar fate in 2022. The entirety of the issue is not known to the authors of this paper, but there is an understanding that a mandate (European Commision, 1998) was provided to CEN by the European Commission which listed the essential characteristics of different products. The mandate did not contain any information regarding the rheological performance of asphalt binders. This implies that any rheology-based specification cannot currently be added to the standards and there is unfortunately no opportunity for harmonisation at present. It should be noted that the recalled revised drafts of both EN 12591 and EN 14023 were proposed with specifications similar to Superpave (American Association of State Highway and Transportation Officials, n.d.), widely used in North-America. The revised drafts included methods such as the Dynamic Shear Rheometer (DSR) measurements for high and intermediate temperature performance and Bending Beam Rheometer (BBR) measurements for low-temperature performance. Although the BBR and DSR devices are expensive, they have become common also in European laboratories.

During the past several decades many new low-temperature test methods have been developed. The most effort in developing a new low-temperature specification was perhaps put in the United States in the 1980s and 1990s where a new specification system called Superpave was introduced. Two low-temperature tests were included in the specification, the Direct Tension Test (DTT) and the BBR test. The former is not widely used anymore, but the BBR approach has been implemented in parts of the United States and in other regions around the world where Superpave principles have been adopted. In the Superpave system, asphalt binders are graded according to their Performance Grade (PG). In this system, each geographic location has been assigned a binder grade with a confidence level of at least 95%.

Canadian based researchers further developed the BBR test to include the reversible aging phenomenon, which has a significant effect on some binders (Evans et al., 2011). The phenomenon of reversible hardening is taken into account by conditioning the asphalt binder samples for an extended period of time to let the structure to form. The improved protocol was termed the Extended Bending Beam Rheometer (EBBR) test and was published as the laboratory standard LS-308 and later as AASHTO TP 122-16. Principally the newer method results in a warmer temperature than the regular BBR test if the tested binder is prone to reversible aging. Previous research has shown that the EBBR results exhibit a good correlation with low temperature cracking in the field (Jing et al., 2020; Gražulite, 2019; Hesp et al., 2009; Li & Hesp, 2022). One disadvantage identified with this method is that the samples need to be conditioned for 72 h which means that the tests need to be run over a period of four days. Another is that the method needs a significant amount of binder to conduct the test. For bulk tank samples this is usually not a problem, but it could become an issue for samples that require extraction from asphalt mixtures. Due to the identified disadvantages, the EBBR method has not been widely adopted in the industry. This has driven researchers to develop easier and quicker test methods for low-temperature performance specification. Most commonly the DSR test has been explored as it is already used in many regions for high-temperature testing. Methods adopting the DSR apparatus require a much smaller volume of binder and in most cases the tests can be finished within several hours.

One method for determining the low-temperature performance by means of the DSR is the measurement of the phase angle. Asphalt binders are a viscoelastic substance and therefore both the elastic and time dependent irrecoverable strain components need to be considered. The DSR is capable of measuring the storage modulus that reflects the elastic component and the loss modulus that reflects the viscous component. Phase angle is the parameter that links these two together. The relationships between storage modulus, loss modulus and phase angle are well described by Li and Hesp (2022). A material with a phase angle of 90° is considered to be in a fully fluid state and hence deformation is completely governed by irrecoverable viscous behaviour. Whereas a material with a phase angle of 0° is governed by solid state elastic recoverable behaviour.

One of the first to assess binders via the phase angle were Migliori et al. (1993), Migliori et al. (1999) and Widyatmoko et al. (2005), who looked at temperatures corresponding to a phase angle of 45°. This criterion is known as the Viscous to Elastic Transition temperature (VET). Migliori et al. (1999) and Soleimani et al. (2009) studied the phase angles for different binders at a temperature of 0 °C and concluded that the phase angles ranged between 27° and 28° which was also in agreement with cold temperature cracking in the field. More recently, researchers have looked at how the phase angle can rank asphalt binders related to other low-temperature methods (Angius et al., 2018; Li et al., 2021), as well as the precision of this method (Khan et al., 2020).

Previous research (Lill et al., 2020) assessed the temperatures where the phase angle equalled 30° ($T(30°)$) and EBBR results on a larger set of samples ($N = 36$) sourced from around the world. A correlation was proposed (Equation (1)) with a coefficient of determination $R^2 = 0.85$.

$$YY_{\text{EBBR}} = 0.8 \cdot YY_{T(30^\circ)} - 26. \tag{1}$$

The goal of this study is to assess different test methods that could potentially substitute the troublesome Fraass breaking point test for low temperature asphalt binder specification purposes in cold regions. One potential substitute is the EBBR test, but as it is a time-consuming test an additional goal of the study is to validate the suitability of the previously proposed correlation presented in Equation (1), which uses a quick and reliable DSR measurement for predicting the EBBR results. A fundamental difference between this study and previously conducted research is that the current study focuses solely on the North-Eastern European asphalt binder market. This is necessary for the development of new and improved asphalt binder specifications in this region.

1. Experimental

1.1. Materials

A total of 13 asphalt binders were sourced from different asphalt or emulsion plants in Estonia. Sampling took place between 2020 and 2021. Table 1 presents the binder grades and sampling location. Since

Table 1. Sample information

Sample No.	Penetration grade	Sampling location
1	100/150	Asphalt plant
2	70/100	Asphalt plant
3	160/220	Asphalt plant
4	70/100	Asphalt plant
5	100/150	Asphalt plant
6	100/150	Asphalt plant
7	100/150	Asphalt plant
8	160/220	Emulsion plant
9	100/150	Asphalt plant
10	100/150	Asphalt plant
11	100/150	Asphalt plant
12	100/150	Asphalt plant
13	160/220	Asphalt plant

there are no oil refineries in Estonia, the asphalt binders originate from external refineries. The most common refineries supplying the Estonian asphalt industry with asphalt binders are located in Belarus, Lithuania, Poland, Russia and Sweden. As the binders are delivered to the asphalt mixture or emulsion plant via different intermediate suppliers, it is not always known at which refinery the binder was produced. Also adding to the uncertainty of the binder origin, the oil industry in the whole region is experiencing geopolitical perturbations related to sanctions imposed on some of the crude oil supplying countries, thus making the origins of the crude oil arriving at the refineries even more obscure. It is known that sample No 8 is produced from Venezuelan crude and the rest most likely from Russian crude. Sample No 8 is currently not available on the market, but until recently it has been widely used in the region. All of the studied binders are marketed according to their penetration grade. These range from 70/100, which is the most common within the Estonian market, to 160/220 which is frequently used within the Nordic market. It should be noted, however, that the 160/220 grade has also been used on a selected number of road sections within Estonia due to its low temperature performance.

1.2. Methods

Laboratory aging

Depending on the implemented test, binders with different aging conditions needed to be tested. Laboratory aging was achieved with two methods. Firstly, short-term aging of the sample using the Rolling Thin Film Oven Test (RTFOT), according to EN 12607-1 (European Committee for Standardization, 2014), was conducted for 85 min at a temperature of 163 °C and an air flow of 4.0 l/min. Part of the RTFOT aged sample from each binder was stored for testing and the rest was additionally subjected to long-term aging in the Pressure Aging Vessel (PAV), according to EN 14769 (European Committee for Standardization, 2012), for 20 h at 100 °C under a dry air pressure of 2.1 MPa. The PAV simulates the aging of asphalt binder that occurs during 8–10 years of service life in the pavement. It is stated which sample aging condition was used for conducting each method.

Needle penetration

The needle penetration test method, adopting a Matest semi-automatic penetrometer, was conducted on all of the unaged samples in accordance with EN 1426 (European Committee for Standardization, 2015) in order to check their penetration grade. The method adopted a standard penetration needle with a weight of 100 g which was penetrated into the sample for 5 sec while conditioning the sample at 25 °C.

Fraass breaking point

According to the product standard EN 12591 (European Committee for Standardization, 2009), the Fraass breaking point (European Committee for Standardization, 2015) method is conventionally employed on unaged binders. However, for this study, it was utilised on the RTFOT aged samples. The method involves covering small metal plates with a thin layer of binder and then installing them into the Fraass apparatus to undergo bending at varying temperatures. The Controls manually operated apparatus was adopted for the purpose of conducting the measurements. The temperature was then lowered at a rate of 1 °C/min until cracking of the binder occurred. Visual inspection of the sample to check for cracking was conducted at a resolution of one per minute after each incremental drop in temperature and post plate flexure cycle. At least two samples were tested per binder sample with the mean value allocated as the Fraass breaking point. If the two results deviated from each other by more than 3 °C another sample needed to be tested.

Dynamic shear rheometer test

Unaged and RTFOT aged binder samples were tested using the Anton Paar MCR302 Dynamic Shear Rheometer (DSR). According to the Superpave specifications, the high temperature grades can be determined via this method. Samples that were aged in the PAV were tested at different temperatures to determine the temperature equal to a phase angle of 30°. An angular velocity of 10 rad/s was adopted for the measurements. The temperature range was selected to cover the entire range of interest allowing for interpolation between points where possible. A total of 5 temperature increments was explored, ranging between 34 °C and −8 °C.

Bending beam rheometer and extended bending beam rheometer test

These two tests were done simultaneously on the same sample using the infraTest bending beam rheometer. The AASHTO TP 122-16 method (American Association of State and Highway Transportation Officials, 2016) is an extension of the Superpave method for determining the low-temperature grade. In the Superpave method, the asphalt binder beams are tested after a conditioning time of 1 hour at the desired testing temperatures. In the AASHTO TP 122-16 protocol, the EBBR samples are tested additionally after being conditioned for 24 and 72 hours from the start of the test. For the purpose of this study, the conditioning increment of 24 hours was skipped as it was deemed that the 72-hour increment would yield more valuable data. The temperatures were calculated where the stiffness equalled 300 MPa and m-value equalled 0.300 at a loading time of 60 seconds. The difference between the EBBR and Superpave low-temperature true grades is considered the grade loss.

2. Results and discussion

2.1. Penetration grading and Superpave grading

All of the samples were tested to verify their penetration grade and Superpave grade without the intermediate grade. As per EN 1426, the needle penetration was tested on the unaged binder. The Superpave high-temperature was carried out on both the unaged and RTFOT aged binders, respectively. The temperatures corresponding to the function G*/sinδ equalling 1.0 kPa for the unaged binder and 2.2 kPa for RTFOT aged binder were determined. The Superpave low-temperature was determined using the regular BBR test, where the limiting temperatures according to stiffness and m-value were determined after 1 h of

Table 2. Penetration and superpave grading

Sample No.	Needle penetration, dmm	Superpave performance grade (XX–YY), °C
1	114	52 – 22
2	78	64 – 28
3	175	52 – 28
4	81	64 – 28
5	121	58 – 28
6	125	58 – 28
7	105	58 – 22
8	159	58 – 28
9	129	58 – 28
10	124	58 – 28
11	111	58 – 28
12	126	58 – 28
13	184	52 – 28

conditioning. Depending on the binder, the limits for stiffness and m-value can result in substantially different temperatures, but the highest of the two is used to determine the Superpave low-temperature grade. The results for penetration and Superpave grading are presented in Table 2.

All but one of the samples needle penetration measurements fall within the specification limit according to their respective grade. Only sample No. 8 falls short by 1 dmm. This could be the result of a longer storage time as this sample was taken from the emulsion plant warehouse where it had been stored for a few years in a sealed 20 L bucket. As this study aims to compare different low temperature test methods, then this non-compliance does not mean the removal of the sample from the study.

The Superpave high-temperature grades range from 52 to 64 °C and the low-temperature grades range between −22 and −28 °C. The Superpave high-temperature results are as expected with the penetration grade 70/100 binders resulting in PG 64. Binders with the penetration grade 160/220 are PG 52 except for sample No. 8 which meets the PG 58 criteria. All but one of the 100/150 binders resulted in a PG 58 grade with sample No. 1 being the exception and missing the PG 58 grade by 0.2 °C.

2.2. Low temperature grading according to European specifications, Superpave and AASHTO TP 122-16

The Superpave low-temperature true grades are presented in Table 3. Eleven of the thirteen samples display a low-temperature PG grade of −28. Only samples No. 1 and No. 7 do not conform to this grade, but they fall short by only 0.1 °C. Interestingly, this means that they have a very similar low-temperature performance according to Superpave, despite being classified as three different penetration grades. The true grades in Table 3 show that these samples expand over only 5.8 °C. Table 3 also displays the Fraass breaking point results of the RTFOT-aged residue showing a poor correlation with the Superpave results. This was also observed in previous studies (Lill et al., 2020). It is widely accepted that the softer grade binders are adopted for use in climates with more severe low-temperatures. However, based on the asphalt binder samples studied, it appears that a softer grade does not always exhibit a better low-temperature performance.

Table 4 presents the results of the DSR limiting phase angle measurements together with the EBBR test low-temperature true grades and the grade loss. Grade loss is defined as the difference between the Superpave low-temperature true grade and the low-temperature true

Table 3. Fraass breaking point and superpave low temperature true grade

Sample No.	**Fraass breaking point (RTFOT residue), °C**	**Superpave low temperature true grade, °C**
1	–15	–27.9
2	–17	–28.1
3	–17	–31.4
4	–17	–28.5
5	–17	–30.0
6	–18	–31.0
7	–15	–27.9
8	–18	–33.7
9	–12	–29.9
10	–16	–29.7
11	–15	–30.3
12	–16	–29.2
13	–	–29.7

Table 4. $T(30°)$ and extended bending beam rheometer test results

Sample No.	**$T(30°)$**	**EBBR true grade, °C**	**Grade loss, °C**
1	3.7	–22.4	5.5
2	4.3	–23.4	4.7
3	−0.5	–25.7	5.7
4	4.6	–23.5	5.0
5	1.3	–24.6	5.4
6	1.4	–25	6.0
7	3.4	–22.2	5.7
8	–5.3	–32.5	1.2
9	1.0	–24.4	5.5
10	2.3	–24.1	5.6
11	3.2	–24	6.3
12	2.8	–23.9	5.3
13	1.9	–23.9	5.8

grade after 72 h of conditioning. A higher grade loss means a bigger drop in the confidence level that no damage will occur to the pavement during its operational lifespan. Ontario, Canada based researchers have stated that binders with a grade loss below 3.0 °C should be acceptable and above this limit they should not be acceptable. The data show that all but one sample fail this specification. These measurements imply that longer conditioning times are important when assessing the performance of asphalt binders for use in the Baltic region where severe cold conditions and prolonged exposure can affect the performance. The only sample passing the criteria with a grade loss of 1.2 °C was sample No. 8 from Venezuelan crude. This means that asphalt binders from Venezuelan crude display less reversible aging when compared to the other crude sources. The proposed 3.0 °C limit cannot be used in the North-Eastern European region at the moment as this would result in the restriction of all the currently studied and still available binders.

The $T(30°)$ measurement is a less tedious method compared to the AASHTO TP 122-16 EBBR test approach. Not considering the sample preparation phases, the DSR method takes around 1 hour to finish while the EBBR test extends over a period of four days. As $T(30°)$ is still an experimental low-temperature testing method, there are no standardised industry criteria set at the moment. As the phase angle defines the viscous and elastic components of the tested sample, it can be used for defining binder performance. For instance, at low-temperatures a bituminous binder behaving in a more viscous than elastic state is preferred. Therefore, a lower $T(30°)$ criterion is considered superior. From the results, it can be concluded that sample No. 8, a 160/220 grade binder, has the best low-temperature performance with a $T(30°)$ result of −5.3 °C. Another 160/220 binder (sample No. 3) comes second with a result of −0.5 °C. The two 70/100 grade binders (samples No. 2 and No. 4) show the highest $T(30°)$. All other samples fall in between, with one contradicting result (sample No. 13). This sample is also a 160/220 binder, but has a $T(30°)$ of 1.9 °C which is higher than the results of some of the 100/150 grade samples. This is interesting as the needle penetration for this sample is the highest and it would be expected that the softest binder would have the best low-temperature performance.

2.3. The correlation between low-temperature grading methods

Based on literature and the measured results, it can be concluded that the reference method should be the AASHTO TP 122-16 EBBR test. Furthermore, sample No. 8 displays considerably different results and as it is not currently available on the local market, the following correlations are presented with the data omitted. Where needed, the correlation together with sample No. 8 is reported in addition.

The correlation between the EBBR and Fraass breaking point results are presented in Figure 1. The Fraass breaking point has negligible correlation with the EBBR results with an $R^2 = 0.08$. One of the reasons being that the samples are tested at different stages of aging and the

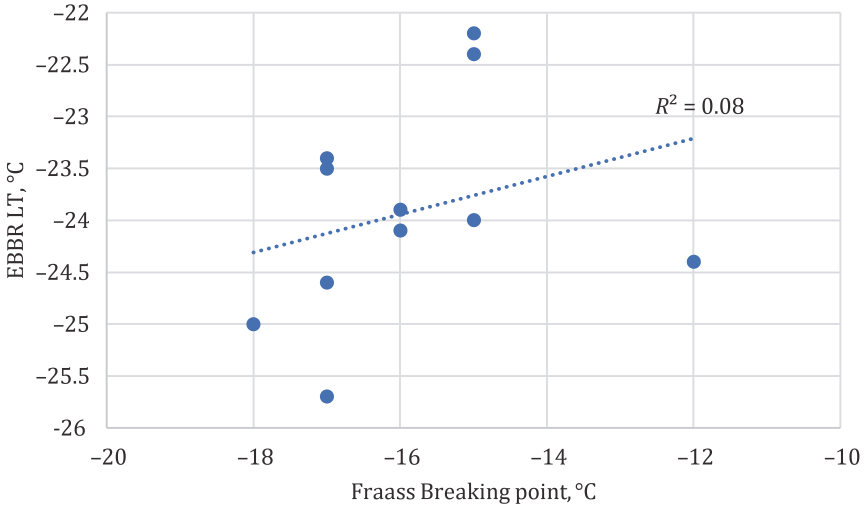

Figure 1. EBBR low temperature vs Fraass breaking point

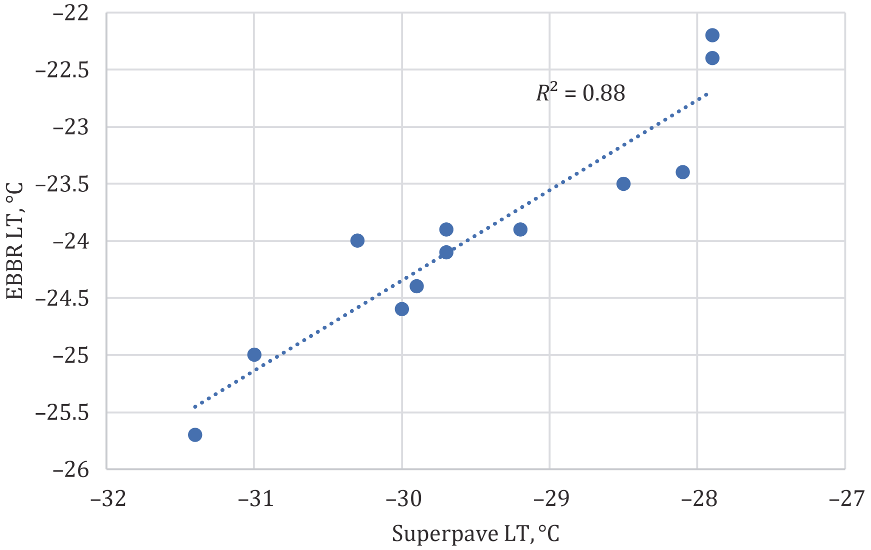

Figure 2. EBBR low temperature vs Superpave low temperature

Fraass breaking point is a failure test, while EBBR is a rheological measurement. This correlation study does not include sample No. 13 as the Fraass breaking point was not measured on this sample.

As can be seen from Figure 2, correlation between the regular BBR and EBBR is high with $R^2 = 0.88$. This does not match previous research conducted (Lill et al., 2020), but this may be due to the number and variation between samples tested. Excluding sample No. 8, the binders

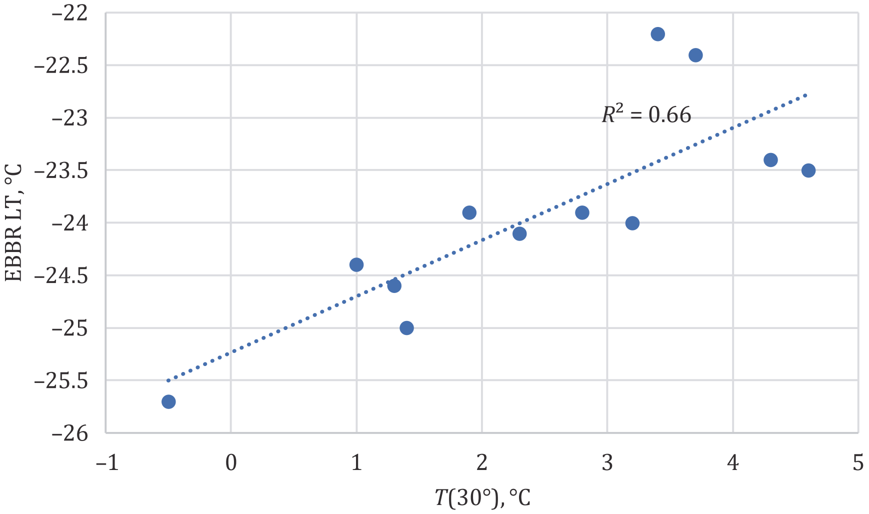

Figure 3. EBBR low temperature vs *T*(30°)

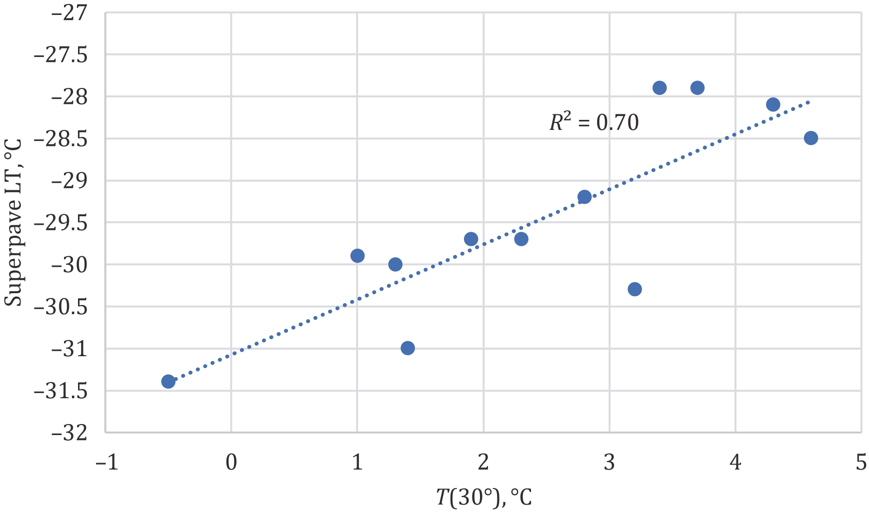

Figure 4. Superpave low temperature vs *T*(30°)

appear to be from very similar crude sources. With the addition of sample No. 8 to the data set, the coefficient of determination (R^2) shows a slight reduction and equals 0.83, which is still a good correlation. This means that in the current market situation in North-Eastern Europe there is no noticeable advantage in using the more cumbersome EBBR method, but in the long run this will lead to grade loss changes going unnoticed.

Figure 3 shows the correlation between the EBBR and $T(30°)$ measurements, whereas Figure 4 demonstrates the correlation between regular BBR and $T(30°)$ measurements. The $T(30°)$ measurement correlates slightly better with regular Superpave BBR results than with the EBBR results. The coefficients of determination are 0.70 and 0.66, respectively. In previous work (Lill et al., 2020), the opposite trend was observed, this might be related to the wider range of binder samples studied. It may be stated that the $T(30°)$ method is satisfactory if the low-temperature performance needs to be approximated relatively quickly. However, the valuable information pertaining to the grade loss would be lost.

2.4. Applicability of the correlation found in previous work

In a previous study (Lill et al., 2020), a correlation between $T(30°)$ and the EBBR methods was found for a larger sample population, which was collected from a more diverse sample pool from around the world. The correlation is presented in Equation (1). Given the limited sample population and poorer correlation within this study, a check was conducted to assess Equation (1) against the EBBR results from the $T(30°)$ data of this sample population. The measured EBBR results and the predicted EBBR values according to Equation (1), as well as the difference is presented in Table 5. It can be seen that for almost all the samples the differences are small, being up to ±1 °C. Sample No. 8 appears to be the exception with a predicted EBBR value that is 2.3 °C lower than the measured. In the case of sample No. 8, the calculated low-temperature performance is lower than measured, meaning that there would be no risk to actual field performance, but there is the issue that the performance of this sample is being underestimated. This could, however, lead to a situation where this binder cannot be used for an asphalt mixture although it would perform adequately. On the other hand, the correlation could also overestimate the performance of asphalt binders. As a result, the use of the correlation is proposed as a crude tool for approximating the low-temperature performance of asphalt binders. Nonetheless, it is still advisable to conduct the EBBR test to obtain an accurate low-temperature performance assessment.

Table 5. Comparison between the measured EBBR results and the predicted values according to Equation (1)

Sample No.	EBBR low temperature true grade, °C	EBBR low temperature true grade prediction according to Equation (1), °C	Difference, °C
1	–22.4	–23.0	0.6
2	–23.4	–22.6	–0.8
3	–25.7	–26.4	0.7
4	–23.5	–22.3	–1.2
5	–24.6	–25.0	0.4
6	–25.0	–24.9	–0.1
7	–22.2	–23.3	1.1
8	–32.5	–30.2	–2.3
9	–24.4	–25.2	0.8
10	–24.1	–24.2	0.1
11	–24.0	–23.4	–0.6
12	–23.9	–23.8	–0.1
13	–23.9	–24.5	0.6

Conclusions

Considering the results and discussion presented, the following conclusions can be made:

- The Superpave high-temperature grade results are as expected with 70/100 penetration grade binders yielding PG 64 and the softer penetration grades 100/150 and 160/220 being divided between PG 52 and PG 58;
- The European low-temperature specification of Fraass breaking point has negligible correlation with EBBR;
- Although the samples are from three different penetration grades, their low-temperature performance according to Superpave is very similar;
- Asphalt binders derived from Venezuelan crude display less reversible aging;
- The BBR and EBBR results correlated well, and it might be explained by a similar origin of the samples;
- Due to a smaller set of samples, the correlation between $T(30°)$ and BBR and $T(30°)$ and the EBBR methods are less pronounced than found in previous work;

– The correlation found in previous work can be used to approximate the low-temperature performance of asphalt binders, but for more accurate results the EBBR should be conducted.

All but one sample were from a similar crude source, the different low-temperature specifications, except the Fraass breaking point, correlated well when focusing on North-Eastern European asphalt binders. Of the studied methods EBBR is the only that considers the reversible aging phenomenon and for this reason, it should be used as the reference method and the $T(30°)$ can be used as a quick approximation of asphalt binder low-temperature performance.

Obviously, these findings are based only on the three penetration grades 70/100, 100/150 and 160/220 obtained from a limited number of suppliers. It would be beneficial to add other grades and also from more suppliers to the studied samples. Additionally, although asphalt binder is the main contributor to the low temperature performance of asphalt pavements, future research could focus on the whole asphalt mixture to include the effects of aggregate and additives.

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Appendix 3

Publication III

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DETERMINING ASPHALT BINDER AGING BY USING LIMITING PHASE ANGLE TEMPERATURE

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Abstract. This paper discusses the applicability of using limiting phase angle temperatures, measured in the Dynamic Shear Rheometer, to assess the aging of asphalt binders. In terms of pavement management, it is of high value for road owners to know the aging condition of the binder present in a pavement. For this purpose, 37 binder samples were collected, including tank samples and others extracted from loose mixture or core samples. Based on these samples, the findings indicate that the T(30°) parameter can successfully be used to measure the aging of asphalt binders. A key discovery was that the T(30°) parameter was able to detect a substantial change in asphalt binder quality which was missed by the current European asphalt binder specification. Newer binders are found to be more elastic than binders used around five years ago. This indicates that the binder in older pavements is of high value and the recycled asphalt pavement obtained from these pavements could even enhance the performance of new mixtures. Additionally, the effect of different aggregate types to the aging of asphalt binders was studied, but no definitive conclusions could be drawn. Furthermore, it was found that, in most cases, the Rolling Thin Film Oven Test resulted in more severe aging of asphalt binder compared to aging in the asphalt

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mixing plant. A correlation between the T(30°) parameter and defects on the pavement was investigated, but correlation was found to be negligible.

Keywords: asphalt binder, binder aging, dynamic shear rheometer, igneous rock, limestone, pavement defects, phase angle, rolling thin film oven test.

Introduction

The most common binder used in pavements today is asphalt binder, and Estonia is no exception. Asphalt binder has been used as the binding agent on approximately 12 000 km of national roads, and Portland cement on only 2 km. Typically asphalt pavements consist of two or three layers of asphalt: base layer asphalt concrete (AC) and surface layer AC or stone mastic asphalt (SMA). If additional bearing capacity is needed, there is a binder course AC between the base and surface layer. The top layer is designed to withstand around 10 years in service and the bottom layers are designed with a life expectancy of 20 years. However, it is not uncommon that the expected lifetime is not met and the pavement deteriorates prematurely. There are numerous reasons for pavement failure, such as loss of bearing capacity of the subgrade, deterioration of base course aggregates due to traffic loads, and other factors. One significant issue is the aging of the asphalt binder. As the binder ages, its stiffness increases, which, on the one hand, provides better resistance to deformation, but, on the other, reduces resistance to fatigue and low-temperature cracking. This is why it is advisable to monitor the aging process also during service life.

Another topic to consider is the development of the oil refining industry. While there have always been empirical requirements for asphalt binders, there have never been strict rules regarding their composition. Consequently, the existing specifications cannot eliminate the possibility of undesirable modifications. The oil refining industry as any other industry continuously advances its production processes to enhance profitability, which can lead to variations in the composition of asphalt binders. On some occasions the changed chemical composition can result in a shortened life-expectancy of the binder, even when the used empirical specifications are met. As the asphalt binder has a huge effect on the performance of asphalt pavements, it is crucial that the binder has adequate performance, because premature pavement failures are costly. At the beginning of the current millennium, researchers (Anderson et al., 2000) in the United States reported that asphalt paving accounted for 0.2% of the country's gross domestic product.

Only recently the Estonian asphalt mixture production market has started incorporating recycled asphalt pavement (RAP) in their mixtures. There has been very little focus on the use of RAP because the specifications for asphalt mixtures are very narrow, and producers do not want to risk fines due to inconsistent RAP. However, the supply of asphalt binder has become more difficult due to geopolitical issues in Eastern Europe (Lill et al., 2023), and an increased focus on sustainability has created the need to use higher amounts of RAP. In Estonia, there are currently only asphalt binder specifications for tank samples, but no requirements for samples extracted from the asphalt mixture. The addition of RAP has necessitated the need to monitor the asphalt binder after it has been mixed into the asphalt mixture and also during service life.

All of the above brings us to the point where we need a quick, easy and reliable method to determine the properties of the binder once it has already been mixed into the mixture. This study focuses on a method where the asphalt binder is extracted from the asphalt pavement and then tested with a Dynamic Shear Rheometer (DSR) to measure the temperature where the phase angle equals 30° (T(30°)). The method can also be used with laboratory-aged materials. The method has been proven to be reliable, with good reproducibility and repeatability (Khan et al., 2020). Previous research has proven that T(30°) can be used to approximate the low-temperature performance of binders (Lill et al., 2023; Li & Hesp, 2022). The aim of this study is to examine whether the T(30°) parameter could be used for monitoring the aging of the binder in the pavement.

1. Background

Aging of asphalt binder

It is known that organic asphalt binders age, which can cause a multitude of pavement defects. This has been a research topic for decades or longer throughout the world (Bell, 1989; Richardson, 1905). There have been comprehensive reviews about binder aging (Ahmad et al., 2024; Hamzah et al., 2015; Wang et al., 2020; Zhang et al., 2020) and specific studies focusing on narrow topics regarding asphalt aging (Gamarra & Ossa, 2018; Kleizienė et al., 2019; Liu et al., 1998; Li et al., 2020; Ma et al., 2021; Sreedhar & Coleri, 2022). Many papers have looked into the possibility of rejuvenating the binder (Kuang et al., 2019; Mohammadafzali et al., 2017; Zhang et al., 2019) or using anti-aging additives (Camargo et al., 2021; Fu et al., 2024; Gawel et al., 2016; Malinowski et al., 2024). Due to the immense number of scientific papers and reports on the topic of asphalt binder aging, it is impossible to familiarise oneself with all of them.

Aging occurring during the production, transportation and laying of asphalt mixture is called short-term aging. During this stage, the primary contributor to asphalt binder aging is the loss of volatiles (Fernandez-Gomez et al., 2013). Additionally, long-term aging of the binder occurs during exploitation in the field. This happens mainly due to the asphalt binder's chemical reaction with oxygen, resulting in oxidation (Liang et al., 2019), but there are other factors like additional loss of volatiles and ultra-violet radiation that causes aging (Hunter et al., 2015).

In both aging stages, the binder becomes stiffer and its viscosity increases, which to some extent is preferable as the mixture containing the aged binder is more durable against permanent deformation. However, at some point, the binder becomes too brittle, making it more susceptible to both low-temperature and fatigue cracking.

While the long-term aging process is controlled by the climatic conditions where the pavement is situated, short-term aging is significantly affected by the mixing temperature in the asphalt mixture production plant. The effect of mixing temperature on the performance of the binder is well described by scholars (Corbett, 1969; Hofko et al., 2017; Liang et al., 2019), where the Rolling Thin Film Oven Test (RTFOT) was used to simulate short-term aging. To simulate the long-term aging, the Pressure Aging Vessel (PAV) was developed, which was expected to simulate aging that occurs during 8 years of exploitation (Bahia & Anderson, 1995).

Measurement of aging

In the following sections, different methods for determining the aging of asphalt binder are discussed.

Asphalt binders consist of two main components: asphaltenes and maltenes. Maltenes are further divided into saturates, aromatics and resins (Tauste et al., 2018). Together, these are called the SARA fractions. One method of measuring the aging of asphalt binder is by this chemical composition. During short-term aging, the saturate fraction decreases due to the loss of volatiles, while during long-term aging they remain unchanged (Liang et al., 2019). As aging progresses, aromatics convert into resins, and resins into asphaltenes, leading to the most significant increase in the asphaltene fraction (Liang et al., 2019). This method for monitoring asphalt binder aging was developed in the 1960s (Corbett, 1969).

Another chemical measure used for analysing the aging of asphalt binder is the investigation of the carbonyl area index. This has been used in many studies as a measure of asphalt binder aging, but research in Louisiana has found correlation between the carbonyl area index and both transverse and alligator cracking (Islam et al., 2024).

Since the early 1990s, following the conclusion of the SHRP program, the DSR has been widely utilised to study the performance of asphalt binders. Initially it started in the United States, but it is now used globally. Even in countries without specific binder specifications for DSR measurements, most research facilities specialising in asphalt binder studies utilise this apparatus.

The DSR was first used to measure the high and intermediate temperature properties of asphalt binders. However, due to its versatility and the small amount of material required for testing, the device has become widely used for assessing various parameters. Many have used the DSR for testing the fatigue resistance of asphalt binders. Most have used time sweep tests where the sample is loaded cyclically for long periods of time until a drop in complex modulus is achieved (Hintz & Bahia, 2013).

In the pursuit of quicker testing methods, significant efforts have been made at Technische Universität Braunschweig, where the Accelerated Dynamic Shear Rheometer Fatigue Test (ADFT) was developed (Kim et al., 2021). Additionally, the same university has also worked on the Binder-Fast-Characterization-Test with the abbreviation BTSV, derived from the German title "Bitumen-Typisierungs-Schnell-Verfahren" (Schrader & Wistuba, 2019). This method has been used for monitoring the aging of asphalt binder at high temperatures.

Another university that has extensively studied the effect of aging on asphalt binder is the University of Nottingham. They have found it is crucial to monitor the aging across the whole temperature spectrum that the binder will endure during its service life (Hu et al., 2023). Regarding fatigue cracking, they refer to the Glover-Rowe parameter (Rowe et al., 2014), which is calculated from DSR frequency sweep data. Rowe et al. (2014) proposed limits of 180 kPa as the onset point of fatigue failure and 450 kPa as the significant propagation point of failure.

Effect of aggregate type on binder aging

Estonia is situated in an area where locally available aggregates consist mainly of limestone, gravel, and sand. However, due to the allowance of studded tires and the use of NaCl for de-icing, the surface layers of asphalt require igneous rock. Local limestone and gravel are commonly used in base course asphalt mixtures. This has started the discussion if the different types of aggregate have an effect on the aging of asphalt binder.

Anderson et al. (1994) stated that aggregate with smaller adsorption of highly polar fractions, such as granite, show higher catalytic effect compared to aggregate with higher adsorption, like limestone. This effect was supported by Wu et al., (2014) who found that binders extracted from mixtures with limestone aggregate showed lower stiffness than those from mixtures with granite aggregate. Additionally, Petersen et al. (1974) discovered that the adsorption of certain polar components onto limestone aggregate might be irreversible, remaining on the aggregate even after extraction.

Furthermore, Wu et al. (2014) mentioned the possibility of oily fractions being absorbed into the aggregate and thereby being protected from oxidation. Consequently, it is expected that the binder in mixtures with limestone aggregate ages slower compared to those with granite.

Viscoelasticity of asphalt binders

Asphalt binders are viscoelastic materials, which means that they have both viscous and elastic responses at the same time. When talking about oscillatory rheology testing then it is the phase angle that is the parameter that is able to distinguish the proportions between viscous or elastic responses (Hunter et al., 2015). A material with a phase angle of 0° is completely elastic and a material with a phase angle of 90° is absolutely viscous. Depending on the binder, its aging condition and the testing temperature, the measured phase angle will lie within these extents.

As a binder ages, the viscous and elastic proportions are altered. With the increase in aging a binder becomes more elastic and less viscous, i.e., the phase angle drops when measuring at the same temperature. Because the T(30°) parameter is based on the phase angle, it is highly likely that it can be used to monitor the aging of binders, thus becoming the subject of this research.

2. Experimental

2.1. Materials

The samples tested in this research were all asphalt binders. Some of them were collected as tank samples from asphalt mixing plants. Other samples were extracted from asphalt mixtures that were taken from the pugmill of the asphalt paver. Some binders were extracted from asphalt core samples. All the asphalt mixture samples were taken from mixes

Table 1. Sample details

Sample no.	Site	Mixture	Rock type	Year of construction	Pavement age	Penetration grade	Origin
1	T92 Kanaküla	AC 16 surf	Igneous	2020	-	100/150	Tank
2					-	100/150	Mixture
3	T65 Veriora (ref)	AC 16 surf	Igneous	2020	-	70/100	Tank
4					-	70/100	Mixture
5					1	70/100	Core
6	T65 Veriora (soft)	AC 16 surf	Igneous	2020	-	160/220	Tank
7					-	160/220	Mixture
8					1	160/220	Core
9	T92 Rihkama	AC 16 surf	Igneous	2020	-	70/100	Tank
10					-	70/100	Mixture
11	T52 Metsla (base)	AC 20 base	Limestone	2020	-	100/150	Tank
12					-	100/150	Mixture
13					1	100/150	Core
14	T52 Metsla (surf)	AC 16 surf	Igneous	2020	-	100/150	Tank
15					-	100/150	Mixture
16					1	100/150	Core
17	T60 Nätsi	AC 16 surf	Igneous	2020	-	100/150	Tank
18					-	100/150	Mixture
19	T52 Holstre (base)	AC 20 base	Limestone	2021	-	100/150	Tank
20					-	100/150	Mixture
21	T52 Holstre (surf)	AC 16 surf	Igneous	2021	-	100/150	Tank
22					-	100/150	Mixture
23	T19201 Vahenurme (bin)	AC 12 bin	Limestone	2021	-	100/150	Tank
24					-	100/150	Mixture
25	T19201 Vahenurme (surf)	AC 16 surf	Igneous	2021	-	100/150	Tank
26					-	100/150	Mixture
27	T81 Käina	AC 16 surf	Igneous	2021	-	160/220	Tank
28					-	160/220	Mixture
29	T6 Riitsaare	AC 16 surf	Igneous	2016	4	100/150	Core
30	T6 Kilingi-Nõmme	AC 16 surf	Igneous	2015	5	70/100	Core
31	T6 Atika	AC 16 surf	Igneous	2016	4	70/100	Core
32	T55 Mõisaküla	AC 20 surf	Igneous	2017	3	160/220	Core
33	T52 Viiratsi (base)	AC 20 base	Limestone	2016	5	70/100	Core
34	T52 Viiratsi (surf)	AC 16 surf	Igneous	2016	5	70/100	Core
35	T52 Kangilaski	AC 20 surf	Igneous	2016	5	160/220	Core
36	T11152 Kirdalu	AC 20 surf	Igneous	2018	3	160/220	Core
37	T11240 Kiisa	AC 16 surf	Igneous	2018	3	70/100	Core

that contained the binder sample taken from the tank. For sites which have data from the tank, mixture and drill cores, the drill cores were sampled approximately one year after construction. Basic details about the samples are presented in Table 1. It is also known that no mixtures included in the study contained RAP.

2.2. Methods

Binder extraction and recovery

The binders from both the loose mixture samples and core samples were extracted with dichloromethane using a semi-automatic extraction device according to EN 12697-1 (CEN, 2020). The solution of asphalt binder and dichloromethane was collected and subsequently added to a rotary evaporator to remove the solvent from the binder. This was done according to EN 12697-3 (CEN, 2018).

Laboratory aging

Both short-term and long-term aging were used to condition the samples. The short-term aging was done by the RTFOT test according to EN 12607-1 (CEN, 2014), where 35 g of binder are poured into class bottles and the bottles were rotated in the RTFOT oven at 163 °C for 85 min with an air flow of 4.0 l/min. Depending on the conducted test some samples were tested with only short-term aging, but others were additionally long-term aged. The long-term laboratory aging was achieved with the Pressure Aging Vessel (PAV) according to EN 14769 (CEN, 2012). 50 g of samples were poured onto pans and the pans were conditioned for 20 h at 100 °C under a dry air pressure of 2.1 MPa. Also, the binder samples obtained by extraction from loose asphalt mixture were PAV aged.

Penetration grading

The tank samples were tested for their needle penetration and softening point to check the compliance with requirements in EN 12591 (CEN, 2009). The needle penetration was determined according to EN 1426 (CEN, 2015a) and the softening point with the Ring and Ball method according to EN 1427 (CEN, 2015b). The needle penetration is measured by penetrating a standard needle with a weight of 100 g for 5 s into the sample that is conditioned at 25 °C. A manual penetrometer was used in this study. In the Ring and Ball method, the binder is poured into two brass rings and the rings placed in the ring holder. Steel balls with a weight of

3.5 g are centred on top of the filled rings. The assembly is placed in a glass beaker, which is filled with 5 °C water. The water is heated by a hot-plate with a rate of 5 °C/min, and the softening point is the average temperature where the two specimens drop 25 mm from their initial plane. An automatic Ring and Ball device was used in this research.

Performance grading

The grade was determined according to the standard AASHTO M320 (AASHTO, 2010). The high temperature grade was determined with the DSR. It was measured both for unaged and RTFOT aged sample. The temperatures where $G^*/\sin\delta$ equalled 1 kPa for unaged and 2.2 kPa for RTFOT aged samples were measured. The lowest of the two determines the high temperature grade. An Anton Paar MCR302 rheometer was used in this study. The low temperature grade was measured with the Bending Beam Rheomerer (BBR). The stiffness and m-value were determined at different temperatures and their respective low temperature values were calculated. The highest of the two determines the low temperature grade. An InfraTest BBR was used to test the samples in this research.

T(30°) measurement

The T(30°) was also determined with the help of the DSR. Phase angles were measured at different temperatures at an angular velocity of 10 rad/s. Using the data achieved from different temperatures, the temperature where the phase angle equalled 30° was determined by interpolation. Extrapolation was allowed when the determined T(30°) was less than 1 °C outside of the data points.

Sum of pavement defects

The Transport Administration of Estonia orders the inspection of the country’s national roads periodically. The work is done in 100 m sections. For each section the defects recorded are the amount of transverse cracks, length of longitudinal and joint cracks, area of alligator cracking, the amount of potholes and the area of ravelling. Using this data the proportion of pavement with defects, which is also known as the sum of pavement defects, is calculated. This is done according to the guidebook (Estonian Transport Administration, 2021) of the Estonian Transportation Administration. For the purpose of this research, the data was obtained from a publicly held register, and an average was calculated by summing the results from all 100 m sections of the investigated road sections and dividing with the length of the section in kilometres. This resulted in an average sum of pavement defects per kilometre.

3. Results and discussion

Penetration and Superpave grading

To check the compliance of the samples to the European standard EN 12591 (CEN, 2009), the needle penetration and softening point tests were conducted on the tank samples. Additionally, for informational purposes, the Superpave performance grade was determined according to AASHTO M320 (AASHTO, 2010). These results can be seen in Table 2.

Since all of these samples are marketed based on their penetration grade, it can be seen that the needle penetration falls within their respective grade. Also, when viewing the softening point then all of the samples fall within standard requirements.

The most prevalent PG grade among the studied binders is PG 58-28, which is suitable for most areas in Estonia. However, it should be noted that the Estonian PG design temperatures have been calculated differently than the usual Superpave system. Previous studies have

Table 2. Tank sample basic properties

Sample no.	Penetration, dmm	Softening point, °C	Superpave performance grade (XX-YY), °C
1	114	41.2	52–22
3	78	48	64–28
6	175	39.2	52–28
9	81	47.8	64–28
11	121	43	58–28
14	125	42.6	58–28
17	105	43.2	58–22
19	129	41.4	58–28
21	124	41.9	58–28
23	126	41.6	58–28
25	126	41.6	58–28
27	184	40.6	52–28

indicated that the Superpave system tends to underestimate high-temperature and overestimated low-temperature design temperatures (Kontson et al., 2023).

Limiting phase angle temperatures

As the research focuses on the feasibility of using T(30°) as a parameter to monitor the aging of asphalt binders, the most crucial aspect was the measurement of T(30°). The results are presented in Table 3. It should be noted that the tank samples were laboratory aged first with the RTFOT and then the PAV before the T(30°) was measured. Samples that were extracted from the mixture were aged with the PAV prior to testing and the binders derived from core samples were tested after extraction without any additional aging.

Figure 1 shows the average T(30°) results for the different binder grades of the tank samples included in the study. It can be seen that the

Table 3. T(30°) results

Sample no.	T(30°), °C	Sample nr.	T(30°), °C
1	3.7	20	3.8
2	3.7	21	2.3
3	4.3	22	3.3
4	4.7	23	2.8
5	0.6	24	5.6
6	−0.5	25	2.8
7	−0.8	26	2.1
8	−4.7	27	1.9
9	4.6	28	1.1
10	4.1	29	−5.7
11	1.3	30	−3.5
12	1.9	31	−2.3
13	−0.7	32	−7.6
14	1.4	33	−3.1
15	1.4	34	−4
16	−3.7	35	−8.3
17	3.4	36	−8.8
18	2.8	37	−2.4
19	1.0	-	-

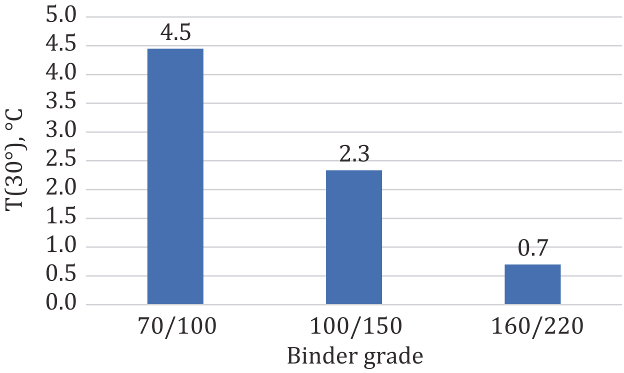

Figure 1. T(30°) average of different binder grades for tank samples

T(30°) parameter is higher for the stiffer 70/100 binders and with the increase in penetration grading the T(30°) parameter decreases. This is very much expected as T(30°) shows the viscous and elastic proportions of materials and stiffer binders achieve the same level on viscous and elastic proportions at higher temperatures compared to softer binders. The same trend applies with the increase in stiffness due to binder aging.

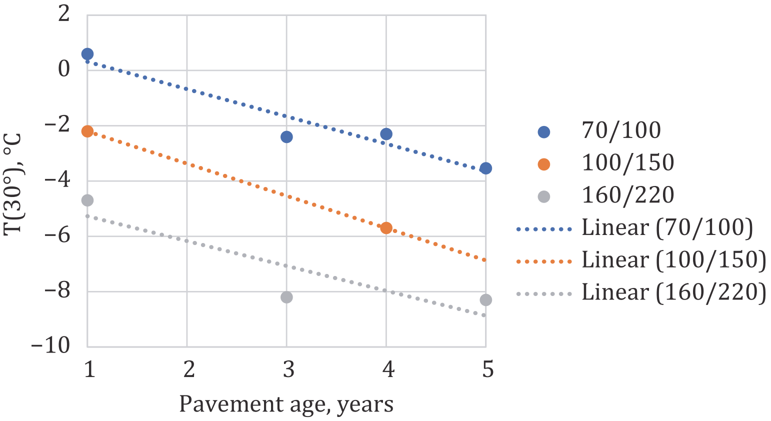

Figure 2. Development of T(30°) with pavement age

T(30°) trend with pavement age

The average T(30°) results of different binder grades and pavement ages, together with a linear trend line for each binder grade, are presented in Figure 2. The obtained results are controversial. As previously mentioned, T(30°) typically increases with the aging of the binder. However, the data collected for this study shows the opposite trend across all binder grades. It is important to note that the data set is very limited in this comparison. Nonetheless, confidence is gained as different binder grades show very similar slopes on their trend lines, and the trend lines are parallel to each other, with stiffer 70/100 binders exhibiting higher T(30°) results than the softer grades.

The most likely cause of this discrepancy is the change in binder sources in the region. In the authors' previous research (Lill et al., 2023), it was found that asphalt binders derived from Venezuelan crude had noticeably lower T(30°) values. However, this binder source became unavailable due to geopolitical issues around 2019. The cored pavements, which were older than one year during coring, are all from the time when this source was still widely used. To confirm this hypothesis, future research should focus on monitoring the aging of certain pavement sections throughout a period of many years, rather than taking samples from different sections with different ages.

It is noteworthy that this change in performance is not evident with regular test methods as all the tank samples showed results that meet the European standard EN 12591 (CEN, 2009). Additionally, after the removal of the Venezuelan binder from the market, frequent complaints about the quality of the supplied binders were noted.

It can be stated that T(30°) is able to identify such fundamental changes in asphalt binder quality like the change in viscous to elastic proportions. As it is favourable for an asphalt binder to retain as much of

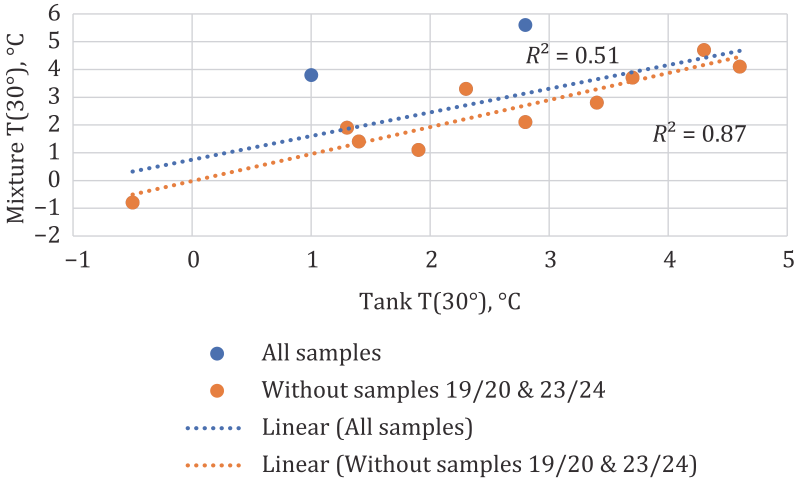

Figure 3. T(30) correlation between tank and mixture samples

its viscous component as possible with the increase in age, it can be said that older binders included in the study are superior compared to newer binders. This means that the elastic component is higher for newer binders, and, therefore, newer pavements can potentially start to crack due to fatigue and low temperature at an earlier stage of their lifetime.

This leads to a hypothesis that the binder contained in RAP from pavements constructed prior to 2020 could be of higher quality than newly refined asphalt binder. This needs to be acknowledged by stakeholders, as it could be beneficial to start using higher amounts of RAP.

T(30°) correlation between tank and mixture samples

When considering all test sites where T(30°) results for tank and mixture samples are available, the correlation is not great with R^2 equalling 0.51 as can be seen in Figure 3. However, there are two outliers, which significantly affect the correlation. These outliers are the tank samples 19 and 23 and their respective mixture samples 20 and 24. If these samples are excluded from the correlation study, then the R^2 jumps to 0.87, which is a remarkable improvement.

The issue seems to lie in the results of the mixture samples, as they seem to be abnormally high. It is crucial to understand if the test results for the outliers are incorrect or not.

While this paper does not primarily focus on the high-temperature performance of the binders, there is available data for high-temperature performance grading for both tank and mixture samples, as presented in Table 4. Looking at the data, it becomes apparent that for almost all samples, the Superpave high-temperature true grade for the recovered binder is lower than for the RTFOT aged sample. This suggest that the aging caused by the RTFOT appears to be more severe than what occurs in the asphalt mixing plant.

However, there are exceptions to this trend. For three samples, the true grade after recovery is higher than after the RTFOT. One such occasion is for the site T65 Veriora (ref) where the true grade after recovery is 1.1 °C higher than for the laboratory-aged samples. This can be easily explained because when sampling for this mixture sample, there was a breakdown of the asphalt paver, and due to this the mixture to be sampled stayed in the truck trailer for around 4 h longer than planned. This additional time at elevated temperatures caused excessive aging.

The other two samples are the previously mentioned outlying mixture samples 20 and 24. The most probable reason for their excessive aging is the fact that these samples were mixed using limestone aggregate. Although previous research (Anderson et al., 1994; Petersen et al., 1974; Wu et al., 2014) has shown that aging in mixtures with

Table 4. Superpave high temperature true grades

Site	Sample no.	Superpave high temperature true grade for tank sample (after RTFOT), °C	Sample no.	Superpave high temperature true grade for mixture sample (after extraction and recovery), °C
T92 Kanaküla	1	59.4	2	58.5
T65 Veriora (ref)	3	65.8	4	66.9
T65 Veriora (soft)	6	55	7	53.1
T92 Rihkama	9	67.1	10	65.3
T52 Metsla (base)	11	60.1	12	58.7
T52 Metsla (surf)	14	59.3	15	57.7
T60 Nätsi	17	60.1	18	58
T52 Holstre (base)	19	58.8	20	63.2
T52 Holstre (surf)	21	59.3	22	59.5
T19201 Vahenurme (bin)	23	60.1	24	63.8
T19201 Vahenurme (surf)	25	60.1	26	57.3
T81 Käina	27	54.9	28	53.1

limestone aggregate is slower compared to granite, the key issue in terms of the current research is temperature. Limestone is much more porous compared to igneous rock, and if the aggregate is wet in the stockpile, and not wanting to compromise production rate, then the asphalt mixing plant has to heat the limestone more than igneous rock to dry it prior to mixing with the asphalt binder. This additional heat is likely the cause of the higher aging degree seen in the results. However, it is noteworthy that sample 12, mixed with limestone aggregate, did not show any additional aging.

Coming back to the T(30°) correlation between tank and mixture samples, it can be concluded that the measurements for samples 20 and 24 are correct, and the lower correlation is valid. These results emphasise that the RTFOT is an empirical short-term aging procedure, which can be used to compare different binders, but it cannot always accurately simulate the aging that occurs in the mixing plant as there are numerous factors that can affect the aging magnitude in the plant.

Usability of T(30°) as an aging monitoring tool

For three test sections with a total of four different asphalt mixtures, there is T(30°) data available starting from the tank sample to the loose asphalt mixture sample, as well as the core sample. The T(30°) results obtained from the core samples, along with their respective tank sample results and their differences, are presented in Figure 4. Given that the pavements were roughly the same age (one year) when coring took place, the results should exhibit similar trends. This is true for three of the four mixtures.

The surface course mixture from T52 Metsla and both the reference mixture and softer mixture containing 160/220 binder from T65 Veriora showed similar differences between the core and tank samples ranging from 3.7 to 5.1 °C. However, notably different was the difference for the base course mixture from T52 Metsla, where the difference was only 2 °C. This indicates that the binder in this mixture has aged more severely and could reach its end-of-life sooner compared to the other mixtures.

Identifying the reason for this excessive aging is challenging, but one potential factor could be the fact that this mixture is produced with limestone aggregate. Although the difference in T(30°) between the tank sample and loose mixture was only 0.6 °C, indicating no indication of excessive heating of the mixture, it is possible that there could have been absorption of lighter fractions of the binder into the porous limestone aggregate, which remained in the pores during the extraction process, although this contradicts the findings of Wu et al. (2014). This is something that needs further investigation, but there is not enough data in the dataset of this research to draw definitive conclusions.

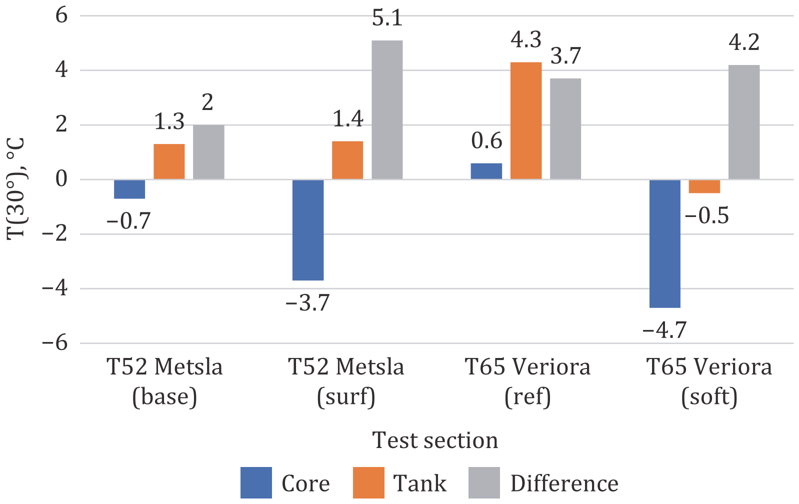

Figure 4. Development of T(30)

However, even for this limited set of data, it is evident that T(30°) has potential to be a parameter to monitor the aging of asphalt binder in mixtures. If the T(30°) of the tank sample, which has been both short-term and long-term aged, is known, then it is possible to drill only one core from the pavement that needs to be inspected, and the binder extracted from this core is sufficient to test the T(30°). The probability of the road deteriorating due to the aging of the binder increases as the T(30°) of the drill core approaches or surpasses the T(30°) of the tank sample. Furthermore, the T(30°) result from tank samples can be set as the limit, even if recycled asphalt pavement is being used in the asphalt mixtures.

Correlation of T(30°) and pavement defects

Estonia employs the sum of defects as one measure to monitor pavement condition. Given the availability of this data, an analysis was undertaken to explore any potential correlation between the T(30°) temperature and the sum of defects per kilometre of road. The pavement defect data was extracted in early 2024, utilising the most recent available data for each section. The inspections for all but one section were conducted in 2023, with T11152 Kirdalu being inspected in 2022.

As depicted in Figure 5, the correlation between the sum of defects and T(30°) is notably small with R^2 being only 0.13. This outcome is not unexpected, as T(30°) primarily measures the aging degree and low-temperature performance of the asphalt binder, whereas not all road defects can be attributed solely to these factors. Furthermore, the test

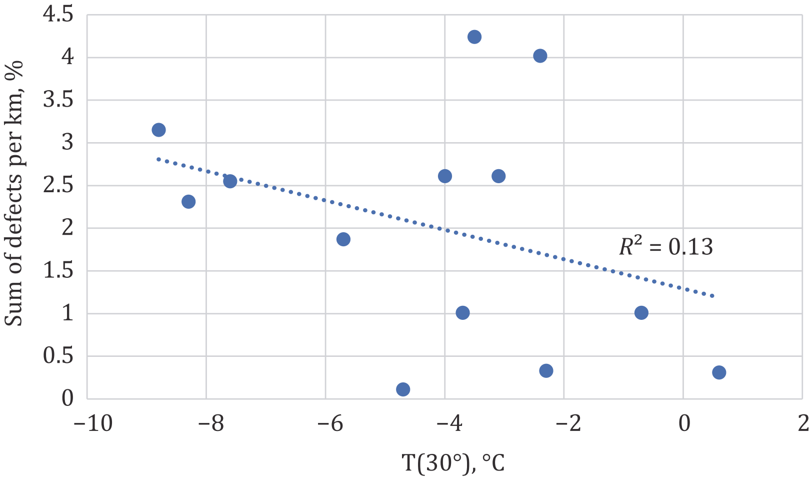

Figure 5. Correlation between sum of defects per km vs T(30)

sections vary in design due to their location and Annual Average Daily Traffic.

It is important to note that the sum of defects per kilometre is relatively low, ranging from 0.11 to 4.24. Although there are no specified limits in the current specifications in Estonia, reports from the early 2000s (Kaal, 2003) suggest that if the sum of defects is below 1, the pavement condition is considered very good, and between 2 to 5, it is considered good. Thus, all inspected pavements included in the current study are categorised as either good or very good.

To potentially enhance future studies, it might be beneficial to include data from pavements older than five years, as the current dataset focuses on a narrow range of only one to five-year-old pavements. Additionally, future studies could consider examining defects specifically related to asphalt binder aging rather than focusing solely on the sum of defects.

Conclusions

Based on the tested samples and the discussion presented, the following conclusions can be made:

- The T(30°) parameter effectively distinguishes between softer and stiffer binders, with higher values observed for stiffer binders and lower values for softer binders;
- T(30°) detected deterioration in binder quality that regular EN standards failed to identify;
- Recycled asphalt pavement from pavements constructed before 2020 may contain higher-quality asphalt binder than newly refined binder;
- The Rolling Thin Film Oven Test cannot consistently predict the aging occurring in the asphalt mixing plant due to fixed parameters;
- Binders in mixtures with limestone aggregate generally age faster, possibly due to higher mixing temperatures used to dry the porous aggregate and/or limestone absorbing lighter fractions of the binder;
- T(30°) is a suitable measure for monitoring asphalt aging, but a reference value from the tank sample is necessary. It also holds potential for use with mixtures containing RAP;
- In the limited number of road sections analysed in this study, there was almost no correlation between the T(30°) temperature and the sum of defects on the pavement. This lack of correlation was expected due to the different nature of these parameters.

In this research, an extensive number of samples were tested to investigate the T(30°) parameter ability to identify the change in asphalt binder performance and also to check its ability to monitor the aging of pavements. The samples included tank samples, but also samples extracted from loose asphalt mixture and from core samples. The obtained results show strong potential for T(30°) to be used for both purposes.

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Kannenberg, K. (2024) Asfaltsegu terakoostise ja sideaine sisalduse laborisisene varieeruvus ning selle mõju kvaliteedi hinnangule [Intralaboratory variability of binder content and gradation testing of bituminous mixtures and its' impact to quality assessment results]. Supervised by Kontson, K; **Lill, K.** Master's thesis. Tallinn, Estonia: Tallinn University of Technology.

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