THE BALTIC JOURNAL OF ROAD AND BRIDGE ENGINEERING 2023/18(4)

ISSN 1822-427X/eISSN 1822-4288 2023 Volume 18 Issue 4: 166–184 https://doi.org/10.7250/bjrbe.2023-18.623



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

KRISTJAN LILL*, KARLI KONTSON, ANDRUS AAVIK

Department of Civil Engineering and Architecture, Road Engineering and Geodesy Research Group, Tallinn University of Technology, Tallinn, Estonia

Received 16 June 2023; accepted 2 November 2023

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 highquality 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

* Corresponding author. E-mail: kristjan.lill@taltech.ee

Kristjan LILL (ORCID ID 0000-0002-2095-806X) Karli KONTSON (ORCID ID 0000-0002-4029-3245) Andrus AAVIK (ORCID ID 0000-0002-8672-0345)

Copyright © 2023 The Author(s). Published by RTU Press

This is an Open Access article distributed under the terms of the Creative Commons Attribution License (http://creativecommons.org/licenses/by/4.0/), which permits unrestricted use, distribution, and reproduction in any medium, provided the original author and source are credited.

The Applicability of Limiting Phase Angle Temperatures for Specifying Asphalt Binder Low Temperature Performance

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 lowtemperature 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

The Applicability of Limiting Phase Angle Temperatures for Specifying Asphalt Binder Low Temperature Performance

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

THE BALTIC JOURNAL OF ROAD AND BRIDGE ENGINEERING 2023/18(4)

of samples (N = 36) sourced from around the world. A correlation was proposed (Equation (1)) with a coefficient of determination $R^2 = 0.85$.

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

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

Table 1. Sample information

The Applicability of Limiting Phase Angle Temperatures for Specifying Asphalt Binder Low Temperature Performance

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 semiautomatic 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 lowtemperature grade. In the Superpave method, the asphalt binder beams

The Applicability of Limiting Phase Angle Temperatures for Specifying Asphalt Binder Low Temperature Performance

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 $G^*/\sin\delta$ 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

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

Table 2. Penetration and superpave grading

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

The Applicability of Limiting Phase Angle Temperatures for Specifying Asphalt Binder Low Temperature Performance

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 3. Fraass breaking point and superpave low temperature true grade

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

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

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 $T(30^{\circ})$ 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 $T(30^\circ)$ 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 $T(30^\circ)$ 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 $T(30^\circ)$ 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 $T(30^\circ)$. All other samples fall in between, with one contradicting result (sample No. 13). This sample is also a 160/220 binder, but has a $T(30^\circ)$ 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

The Applicability of Limiting Phase Angle Temperatures for Specifying Asphalt Binder Low Temperature Performance

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°)

The Applicability of Limiting Phase Angle Temperatures for Specifying Asphalt Binder Low Temperature Performance

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 $T(30^\circ)$ measurements, whereas Figure 4 demonstrates the correlation between regular BBR and $T(30^\circ)$ measurements. The $T(30^\circ)$ 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 $T(30^\circ)$ 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 $T(30^\circ)$ 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 $T(30^{\circ})$ 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.

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

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

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 $T(30^\circ)$ and BBR and $T(30^\circ)$ and the EBBR methods are less pronounced than found in previous work;

The Applicability of Limiting Phase Angle Temperatures for Specifying Asphalt Binder Low Temperature Performance

 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 $T(30^\circ)$ 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.

Acknowledgements

The authors would like to express their appreciation to Prof. Simon A. M. Hesp for giving feedback on the content of the paper and to Dr Christopher Davies for providing proof reading support. Additionally, the authors would like to thank the reviewers of this manuscript.

Funding

The testing for this paper was funded by the Estonian Transport Administration.

REFERENCES

- Aliha, M. R., & Shaker, S. (2020). Effect of bitumen type, temperature and aging on mixed I/II fracture toughness of asphalt binders-experimental and theoretical assessment. *Theoretical and Applied Fracture Mechanics*, 110, Article 102801. https://doi.org/10.1016/j.tafmec.2020.102801
- American Association of State and Highway Transportation Officials. (2016). Determination of performance grade of physically aged asphalt binder using extended bending beam rheometer (BBR) method.
- American Association of State Highway and Transportation Officials. (n.d.). AASHTO M320 – Performance-graded asphalt binder.

- Angius, E., Ding, H., & Hesp, S. A. (2018). Durability assessment of asphalt binder. *Construction and Building Materials*, 165, 264–271. https://doi.org/10.1016/j.conbuildmat.2018.01.037
- Baumanis, J., Riekstins, A., & Balodis, A. (2021). Investigation of comparability of TSRST and SCB cracking tests for evaluation of low-temperature properties in asphalt mixtures and use in quality control. *IOP Conference Series: Materials Science and Engineering*, 1202, Riga, Latvia. https://doi.org/10.1088/1757-899X/1202/1/012022
- Besamusca, J. T., Willemsen, T., & van de Ven, M. F. (2011). A plea for the introduction of performance related bitumen specifications. *Road Materials And Pavament Design*, 11(sup1), 355–377. https://doi.org/10.1080/14680629.2010.9690338
- Bueno, M., Hugener, M., & Partl, M. N. (2014). Fracture toughness evaluation of bituminous binders at low temperatures. *Materials and Structures*, 48, 3049–3058. https://doi.org/10.1617/s11527-014-0378-4
- European Commission. (1998). Mandate M/124 Mandate to CEN/CENELEC concerning the execution of standardisation work for harmonized standards on road construction products. Brussel.

https://law.resource.org/pub/eu/mandates/m124.pdf

- European Committee for Standardization. (2009). EN 12591 Bitumen and bituminous binders. Specifications for paving grade bitumens. https:// standards.iteh.ai/catalog/standards/cen/38a7da38-1c54-48f3-a0f3-1a45fb81c414/en-12591-2009
- European Committee for Standardization. (2010). EN 14023 Bitumen and bituminous binders – Framework specification for polymer modified bitumens. https://standards.iteh.ai/catalog/standards/cen/b9736364-314a-4743-8ffaa95b53079407/en-14023-2010
- European Committee for Standardization. (2012). EN 14769 Bitumen and bituminous binders – Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV). https://standards.iteh.ai/catalog/standards/ cen/7e60dbee-bd26-4cef-812f-1e5c77446899/en-14769-2012
- European Committee for Standardization. (2014). EN 12607-1 Bitumen and bituminous binders – Determination of the resistance to hardening under influence of heat and air. RTFOT method. https://standards.iteh.ai/catalog/ standards/cen/5e1f0c0a-99bc-4c99-a191-cf07c1482527/en-12607-1-2014
- European Committee for Standardization. (2015). EN 12593 Bitumen and bituminous binders – Determination of Fraass breaking point. https:// standards.iteh.ai/catalog/standards/cen/caa4d7eb-7717-4987-b0ce-457b119aeafe/en-12593-2015
- European Committee for Standardization. (2015). EN 1426 Bitumen and bituminous binders – Determination of needle penetration. https://standards. iteh.ai/catalog/standards/cen/853455a7-1455-4f3e-a2b8-60d17bfdf3df/ en-1426-2015
- Evans, M., Marchildon, R., & Hesp, S. A. (2011). Effects of physical hardening on stress relaxation in asphalt cements: Implications for pavement performance. *Transportation Research Record: Journal of the Transportation Research Board*, 2207(1), 34–42. https://doi.org/10.3141/2207-05

The Applicability of Limiting Phase Angle Temperatures for Specifying Asphalt Binder Low Temperature Performance

- Gražulite, J. (2019). Bitumen stress relaxation modulus as an indicator of asphalt pavements resistance to low temperature cracking [Doctoral dissertation, Vilnius Gediminas Technical University]. http://dspace.vgtu.lt/handle/1/3824
- Gražulyte, J., & Vaitkus, A. (2017). Analysis of methods and criteria for evaluation of bitumen performance at low temperatures. "Environmental Engineering" 10th International Conference, Vilnius. https://doi.org/10.3846/enviro.2017.137
- Hesp, S. A., Soleimani, A., Subramani, S., Phillips, T., Smith, D., Marks, P., & Tam, K. T. (2009). Asphalt pavement cracking: analysis of extraordinary life cycle variability in easternand northeastern Ontario. *International Journal of Pavement Engineering*, 10(3), 209–227.

https://doi.org/10.1080/10298430802343169

Jing, R., Varveri, A., Liu, X., Scarpas, A., & Erkens, S. (2020). Rheological, fatigue and relaxation properties of aged bitumen. *International Journal of Pavement Engineering*, 21(8), 1024–1033.

https://doi.org/10.1080/10298436.2019.1654609

- Khan, A. N., Akentuna, M., Pan, P., & Hesp, S. A. (2020). Repeatability, reproducibility, and sensitivity assessments of thermal and fatigue cracking acceptance criteria for asphalt cement. *Construction and Building Materials*, 243, Article 117956. https://doi.org/10.1016/j.conbuildmat.2019.117956
- Li, Y., & Hesp, S. A. (2022). On the use of empirical phase angle limits for the grading of asphalt binder. *Construction and Building Materials*, 346, Article 128413. https://doi.org/10.1016/j.conbuildmat.2022.128413
- Li, Y., Ding, H., Nie, Y., & Hesp, S. A. (2021). Effective control of flexible asphalt pavement cracking through quality assurance testing of extracted and recovered binders. *Construction and Building Materials, 273*, Article 121769. https://doi.org/10.1016/j.conbuildmat.2020.121769
- Lill, K., Khan, A. N., Kontson, K., & Hesp, S. A. (2020). Comparison of performance-based specification properties for asphalt binders sourced from around the world. *Construction and Building Materials, 261,* Article 120552. https://doi.org/10.1016/j.conbuildmat.2020.120552
- Lill, K., Kontson, K., Khan, A., Pan, P., & Hesp, S. A. (2019). Comparison of physical and oxidative aging tendencies for Canadian and Northern European asphalt binders. *Proceedings of Canadian Technical Asphalt Association*, (pp. 24–28). https://www.ctaa.ca/download/abstracts-2019/Abstract-2019-020-Lill.pdf
- Lu, X., & Isacsson, U. (1997). Characterization of styrene-butadiene-styrene polymer modified bitumens – Comparison of conventional methods and dynamic mechanical analyses. *Journal of Testing and Evaluation*, 25(4), 383–390. https://doi.org/10.1520/JTE11874J
- Lu, X., Isacsson, U., & Ekbald, J. (2003). Influence of polymer modification on low temperature behaviour of bituminous binders and mixtures. *Materials and Structures*, 36, 652–656. https://doi.org/10.1007/BF02479497
- Lu, X., Uhlback, P., & Soenen, H. (2017). Investigation of bitumen low temperature properties using a dynamic shear rheometer with 4 mm parallel plates. *International Journal of Pavement Research and Technology*, 10(1), 15–22. https://doi.org/10.1016/j.ijprt.2016.08.010

- Migliori, F., Ramond, D., & Pastor, M. (1993). Etude statistique de quelques cas de fissurations thermiques. *Proceedings, Fifth Eurobitume Congress, 1,* 724–728.
- Migliori, F., Ramond, G., Ballie, M., Brule, B., Exemelin, C., Lombardi, B., Samanos, J., Ferraro Maia, A., Such, C., & Watkins, S. (1999). Correlation between the thermal stress cracking of bituminous mixes and the binders' rheological characteristics. *Eurobitume Workshop 99 – Performance Related Properties of Bituminous Binders*. Luxembourg.
- Shaker, S., Aliha, M., & M, K. (2019). Aging effect on combined mode fracture resistance ofbitumen. Fatiguw & Fracture of Engineering Materials & Structures, 42(7), 1609–1621. https://doi.org/10.1111/ffe.13003
- Siroma, R., Lan, N. M., Hornych, P., & Chailleux, E. (2021). A literature review of bitumen aging: From laboratory procedures to field evaluation. *Proceedings* of the RILEM International Symposium on Bituminous Materials, 27, 89–95. https://doi.org/10.1007/978-3-030-46455-4_11
- Soleimani, R., Walsh, S., & Hesp, S. (2009). Asphalt cement loss tangent as a surrogate performance indicator for control of thermal cracking. *Journal of the Transportation Research Board*, 2126(1), 39–46. https://doi.org/10.3141/2126-05
- Turk, M. R., & Tušar, M. (2018). Low temperature properties of paving grade bitumen. Proceedings of the 5th International Conference on Road and Rail Infrastructures. Zadar, Croatia. https://doi.org/10.5592/CO/cetra.2018.876
- Widyatmoko, I., Heslop, M., & and Elliott, R. (2005). The viscous to elastic transition temperature and the in situ performance of bituminous and asphaltic materials. *Journal of the Institute of Asphalt Technology, Asphalt Professional No 14*, 3–7. https://www.researchgate.net/ publication/288823733_The_Viscous_to_Elastic_Transition_Temperature_ and_the_In_Situ_Performance_of_Bituminous_and_Asphaltic_Materials
- Yee, P., Aida, B., Hesp, S. A., Marks, P., & Tam, K. K. (2006). Analysis of premature low-temperature cracking in three Ontario, Canada, Pavements. *Transportation Research Record: Journal of the Transportation Research Board*, 1962(1), 44–51. https://doi.org/10.1177/0361198106196200106