

# 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  $T(30^\circ)$  parameter can successfully be used to measure the aging of asphalt binders. A key discovery was that the  $T(30^\circ)$  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  $T(30^\circ)$  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  $30^\circ$  ( $T(30^\circ)$ ). 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  $T(30^\circ)$  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  $T(30^\circ)$  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-Typisierung-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  $0^\circ$  is completely elastic and a material with a phase angle of  $90^\circ$  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  $T(30^\circ)$  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 Rheometer (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).

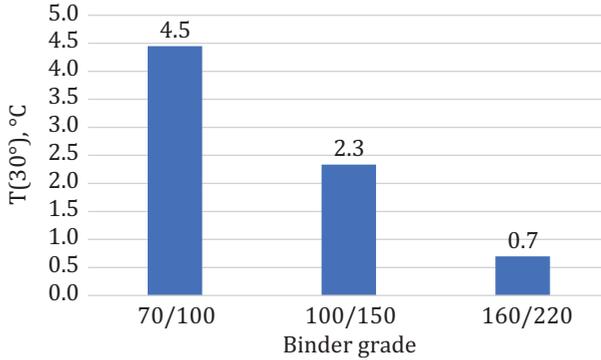
### Limiting phase angle temperatures

As the research focuses on the feasibility of using  $T(30^\circ)$  as a parameter to monitor the aging of asphalt binders, the most crucial aspect was the measurement of  $T(30^\circ)$ . 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  $T(30^\circ)$  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  $T(30^\circ)$  results for the different binder grades of the tank samples included in the study. It can be seen that the

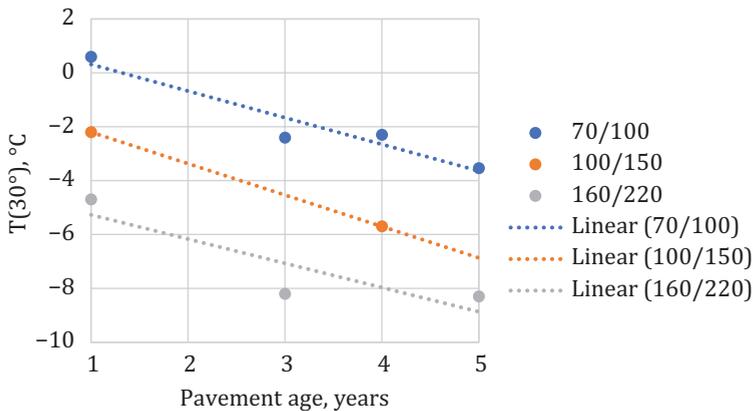
Table 3.  $T(30^\circ)$  results

Sample no.	$T(30^\circ)$ , °C	Sample nr.	$T(30^\circ)$ , °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

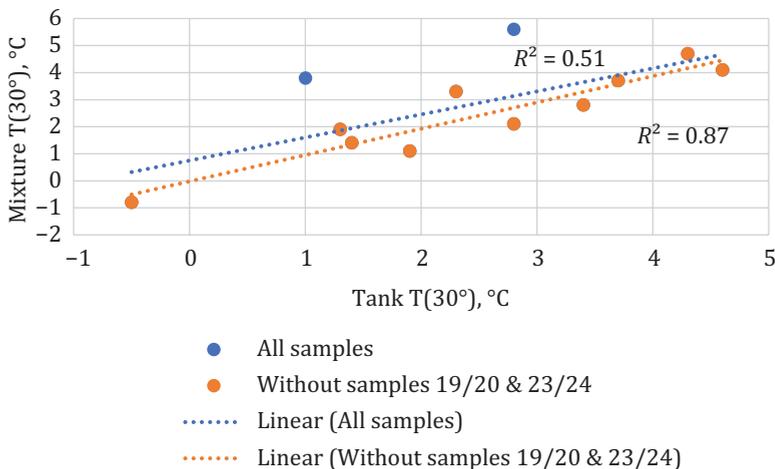
The average T(30°) results of different binder grades and pavement ages, together with a linear trend line for each binder grade, are presented in Figure 2. The obtained results are controversial. As

previously mentioned,  $T(30^\circ)$  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  $T(30^\circ)$  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  $T(30^\circ)$  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  $T(30^\circ)$  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.**  $T(30^\circ)$  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 suggests 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  $T(30^\circ)$  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  $T(30^\circ)$  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.

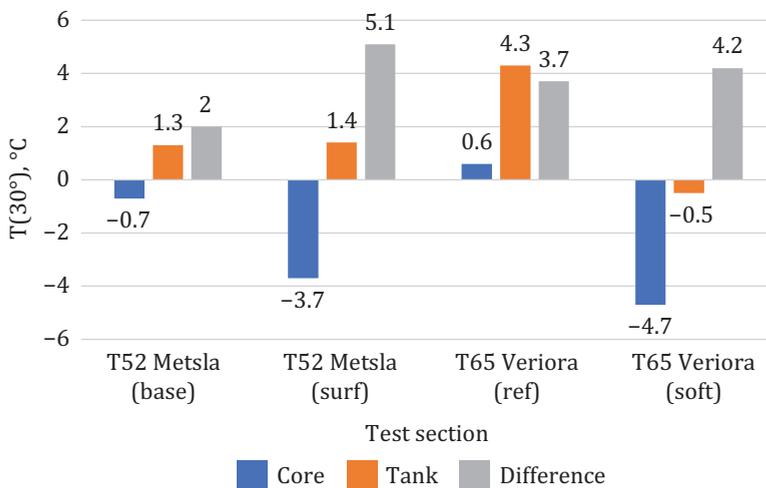


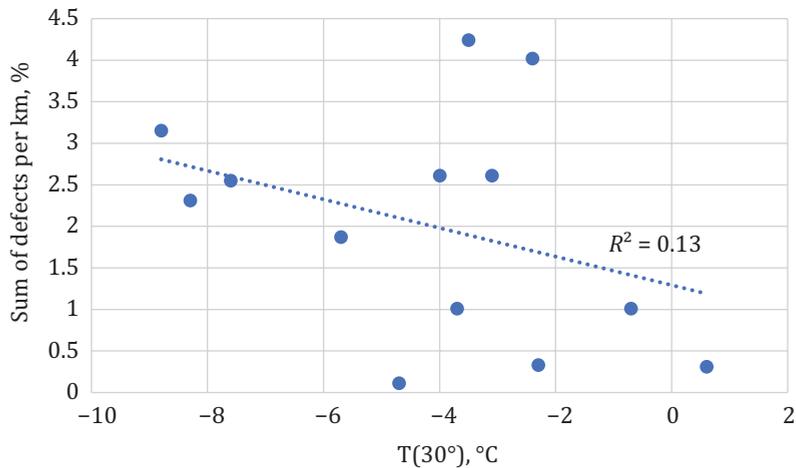
Figure 4. Development of  $T(30)$

However, even for this limited set of data, it is evident that  $T(30^\circ)$  has potential to be a parameter to monitor the aging of asphalt binder in mixtures. If the  $T(30^\circ)$  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  $T(30^\circ)$ . The probability of the road deteriorating due to the aging of the binder increases as the  $T(30^\circ)$  of the drill core approaches or surpasses the  $T(30^\circ)$  of the tank sample. Furthermore, the  $T(30^\circ)$  result from tank samples can be set as the limit, even if recycled asphalt pavement is being used in the asphalt mixtures.

### Correlation of $T(30^\circ)$ 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  $T(30^\circ)$  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  $T(30^\circ)$  is notably small with  $R^2$  being only 0.13. This outcome is not unexpected, as  $T(30^\circ)$  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  $T(30^\circ)$

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  $T(30^\circ)$  parameter effectively distinguishes between softer and stiffer binders, with higher values observed for stiffer binders and lower values for softer binders;
- $T(30^\circ)$  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;
- $T(30^\circ)$  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  $T(30^\circ)$  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  $T(30^\circ)$  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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