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Impact of distillation temperature on the solvent residue and viscoelastic properties of asphalt binders

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Asphalt used as a paving material is a mix of aggregates and bituminous binder. Due to reasons of acceptance tests after paving or for fault analysis, it is necessary to separate asphalt mixes into their components. Therefore, different organic solvents are commonly used. The European Standard defines a procedure for both, recovery of aggregates and the distillation of binder from solvent mixture. Within the distillation procedure, the applied maximum temperature and the time of its application are not defined clearly. This research is an approach to define the distillation temperature depending on the viscosity of the binder. Four different binder samples (unmodified, polymer-modified) were used to determine the impact of distillation temperature on solvent residue and the recovered binder and its rheological properties. The solvent residue was calculated by weight and the binder properties were obtained by a dynamic shear rheometer and rotational viscometer. These results show a dependence of the solvent residue on the viscosity at distillation temperature. Thus, it is possible to choose reasonable distillation temperatures with regard to binder viscosity instead of setting one temperature for all binders. It was also found that there is no impact of tetrachloroethene as solvent on the binder properties if its residue is low enough ($< 1\%$ w/w).

Keywords: bitumen; RTFOT; recovery; distillation; DSR; tetrachloroethene

1. Introduction

Asphalt mixtures used as a paving material consist of aggregates, bituminous binder, air voids and depending on the mix design also of different additives to change the chemical or physical properties. Due to reasons of acceptance tests after paving, for fault analysis or research purposes it is necessary to separate asphalt mixes into their components again. Thus, it is possible to look into the properties of each of the asphalt components in detail. Regarding the bituminous binder there are two reasons for the separation: first, to determine the binder content in the asphalt mixture and, second, to determine changes in the binder properties due to improper handling in the construction process (e.g. excessive heating etc.) or ageing during in-service life. Binder recovery is also necessary for research purposes such as the development of new long-term ageing procedures. Subsequently, conventional and performance-related binder tests are carried out on recovered binder samples. Thus, for the recovery of the binder from asphalt mixes organic solvents and recovery procedures are necessary that cause no significant changes to the properties of the recovered binder.

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A number of solvents have been used since the early 1900s starting with carbon disulphide (CS₂) (Bateman & Delp, 1927) and followed by benzene (C₆H₆). In the 1950s and 1960s, chlorinated solvents like trichloroethene (TCE, C₂HCl₃), 1-1-1-trichloroethane (C₂H₃Cl₃) and dichloromethane (CH₂Cl₂) became very popular (Cipione, Davison, Burr, Glover, & Bullin, 1991). Because benzene has been proven carcinogenic (GHS 1A), its use has been phased out. GHS is the abbreviation for Globally Harmonised System of Classification, Labelling and Packaging of Chemicals and has been implemented in European Law within Regulation (EC) No. 1272/2008, also known as CLP (Classification, Labelling and Packaging) regulation. Based on the research of Abson and Burton (1960), TCE was found as effective as benzene and, thus, it became the main replacement for benzene. Traxler (1967) found that the addition of about 10–15% of ethanol or methanol to TCE removes more binder from the aggregates. This has become popular among many researchers at least in the US (Cipione et al., 1991). In 2001, TCE was declared carcinogenic too (GHS 1B) and should be replaced by the industries by April 2016 due to Regulation (EU) No. 348/2013. The use of TCE is still allowed in closed-loop systems, but it is merely a matter of time until TCE gets completely banned by the legislature and industries have to replace it. Thus, dichloromethane (GHS 2), toluene (–) and tetrachloroethene (systematic name: perchlorethylene PCE, GHS 2) are becoming the commonly used solvents today. Metz and Wörner (2005) found out that binders recovered from lab mixtures by TCE and toluene have the same needle penetration and softening point. In Germany, the use of 1-1-1-trichloroethane for asphalt binder extractions is prohibited because of its highly ozone-depleting chemical structure. Bitumen is also less soluble in 1-1-1-trichloroethane than in the other solvents (EN 12697-3, 2013). Since the middle of the 1990s, the search and development of alternative, non-toxic and environmental friendly solvents began. In 2001, the Minnesota Department of Transportation published a research report (McGraw, Iverson, Schmidt, & Olson, 2001) based on Stroup-Gardiner and Nelson (2000) and Collins-Garcia, Mang, Roque, and Choubane (1999) asserting *n*-Propyl Bromide (*n*PB, C₃H₇Br) as a suitable solvent alternative. This is already outdated because in the EU *n*PB has been classified as a reproductive toxicant (GHS 1B), which makes it a ‘substance of very high concern’. In 2013, also the US National Toxicology Program recommended that *n*PB to be classified as reasonably anticipated human carcinogens (US Department of Health and Human Services, 2013). The most promising alternative solvent candidates are caproic acid methyl ester (C₇H₁₄O₂) and caprylic acid methyl ester (C₉H₁₈O₂). Research on different alternative solvents including caprylic acid methyl ester has been done by the DAI e.V. (Weingart & Krüger, 2012), a research association of Germany’s asphalt industry. They concluded that caprylic acid methyl ester is a suitable solvent for binder recovery from asphalt mixtures. Another German study employing an InfraTest Verte fully automated hot-extracting device confirmed a good applicability of caprylic acid methyl ester for binder recovery (Alisov & Wistuba, 2016).

In the EU there are two different state-of-the-art methods for binder recovery from solvent mixtures: EN 12697-3: *Bitumen recovery: Rotary evaporator* (2013) and EN 12697-4: *Bitumen recovery: Fractionating column* (2015). In the US, also two different methods are used for the binder recovery: ASTM D1856: *Standard test method for recovery of asphalt from solution by Abson method* (ASTM, 2015), introduced in 1933 and ASTM D5404: *Standard practice for recovery of asphalt from solution using the rotary evaporator* (ASTM, 2012). The rotary evaporator method is the most used method in the EU and US today. Peterson, Soleymani, Anderson, and McDaniel (2000) showed that the rotary evaporator method has to be preferred when it comes to binder tests.

Several studies have been published in the past stating severe changes to the binder properties after applying solvents. Most of them are from the 1960s to the 1990s applying completely

different procedures than what is state-of-the-art today. Differences are concerning the distillation temperature, the applied vacuum and the sample preparation. However, most of the results and conclusions were obtained from tests with virgin binders. In practice, binder recovery is applied on asphalt mixes only. Thus, virgin binders will never get in touch with organic solvents. Abu-Elgheit, Hancock, and Traxler (1969) and Abu-Elgheit and Ijam (1982) found a high sensitivity to hardening applying chlorinated and also non-chlorinated solvents. Abson and Burton (1960) and Burr, Davison, Jemison, Glover, and Bullin (1991) confirmed this, stating that all solvents cause about the same hardening effect except tetrachloroethene, which leads to even worse results. Burr, Jemison, Kyle, and Cipione (1989) concluded that binders specified by Strategic Highway Research Program are susceptible to hardening caused by the solvent close to short-term ageing employing an rolling thin film oven (RTFO), but also found softening effects to binders while they are in dilute solution and exposed to high temperatures (Burr, Davison, Glover, & Bullin, 1994). A high sensitivity to hardening of virgin binders was also found in preliminary tests of this study. This high sensitivity to hardening makes a pretreatment in kind of short-term ageing necessary. Burr, Glover, Davison, and Bullin (1993) dissolved RTFO-aged bitumen samples in toluene and TCE with 15% ethanol, distilled the solution by rotary evaporator method and found a decent hardening effect only. Burr, Davison, Glover, and Bullin (1990) also treated RTFO-aged samples with TCE only and found no hardening effects. They concluded that RTFO-aged samples return to their original viscosities, because their volatiles were already removed in the RTFO. In addition to the loss in volatiles through evaporation, changes in the chemical composition caused by oxidation have to be considered too. Peterson et al. (2000) found no significant hardening effect to an RTFO-aged PG 64–22 binder employing *p*PB as a solvent.

New state-of-the-art procedures and uncertainties applying tetrachloroethene as a suitable solvent for binder recovery from mixtures are the key reasons for this study. The main focus is on how a specific solvent (tetrachloroethene) commonly used for distillation affects different binder types and how sensitively different binder types react to solvent residue. The study shows the crucial impact of solvent residue on the viscoelastic behaviour and gives recommendations for a smooth distillation. It will further show that the current specification in the European Standard is not sufficient to ensure solvent-free binder samples after distillation.

2. Materials and methods

2.1. Materials

Four different binders, both non-modified and polymer-modified, are investigated in this study. The samples are graded by their needle penetration value and the polymer-modified binders additionally by their softening point according to EN 1426 (2007) and EN 1427 (2007). Four different binders, all produced from ‘Arab heavy’ crude oil have been selected to cover a wide viscosity range: 50/70, 160/220, PmB 45/80-65 and PmB 25/55-65. Both PmBs are styrene-butadiene-styrene (SBS) modified bitumen. The 50/70 paving grade bitumen is commonly used for asphalt pavements in central European countries. 160/220 bitumen is only used for surface layers of rural roads with very low volume of heavy goods vehicle traffic. However, 160/220 was included in the study due to its low viscosity. With four different binders at hand the coverage of a wide viscosity range is achieved to test the hypothesis that a binder’s viscosity has a crucial effect on the solvent residue after distillation. Modified bitumen is mainly used in surface layers of highways and other high-level roads. All samples were stored at room temperature in sealed metal cans. Conventional as well as performance-related results for these four bitumen are shown in Table 1. This table shows that the softening point does not work properly for PmBs. While the

Table 1. Binder properties.

Grading	Ageing	Pen 1/10 mm	R&B °C	Dyn. viscosity 135°C mPas	Elast. rec. %
50/70	Virgin	64	51.2	420	—
	RTFOT	40	56.6	700	—
160/220	Virgin	165	42.0	220	—
	RTFOT	89	44.8	294	—
PmB 45/80-65	Virgin	67	70.6	1854	92
	RTFOT	50	67.4	2408	87
PmB 25/55-65	Virgin	50	77.6	2242	81
	RTFOT	30	77.8	3700	78

needle penetration drops for both PmBs after RTFO-ageing, the softening point drops as well for the PmB 45/80-65 or stays almost the same for the PmB 25/55-65. For paving grade bitumen, a dropping penetration always causes a rising softening point. Compared with paving grade bitumen, PmBs are affected by two different mechanisms during ageing: first, the oxidation of the base bitumen and second the degradation of the polymer. While the oxidation of the bitumen causes a rising softening point, the degradation of the polymer can cause the opposite depending on the type of polymer used as a modifier. SBS, a thermoplastic elastomer, develops some kind of elastic network within the bitumen. This network supports the bitumen at elevated temperatures and causes higher stiffness and elastic properties. Degradation of SBS polymers means the increasing destruction of this network. Thus, the softening point is affected in two different ways. On one hand the softening point is rising because of the ageing of the base bitumen and on the other hand the softening point is dropping because of the SBS degradation. It can be concluded that the softening point is not suitable to characterise the effects of ageing for PmBs. Thus, the softening point is given as basic information in Table 1 only and is not further employed in this study.

A fully automated hot-extracting device using PCE is employed for binder recovery. PCE of $\geq 99.5\%$ purity grade for chemical synthesis was selected as a solvent in this study.

2.2. Methods

2.2.1. Sample preparation

The sample preparation is carried out in three steps.

Step 1 consists of short-term ageing of all four binders. Therefore, a RTFO was employed. This step was applied because a preliminary study showed a high sensitivity of virgin binders to high temperatures during distillation. Another important fact is that in practice binder recovery is applied on asphalt mixes only. Thus, virgin binders will never get in touch with organic solvents like PCE used in this study. The short-term ageing was performed in accordance to EN 12607-1 (2007).

Step 2 was to dissolve the binder samples in PCE. Therefore, the binder was heated to 180°C and about 12.5 g were poured into a 500 mL Erlenmeyer flask. After cooling down to room temperature, the flask with the bitumen was weighed and PCE was filled to 500 mL into the flask. Then, a hypersonic bath was applied for 30 min to dissolve the binder in a timely manner and to obtain about the same time of contact between binder and solvent as in the hot-extraction device. The mentioned amount of binder was chosen to achieve a binder-concentration in solvent similar to what can be expected from hot-extraction using a fully automated Infracore type

Table 2. PCE distillation guideline.

Solvent	Standard	1st Phase		2nd Phase		Extra
		Temp. T ₁ (°C)	Pressure P ₁ (mbar)	Temp. T ₂ (°C)	Pressure P ₂ (mbar)	Temp. T ₃ (°C)
Tetrachloroethene	EN 12697-3	110	400	160	20	180
Tetrachloroethene	TUV-modified	110	300	120, 140, 160, 180	20	–

ecoTest 20-1100 hot-extraction device. Another reason for this amount was the maximum weight capacity of the high precision scale available for this study.

Step 3 was the separation of binder and solvent by distillation. Therefore, a rotary evaporator was employed (Heidolph Laborota 4000 efficient with ILMVAC GmbH 4000482 vacuum pump). All distillations were mainly done according to EN 12697-3 (2013). This European Standard defines several temperature/pressure combinations depending on the solvent used for extraction. Phase 1 of the distillation guideline from EN 12697-3 (2013) was applied on all samples. This phase was modified regarding the applied pressure. A lower pressure of 300 mbar instead of 400 mbar speeds up the distillation significantly and thus avoids possible ageing effects to the binder due to heat. At a pressure of 300 mbar there is still no danger of boiling over when lowering the pressure stepwise between 400 and 300 mbar. Phase 2 was modified in terms of the target or maximum temperature. Maximum temperatures used within this study are 120°C, 140°C, 160°C and 180°C. 180°C was reached in one go without an intermediate step. All applied temperatures were held for 10 min. In comparison, the standard procedure described in the EN 12697-3 (2013) requires a temperature of 160°C in Phase 2 for 10 min. If the bitumen is still bubbling after 10 min at 160°C, the extra temperature of 180°C has to be applied for another 10 min after the bubbling has stopped. This makes the distillation a (timely) inaccurate procedure because it is a subjective decision when bubbling has stopped. In Table 2 details are given with respect to the applied distillation scheme according to EN 12697-3 (2013) and also the modified procedure used for this study named ‘TUV-modified’.

Due to the weight capacity of the high precision scale available for this study, a 250 mL round-bottomed flask was used as distillation flask. This is contrary to the European Standard which requires a 1000 mL flask. The volume of the flask has a major influence on the distillation duration. Larger flasks cause a higher evaporation rate and therefore shorter distillation durations, which is preferable because of ageing effects to the binder due to heat. A 250 mL flask was also chosen to obtain distillation durations similar to what can be expected in practice, both with higher amounts of solution and 1000 mL distillation flask. In total, each sample was in solution for about 3 h (from that about 2–2.5 h in dilute solution). Before weighing, the round-bottomed flask with the distilled bitumen was cleaned off the oil from the rotary evaporators’ oil bath with acetone. With the weight of the bitumen at hand before and after the distillation it is possible to calculate the solvent residue.

In this study, a triple determination was carried out to achieve a limited statistical certainty. This leads to 30 samples of each binder and thus a total of 120 samples were prepared for this study.

The rheological binder properties are obtained by rotational viscometer (RV) and dynamic shear rheometer (DSR). The RV was employed to measure the high-temperature viscosity whereas the DSR was employed to measure the linear viscoelastic (LVE) binder properties at elevated in-service temperatures. These properties are the dynamic modulus $|G^*|$ and phase lag δ .

2.2.2. Rotational viscometer (RV)

A RV is a device for the determination of the dynamic viscosity of bituminous binders. Therefore, torque is applied to a spindle (a cylinder) rotating in a special sample container containing the test sample. This sample container consists of a cylindrical aluminium pot and is directly put into the electrical heating unit. The applied torque measures the relative resistance of the spindle to rotation and provides a measure of the dynamic viscosity of the sample.

In addition to viscosity measurements at all distillation temperatures from 120°C to 180°C, steps of 10 K were selected to obtain a smooth viscosity-curve. The measurements were conducted by applying a constant spindle velocity of 20 RPM. To achieve a limited statistical certainty, a triple determination was carried out. In total, 12 tests were performed in accordance with EN 13302 (2010) using a Brookfield DV-III RV with two different spindles (SC4-21 and SC4-27).

2.2.3. Dynamic shear rheometer (DSR)

For this study a DSR was chosen to obtain the properties of all binder samples used in this study. The main reason to employ a DSR was the small binder amount needed for each test. DSRs are capable of measuring the rheological properties of bituminous binders under sinusoidal load. Therefore, the LVE properties are obtained over a wide range of temperatures and frequencies. The LVE properties are expressed by dynamic modulus $|G^*|$ and phase lag δ . Both, the dynamic modulus and the phase lag are used to calculate performance-related criteria (Performance Grade) in accordance with the AASHTO Standard M320 (AASHTO, 2010).

In this study, the upper SUPERPAVE measurement scheme was selected according to (AASHTO, 2010). This scheme consists of a parallel-plate geometry with a diameter of 25 mm (PP25), a specimen height of 1 mm, a sinusoidal loading frequency of 10 rad/s (1.592 Hz) and a temperature sweep from 46°C to 82°C in steps of 6 K. There are two loading modes available: stress-controlled and strain-controlled. In this study, the strain-controlled mode with a total strain of 1% of the specimen height was preferred to stay within the LVE limits of the different binders. In total, 120 tests were performed in accordance with EN 14770 (2005) using a Thermo Scientific HAAKE MARS II DSR. This rheometer consists of a thermoelectrically tempered lower plate (type TM-PE-P) and actively tempered hood (type TM-EL-H) to ensure a uniform temperature distribution within the whole specimen.

3. Results and discussion

3.1. Dynamic viscosity

In Figure 1, the results of dynamic viscosity measurements of four RTFO-aged binders are shown. It was necessary to cover a wide viscosity range to verify the assumption that the solvent residue after distillation is more dependent on binder viscosity than on distillation temperature. With these four binders it is possible to cover a viscosity range starting from 59 mPas at a test temperature of 180°C to 11,988 mPas at 120°C. In Figure 1 the mean values of the obtained results are plotted twice. *Y*-axis one (black coloured) shows the results in logarithmic and *Y*-axis two (grey coloured) in linear scaling. Based on all viscosity measurements of RTFO-aged binders a triple determination resulted in a maximum coefficient of variation of 2.8%. In addition to measurements on RTFO-aged binders also measurements on virgin binders have been carried out. Figure 2 shows the gain in dynamic viscosity due to short-term ageing employing an RTFO in comparison to the dynamic viscosity of the virgin binders. The ageing is between 9% and 81% depending on binder and RV test temperature. It is possible to indicate the test temperature as arrows because the order from 120°C to 180°C in steps of 10 K is the same for all binders tested

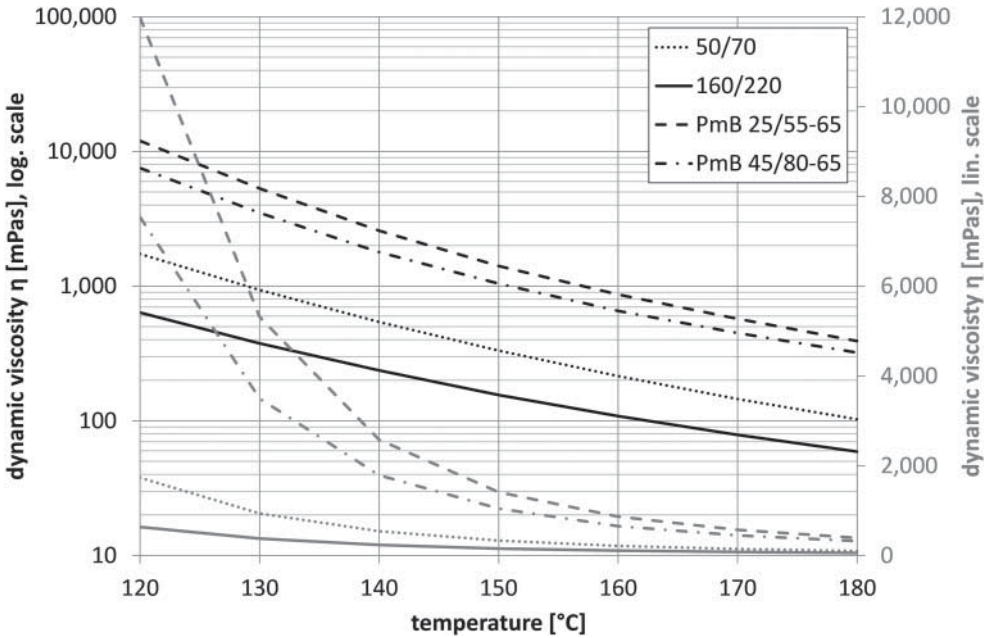


Figure 1. Dynamic viscosity of four RTFO-aged binders.

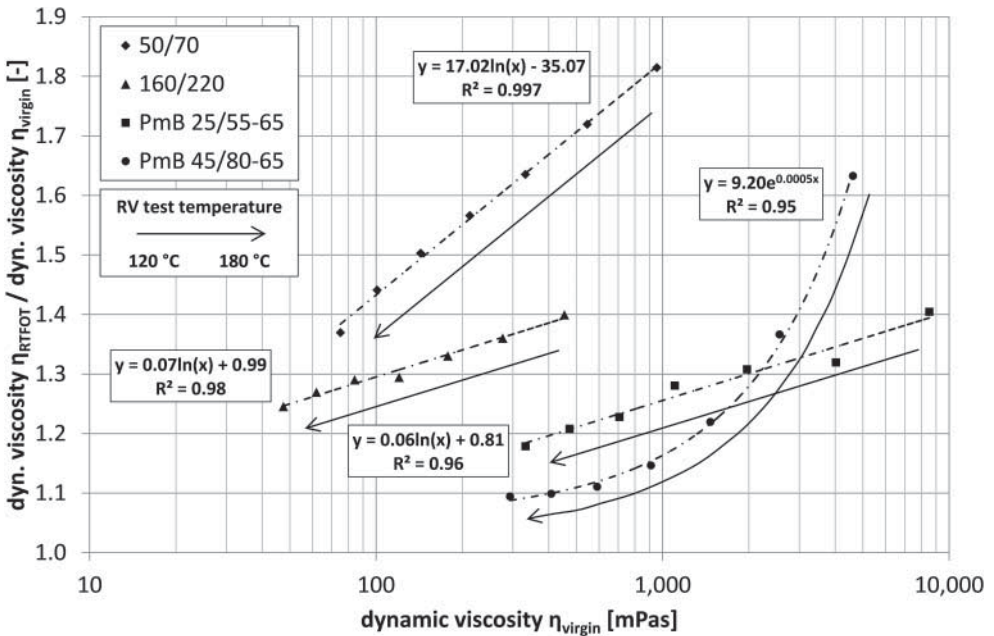


Figure 2. Gain in dynamic viscosity of four binders caused by rolling thin film oven test (RTFOT).

in this study. It can be seen that the lower the test temperature, the higher is the impact on the resulting viscosities but in detail, the ageing affects the viscosity of each binder in a very different way. Further, the gain in dynamic viscosity and thus the ageing is not dependent on the dynamic viscosity of the virgin binder. With a viscosity gain of 37–81% in comparison to the virgin sample, the paving grade bitumen 50/70 is more susceptible to RTFO-ageing than the others. Except the PmB 45/80-65, the other three binders show a logarithmic correlation between the dynamic viscosity of each virgin binder to the gain in dynamic viscosity after RTFO-ageing. The behaviour of the PmB 45/80-65 can be expressed as an exponential function and is possibly caused by the susceptibility of the base bitumen to RTFO-ageing or the polymer used for modification. Both, the logarithmic and exponential functions give good coefficients of determination of at least $R^2 = 0.95$. A second measurement has been conducted to ensure that this exponential behaviour is not due to non-linearity effects caused by the applied loading mode (constant velocity). For the second measurement, the same torque has been applied as resulted with the virgin sample at a constant velocity of 20 RPM. Both measurements delivered the same results. Thus, it is concluded that the observed behaviour is based on an ageing-related phenomenon and not due to non-linearity.

3.2. Results after binder distillation

Within the sample preparation procedure the RTFO-aged binders are dissolved in PCE, distilled at temperatures varying from 120°C to 180°C and the solvent residue is calculated as described in Section 2.2.1. Figure 3 shows the solvent residue plotted against the distillation temperature (applied maximum temperature). It can be clearly seen that lower distillation temperatures cause higher solvent residues in the recovered binder. There is almost no change for the 160/220 sample. This is due to the already very low viscosity at 120°C. Figure 3 also shows that it is necessary to apply a distillation temperature of at least 160°C for paving grade and 180°C for polymer-modified bitumens to reach a maximum solvent residue of < 1% w/w.

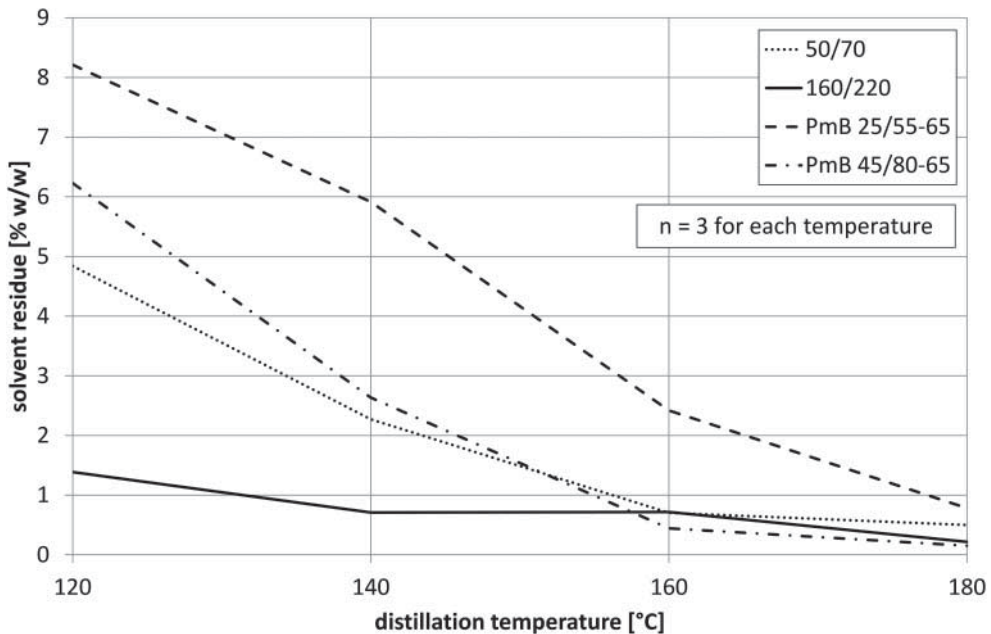


Figure 3. Distillation temperature dependency of solvent residue.

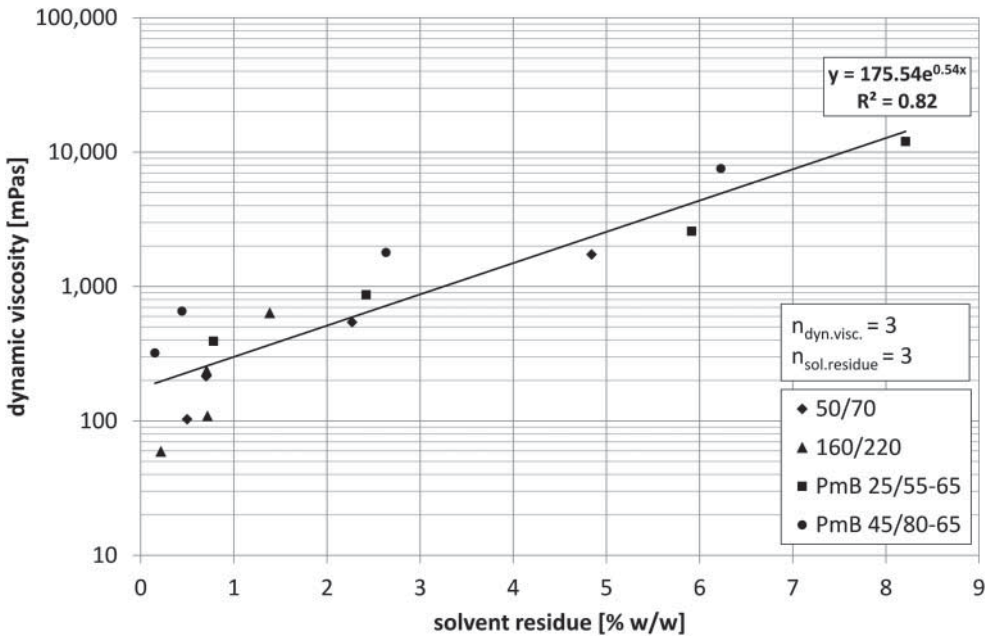


Figure 4. Comparison of solvent residue and dynamic viscosity of four binders.

Figure 4 is a combination of the results from Figures 1 and 3. It shows the solvent residue plotted against the dynamic viscosity of all four binders at the same temperature. Each data point consists of a triple determination of both, the dynamic viscosity and the solvent residue. It can be clearly seen that the solvent residue is highly dependent on binder viscosity of both, paving grade and polymer-modified bitumen. Thus, the solvent residue is a function of the binders' dynamic viscosity at distillation temperature independent from binder grade, modification and boiling point of the solvent. With low concentrations of solvent left in the recovered bitumen, the impact of the boiling point is no longer the main parameter. Instead, the diffusion and molecular associations between bitumen and solvent become more relevant. The trend line expressed as an exponential function gives a good coefficient of determination of $R^2 = 0.82$. Regarding the predefined distillation temperatures in EN 12697-3 (2013) it is preferable to define the maximum distillation temperature according to the binder viscosity instead of 'rising bubbles' from binders' surface in the distillation flask. However, it is difficult to predict the dynamic viscosity of recovered binders before they have been recovered from asphalt mixture.

Each binder sample prepared for this study has been tested by DSR applying the upper SUPERPAVE measurement scheme according to the AASHTO Standard M320 (AASHTO, 2010). Thus, it is possible to show the impact of solvent residue on the performance-related properties of recovered asphalt binders. For example, Figures 5 and 6 show the impact of solvent residue on both, the dynamic modulus and the phase lag of a PmB 45/80-65 obtained by DSR at 64°C and 1.592 Hz. In both figures, grey coloured data points are single distillations and black coloured ones the mean value at one single temperature. The error bars show the standard deviation of both, dynamic modulus/phase lag and solvent residue. The trend line expressed as a linear function gives a very good coefficient of determination of $R^2 = 0.99$ for the dynamic modulus. Figure 5 clearly shows that the higher the solvent residue the lower the dynamic modulus. In addition to the four applied distillation temperatures (120°C, 140°C, 160°C, 180°C) the very left data points show the results from an untreated RTFO-aged binder and also results from

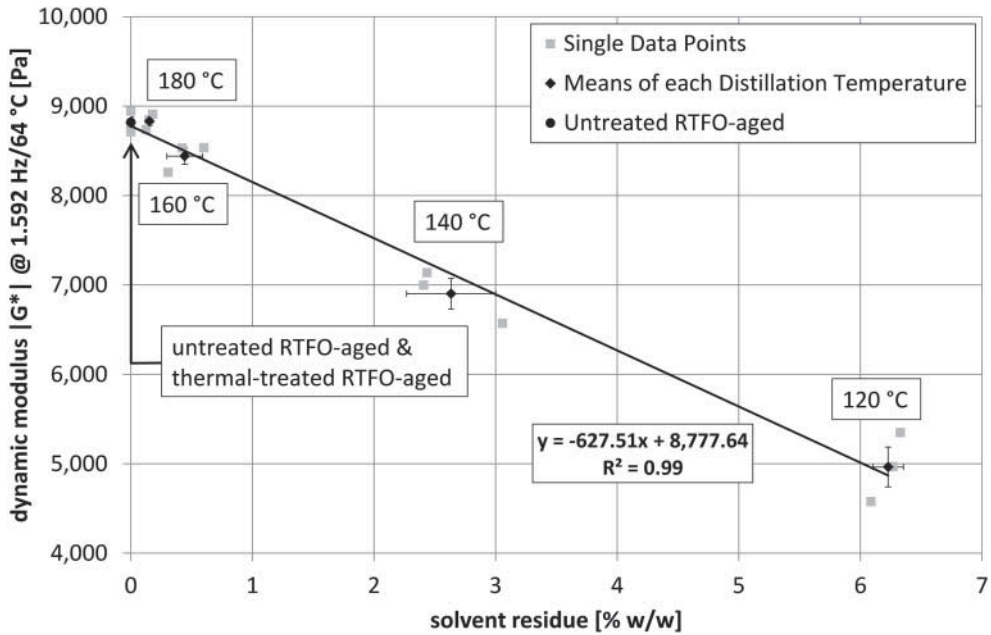


Figure 5. Impact of solvent residue on dynamic modulus $|G^*|$ of PmB 45/80-65 at 1.592 Hz and 64°C.

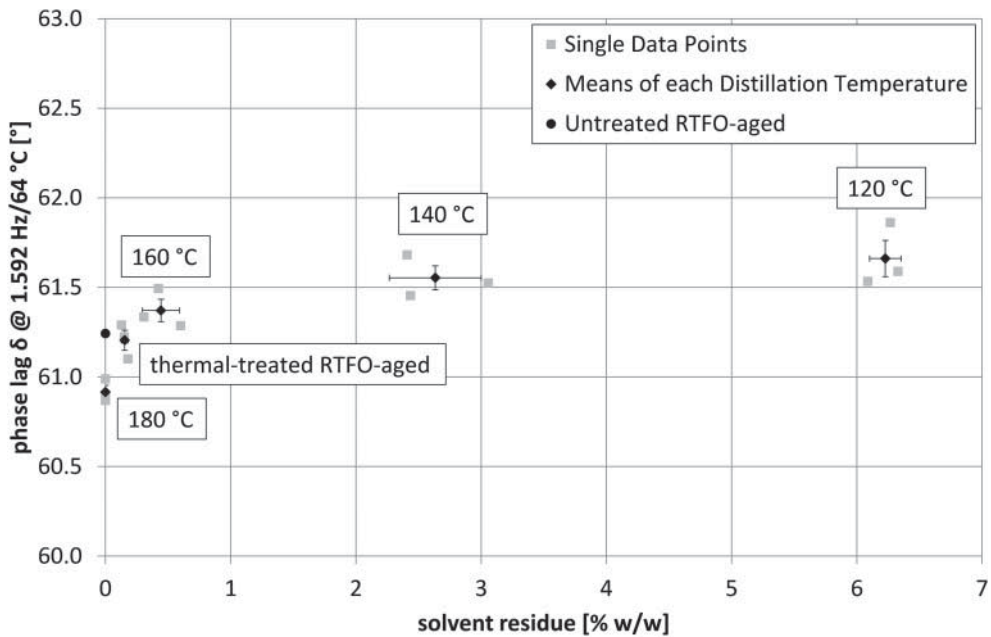


Figure 6. Impact of solvent residue on phase lag δ of PmB 45/80-65 at 1.592 Hz and 64°C.

a pseudo-distillation without solvent (thermal-treated RTFO-aged). Therefore, the binder was put into a distillation flask and treated at the highest applied temperature (180°C) for the same time as real distillations with solvent involved. This was done to find out the influence of the heating process itself during distillation. The results of untreated RTFO-aged and thermal-treated

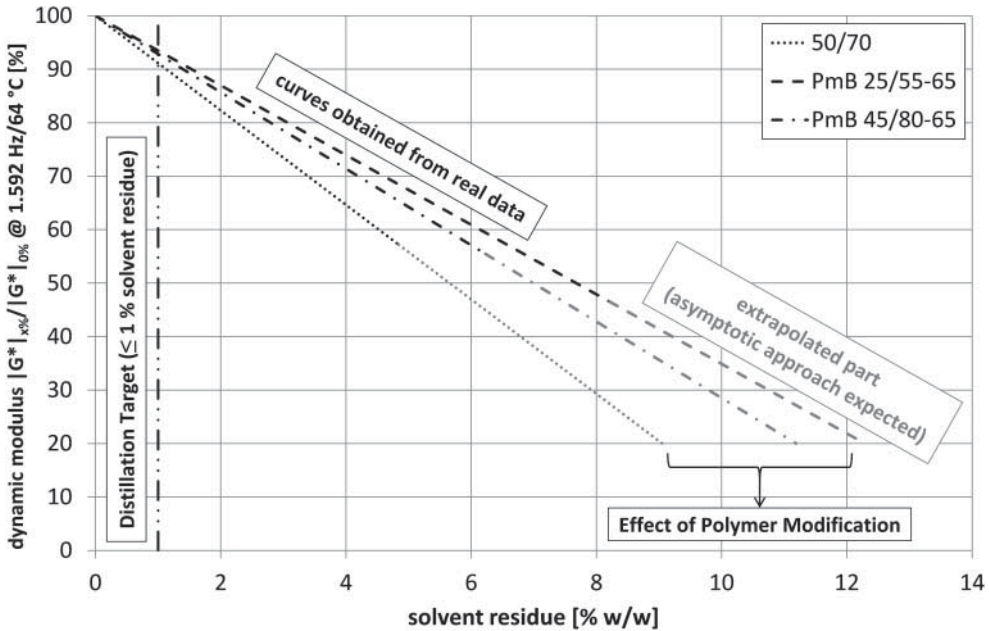


Figure 7. Impact of solvent residue on dynamic modulus $|G^*|$ of three binders at 1.592 Hz and 64°C.

RTFO-aged bitumen are so close together that it is not possible to see a difference in Figure 5. This means that there is no change of the binder properties due to heating during distillation. From laboratory experience, there is only one important requirement to achieve this result: all ground glass joints of the rotary evaporator have to be tightly sealed with grease ('A transparent joint is a tight joint.') or polytetrafluoroethylene (PTFE) sleeves. This avoids air (oxygen) from getting into the evaporator acting there as an oxidation agent. The plotted results in Figures 5 and 6 also indicate that there is no change within the binder properties caused by the use of PCE to solve the binder. All three samples, untreated RTFO-aged, thermal-treated RTFO-aged and PCE-treated RTFO-aged binder distilled at 180°C with solvent residue of < 0.2% w/w show the same dynamic modulus of about 8820 Pa. Even more, Figure 6 shows no impact to the phase lag (pay attention to the scaling of the Y-axis). Thus, the SBS modification is fully effective independently from the solvation in PCE due to the recovery and from the amount of the solvent residue in the bitumen. The bitumen is getting softer but the ratio between the storage and the loss modulus is almost the same.

While Figure 5 shows the change of dynamic modulus in absolute terms of one single binder, Figure 7 shows the relative change of the dynamic modulus of three of the four employed binders in relation to each untreated RTFO-aged sample (control sample). The presented curves consist of a black coloured and a grey coloured part. The black coloured part is calculated from actual data obtained within this study, the grey coloured part is linear extrapolated up to a dynamic modulus of zero. Close to zero some kind of asymptotic approach is expected. This extrapolation was done to show the difference between paving grade bitumen and polymer-modified ones. While both polymer-modified binders have about the same gradient, the 50/70 is a little bit steeper. A reason for this behaviour could be the polymers sticking the bitumen together and thus reducing the softening effect of the solvent residue. No results of the 160/220 paving grade bitumen are shown in Figure 7 because the solvent residues are too close together to show a reasonable curve. Even at a maximum distillation temperature of 120°C the solvent residue is close to 1% because

of the low viscosity. Following the results presented in Figure 7, it is highly recommended to remove as much solvent as possible within the distillation. Even 1% w/w of solvent residue in the bitumen reduces the dynamic modulus (64°C, 1.592 Hz) up to 10% in comparison to untreated RTFO-aged bitumen. Therefore, 1% w/w of solvent residue has to be the minimum target after distillation to ensure that the change in dynamic modulus stays below the reproducibility given by EN 14770 (2005).

4. Conclusion and recommendations

The main target of this study was to show the impact of the distillation temperature on the solvent residue and mechanical binder properties to give a recommendation for a reasonable distillation temperature. In European countries, tetrachloroethene (PCE) is widely used and thus this solvent was applied in this study. A RV and DSR was employed to obtain the properties of both, RTFO-aged binders and PCE-treated RTFO-aged binders. The distillation was done by a rotary evaporator.

The following findings can be derived from the study:

- There is no change of the binder properties caused by the influence of temperature during distillation, as long as the ground glass joints of the rotary evaporator are tightly sealed with grease or PTFE sleeves. This avoids oxygen from getting into the evaporator acting there like an oxidation agent.
- There is also no change within the binder properties obtained by DSR caused by the influence of PCE as long as the solvent is completely removed ($< 1\%$ w/w).
- The solvent residue in the recovered binder is highly dependent on the dynamic viscosity of the binder at distillation temperature and not primarily dependent on the distillation temperature by itself or the binder grading, modification or boiling point of the solvent.
- 1% w/w of solvent residue causes a reduction of up to 10% in dynamic modulus $|G^*|$ at 64°C and 1.592 Hz.
- Due to the fact that there is no change within the binder properties caused by the influence of heat, it is recommended to apply a distillation maximum temperature of 180°C for every binder, regardless of whether it is modified or unmodified.
- Due to additional laboratory experience with solvent amounts of 4–6 L in practice, it is highly recommended to use of 2000 or 3000 mL flasks instead of 1000 mL ones as required in the European Standard. Most oil baths are capable of these large flasks. This will reduce the distillation duration by up to half.
- There is no need to do an intermediate step at 160°C before applying the extra temperature of 180°C. As shown in the study there is no harm to the binder due to heat and thus the extra temperature can be applied in one single go. The recommended application time is 10 min.

Disclosure statement

No potential conflict of interest was reported by the authors.

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