# ANOTHER LOOK AT ACCELERATED AGING OF ASPHALT CEMENTS IN THE PRESSURE AGING VESSEL

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## ABSTRACT

*Pavement performance and durability are determined in large part by the way in which the rheological properties of the bitumen change over time. In the late 1980s, the U.S. Strategic Highway Research Program adopted the Pressure Aging Vessel (PAV) for the accelerated aging of bitumens. The PAV was meant to produce materials for low temperature and fatigue performance grading with properties similar to those reached after 8-10 years of service. Despite a vast amount of literature that discusses the chemical aging of bitumens, the currently used PAV protocol still fails to replicate the aging process as it occurs in service. Few studies use a comprehensive approach by comparing laboratory and field aging for a sufficiently large sample set. This paper discusses how relatively simple changes to the PAV protocol can lead to significant improvements in performance grading. Materials from a pavement trial and regular contracts in Ontario, Canada, are used to demonstrate the major benefits of aging in thinner films and for longer periods. Low temperature grade losses ranged from modest amounts for good quality bitumens to highs of 10°C for certain modified materials known to suffer from premature, severe, and excessive failures in service.*

**Keywords:** pressure aging vessel, accelerated aging, bitumen, specification grading, cracking, field validation

## 1. INTRODUCTION

## 1.1 Development of the Rolling Thin Film Oven and Pressure Aging Vessel

The Rolling Thin Film Oven (RTFO) and Pressure Aging Vessel (PAV) were adopted by researchers funded through the U.S. Strategic Highway Research Program (SHRP) to provide a convenient and rapid method for the chemical aging of bitumens in the laboratory [1-5]. The PAV prototype varied only slightly from what had been used to study bitumen oxidation in Iowa during the late 1960s [6, 7]. While the original method called for pure oxygen at 300 psi and 60°C, and a 144 hour aging time, the final American Association of State Highway and Transportation Officials (AASHTO) standard R28 specified compressed air, a temperature of 90°C, 100°C, or 110°C (depending on the climate), and a time of 20 hours [8]. For safety reasons the oxygen was changed to air. To facilitate the wide acceptance of the protocol, the aging time was shortened to 20 hours by increasing the temperature from 60°C to around 100°C. The 50 g of bitumen per PAV pan was dictated by the desire for a homogeneous film thickness and a sufficient amount of material for further testing. It was recognized by SHRP researchers that the compromises made would sacrifice some accuracy, but this loss was considered acceptable.

The PAV-aged bitumen is used in the Superpave™ specification, as now embodied in AASHTO standard M320 [9], for performance grading, with the aim of controlling cracking. Early validation attempts for the tests and specification criteria have demonstrated that AASHTO M320 is deficient [10-13]. It fails to adequately control fatigue and thermal cracking, and on more than a few occasions bitumens of identical grade have shown anywhere from best-case to worst-case performance in service [14-18].

A significant part of the problem with the specifications originates from the inability of the PAV protocol to replicate chemical aging as it occurs in service. Early efforts by SHRP researchers looked at the effect of film thickness, aging temperature, and aging time on the performance-based properties as measured in the penetration test [1] and Dynamic Shear Rheometer (DSR) test [5]. These studies revealed that the variables investigated could have a significant influence on the properties measured, especially in the case of poor quality bitumens. However, after these early laboratory studies, there do not appear to have been any attempts at improving the PAV protocol with the aid of field validations. In large part, this dearth of studies may be due to the significant inertia that occurs once a standard is established. The consequences of an imperfect aging method are demonstrated in the photographs in Figure 1. These images show two regular Ontario pavement contracts with opposite performance. Similar variations have been found in numerous other contracts around Ontario [15]. Hence, the problem is of a magnitude that justifies further study on how to improve the AASHTO R28 standard.

**FIGURE 1 – Performance variation for bitumens of nearly identical Superpave grades but different rheological type ((a) sol-type versus (b) gel-type) [15].**

*Note:* These are representative 2009 photographs. The first 30 km contract was constructed in 1999 on Highway 11 with unmodified sol-type bitumen. The left image is right above a major culvert. The second 12.4 km contract was constructed in 1998 on Highway 41 with gel-type bitumen (straight bitumen blended with waste engine oil residue (WEO)). The only explanation for this performance difference was found to be the difference in bitumen rheological type and thus chemical aging tendency and quality (WEO gels the bitumen) [16].

### 1.2 Study Objectives

The objective of the research described herein was to aid in the development of an improved PAV aging method for bitumen. It was decided to aim for only simple changes to the current approach in order to facilitate the wide acceptance of the outcome of this research. A previous publication by the authors describes the chemical findings of this study [17]. Described here are the effects of film thickness, the presence of moisture during aging, and aging time on the high and low temperature rheological properties.

## 2. MATERIALS AND EXPERIMENTAL METHODS

### 2.1 Materials

The materials investigated in this study were obtained from different sources. Seven bitumens from six suppliers were sampled during the construction of a 2003 pavement trial on Highway 655 north of Timmins, Ontario (T1-T7) [14]. Four additional materials were obtained during the construction of a warranty contract in northern Ontario (W1), and during the construction of a number of regular contracts in Edmonton, Alberta (E1-E3).

The Highway 655 trial materials graded in a narrow range between -35°C and -36°C, just above the -37°C to -38°C required to guarantee a 98% confidence level that no damage occurs in any given winter for the trial location [14]. The Ontario warranty material graded as a PG 58-34, while all three Edmonton materials graded as PG 70-28. Further pertinent information is provided in Table 1.

**TABLE 1 – Investigated Bitumen Compositions**

| Bitumen Code | Crude Source | Technology | Superpave Grade, °C | Grade Span, °C |
|---|---|---|---|---|
| T1 | Lloydminster | RET + PPA | 64-34 | 101 |
| T2 | Unknown | Air Blown + SB | 64-34 | 101 |
| T3 | Unknown | SB | 64-34 | 101 |
| T4 | Unknown | SB + WEO | 64-34 | 102 |
| T5 | Unknown | SB | 64-34 | 101 |
| T6 | Unknown | Air Blown + P31 + SB | 58-34 | 94 |
| T7 | Unknown | WEO | 52-34 | 89 |
| W1 | Unknown | SB | 58-34 | 94 |
| E1 | Montana | SB | 70-28 | 100 |
| E2 | Western Canadian | SB | 70-28 | 101 |
| E3 | Western Canadian | Unknown | 70-28 | 103 |

*Note:* P31 = either PPA or $H_3PO_4$ used as a catalyst for the air blowing, PPA = Polyphosphoric Acid as a catalyst for RET modification, RET = Reactive Elastomeric Terpolymer (DuPont Elvaloy®), SB = Styrene-Butadiene or Styrene-Butadiene-Styrene copolymer, and WEO = Waste Engine Oil residue. Sample E3 was found to be free of SB, PPA, or other known additives.

### 2.2 Experimental Procedures

All bitumens were aged according to RTFO (AASHTO T240 [19]) and PAV (AASHTO R28 [8]) protocols, and their grades were determined by using Superpave criteria (AASHTO M320 [9]). In addition, modified aging protocols were investigated as summarized in Table 2.

Field-aged bitumens were obtained from cores taken from between the wheelpaths of each test section on Highway 655 [14, 17]. The surface lift of each core was separated and broken into small pieces. The material was immersed in toluene for 24 hours, after which the solvent was decanted. The aggregate was rinsed an additional four to five times with toluene and twice with a toluene/ethanol (80/20) mixture. A total of 6 L of solvent was used to extract 200 g of bitumen from 4 kg of asphalt. The bulk of the solvent was removed at a temperature of about 100°C over several hours using a rotary evaporator. Once the last drops of solvent were transferred to the receiving flask, the temperature of the oil bath was raised to 160-170°C and the vacuum was reduced to 20-60 mbar for an additional hour to remove remaining traces of solvent. Infrared spectroscopy was used to check that no additional oxidation occurred during the recovery process.

Materials were tested in the Bending Beam Rheometer (BBR, AASHTO T313 [20]) and the Dynamic Shear

Rheometer (DSR, AASHTO T315 [21]) according to standard procedures.

**TABLE 2 – Modified Pressure Aging Vessel Protocols**

| Aging Protocol | Aging Time, h | Bitumen Weight/Pan, g | Oxidizing Agent |
|---|---|---|---|
| $PAV_{20\text{-}50}$ | 20 | 50 | Extra Dry Air |
| $PAV_{20\text{-}25}$ | 20 | 25 | Extra Dry Air |
| $PAV_{20\text{-}12.5}$ | 20 | 12.5 | Extra Dry Air |
| $PAV_{20\text{-}12.5\text{-}W}$ | 20 | 12.5 | Moist Air |
| $PAV_{40\text{-}50}$ | 40 | 50 | Extra Dry Air |

*Note:* All used regular RTFO residues. $PAV_{20\text{-}50}$ followed the protocol provided in AASHTO R28 [8]. The moisture was introduced in $PAV_{20\text{-}12.5\text{-}W}$ by placing an extra pan with 25 mL of water inside the vessel.

## 3. RESULTS AND DISCUSSION

### 3.1 High Temperature Rheology

The data obtained in the DSR analysis of the bitumens used in the Highway 655 test sections are provided in Figure 2. Black space diagrams of log G* versus the phase angle are used for the analysis, as they provide a comprehensive picture of the rheological state of a material without having to rely on time-temperature shifting.

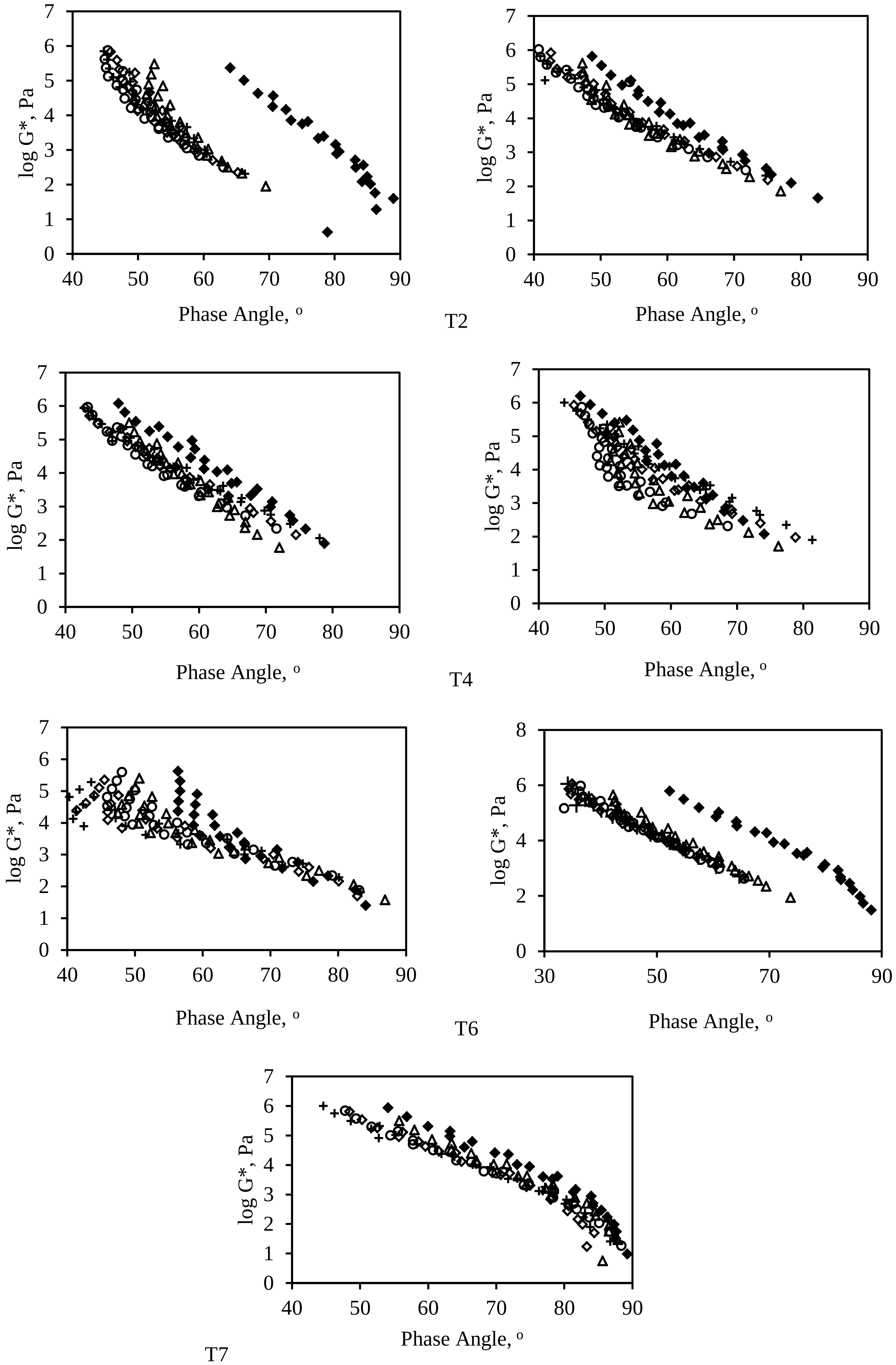
The diagrams reveal that the phase angle is shifted to higher values (less elastic and more viscous behaviour) for several of the recovered samples compared to their corresponding PAV residues. This shift could be explained in several different ways. It is possible that the extraction and recovery processes either failed to recover the polymer or somehow changed the polymer and/or base bitumen structure.

Care was taken to ensure that no additional oxidation occurred during the evaporation and that all the solvent was removed from the evaporator. Our previous publication on the spectroscopic findings shows that the styrene indices for the recovered residues were close to those of the unaged and laboratory-aged materials (meaning that at least the polystyrene parts of the SB copolymers were largely recovered) [17]. In contrast, a significant deficit for the butadiene indices was found, with several recovered bitumens showing little or no sign of the original polymer component. This could be explained through a significant degree of polymer degradation for the SB-type modifiers in service. The butadiene backbone is prone to oxidative cleavage, which in turn would show up as an increase in phase angle at high temperatures. This effect could be positive if it allowed the bitumen to flow more readily and heal damage during the summer. However, as will be discussed in the following section, the cracking distress is largely determined by the low temperature properties, and when these are under-designed, any benefits of healing will largely be overshadowed by the detrimental effects of the prior cracking events.

For the RET modified bitumen, the cause of the rather large shift to higher phase angles is likely due to an inability to recover all the modifier. This epoxy-type polymer reacts with the asphaltenes to a degree that it becomes insoluble in solvents like toluene.

Finally, the shift could be due to a limited degree of reversible structuring in the laboratory aged materials. Asphaltenes will be less strongly associated just after solvent recovery, and hence in such circumstances the phase angle may be higher. However, this steric hardening process is considered to be reversible [22].

Figure 2 shows that the harsher PAV aging protocols produce stiffer materials which more closely match the stiffness properties recovered from the trial sections during summer 2011. The materials move towards lower phase angles and higher complex moduli when aged in thinner films, for longer times, or in thinner films in the presence of moisture. Hence, the modified PAV protocols appear to provide a net benefit.



T1 T2 T3 T4 T5 T6 T7

**FIGURE 2 – Black space diagrams for PAV and recovered residues from Highway 655 test sections.**
*Note:* Measurements were obtained at temperatures of 34, 46, 58, 70, and 82°C and frequencies of 0.1, 0.3162, 1, 3.162 and 10 rad/s. Δ = $PAV_{20\text{-}50}$, ◊ = $PAV_{20\text{-}12.5}$, + = $PAV_{20\text{-}12.5\text{-}W}$, ○ = $PAV_{40\text{-}50}$, ♦ = recovered.

### 3.2 Low Temperature Rheology

The findings for the recovered bitumens and various PAV residues are provided in Table 3. The grade temperatures for the recovered bitumens were determined in 2008, 2009 and 2011, after five, six, and eight years of service, respectively. The PAV results for bitumens W1 and E1-E3 are provided for comparison, as these were likely produced from superior quality, western Canadian crude sources. The individual PAV$_{20-50}$ aging errors for each section in Table 3 are determined by subtracting the recovered grades after eight years of service (column 4) from the regular AASHTO R28 grades (column 5).

**TABLE 3 – Limiting BBR Temperatures for Recovered and PAV-Aged Bitumens and AASHTO R28 Errors**

| Code | Recovered Bitumens | | | PAV-Aged Bitumens | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 2008 | 2009 | 2011 | PAV$_{20-50}$ | Error, °C | PAV$_{20-25}$ | PAV$_{20-12.5}$ | PAV$_{20-12.5-W}$ | PAV$_{40-50}$ |
| T1 | -38.5 | -34.3 | -33.5 | -35.7 | -2.2 | -34.4 | -33.9 | -34.3 | -32.8 |
| T2 | -34.2 | -34.0 | -29.3 | -35.7 | -6.4 | -29.4 | -25.2 | -26.8 | -25.9 |
| T3 | -34.2 | -29.6 | -28.3 | -37.0 | -8.7 | -29.1 | -28.8 | -31.2 | -30.0 |
| T4 | -25.0 | -26.7 | -23.3 | -36.0 | -12.7 | -31.6 | -26.8 | -25.1 | -28.4 |
| T5 | -31.1 | -35.8 | -32.5 | -35.4 | -2.9 | -31.4 | -29.3 | -29.6 | -29.9 |
| T6 | -30.4 | -26.6 | -30.5 | -34.6 | -4.1 | -28.8 | -24.6 | -23.8 | -27.2 |
| T7 | -34.3 | -27.5 | -28.6 | -34.7 | -6.1 | -30.5 | -28.1 | -23.9 | -27.7 |
| W1 | - | - | - | -32.9 | - | -32.1 | -31.0 | - | -28.3 |
| E1 | - | - | - | -29.6 | - | -27.6 | -26.8 | - | -26.8 |
| E2 | - | - | - | -30.6 | - | -29.1 | -27.5 | - | -28.7 |
| E3 | - | - | - | -33.5 | - | -32.3 | -31.3 | - | -31.8 |

*Note:* The recovery of the trial bitumens in 2008 and 2009 was done with tetrahydrofuran (THF) as described previously [14], while the recovery in 2011 was done with toluene followed by toluene/ethanol as described earlier. Aging experiments with moist air were done only for Highway 655 materials.

The data are further analysed in Figure 3 where both limiting S(60 s) and m(60 s) temperatures are plotted versus aging protocol. In addition, Figure 4 provides the average changes versus the regular AASHTO R28 protocol as well as the average errors and root mean square deviations (residuals) for each aging protocol.

Figure 3 shows that there appear to be two groups of materials. Group A bitumens T2-T7 and W1 rapidly lose their relaxation ability upon harsher aging in thinner films, for longer times, and due to the presence of moisture. The m-value decreases, with some bitumens losing as much as 10°C from their low temperature grade. These are gel type materials with low stiffness and a zero shear viscosity approaching infinity.

In contrast, bitumens T1 and E1-E3 in group B suffer only modest losses in both limiting stiffness and m-value temperatures upon harsher aging. These materials were likely made with Lloydminster or Cold Lake bitumens, which are known to provide excellent durability in service due to their high contents of naphthenic hydrocarbons and low contents of linear paraffins. They suffer little hardening from the formation of additional asphaltenes [23].

It is also interesting to note that the worst performers were bitumens T2, T4, T6, and T7. These materials were either air blown (T2 and T6) or modified with WEO (T4 and T7). This result clearly shows that the AASHTO M320 specification, as implemented in much of North America, rewards the wrong modification technologies.

The material used in the warranty contract (W1, ▲) falls within group A and hence is unlikely to provide the durability desired. However, it should be noted that its loss in going from regular PAV$_{20-50}$ aging to PAV$_{20-12.5}$ is only 1.9°C and to PAV$_{40-50}$ still a respectable 4.6°C.

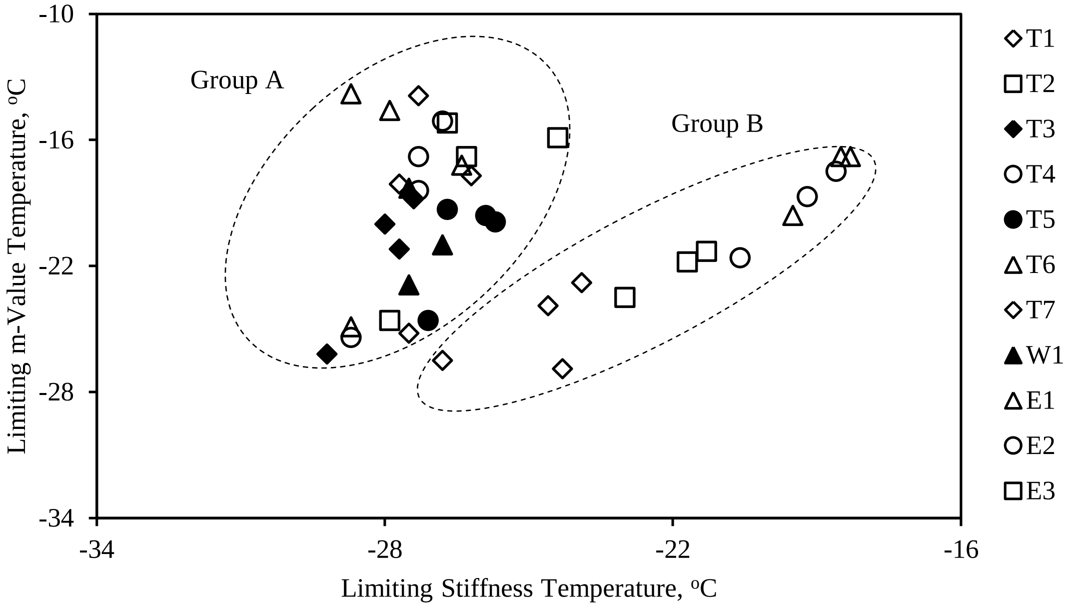


**FIGURE 3 – Limiting stiffness and m-value temperatures versus PAV aging protocol.**
*Note:* The coldest limiting temperatures for each material represent the AASHTO R28 protocol, while the warmest typically represent either $\mathrm{PAV}_{20\text{-}12.5\text{-W}}$ or $\mathrm{PAV}_{40\text{-}50}$ (see Table 3).

The error analysis in Figure 4 shows that the average losses for the modified protocols are significant, ranging from 4.8°C to 7.8°C. Such potential errors can explain the opposite performance as shown in Figure 1 and the variable cracking in the Timmins trial. The average deviation from the recovered grades after eight years of service is -6.2°C for the AASHTO R28 protocol. Hence, the current method falls short by more than a full grade (6°C), an amount that typically reduces the confidence that no damage occurs in any given winter from the intended 98% to less than 50% [24]. The data show that the modified protocols all do much better than the current one, with average deviations from the recovered grades ranging from -1.3°C to 1.6°C.

However, average deviations only tell part of the story, as producers are more interested in the root mean square deviations since they better account for over-designs when a laboratory protocol ages the material by too much. Such analysis provides similar conclusions with significant improvements under harsher aging conditions.

Finally, user agencies are most concerned about situations in which the pavement is under-designed, as these can produce early cracking [15, 16]. Hence, for users the most desirable protocol is the one that penalizes the poor performers most while leaving the good performers relatively untouched. It is obvious that for user agencies the $\mathrm{PAV}_{20\text{-}12.5\text{-W}}$ is the best option in this respect.

### 3.3 Pavement Trial Performance

The performance of the pavement trial sections on Highway 655 has been reviewed on a number of prior occasions [14, 17]. In brief, bitumens used in this trial can be divided into three groups. The material in Section T1 has performed as desired, with virtually no cracks showing after eight years of service. The material in Section T5 has cracked by a moderate amount, with intermittent full width transverse cracks of moderate severity. The materials used for the remaining sections have all experienced severe and excessive distress, with numerous longitudinal and transverse cracks showing throughout. Figure 5 shows the approximate cracking counts for each section as a function of the original and recovered 2011 grades. It is obvious that there is a strong correlation between cracking distress and the tendency to harden.

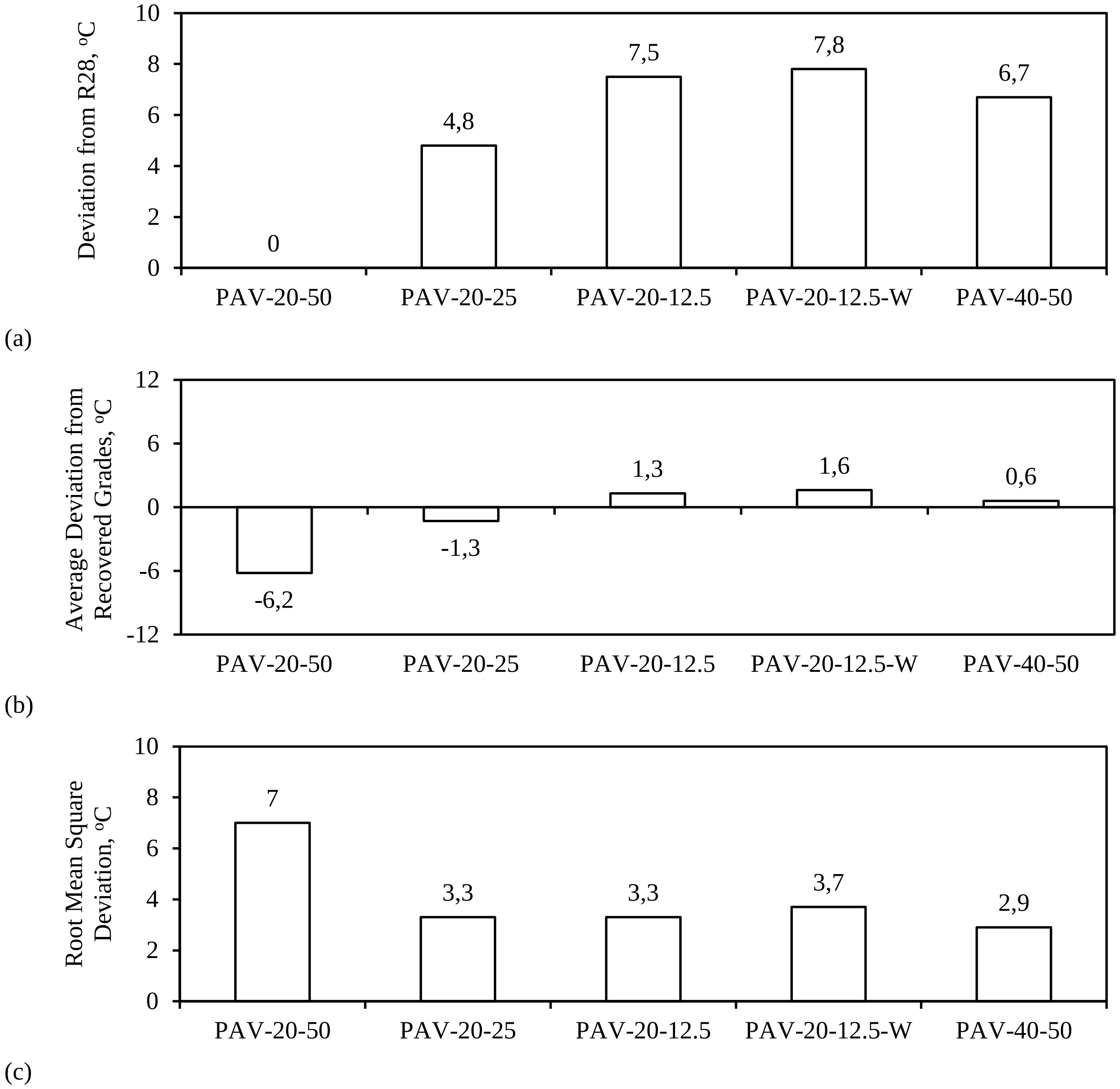


**FIGURE 4 – Average grade deterioration and errors in modified pressure aging vessel protocols: (a) changes from regular AASHTO R28 protocol; (b) average deviations from recovered 2011 grades; and (c) root mean square deviations from recovered 2011 grades.**

Data in Table 3 combined with the cracking distress counts of Figure 5 suggest further changes will occur. Sections 6 and 7 will likely age more in years to come, and their cracking distress will increase correspondingly. The high correlation between crack counts and recovered grades in Figure 5 is probably to some extent accidental, since total crack lengths and pavement condition indices would provide different correlations. Irrespective, the $\text{PAV}_{20\text{-}12.5\text{-W}}$ protocol provides a meaningful improvement over AASHTO R28.

### 3.4 Relationship between Chemical and Physical Hardening

The bitumens investigated in this study were also assessed for their tendency to physically harden during conditioning for three days at a temperature of -20°C [25]. Figure 6 shows a graph of the loss in low temperature grade after $\text{PAV}_{40\text{-}50}$ aging versus the loss in grade due to conditioning at -20°C. This result suggests that the same bitumen constituents must be responsible for increases in stiffness due to additional conditioning in the BBR and aging in the PAV. This explanation was suggested by several authors, starting with the work of Royal Dutch/Shell researchers [23, 26, 27]. Labout stated in 1950 [23], "Owing to their aromatic structure, asphaltenes are more or less polarisable, which may induce mutual attraction. This attraction may also be promoted by the presence of polar groups, derived from sulphur, oxygen or nitrogen atoms in the asphaltenes. The mutual attraction results in the formation of a gel structure if the surface of the particles is heterogeneous; hence attraction is limited to relatively few places."

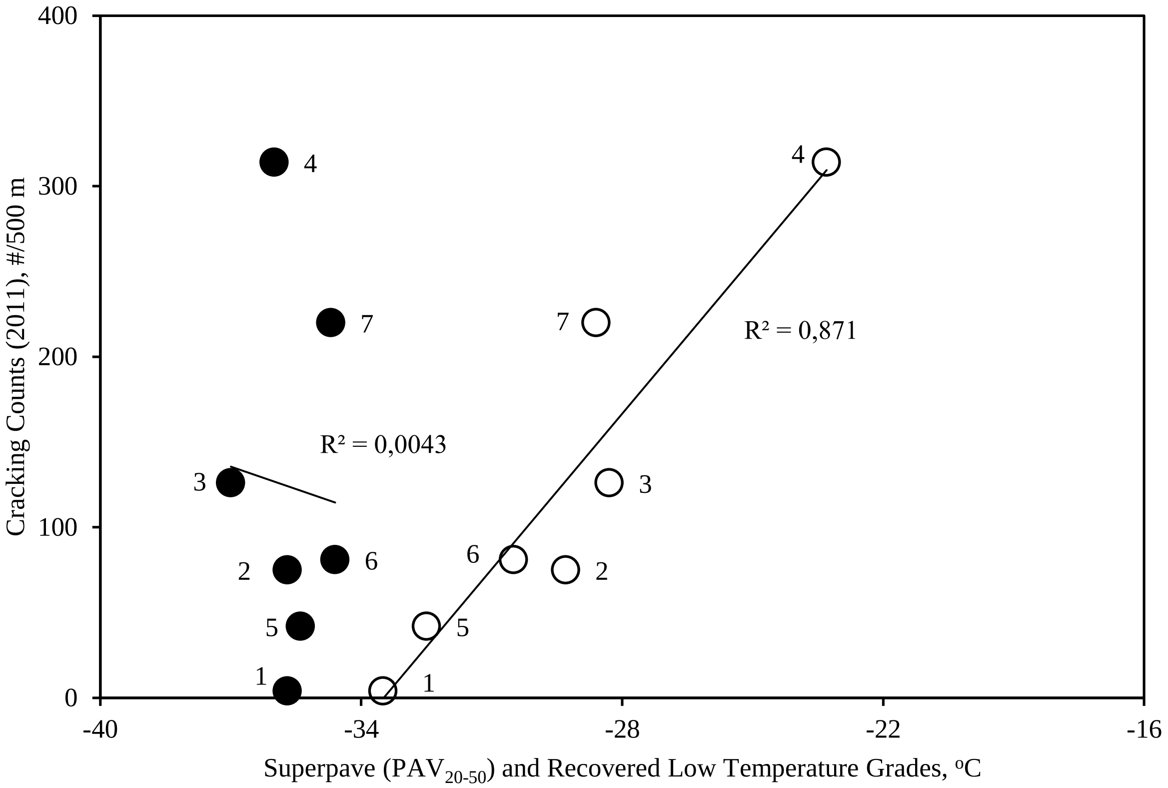


**Figure 5 – Cracking distress versus Superpave (filled symbols) and recovered (open symbols) grades [17].**
*Note:* Compositions are provided in Table 1.

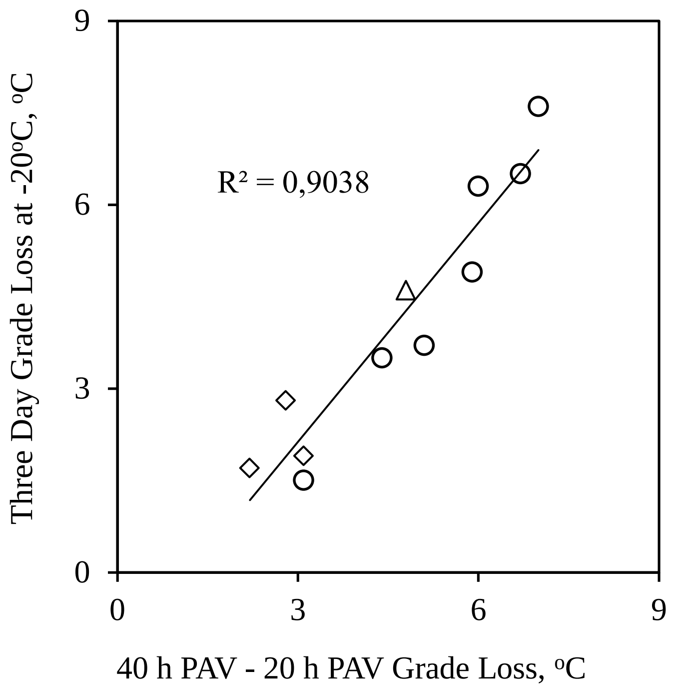


**Figure 6 – Physical versus chemical hardening tendencies [28].**
*Note:* Circles are for Highway 655 materials, diamonds are for E1-E3, and triangle is for W1.

This comparison shows that the effects of physical and chemical hardening are very similar. Bitumens that physically harden are also likely to suffer from premature oxidative hardening. Both processes originate from the imbalance between the solubility properties of the asphaltenes and the maltenes [22, 23, 27]. When maltenes lack the aromaticity to solvate or peptize asphaltenes, a gel type structure is formed with a high viscosity (low m-value) at low temperatures, leading to premature and excessive thermal cracking [14-18, 29].

## 4. SUMMARY AND CONCLUSIONS

Given the findings of this research, the following summary and conclusions are offered:

- AASHTO R28 is deficient as it fails to age bitumen to a degree that reflects 8-10 years of service.
- Gel type bitumens that are air blown or modified with waste engine oil residue appear to be particularly prone to premature, severe, and excessive cracking. This tendency is likely due to the early formation of a skin on the bitumen that hinders the free diffusion of oxygen during PAV aging.
- Aging for longer times, or in thinner films, and/or in the presence of moisture provides significant improvements, as poor quality bitumens are penalized more than good quality ones. Grade losses ranged from small amounts for good quality bitumens from western Canadian sources to 10°C for modified materials known to fail in service.
- Extended conditioning prior to BBR testing provides similar grade losses to those from additional chemical aging in the PAV. This result suggests that the same bitumen constituents are involved in both hardening processes.

It is up to the producers and users of bitumen to implement the changes proposed. Minor changes in the PAV protocol can provide major benefits by preventing the occurrence of premature failures, thus allowing user agencies to better pay for performance.

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## DISCLAIMER

None of the sponsoring agencies necessarily concurs with, endorses, or agrees to adopt the findings, conclusions, or recommendations either inferred or expressly stated in subject data developed in this study.

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