3rd International Conference on Road and Rail Infrastructure
28–30 April 2014, Split, Croatia

Road and Rail Infrastructure III
Stjepan Lakušić – EDITOR
ORGANISATION

CHAIRMEN

Prof. Stjepan Lakušić, University of Zagreb, Faculty of Civil Engineering
Prof. Željko Korlaet, University of Zagreb, Faculty of Civil Engineering

ORGANIZING COMMITTEE

Prof. Stjepan Lakušić
Prof. Željko Korlaet
Prof. Vesna Dragčević
Prof. Tatjana Rukavina
Assist. Prof. Ivica Stančerić
dr. Maja Ahac
Ivo Haladin
dr. Saša Ahac
Josipa Domitrović
Tamara Džambas

All members of CETRA 2014 Conference Organizing Committee are professors and assistants of the Department of Transportation, Faculty of Civil Engineering at University of Zagreb.

INTERNATIONAL ACADEMIC SCIENTIFIC COMMITTEE

Prof. Vesna Dragčević, University of Zagreb
Prof. Isfendiyar Egeli, Izmir Institute of Technology
Prof. Rudolf Eger, RheinMain University
Prof. Ešref Gačanin, University of Sarajevo
Prof. Nenad Gucunski, Rutgers University
Prof. Libor Izvolt, University of Zilina
Prof. Lajos Kisgyörgy, Budapest University of Technology and Economics
Prof. Željko Korlaet, University of Zagreb
Prof. Zoran Krakutovski, University of Skopje
Prof. Stjepan Lakušić, University of Zagreb
Prof. Dirk Lauwers, Ghent University
Prof. Zili Li, Delft University of Technology
Prof. Janusz Madejski, Silesian University of Technology
Prof. Goran Mladenović, University of Belgrade
Prof. Otto Plašek, Brno University of Technology
Prof. Vassílios A. Profillidis, Democritus University of Thrace
Prof. Carmen Racanel, Technical University of Civil Engineering Bucharest
Prof. Tatjana Rukavina, University of Zagreb
Prof. Andreas Schoebel, Vienna University of Technology
Prof. Mirjana Tomičić-Torlaković, University of Belgrade
Prof. Audrius Vaitkus, Vilnius Gediminas Technical University
Prof. Nencho Nenov, University of Transport in Sofia
Prof. Marijan Žura, University of Ljubljana
ASSSESSMENT OF AN APPROPRIATE MODIFIER CONTENT IN MODIFIED BITUMEN BASED ON THE MULTIPLE STRESS CREEP RECOVERY TEST

Jan B. Król, Piotr Radziszewski, Karol J. Kowalski, Michał Sarnowski
Warsaw University of Technology, The Faculty of Civil Engineering, Poland

Abstract

Construction of a durable bituminous pavements requires high quality materials such as bituminous binders with extended viscoelastic properties. The main objective of this paper was to compare and analyse high temperature viscoelastic properties of polymer modified bitumen (PMB) in order to assess appropriate modifier content. In this paper, there is described multiple stress creep recovery (MSCR) procedure in accordance to Superpave PG specification. Using the MSCR procedure, comparison with a traditional PG criteria was discussed. PG criteria and MSCR procedure were used to asses properties of polymer modified bitumens produced in Europe and to classified them in accordance to European standards. Additionally, new MSCR procedure was used to verify properties of binders used for construction of high traffic load pavements. Based on a conventional tests (penetration, softening point, elastic recovery and Fraass breaking point) as well as recovery and non-recoverable creep compli-ance obtained from MSCR test, amount of an appropriate modifier content was analysed. It was concluded, that although bituminous binders complies with European specification requirements, they are significantly different in terms of their rheological properties due to the modifier content. It was found, that polymer modified bitumen within the same hardness group exhibit different high temperature properties. This work confirmed poor correlation between elastic recovery in ductility test versus recovery from MSCR test.

Keywords: polymer modified bitumen (PMB), multiple stress creep recovery (MSCR), modification

1 Introduction

Superpave specification was developed by the Strategic Highway Research Program (SHRP) in the United States about 20 years ago. Research phase was launched in 1987, lasted 5 years and costs about 150 million dollars. Implementation stage of the program began in 1993 and up to now the requirements of Superpave are subjected to verification and changes [1]. The Superpave specification is using climatic zones to determine the performance grade of asphalt binders. Binders should comply the requirements for specific climate zone and at the same time, Superpave specification does not impose requirements on the composition and origin of the binders. Manufacturers of asphalt binders in the United States in order to increase the functional scope of the use of binders commonly modifies asphalt binders. Superpave requirements in the original assumption were developed taking into account the average traffic speed and load. For heavy and/or slow traffic Superpave recommends increasing upper PG (performance grade) to the next level. As the result of this recommendation, manufacturers stiffer the binder by high polymer level modification. It hence increases the costs and raise the technological temperature of binder application.
The second major issue arriving when increasing PG is the lack of “sensibility of the test method” on the type of modification. In the United States for the purpose of stiffening binders and raise the type of PG, different types of modifiers (not polymer type) were started to be used. It has led to meet Superpave requirements, but it not improved pavement properties. The value of the parameter $G^*/\sin\delta$ is overwhelmingly dependent on the stiffness modulus value and in much lesser extent depends on the value of the phase angle. While the properties of bitumen modified with elastomers (e.g. SBS type) advantages over traditional road bitumens in an increase of the elasticity (that is not dependent on the stiffness of the binders), it is strongly dependent on the value of phase angle [2].

In response to the verification of the requirements of Superpave to adapt them to changing material modification trends and with take into account the nature of the traffic, the new Superpave requirements introduced the test method of multiple stress creep recovery (MSCR) [3].

2 Materials and testing methods

2.1 Materials

Binder samples used in this study were obtained from asphalt plants located in north-east, central, south and south-west parts of Poland. Two types of polymer modified bitumens PmB 25/55-60 (five samples) and PmB 45/80-55 (five samples) from two binder producers, were acquired. Each sample was obtained according to EN 58 (Bitumen and Bituminous Binders – Sampling Bituminous Binders) specification during binder unloading when transported to the asphalt mixture plant. Modified binders produced in Poland are obtained from Russian crude oil and modified with SBS polymer. It has to be noted, that according to the European requirements, bituminous producers providing PmB does not have to characterize content of the polymer used for modification (typical value of the SBS content in Polish market is about 3-5%).

2.2 Conventional testing methods

All binders in this study were tested with conventional classification test such as penetration at 25°C according to EN 1426, softening point according to EN 1427 and elastic recovery according to EN 13398. Additionally, samples were tested using dynamic shear rheometer according to EN 14770 and bending beam rheometer according to EN 14771 in order to analyze rheological properties and for the PG classification. Properties of polymer modified bitumen were obtained for non-aged as well as short-term (RTFO – Rolling Thin Film Oven) and long-term (PAV – Pressure Ageing Vessel) aged samples in accordance to Superpave classification. Properties of modified binders were evaluated according to EN 14023:2010 specifications (with Polish normative appendix NA). Test results obtained from BBR (bending beam rheometer) and DSR (dynamic share rheometer) apparatus were evaluated according to the US specifications (ASTM D6373 Standard Specification for Performance Graded Bituminous Binder).

2.3 Multiple stress creep recovery test method (MSCR)

Multiple stress creep recovery (MSCR) procedure allows to characterize high-temperature properties of bitumen and is used to designate the upper range of functional PG according to the AASHTO MP 19 [4]. This test shall be carried out in dynamic shear rheometer (DSR), using the measuring system parallel plates with a diameter of 25 mm and 1 mm gap in accordance to the procedure described in the AASHTO TP 70 [5]. The test procedure requires the repeated shear load per one second at constant stress applied to the bitumen sample after RTFO. Each load cycle is followed by a 9-second relaxation of material. Single cycle lasts 10 seconds and is repeated ten times on the 0.1 kPa and 3.2 kPa stress level. In each cycle of creep and re-
laxation, the strain change is recorded in the first and tenth of a second. Deformation analysis
during the cycle can be separated for the elastic deformation (recoverable) and permanent
deformation (non-recoverable). The result of the MSCR test is the percentage average elastic
recovery $R$, calculated according to the formula (1) and (2) and non-recoverable creep compli-
cance $J_{nr}$ calculated according to the formula (3) and (4).

\[
R = \frac{\varepsilon_1^{10} - \varepsilon_1}{10} \quad \text{(1)}
\]

\[
\varepsilon_r = (\varepsilon_1 - \varepsilon_{10}) \cdot 100 / \varepsilon_1 \quad \text{(2)}
\]

\[
J_{nr} = \frac{\varepsilon_1^{10} - \varepsilon_{10}^1}{10} \quad \text{(3)}
\]

\[
J_{nr}(\tau, N) = \varepsilon_{10} \tau \quad \text{(4)}
\]

where:
- $\tau$ stress;
- $N$ number of cycles;
- $\varepsilon_1$ strain increment after the first second cycle (at the end of the phase of creep);
- $\varepsilon_{10}$ strain increment after the 10 second cycle (at the end of the relaxation phase).

The average percentage of elastic deformation $R$ specifies the elastic properties of binders [6],
and non-recoverable creep compliance $J_{nr}$ specifies the permanent deformation sensitivity of
the material. The increasing of $J_{nr}$ value affects decrease resistance to permanent deformation
[7, 8]. The requirements of AASHTO MP 19 [4] bring additional classification within a single
performance grade. Standard sets four classes depending on traffic load (ESAL) includes:
standard – S, high – H, very high – V and extremely high – E (see table 1) [6].

<table>
<thead>
<tr>
<th>Classes of traffic load</th>
<th>Standard S</th>
<th>High H</th>
<th>Very high V</th>
<th>Extremely high E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Traffic characteristics</td>
<td>≤70 km/h</td>
<td>≥70 km/h</td>
<td>≥20 km/h</td>
<td>&lt;20 km/h</td>
</tr>
<tr>
<td>Requirement (binder after RTFOT)</td>
<td>$J_{nr}^{3,2} \leq 4,0$ [kPa⁻¹]</td>
<td>$J_{nr}^{3,2} \leq 2,0$ [kPa⁻¹]</td>
<td>$J_{nr}^{3,2} \leq 1,0$ [kPa⁻¹]</td>
<td>$J_{nr}^{3,2} \leq 0,5$ [kPa⁻¹]</td>
</tr>
</tbody>
</table>

### 3 Tests results

During this study basic classification and advanced rheological test were conducted on diffe-
rent samples of modified binders to compare the repeatability of properties of binders from
Polish market. Results of binder penetration tests conducted at 25°C, softening point tem-
perature by R&B method, Fraass breaking point and elastic recovery are shown in Table 2 for
25/55-60 and 45/80-55 polymer modified bitumen.
Table 2  Basic properties of polymer modified bitumen

<table>
<thead>
<tr>
<th>Sample of PMB binder</th>
<th>Penetration at 25°C [0.1mm]</th>
<th>Softening point temperature by R&amp;B [°C]</th>
<th>Fraass breaking temperature [°C]</th>
<th>Recovery elastic [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>25/55-60_1</td>
<td>33</td>
<td>64.8</td>
<td>-11</td>
<td>68</td>
</tr>
<tr>
<td>25/55-60_2</td>
<td>36</td>
<td>66.2</td>
<td>-16</td>
<td>82</td>
</tr>
<tr>
<td>25/55-60_3</td>
<td>25</td>
<td>69.6</td>
<td>-17</td>
<td>75</td>
</tr>
<tr>
<td>25/55-60_4</td>
<td>32</td>
<td>59.4</td>
<td>-10</td>
<td>81</td>
</tr>
<tr>
<td>25/55-60_5</td>
<td>41</td>
<td>60.8</td>
<td>-22</td>
<td>76</td>
</tr>
<tr>
<td>min value</td>
<td>25</td>
<td>59.4</td>
<td>-22</td>
<td>68</td>
</tr>
<tr>
<td>max value</td>
<td>41</td>
<td>69.6</td>
<td>-10</td>
<td>82</td>
</tr>
<tr>
<td>SD</td>
<td>5.47</td>
<td>3.72</td>
<td>4.35</td>
<td>5.30</td>
</tr>
<tr>
<td>45/80-55_1</td>
<td>46</td>
<td>58.0</td>
<td>-14</td>
<td>77</td>
</tr>
<tr>
<td>45/80-55_2</td>
<td>49</td>
<td>56.2</td>
<td>-11</td>
<td>79</td>
</tr>
<tr>
<td>45/80-55_3</td>
<td>53</td>
<td>62.0</td>
<td>-18</td>
<td>84</td>
</tr>
<tr>
<td>45/80-55_4</td>
<td>46</td>
<td>56.4</td>
<td>-11</td>
<td>62</td>
</tr>
<tr>
<td>45/80-55_5</td>
<td>56</td>
<td>62.0</td>
<td>-15</td>
<td>86</td>
</tr>
<tr>
<td>min value</td>
<td>46</td>
<td>56.2</td>
<td>-18</td>
<td>62</td>
</tr>
<tr>
<td>max value</td>
<td>56</td>
<td>62.0</td>
<td>-11</td>
<td>86</td>
</tr>
<tr>
<td>SD</td>
<td>3.67</td>
<td>2.62</td>
<td>2.64</td>
<td>8.63</td>
</tr>
</tbody>
</table>

It can be noticed, that for the same binder type (25/55-60 or 45/80-55) results of penetration and softening point are diversified but except one case for 25/55-60 sample, binders met requirements (table 3).

Table 3  Requirements of polymer modified bitumen in according to PN-EN 14023:2010 (Appendix NA)

<table>
<thead>
<tr>
<th>PMB type</th>
<th>Penetration at 25°C [0.1mm]</th>
<th>Softening point temperature by R&amp;B [°C]</th>
<th>Fraass breaking temperature [°C]</th>
<th>Recovery elastic [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>25/55-60</td>
<td>25/55</td>
<td>≥ 60</td>
<td>≤ -10</td>
<td>≥ 50</td>
</tr>
<tr>
<td>45/80-55</td>
<td>45/80</td>
<td>≥ 55</td>
<td>≤ -12</td>
<td>≥ 50</td>
</tr>
</tbody>
</table>

For analysed binders large diversification of test results was recognized in Fraass breaking point with a differences of up to 10°C and in elastic recovery over 20%. According to the Polish specifications (Appendix NA for EN 14023), for the polymer modified bitumen the required values are: 50% for elastic recovery and -10°C or -12°C for breaking point for PMB 25/55-60 and PMB 45/80-55 respectively. This fact shows significant differences between similar types of binders.

3.1 Assessment of the PG gradation

Based on the G*/sinδ value and Superpave requirements, upper limit of the performance grade temperature has been determined for polymer modified bitumen based on the original and RTFO aged binder. In accordance to Superpave requirements, the critical temperature of PMBs were determined using BBR apparatus based on the stiffness modulus and m parameter on PAV aged samples. PG gradation of tested polymer modified bitumen containing low and high temperature limit are shown in Figure 1.
Based on the PG gradation of tested binders it can be concluded, that all modified bitumens shown upper limit of the performance grade temperature above 70°C. Higher temperature (about 82°C) were obtained for PMB 25/55-60. High upper limit of the performance grade temperature, determined using DSR apparatus, indicate high rutting resistance.

Based on the conducted analysis of low BBR temperature, it was concluded that PMBs does not exhibit favorable low temperature properties, especially when compared with the typical critical winter pavement temperatures in Poland (from 28°C to -34°C).

3.2 Assessment of multiple stress creep recovery and elastic recovery

Large diversification of test results of polymer modified bitumens both in classification tests according to European Standards and obtained a significant stiffness of binder on the basis of assessments by PG grade, demonstrates difficulties in the proper evaluation of the polymer modified bitumen and, in particular, the contents of the polymer. The MSCR test method allows the assessment of appropriate level of polymer modification of bitumen based on the elastic recovery R with respect to the non-recoverable creep compliance $J_r$ of the binders and to the traffic load. Figures 2 and 3 shows test results for PMB 25/55-60 and PMB 45/80-55, respectively, depending on the elastic recovery and the $J_r$ parameter from MSCR test with 3,2 kPa stress level. A straight line with dots in figures presents the empirical curve for proper polymer content in bitumen. Results located over the curve indicate the correct amount of polymer.
On the basis of an analysis of the test results obtained from MSCR test, some of the modified binders are located below the curve. For PMB 45/80-55 sixty percent of tested binders are located below the reference curve, which proves that they contain insufficient amount of polymer. Analysis of the results of non-recoverable creep compliance \( \lnr \) shows higher stiffness of the binder from group PMB 25/55-60. For most of these binders non-recoverable creep compliance not exceeded 0.6 kPa\(^{-1}\) value. It can be noticed, that in spite of the insufficient level of polymer modification, most binders complies the requirements of the EN standards and there is possible to evaluate the PG grade according to Superpave (AASHTO M 320-09 Performance-Graded Asphalt Binder).

Figure 3  Relationship between elastic recovery \( R \) and the \( \lnr \) parameter from MSCR with 3,2 kPa stress level for PMB 45/80-55

Figure 4  Relationship between elastic recovery from ductility and elastic recovery from MSCR

In order to compare methods used to evaluate elasticity of modified binder, in Figure 4 there is presented correlation between elastic recovery from ductility and elastic recovery from MSCR test. Based on the results presented in Figure 4, no correlation can be observed for both the 64°C and 70°C tests temperature (\( R^2 < 0.35 \)).
4 Conclusions

Based on the tests conducted and analysis of the polymer modified bitumen produced in Poland, the following conclusions can be drawn:

1. The bituminous binders exhibit large diversification of test results, but, except few cases, meet all of the current European requirements.

2. Based on the stiffness modulus and m parameter obtained from BBR and on the G*/sinδ parameter from DSR tests, it can be noticed that all bituminous binders demonstrate very good performance in high temperature with limited resistance to low thermal cracking.

3. All of the tested modified binders exhibit over 50% value of the ductility elastic recovery, but only half of tested modified binders exhibit relevant polymer content in accordance to MSCR test method.

4. According to some reports [9,10] published in recent years about difficulties in the application of the polymer modified bitumen and not sufficient quality, it seems necessary to incorporate an additional assessment by MSCR methods. Multiple Stress Creep Recovery method allows for assessment of appropriate amount of elastomer used for modification and to eliminate binder with weak elastic properties.

References


[5] AASHTO TP 70-09 Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)


