EFFECTIVENESS OF ASPHALT PRESERVATIVES

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1 Introduction

1.1 Background

Historically, maintenance work on the Highways Agency (HA) network has involved removal of defective asphalt layers and replacement with a new material of similar performance or better. Following the recent decision from the UK government to reduce public spending, the current maintenance strategy may need to be revisited to cost-effectively maintain and preserve the strategic road network.

A number of pavement preservation systems are being marketed commercially, including some which have gained HAPAS (Highway Authorities Product Approval Scheme) approval, that claim to preserve and extend the life of asphalt surfacing. The predominant way these systems are assumed to work is either by the rejuvenation of the binder through modification of its chemical composition, or by retarding the ageing process of the binder constituent of the asphalt mixture. All the processes involve the application of a liquid agent to the existing asphalt. Some include a mixing of the agent with the asphalt in situ. Such products, in principle, have the potential to fulfil HA’s objective of extending life at reduced cost and preserving the pavement asset.

1.2 Scope of the Study

In recent times, the HA has allowed some departures to permit trial applications of asphalt preservatives but there is little objective evidence to confirm the effectiveness of these treatments or to identify the materials that have been applied.

In October 2010, HCG/HRG were awarded a project by the HA (2/1308 Task 395 (1308) MOTT) to carry out a review of the use of pavement preservation material in the UK and provide a comparative study under laboratory conditions of the effectiveness of asphalt preservatives. The long term aim is to enable guidance to be developed for their use on the HA’s road network.

The key objectives of this project were:

1. To measure under laboratory condition the effects of asphalt preservatives on binders, prepared mixes and specimens of aged existing pavements;
2. To investigate what further work (if any) will be required to produce guidance and advice on the use of asphalt preservatives;
3. To identify the likely form guidance and advice on asphalt preservatives should take.

Due to budgetary constraints, the first objective of this project was limited to assessing the effectiveness of asphalt preservatives on binder properties. This report presents the finding from the study.
2 Pavement Preservation

There are a number of definitions associated with pavement preservation; for example, the Federal Highways Administration (FHWA) Pavement Preservation Expert Task Group in the US has defined it as “a program employing a network level, long-term strategy that enhances pavement performance by using an integrated, cost-effective set of practices that extend pavement life, improve safety and meet motorist expectations”. In general Pavement Preservation is considered to represent a proactive approach to maintaining existing highways; this includes routine maintenance, preventive maintenance and minor remedial work.

In the UK, suppliers of pavement preservation materials joined together under the umbrella of the Road Surface Treatment Association (RSTA) and formed an informal group known as the Pavement Preservation User Group (PPUG). Amongst the pavement preservation materials marketed in the UK, some products are known to incorporate a “rejuvenating agent”.

In addition to the above, there are a number of pavement preservation systems that claim to have the ability to extend the service life of distressed surface course simply by preserving its condition and protecting it from rapid deterioration. These products are sometimes referred to as “sealers”. These systems include mist spray application of premium/intermediate grade bitumen emulsion followed by gritting application. Other than providing a seal and better appearance of the new surface, these sealers are not claimed to rejuvenate the existing surfacing. The current study focuses upon assessing the effectiveness of bitumen rejuvenators; strictly speaking, therefore, sealers may be considered outside the scope of this study. However, some of these sealers will be included as a part of the laboratory test programme, for comparison purposes, because their ability to maintain and preserve road pavements when applied as surface dressings is proven. Consequently their descriptions are presented in this report.

2.1 Ageing of Bitumen

Bitumen is a complex system that predominantly consists of hydrocarbons and their derivatives. Despite the complexity in the chemical composition of bitumen, a method has been established to separate bitumen into two main chemical groups, asphaltenes and maltenes. Asphaltenes are high molecular weight hydrocarbon, polyaromatic fractions precipitated from bitumen by non-polar solvents such as normal pentane, hexane or heptane, and are soluble in benzene. Maltenes are the relatively high boiling fractions soluble in non-polar solvents, consisting of resins and oils. Maltenes are also sometimes chemically separated into polar aromatics, naphthene aromatics and saturates.

Traditionally, bitumen has been described as being in a colloidal system where the highest molecular weight components (i.e. the asphaltene micelles) are dispersed and sustained by the polar aromatics. It is known that the rheological properties of bitumen can be largely reflected by the stability of its colloidal system [1]. However recent work reported by Redelius [2] suggested that this may not necessarily be the case. Using an alternative model, Hansen thermodynamic solubility model, he suggested that bitumen may be considered as a mixture of millions of different molecules which is kept in solution by its mutual solubility and a continuum of different polarities and molecular weights are necessary for complete stability. The model predicts that if certain fractions are removed from bitumen the stability may be lost resulting in formation of precipitate and phase separations.

In service ageing starts as early as the time of initial delivery from the hot mix plant and continues throughout the life of a bituminous pavement (such as with time, thermal cycles or radiation from the sun). Conversion of naphthene aromatics to polar aromatics, subsequently oxidised to become asphaltenes, takes place during age-hardening of bitumen. Depending on the relative rates of conversion between these components, the content of polar aromatics may either increase or decrease. This could lead to the asphaltenes being no longer in
colloidal dispersion, resulting in agglomeration of asphaltenes and leading to lower ductility, higher viscosity and embrittlement of the bitumen film. These changes are associated with pavement deterioration taking place.

2.2 Rejuvenation Process

"Due to oxidation and to the direct influence of sun, salts, acids and oils, the bitumen in the asphalt surface of a road ages rapidly. At the same time the road surface is subject to increasing heavy mechanical forces from the passing traffic. As a result the bitumen loses its flexibility and adhesive capacity and it becomes brittle." These statements have been used by many pavement preservation material suppliers to present their business case that, in order to preserve their service life, road surfacings require treatment (preservation) after 3-5 years from opening to traffic, and repeated applications are recommended to further extend the service life.

A range of rejuvenating agents are readily available on the market, such as aqueous acrylic emulsions modified with epoxy resin, coal tar emulsions, maltene (oil) based emulsions and natural asphalt fortified based systems, cut back with a hydrocarbon solvent. Rejuvenators can be applied directly without disturbing the existing bituminous surface (cold application), although preheating of the existing surface may be required in some cases (hot application).

It is claimed that these rejuvenators can penetrate into the existing surface to a depth of up to 12mm, rejuvenating, binding, sealing and consolidating it; however, data from Vallerga [3] suggested that the effective depth of rejuvenation was only up to 9mm. Furthermore, based on the work on the hard shoulder of US 99 at Lodi in August 1961, Vallerga found that only cationic “maltene” rejuvenator penetrates and more effectively restores the penetration of the aged bitumen, whilst bitumen emulsion appeared to have less effect; this finding is illustrated in Figure 1.

![Figure 1: Effectiveness of Rejuvenator with Depth in Pavement, Lodi Shoulders Project [3]](image)

Note: Figure 1 uses American units and terminology: oil-resin emulsion contained cationic “maltene” emulsion whilst asphalt emulsion (US) is the same term as bitumen emulsion (UK).
Brownridge [4] reinforced Vallerga’s view that in order for a rejuvenator to penetrate it cannot be retarded by blending in a bitumen emulsion or formulated into a quick dry emulsion, since the effectiveness will cease as soon as the absorption stops (i.e. the emulsion has cured). There are many bitumen emulsions being marketed that claim rejuvenation capability; however, Browridge stated that if “the (bitumen) emulsion breaks or cures on the pavement surface then it is sealing, not rejuvenating”. He further argued that engineered cationic emulsion containing maltene saturates (light fractions), which is wax free, should be used as the base medium for rejuvenator. This engineered emulsion would have better ability to penetrate (‘diffuse’) into the bitumen film that is being rejuvenated, through its solvency effect with the binder; the molecular composition of the maltene base oil used in the formulating provides this solvency without the use of distillate or solvents.

In general, the following criteria have been adopted as measures of the effectiveness of a bitumen rejuvenator, specifically its ability to:

- Restore the binder rheology, e.g. increase the penetration value, reduce viscosity or reduce stiffness in the top portion of the pavement surfacing where rejuvenator was applied;
- Seal the pavement against ingress of moisture and air, reducing the risk of stripping and/or slowing down oxidative hardening of the upper layer and below;
- Improve the durability and extend the service life of the treated surface course due to any or a combination of the above.

### 2.3 Effect on Early Life Friction

Many reports highlight a short-term reduction in friction following the application of preservation materials; an example of this was reported on the 5 No trial sections on California Interstate 5 (I-5) as illustrated in Figure 2 [5].

![Figure 2: Effect of Treatment on Surface Friction over Time on California I-5 Project [5]](image)

The above test sections carried relatively heavy traffic in a moderate climate. These treatments were applied on 25 October 2001 and contained either rejuvenator (i.e. Reclamite, Topein-C and Pass Oil) or sealer (i.e. CSS-1h and CQS-1h). Tests by Dynamic Friction Tester (ASTM E-1911) on these trial sections were carried out before construction and after 1, 42 and 272 days; these sections were repaved shortly after the latest test. There was no explanation given as to why there appeared to be a small increase in the first day friction after...
Reclamite treatment. This could simply be due to the variability of the test method. In practice, therefore, traffic must be strictly controlled until the friction index reaches an acceptable level. There are many factors that may affect the rate of increase in friction such as preservation material type, curing time, or climatic condition. In addition to these, the use of “sand” or grit can improve the early life friction, as shown in Figure 3 [5].

![Early Life Friction, with and without “sanding” (i.e. gritting)](image)

Figure 3: Early Life Friction, with and without “sanding” (i.e. gritting) [5]

The above shows some improvement in the friction index after sanding, even though the improved value remained less than that of the Control.

### 2.4 Effect on Permeability

Another benefit claimed by preservative treatments is sealing the pavement against ingress of moisture and/or air. This benefit may be assessed by laboratory permeability testing such as that carried out on cores taken from Arizona and California projects in 2006 [5]; a summary of the test results is illustrated in Figure 4.
Figure 4: Laboratory Permeability of Cores taken in 2006 Projects [5]

For application over porous surfacing (i.e. AR AFC), the rejuvenators (Pass QB and Reclamite) increased the permeability of the treated surfacing materials whilst that of the sealer (CSS-1) appeared to reduce permeability, compared with the Control. The latter may be regarded as a disadvantage if the reduced permeability had affected the drainage properties of the porous surfacing. It is, however, not clear how it was possible for these rejuvenators to improve the drainage properties of porous asphalt.

For application over dense asphalt and chip seal (“surface dressing”), there appeared to be either a reduction or little change in the permeability after each treatment. Chip seals (sealers/surface dressings) have a much lower permeability than dense-graded asphalt materials during their service life.
3 Laboratory Testing

3.1 Samples for Testing

In order to assess the effect of Pavement Preservation Material (PPM) on binders used in surfacing materials, a suite of laboratory testing was developed. To provide a means of comparison and to identify PPMs a 40/60 paving grade bitumen was selected for use as the binder to be aged and rejuvenated (hereafter the “base binder” or B50), to represent a most commonly used binder in asphalt surfacings including Hot Rolled Asphalt, generic Stone Mastic Asphalt and Thin Surfacing materials. The base bitumen (40/60pen) B50 and PPMs selected for the laboratory testing are summarised in Table 1.

Table 1: Samples for Testing

<table>
<thead>
<tr>
<th>Description</th>
<th>Reference ID</th>
</tr>
</thead>
<tbody>
<tr>
<td>40/60pen Bitumen</td>
<td>B50</td>
</tr>
<tr>
<td>PPM</td>
<td>C</td>
</tr>
<tr>
<td>PPM</td>
<td>D</td>
</tr>
<tr>
<td>PPM</td>
<td>E</td>
</tr>
<tr>
<td>PPM</td>
<td>F</td>
</tr>
<tr>
<td>Base Binder for PPM F</td>
<td>G</td>
</tr>
<tr>
<td>PPM</td>
<td>H</td>
</tr>
<tr>
<td>PPM</td>
<td>I</td>
</tr>
</tbody>
</table>

3.2 Risk Assessment

As a part of routine practices, COSHH and Risk Assessments were carried out on the samples listed in Table 1, which incorporate either emulsion or solvent based bituminous materials. Review of the Manual Safety Data Sheets for these products suggest some samples were relatively volatile and have low flash point. This has resulted to variation in initial risk levels being High to Medium.

In order to further assess the safety aspect of the test program, initial trials were carried out in stages within a confined environment at high temperatures (i.e. oven), prior to starting the Main Test Programme. These trials concluded that introducing a flow of nitrogen (to replace air within the oven), with an active extraction in place, was able to reduce the level of risk from High/Medium to Low; this approach was subsequently adopted in the Main Test Programme.

3.3 Methodology

The literature review highlights the fact that any rejuvenation process would mostly take place by diffusion or gradual absorption of the applied rejuvenator into the binder film of the surface course downwards. This process is very slow and subject to many variables such as surface temperatures, porosities and residual binder properties. Furthermore, successful preservative treatment should not only rejuvenate or restore the properties of aged binder but ideally it should be able to favourably control the subsequent rate of age-hardening such that it is at least similar to, or better than, that of the aged binder.

These considerations make it almost impossible to simulate this rejuvenation process in a laboratory before any further assessment of the effectiveness of the process can be made. For this project, a simplified approach was adopted incorporating the use of a control binder B50, subjected to an accelerated ageing condition, before and after being treated with PPMs. As stated in the previous section, PPMs could be emulsion or solvent (“cutback”) based.
Clause 955 of the Specification for Highway Works (SHW) contains a procedure for recovering emulsion and cutback bitumen (i.e. bitumen incorporating hydrocarbon solvent) and a subsequent accelerated ageing protocol on the binder residue after recovery (see Figure 5). However the protocol is being revised, because the microwave procedure is no longer felt necessary, PAV ageing for emulsions is under review in Europe and experience of this study will improve it. This clause also contains guidance on accelerated ageing of bituminous binders for hot mix asphalt (see Figure 6).

These processes involve the use of the RTFO (Rolling Thin Film Oven) equipment (see Figure 7), where samples in bottles are placed in a moving carousel and subjected to a jet of either nitrogen or air at an elevated temperature (85, 135 or 163°C) for a total duration of up to 24 hours.

### Rapid Recovery Test (RRT)
- **Conditions:**
  - 85 °C
  - 75 mins
  - Nitrogen gas
  - RTFOT but screws and PTFE bottles

### Microwave Procedure
- Establishes number of cycles with weight loss
- Max. 90 °C
- Total weight loss after RRT provides information on binder content (no need for microwave procedure if ‘Recovered Binder’ not required)

### Modified Ageing RTFOT
- **Conditions:**
  - 135 °C
  - Air - jetted at 4000 ml/min
  - Screws
  - 3hrs.
  - 8hrs. – ‘Aged Binder’
  - 22hrs.- Equivalent to PAV/HiPAT

### Stabilising Procedure:
- **Conditions:**
  - 135 °C
  - Nitrogen gas
  - 1 hr. or until weight loss stabilised and volatile oil largely removed.
  - Provides information on volatility and proportion of fluxing oil

### Figure 5: SHW Clause 955 Rapid Recovery and Accelerated Ageing Procedures for Cutbacks/Fluxed and Emulsions
Clause 955 adopts polytetrafluoroethylene (PTFE) in place of the traditional glass bottles used in the standard RTFOT (BS EN 12607-1) and introduces a stainless steel screw in each PTFE bottle, to stir and homogenise the binder sample during ageing. For this study, a new set of aluminium containers, internally coated by PTFE, were manufactured and used. The introduction of the stainless steel screws helps to provide a continuous exposure of fresh binder sample to the air; consequently, the test duration is reduced as the screws accelerate the ageing process and, in the case of testing polymer modified binders, the problems related to ‘skinning’ are minimised.

Typical glass/PTFE (+ stainless steel screw) bottles are shown in Plates 1 and 2.
3.4 The Main Test Programme

The recovery and accelerated ageing protocol currently specified in the SHW Clause 955 was chosen, subjected to a modification to suit the aim of this study. Specifically, the modified protocol can be summarised as follows:

- A combination between the ageing protocol for cutbacks/emulsions (Figure 5) and hot mix asphalt binders (Figure 6) was adopted.

- The first half of the ageing process was carried out to rapidly age the control (base) B50 bitumen, thus the procedure in accordance with Figure 6, but with the Modified Ageing RTFOT ageing protocol for up to 4 hours only.

- The second half of the protocol was to “recover” and further age a blend of control bitumen and PPM, thus the procedure in accordance with Figure 5, but with recovery time at 50°C for 2 hours in nitrogen followed by accelerated ageing at 135°C for another 2 hours in air.
The RTFO was placed under an active extraction unit in the laboratory mixing room and the test was closely monitored by using an electrical viewing device (i.e. webcam); this allowed remote monitoring and prompt action if it was required.

The adopted Main Test Programme is summarised as follows:

a) The base binder B50 is initially subjected to short-term ageing i.e. conditioning at 163°C for 45 minutes in air jetted at 4000 ml/minute;

b) After completion of short-term ageing, the base binder is left to cool down and then subjected to accelerated ageing at 135°C for 4 hours in air jetted at 4000 ml/minute;

c) The bottles are then removed from the oven and placed in the fume cupboard and allowed to cool down to ambient;

d) PPM is then added to the above binder at an appropriate rate. This process involves removal of an amount of base binder which is then replaced by an equivalent amount of the PPM. In this manner, the weight of material within the test container remains constant. Note that the residual binder contents of the emulsion and solvent based PPMs were 60% and 40% by the total weight of PPM, respectively;

e) The bottles containing the PPM are left in the fume cupboard at ambient temperature for 24 hours. This process allows any very low flash point volatile oil to safely evaporate and consequently reduce the amount of hydrocarbon solvent within the bottle;

f) The bottles are transferred to the RTFO oven at 50°C for 2 hours in nitrogen jetted at 4000 ml/minute;

g) The nitrogen supply is then changed to air jetted at 4000 ml/minute;

h) The oven temperature is raised from 50°C to 135°C within 30 minutes;

i) The conditioning at 135°C is continued for another 2 hours in air jetted at 4000 ml/minute;

j) At each stage of assessment, samples are weighed and removed for further assessment.

For clarity, the above protocol is illustrated in Figure 8; the test schedule is shown in Table 2.
Note: Stages 2, 5 and 6 are interim conditioning involving either preheating or cooling of sample and/or oven. Details are presented in the Appendix B.

Figure 8: Modified Rapid Recovery and Accelerated Ageing Procedures for Pavement Preservation Materials
### Table 2: Test Schedule

<table>
<thead>
<tr>
<th>Base Binder</th>
<th>Added PPM</th>
<th>Stage of Ageing Protocol*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>B50</td>
<td>None</td>
<td>T</td>
</tr>
<tr>
<td>B50</td>
<td>D</td>
<td>-</td>
</tr>
<tr>
<td>B50</td>
<td>E</td>
<td>-</td>
</tr>
<tr>
<td>B50</td>
<td>F</td>
<td>-</td>
</tr>
<tr>
<td>B50</td>
<td>G</td>
<td>-</td>
</tr>
<tr>
<td>B50</td>
<td>H</td>
<td>-</td>
</tr>
<tr>
<td>B50</td>
<td>I</td>
<td>-</td>
</tr>
</tbody>
</table>

Note:
- Stage of Ageing Protocol refers to that presented in Figure 8.
- T denotes the point at which a sample (1 bottle) was taken and the binder tested for rheology.
- A denotes the point at which PPM additive was applied to the bottle containing B50 sample.

Stage 1 was intended to simulate the short-term ageing of bituminous binder, to a similar level as that experienced during the manufacturing of an asphalt mixture in the mixing plant. This protocol is exactly the same as that specified in the SHW sub-Clause 955.6 for “Binders for Manufacturing Asphalt or other Hot Mixed Materials”. This treatment was applied to the control base binder B50.

In a previous study completed by SW for the HA [6], hot mixed, modified or non-modified binders, when subjected to the modified ageing RTFO (see Figure 6) for 8 hours would harden to the same level as binder after the High Pressure Ageing Test (HiPAT), PAV85 (BS EN 14769), which simulates ageing in service for 5 – 10 years. The present study required a partly aged binder, say, having similar residual properties as that exposed in service for 3 to 5 years. Consequently, the control binder B50 recovered from Stage 1 was subjected to a further 4 hours ageing in air (which is half of the 8 hours ageing) in Stage 3.

In Stage 4, the binder recovered from Stage 3 was treated by PPM at the respective application rate. Subsequently the treated binder was subjected to a further ageing test in Stages 7 and 8b.

Assessment of the materials recovered from Stages 1, 3, 7 and 8b, was carried out by using an Advanced Dynamic Shear Rheometer (DSR) at varying test temperatures and frequencies. In this test, complex shear (stiffness) modulus and viscoelastic response were specifically determined.

Figure 9 shows the reasoning behind the test protocol. The comparison with a control after addition of the PPM and after accelerated ageing and using an advanced DSR to study rheology (G* and phase angle) provides a robust product identification test.
Test

Stage 1 and Stage 3
Sample of aged bitumen (originally 40/60 penetration grade) prepared as a homogenous thin film in PTFE coated aluminium bottle

Simulates
Aged binder in asphalt after 3 to 5 years

Stage 4
Screws removed
Cooled to ambient (~20°C) to provide hard binder film
PPM added and left for 24hours

Stage 4
Application of PPM to road surface with some loss of volatiles

Stage 7
Heat to 50°C and rotate for 2 hours in nitrogen (no ageing), no mixing (no screws)

Stage 7
Coating, further loss of volatiles and accelerated penetration of the PPM into surface of aged binder

Stage 8b
Heat to 135°C introduce screws, mixing, ageing in air for 2 hours

Stage 8b
In service ageing to show maximum effect of a PPM when compared with the untreated bitumen control

Figure 9: Pavement Preservative Material Identification Test
Results and Discussion

4.1 Ageing Profile of the Base Bitumen (Sample B50)

In order to assess the effectiveness of PPMs, the ageing profile of the base bitumen, i.e. Sample B50, was initially established. Test results on this sample were subsequently used for benchmarking purposes.

The test results are presented in the following figures, together with results previously reported [6] for:

- The Ageing Profile test of a similar bitumen grade (i.e. 40/60 pen) and supplier, tested to SHW Clause 955 for hot mix asphalt binder (Figure 6) (samples with prefix OM143 refer);
- The RTFO (EN) and HiPAT (EN) tests of the same bitumen after being subjected to RTFO and HiPAT protocols to EN12607-1 and EN14769 respectively. These protocols are known to simulate Short Term Ageing (STA) and Long Term Ageing (LTA) of bituminous binders; for simplicity the stage of ageing which takes place between these protocols will be referred as Medium Term Ageing (MTA).

Figure 10: Complex Modulus vs Stage of Ageing of Bitumen Grade 40/60 at 25°C

Figure 10 shows that:

- As expected, the complex modulus of these samples increases in order of short, medium to long term ageing;
- For binders at a similar stage of ageing, the complex modulus values of B50 (current) sample were slightly lower than those subjected to the SHW Clause 955 ageing protocol. This is not unexpected since the SHW protocol would be expected to be slightly more severe than that of the current ageing protocol;
- The binders subjected to the current ageing protocol (Stages 1, 3 and 8b) can be considered as having comparable properties to those after short, medium and long term ageing respectively. Hereafter these stages will also be referred as STA, MTA and LTA.
Note: graphs in colour codes depending upon stage of ageing = green (STA), blue (MTA) and red (LTA).

**Figure 11: Ageing Profile of Sample B50 (Rheology)**

Figure 11 shows that the overall rheology (complex modulus and phase angle) over a range of temperatures of Sample B50 tested after Stages 1, 3 and 8b is comparable to that of the OM143 binders after STA, MTA and LTA protocols respectively. The results demonstrate that as the stage of ageing increases:

- Complex modulus values also increase over the test temperature range. This indicates stiffening or hardening of the binder;
- Phase Angle decreases over the test temperature range. This indicates higher elastic response. This may improve deformation resistance at high temperature, but this also means increased ‘brittleness’ at failure which may lead to reduced resistance to low temperature cracking;
- The above trends are expected for “as supplied” 40/60 penetration grade bitumen.

From the literature review and contact with industries, it is understood that the best time to apply PPM is during the early life of the surfacing material particularly within the first 3 – 5 years after construction. The above findings suggest that application of the ageing protocol to the Base Binder has successfully resulted in residual binder having similar properties as a binder having less than 5 years ageing in service, i.e. a level of hardening between that of STA and LTA. Consequently, the protocol was adopted and the residue of binder after Stage 3 was subjected to PPM application.

Using the same set of data, empirical properties such as penetration, softening point and penetration index were calculated from the rheological data. The results are summarised in Table 3.

**Table 3: Empirical Properties Calculated from Rheology**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Equivalent test</th>
<th>Calculated Penetration (d&lt;sub&gt;100&lt;/sub&gt;) at 25°C</th>
<th>Calculated Softening Point (°C)</th>
<th>Penetration Indices (I&lt;sub&gt;P&lt;/sub&gt;)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B50 Stage 1</td>
<td>RTFOT = STA</td>
<td>33</td>
<td>61.0</td>
<td>0.3</td>
</tr>
<tr>
<td>B50 Stage 3</td>
<td>MTA</td>
<td>25</td>
<td>65.8</td>
<td>0.8</td>
</tr>
<tr>
<td>B50 Stage 8b</td>
<td>LTA = PAV85</td>
<td>18</td>
<td>70.2</td>
<td>0.8</td>
</tr>
</tbody>
</table>
The above test results demonstrate that as the level of ageing increases, the penetration value reduces, the softening point and PI values increase. These trends are as expected showing loss of volatile oils and oxidation.

### 4.2 Effectiveness of PPMs

Using the same principles as that adopted for the base bitumen Sample B50, comparisons have been made for the B50 samples after being treated by PPM. For clarity, summaries of the test results are presented in separate figures with overall comparison shown in Figure 12 and details in Figures 13 to 15. Please note that sample G was the bituminous base used to manufacture the emulsion PPM F.

![Complex Modulus vs Stage of Ageing of Samples at 25°C](image)

Figure 12: Complex Modulus vs Stage of Ageing of Samples at 25°C

Figure 12 shows that the addition of PPM reduced the complex modulus of the B50 binder after MTA, when tested at 25°C at 0.4 Hz. Apart from the binder treated by PPM E, the complex modulus values of the other treated binders, after being subjected to further ageing, remain less than that of the B50 binder after LTA. In addition to this, binders treated by PPM F and binder G show comparable properties after further ageing; this is not unexpected since sample G was the base binder of sample F and this condition was consistent throughout the test temperatures and stages of ageing, as illustrated in Figure 13. However, these conditions varied for different PPM materials, as seen from the illustrations given in Figures 14 and 15.
Note: graphs in colour codes depending upon stage of ageing = green (STA), blue (MTA) and red (LTA).

Figure 13: Ageing Profile of Samples with the Same Base Binder

Note: graphs in colour codes depending upon stage of ageing = green (STA), blue (MTA) and red (LTA).

Figure 14: Ageing Profile of Samples – Complex Moduli
Figure 15: Ageing Profile of Samples – Phase Angle

The above figures show the overall rheology (complex modulus and phase angle) over a range of temperatures of Sample B50 tested after being treated with either solvent or emulsion based PPM. The results show that as the stage of ageing increases:

- Complex modulus values were initially reduced, but this was then followed by increases in complex modulus over the test temperature range. This indicates initial softening after the addition of the PPMs, followed by hardening of the binder, although the rate of hardening was generally less than that for the base bitumen. For example, after LTA, all binders treated with these PPMs, but apart from that treated by PPM E, have lower complex modulus values than that of B50 sample. Sample B50 + E after LTA has significantly higher complex modulus values than B50 sample after LTA;

- Phase Angle generally decreases over the test temperature range, although some samples show increased phase angle at lower test temperatures. This may indicate improvement in high temperature properties of the treated samples, i.e. improved deformation resistance at high temperature and improved resistance to low temperature cracking.

The above trends show different effectiveness of the treatment as the binder undergoes further ageing. Using the same set of data, empirical properties such as penetration, softening point and penetration index were calculated from the rheological data. The results are summarised in Table 4.
Table 4: Empirical Properties Calculated from Rheology for Samples with PPM

<table>
<thead>
<tr>
<th>Sample</th>
<th>Calculated Penetration ($d_{100}$) at 25°C</th>
<th>Calculated Softening Point (°C)</th>
<th>Penetration Indices ($I_p$)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>B50 + D Stage 7</td>
<td>35</td>
<td>59.4</td>
<td>0.3</td>
</tr>
<tr>
<td>B50 + D Stage 8b</td>
<td>24</td>
<td>64.8</td>
<td>0.6</td>
</tr>
<tr>
<td>B50 + E Stage 7</td>
<td>61</td>
<td>58.6</td>
<td>1.6</td>
</tr>
<tr>
<td>B50 + E Stage 8b</td>
<td>12</td>
<td>78.4</td>
<td>1.3</td>
</tr>
<tr>
<td>B50 + F Stage 7</td>
<td>27</td>
<td>72.8</td>
<td>2.0</td>
</tr>
<tr>
<td>B50 + F Stage 8b</td>
<td>25</td>
<td>69.6</td>
<td>1.4</td>
</tr>
<tr>
<td>B50 + G Stage 7</td>
<td>31</td>
<td>65.0</td>
<td>1.1</td>
</tr>
<tr>
<td>B50 + G Stage 8b</td>
<td>25</td>
<td>68.8</td>
<td>1.3</td>
</tr>
<tr>
<td>B50 + H Stage 7</td>
<td>51</td>
<td>56.4</td>
<td>0.7</td>
</tr>
<tr>
<td>B50 + H Stage 8b</td>
<td>21</td>
<td>67.2</td>
<td>0.7</td>
</tr>
<tr>
<td>B50 + I Stage 7</td>
<td>53</td>
<td>57.8</td>
<td>1.0</td>
</tr>
<tr>
<td>B50 + I Stage 8b</td>
<td>42</td>
<td>59.8</td>
<td>0.9</td>
</tr>
</tbody>
</table>

Note: *$I_p$ is applicable for unmodified bitumen only; thus a comparison of $I_p$ data between modified/unmodified bituminous materials will not be valid. For completeness however the $I_p$ of PPM treated samples is shown in the above table.

The above test results demonstrate that as the level of ageing increases, the penetration value reduces. These trends are similar to those found for the base bitumen B50. However, there are also unfavourable trends i.e. initial increases in penetration followed by significant reduction of the penetration value after ageing.

The almost identical rheological data for Stage 8b results for PPM F and its base binder G demonstrates that the protocol to remove the water and volatile oil from the emulsion and age that “recovered” binder when compared to ageing the base is robust.

As a part of the test ageing test protocol, weight loss was measured at each stage of testing. The test results suggest that the initial conditioning of 24 hours at ambient (Stage 4) and 2 hours at 50°C in Nitrogen (Stage 7) has driven off most of the water from PPM emulsions (assuming that the water content was around 40%) but only half of the solvent from those PPMs that were not emulsions. Further weight loss was recorded after further 2.5 hours ageing at 135°C in air (Stage 8a & 8b).
5 Lessons Learnt from this Study and the Current Practices

The findings from the current study have successfully demonstrated a suitable test protocol to measure the effectiveness of different PPM treatments. It was found that the effectiveness of PPMs to preserve the properties of the treated binder may be affected by several factors such as the type of PPM and the post-treatment ageing condition.

Currently, the most commonly adopted indicator of the effectiveness of PPMs involves recovering the binder from the existing surface course before and after treatment, by using solvent recovery. Whilst this kind of recovery protocol is a widely adopted standard, there is always the possibility of interaction between the treated binder and the solvent used. This interaction could be quite complex and may have positive or negative effects on the recovered binder properties. Recovered binder is often evaluated soon after the asphalt is treated with the PPM, however this study indicates that consideration should be given to properties of the recovered binder after a few years to properly measure performance.

During their last meeting in 2010, the PPUG have issued a draft specification for ‘Bituminous Pavement Rejuvenation’. This draft specification included a proposal to introduce a performance based payment dependent upon the achieved level of reduction in absolute or complex viscosity and/or complex modulus (stiffness) of the treated material, typically to a level of reduction by not less than 20 or 30 per cent for samples taken from the upper 10mm of the surface course. These parameters were to be determined at a single test temperature i.e. 60°C. For clarity, the draft acceptance criteria proposed by the PPUG are reproduced in Table 5 below.

Table 5: Draft Acceptance Criteria for a Pavement More Than 3 Years in Age (PPUG, 2010)

<table>
<thead>
<tr>
<th></th>
<th>Parameter</th>
<th>Criteria</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Absolute Viscosity at 60°C (Pa.s)</td>
<td>&gt; 30% Decrease</td>
<td>BS EN 12596 or ASTM D2171</td>
</tr>
<tr>
<td>2a</td>
<td>Complex Modulus at 60°C (kPa) 10 rad/s</td>
<td></td>
<td>BS EN 14770 or AASHTO T315</td>
</tr>
<tr>
<td>2b</td>
<td>Complex Viscosity at 60°C (Pa.s) 10 rad/s</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2c</td>
<td>Phase Angle at 60°C (degrees) 10 rad/s</td>
<td></td>
<td>Report</td>
</tr>
</tbody>
</table>

For completeness, the rheological data from the samples tested in the current study were reanalysed to address the parameters stated as items 2a, 2b and 2c in the above table. A summary of the finding is as follows:

- At 60°C:
  - all samples, apart from that treated by sample B50+F, show reduction in complex modulus and complex viscosity by at least 50%. These results may be considered by PPUG as being in compliant with their proposed acceptance criteria (Table 5 refers);
  - only samples treated by samples B50+D and B50+H show an increase in phase angle by 3%;
- At 25°C:
  - all samples, apart from that treated by sample B50+F, show reduction in complex modulus and complex viscosity by at least 30%;
  - samples treated by samples B50+D, B50+I and B50+H show an increase in phase angle by 8 to 11%.

The above analysis shows variations in the level of ‘effectiveness’ of different treatments, depending upon the test temperature.

Please note that the current study was limited to assessment of an “as supplied” (“non-recovered”) binder that was aged and treated with a PPM in a repeatable controlled manner and our assessments were based upon testing at 0.4 Hz at 25°C, instead of 10 rad/s at 60°C (which is approximately 1.6 Hz). Whilst the relative difference in values might be expected to be consistent, the estimates would need to be verified. In the UK the DSR test frequency is generally 0.4Hz as specified in SHW clause 956 which simulates slow moving traffic and has a comparable loading time to the penetration test.
Removing samples to recover binder from the upper 10mm of an aged surface course (or any agreed depth) before and after treatment may not necessarily represent the effective penetration of the PPM into the asphalt and as stated earlier, there is a possibility of interaction between the treated binder and the solvent used during the binder recovery process.

Based upon observations and test results from the current laboratory work, it is suggested that:

- Assessment of the effectiveness of PPMs should be based on measurements over a range of test temperatures since responses of the treated materials may vary between low and high temperatures;
- Accelerated ageing protocol should be a part of the assessment methods, since some results indicated an initial softening effect but followed by an accelerated ageing. The ageing protocol adopted in this study could be used as the basis for assessment of the PPM against a control base binder.

In addition to the above, the literature review highlighted a potential risk with early life friction and reduced permeability. The former may lead to skidding hazard whilst the latter may not be desirable in case of noise or spray reducing surface course.

The present study was limited to assessment of a control paving grade bitumen aged and treated by different types of PPM, whilst current practice has been mostly based on the properties of post-treatment recovered binder. Whilst the assessment method adopted in the current study may have successfully differentiated the effectiveness of different PPMs, there is still a gap between this work and procedures adopted in practice. It is therefore recommended that the findings from the current study should be verified and further work should be carried out. The proposal should include tests of asphalt cores and in situ treated roads to determine benefits including reduction of water permeability.
6 \textbf{Summary}

The laboratory work has demonstrated that different treatments resulted in different level of effectiveness to resist hardening of bituminous binder. For clarity, an overall comparison between the different treatments is reproduced on the same graph, as shown in the following figures.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{complex_modulus_chart.png}
\caption{Comparison Between Different Treatments – Complex Modulus}
\end{figure}
The above figures 16 and 17 demonstrate that the effectiveness of PPM in terms of preserving the binder stiffness and reducing the rate of hardening may vary considerably between products. Some samples treated PPMs were trending towards a much lower phase angle which may imply increased brittleness.

This study suggests that assessment of PPMs should be based on measurement over a range of test temperatures and, more importantly, include ageing protocol. The protocol developed during this study was considered as robust and good for use as product identification test of samples treated by PPM.

Potential risk with early life friction and reduced permeability was highlighted in the literature review; however, work has not been done in this project to verify these aspects.
7 References