QUALITY OF BITUMENS IN ASPHALT HOT-MIXES WITH EMPHASIS ON THE DURABILITY OF CONSTRUCTED PREMIX SURFACINGS

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The chemical and physical properties (quality) of bitumens and the durability of hot-mix surfacings placed in various locations have been investigated over a four-year period. Performance-related properties of the residual bitumen subsequently recovered, were identified and quantified by comparing asphalts from early failures to those which have performed well. There is a significant relationship between ductility and chemical composition of the residual bitumen and the durability of new hot-mix surfacings.

A rapid test for the semi-quantitative estimation of asphaltenes in bitumen is described. The method based on the Oliensis spot test, can be used for on-site bitumen quality control.

INTRODUCTION

The poor performance of asphalt surfacings either during construction or in-service, can be very traumatic to contractor, client, consultant, road-user and ultimately to the taxpayer. The binder is quite often blamed, with or without justification, of being the cause of such unfortunate events. Although the binder in hot-mix asphalt is small in volume when compared to the mineral components, it nevertheless plays a very important role as a binding agent and should continue to do so for an acceptable time. Asphalt technologists' ultimate quest is for performance-related specifications that will ensure the procurement of quality bitumens which could produce asphalts with desirable performance during construction, and which will resist physical deterioration of the asphalt application over a long term of service. It is generally accepted by researchers in the field of asphalt chemistry such as Petersen (1) and Rostler and White (2), that it would be difficult to specify road bitumens on the basis of chemical composition. Limited variations within penetration grades of road bitumens will therefore have to be accommodated in design parameters and construction techniques. In spite of the lack of extended specifications which may result in the production of road bitumens of consistent quality but unaffordable prices, it should be possible to attain the same objective provided the properties of both materials and mixes have been evaluated in the design phase, and quality control by means of simple performance-related tests is exercised during construction.

The objective of this study was to establish performance-related data on the properties of original and aged bitumens, which can be used as references to ascertain why asphalt applications are not performing according to expectations. The field data together with information on the chemical and physical properties of locally produced

road bitumens, are used to develop guidelines for the optimal application of available asphalt materials.

Changes in the chemical composition and physical properties of bitumens from the refinery through to premix surfacings which have performed well over the first four years after construction, are reported as reference data. A chromatographic method (3) based on separation by polarity of chemically related groups i.e. saturates, non-polar aromatic compounds, polar aromatic compounds (resins) and n-hexane insolubles (asphaltenes) were used to determine the chemical composition of the binders.

The residual bitumens recovered from premix surfacings which showed signs of severe cracking and disintegration within the first year of service, were evaluated. The results of the laboratory tests were compared to the design and construction data, in order to assess the contribution of the binder to the distress of the applications.

Durability related properties which could be used as durability prediction criteria, were identified through quantitative comparisons of properties in residual bitumens from early failures and premix surfacings which have performed well. The proposed durability criteria were tested by the ageing of pen grade bitumens with varying chemical compositions by means of the Australian Research Board (ARRB) durability test (4).

These investigations also focused on the need for a rapid field test which can be used to evaluate the quality of bitumen during storage, hot-mix production and construction.

DURABILITY PREDICTION

The durability of a binder can be defined as its potential ability to absorb inherent chemical and molecular structural changes which are accelerated by ageing agents such as heat, ultra-violet radiation and oxidation, and still retain adequate residual adhesive and cohesive properties. The rate and extent to which the chemical compounds of a particular bitumen are transformed from less polar to more polar agglomerates, depend on the chemical composition of the original virgin bitumen which may have had adequate durability qualities as they have been determined by crude source, refining and blending methods. Premature ageing through prolonged exposure to elevated temperatures during storage and especially during the production of the hot-mix where the thin binder film is very susceptible to heat degradation, can reduce these potential durability qualities significantly. Consequently, variations in the performance of asphalts are to be expected. Lombard et al (3) found that the chemical composition of the blown bitumen used for the production of pen grade bitumens, has a marked effect on the binder's resistance to ageing. Pen grade bitumens prepared from heavily blown bitumen had high asphaltenes and low residual ductility after exposure to the rolling thin film oven test (RTFOT), while bitumens of similar penetration but prepared from less severely blown bitumens, had significantly lower asphaltene values and performed better with respect to ductility.

The variable effects of premature ageing of the bitumen on the engineering properties of hot-mixes, added to differences in properties of plant and laboratory-prepared mixes, may cause hot-mix asphalts to perform very differently on the road than

expected. The rheological properties of a bitumen are very relevant to the design of the mix and the construction procedures. For example, unexpectedly high viscosities of an aged bitumen containing severely blown bitumen, may contribute to compaction problems resulting in too high void-contents. Brown (5) concluded that the voids should be below 8 per cent for this reason but not lower than 3 per cent which again may aid rutting and bleeding. In contrast, a bitumen with low asphaltene content and therefore relatively lower viscosity, may lead to rutting. It also has a smaller plasticity range which could lead to deformation at high ambient temperatures and under heavy loads, and embrittlement of the binder at low road temperatures. The ideal binder should have a balance of active to non-reactive compounds, which is reflected in its temperatureviscosity performance and cohesive properties. Van Gooswilligen et al (6) studied the relation between field performance and bitumens which have been produced both commercially and experimentally. Poor molecular distribution and an imbalance in chemical components as found in bitumens with high proportions of blown bitumens, had high stability but unsatisfactory durability. They proposed a laboratory test frame work, the QUALOGON1, which only measures performance-related physical properties of bitumens and asphalt, to predict road performance.

Rostler and White found that the results from the ageing tests they proposed, correlated well with actual performance and durability indices based on the chemical composition of the original bitumens (7). The ageing procedures however, are cumbersome and take too long for routine quality control purposes.

The relation of asphalt chemistry to physical properties and specifications has been extensively reviewed by Halstead (8). Jamieson and Hattingh (9) used the Rostler and Sternberg precipitation method (10) for chemical analyses of bitumens from different sources which were used in a premix surfacing experiment in Durban, Natal. They established the optimum limits of component ratios and penetration/log ductility relationships of bitumens which have performed well in the coastal region climate. They concluded that the chemical method predicted the relative road performance more consistently than the penetration/log ductility ratio.

An extensive study by Chari et al (11) on age hardening of asphalt, focused on the development of performance-related specification criteria. Comparisons were made between data from field specimens of up to 24 months of service, and bituminous materials aged in the laboratory by thin film ovens at varying temperatures, by forced draught oven and by ultra-violet radiation. The results of accelerated weathering in the laboratory do not always correlate with field performance and are costly and time-consuming to implement as specification tests.

RESIDUAL BITUMEN QUALITY AND PERFORMANCE OF PREMIX SURFACINGS

The performance of five asphalt surfacings relative to the quality of the residual bitumen recovered from the road, is described. The road performance of the satisfactory sections was monitored and assessed by visual examinations and deflection

¹ Shell trade mark

measurements. Cores and blocks were removed from the surfacings to determine the engineering properties of the mixes and the properties of the residual binder.

Laboratory testing of asphaltic materials

Specification testing of the original bitumens, RTFOT-residues and the residual bitumen recovered from the hot-mix (12) were determined according to South African Bureau of Standards (SABS) specifications (13). The chemical composition of all the bituminous samples was determined by liquid chromatographic separation (HPLC) (3). The Fraass brittle temperatures were also determined (14).

The binders from the aged cores were recovered discretely from the top (0 -4 mm) and bottom (20 mm) of the cores, as well as all the binder from the top and bottom halves of duplicate cores. The properties of the binder in the upper crust (0-4 mm) are significantly affected by environmental conditions in contrast to the binder in the deeper layers of a well-compacted surfacing. The discrete recovery of residual binders make it possible to differentiate between environmental and characteristic ageing of bitumens of variable original chemical compositions, and to establish whether these differences affect long-term durability. Edler et al (15) used ratios of increases in the high molecular mass constituents, viscosity and oxidation levels to measure the ageing of thin binder films. Since most of the specification tests require a fair sized sample, only the ageing tests and chemical analyses could be performed on the small amounts of binder recovered from the discrete layers. The ratio of large to small molecular size fractions correlated well with asphaltene and viscosity values and was therefore discontinued as an ageing test in this project. Ductility, penetration, softening point and colloidal stability as determined by the Oliensis spot test, were performed on the binders recovered from the top and bottom halves of cores.

An evaluation of the residual bitumen from three sections, A, B and C, placed on the $\,$ N1/ Capetown - Messina national route, and which have performed satisfactorily

In 1987 a research project to determine the quality of bitumen as it is delivered from the refinery, through every stage of handling to the constructed asphalt layer, was initiated. The objective of this study was to establish for each pre-construction phase, the extent of change in the chemical and physical properties (quality) of penetration grade bitumens from different refineries. This information can assist the asphalt industry in decisions on accommodating variability either in the mix design, and application of asphalt, or by changes to the existing specifications. Two 40/50 penetration grade bitumens (A and B) from two different refineries and a 60/70 pen grade (C) from a third refinery, were used.

The virgin bitumens varied considerably with respect to chemical composition. No extraordinary ageing or degradation of the bitumens were found during the preconstruction phase. The properties of the binders recovered from the premix were similar to those of the RTFOT-residues - A aged slightly less and B and C more than predicted by the RTFO-test - the penetration of A was higher, B lower and C fairly similar to the values predicted by the RTFO-test, but the asphaltenes in B and C were substantially higher than the RTFO-residues' asphaltenes. The above results can also

be used as reference in the quality control of binders in hot-mix production. (The road bitumens produced in South Africa at individual refineries are fairly consistent with respect to their chemical composition.)

The aggregate in these asphalt mixes was semi-gap graded. Two 40/50 penetration grade bitumens (A and B) were used on two adjacent sections near Pretoria on the Transvaal highveld where climatic conditions are rather severe with extreme day/night winter temperatures and high ultra violet radiation. Both of these experimental sections are in the slow shoulder. The 60/70 pen bitumen (C) was used near Cape Town where a mild mediterranean climate prevails. This section is in the slow to fast lane. Three separate paving widths of asphalt were placed on this section and cores for laboratory testing were taken from all three. The construction operations on all the sections were closely controlled to ensure that the design specifications with respect to voids and density were met.

These sections have been monitored over the past four years and cores for laboratory testing were taken after three years of service. All the sections are still performing satisfactorily except for very limited reflective cracking from the supporting structure on the B-section. No premature cracking of the surface, flushing, ravelling or loss of chips have occurred.

The results of the original bitumens, RTFOT-residues, binder recovered from the premix and the residual binders (top and bottom) after three years of service, are shown in Tables 1 to 3 for the A, B and C-sections respectively. Only small variations were found in the properties of different cores from the same section, except where cores were taken from the cracked areas in the B-section. Therefore the results from only three representative cores are given.

Discussion of results from sections A (N1/2), B (N1/22) and C (N1/1)

Oxidation levels (O.L.) from the sound surfaces are very similar, irrespective of pen grade and location, except for the binders from the cracks. The higher oxidation and viscosity levels found in the residual binders from the cracks have been caused by exposure. The work by Edler et al (15), showed that O.L.s seemed to reach a finite value and that they do not necessarily correlate with other properties usually associated with ageing, e.g. viscosity increases. No oxidation peaks were present in the deeper levels of asphalt from the uncracked sections of the B-section although their viscosities and chemical compositions changed considerably. The absence of oxidation products could indicate that the section was compacted more effectively because the viscosity of B was lower than that of A (13,5 vs 40,1 kPa.s). Oxidation measurement as a durability prediction criterion was therefore not considered, although it is a valuable tool in the evaluation of aged binders.

There seems to be a characteristic ageing pattern for different bitumens, especially with respect to changes in the chemical composition i.e. shift from the more non-polar to the more polar compounds. Provided the layer is adequately compacted, this pattern is more dependent on the chemical composition of the binder at the time it is placed on the road than on the effect of the environment. Comparisons of the

| | SAMPLE DESCRIPTION | | | | | | | | | | |
|------------------------------------|--------------------|---------|--------|------|----------|---------|-----------|-----------|-----------|-------------|-------------|
| TESTS | Original | RTFOT | Premix | | | Recover | ed from (| cores aft | er 3 year | s | |
| | 40/50 pen | residue | | Тор | cores at | 0 mm/b | ottom cor | res at 20 | mm | _ | |
| | , | | | Тор | Bot | Тор | Bot | Тор | Bot | Top half | Bot half |
| TESTS ON ORIG. AND REC BINDERS: | | | | | | | 17 | | | | |
| Pen 25 °C | 39 | 25 | 31 | | | | | | | 17 | 20 |
| Soft pt (°C) | 57,3 | | 61,1 | | | | | | | 69,2 | 67,0 |
| Fraass (°C) | -7.0 | -6,0 | -8,0 | | | | | | | -3,5 | -4,5 |
| Ductility (cm) 25°C | 100+ | 21 | 48 | | | | | | | 8 | 11,0 |
| Spot test | Neg | | 755 | | | | | | | Neg | Neg |
| Max. xylene value | | | | | | | | | | 25 | 25 |
| Oxidation level | | | | 0,87 | 0,65 | 1,02 | 0,70 | 0,95 | 0,63 | 0,71 | 0,65 |
| Visc @ 50 °C (kPa.s) | | | | 222 | 40,1 | | 5,000,000 | 210 | 120012/2 | 534555 | 2000 |
| Binder content (%) | | | | | - 10 | | | | | 4,98 | 5,28 |
| Chem anal % m/m: | | | | | | | | | | | |
| Saturates | 7,8 | 8,6 | 8,0 | | | | | | | | 3,0 |
| Aromatics | 36,0 | 31,5 | 33,5 | 16,3 | 23,7 | 13,3 | 22,4 | 21,8 | 23,6 | 22,1 | 22,9 |
| Resins | 38,1 | 38,7 | 32,2 | 28,6 | 35,4 | 28,1 | 28,6 | 36,2 | 34,3 | 29,6 | 30,6 |
| Asphaltenes | 20,4 | 24,3 | 24,3 | 30,8 | 27,0 | 30,9 | 29,2 | 30,6 | 27,1 | 31,8 | 31,0 |
| Ageing index | 3,6 | 2,9 | 2,7 | 1,5 | 2,2 | 1,3 | 1,7 | 1,9 | 2,1 | 1,6 | 1,7 |

Table 1 - Properties of original and recovered Binders from A - top and bottom Layers

| | | | | | SAMPLE | DESCR | IPTION | | | | |
|--------------------------------------|--------------|---------|--------|------------------------------------|----------|--------|----------|-----------|------|-------------|-------------|
| TESTS | Original | RTFOT | Premix | Recovered from cores after 3 years | | | | | | | |
| | 40/50 pen | residue | | Тор | cores at | 0 mm/b | ottom co | res at 20 | mm | 1 | |
| | | | | Тор | Bot | Тор | Bot | Тор | Bot | Top half | Bot half |
| TESTS ON ORIG. AND | | 1111 | | | | | | | | | |
| REC BINDERS: | 40 | 0.4 | 00 | | | | | | | 47 | 40 |
| Pen 25 °C | 42 | 31 | 26 | | | | | | | 17 | 19 |
| Soft pt (°C) | 52,2 | 0.5 | 56,9 | | | | | | | 61,5 | 61,1 |
| Fraass (°C) | -4,0 | -3,5 | -4,5 | | | | | | | 0,0 | -0,5 |
| Ductility (cm) 25°C | 100+ | 100+ | 100+ | | | | | | | 62 | 140 |
| Spot test | Neg | | | | | | | | | Neg | Neg |
| Max. xylene value Oxidation level | | | | 0.00 | NODE | 1.00 | 0.74 | 0.00 | NODE | 20 | 20 |
| | | | | 0,96 | NOP* | 1,06 | 0,74 | 0,96 | NOP* | 100 | |
| Visc @ 50 °C (kPa.s) | | | 10 | 264 | 13,5 | 341 | 19,6 | | | 18,9 | 4.04 |
| Binder content (%) | | | | U | | С | | U | | 4,79 | 4,81 |
| Cracked(C)/Uncracked(U) | | | | U | | C | | 0 | | | |
| Chem anal % m/m: | | | 19 | | | | | | | | |
| Saturates | 9,4 | 7.2 | 5,3 | 7.8 | 8,0 | 5.7 | 6,3 | 4,7 | 6.6 | 5,5 | 7,0 |
| Aromatics | 38.3 | 38,9 | 32,9 | 19,5 | 26,5 | 19,2 | 24,1 | 20,8 | 24,5 | 24 | 25 |
| Resins | 36,0 | 34.7 | 44,9 | 32.4 | 43,2 | 29,7 | 38,2 | 33,5 | 37,7 | 40,6 | 40,8 |
| Asphaltenes | 11,9 | 13.8 | 18,7 | 28,2 | 20,3 | 27.1 | 21,8 | 25,5 | 20,0 | 21,8 | 20,5 |
| Ageing index | 6,2 | 5,3 | 4,2 | 1,8 | 3,4 | 1,8 | 2,9 | 2,1 | 3,1 | 3,0 | 3,2 |

^{*} NOP = No oxidation peak

Table 2 - Properties of original and recovered Binders from B - top and bottom Layers

| | SAMPLE DESCRIPTION | | | | | | | | | | |
|--|-----------------------------------|------------------------------------|------------------------------------|------------------------------------|------------------------------------|------------------------------------|------------------------------------|------------------------------------|------------------------------------|--|--|
| TESTS | Original | RTFOT | Premix | Recovered from cores after 3 years | | | | | | | |
| 12010 | 60/70 pen | residue | | Тор | cores at | 0 mm/bo | ottom cor | es at 20 | mm | T . | D-4 |
| | p | | | Тор | Bot | Тор | Bot | Тор | Bot | Top half | Bot half |
| TESTS ON ORIG. AND REC BINDERS: Pen 25 °C Soft pt (°C) Fraass (°C) Ductility (cm) 25°C Spot test Max. xylene value Oxidation level Visc @ 50 °C (kPa.s) Binder content (%) | 59 50,0 -9,5 100+ Neg | 36 -8,0 100+ | 37 52,7 -7,5 100+ | 1,01 83,2 | 0,77 | 0,96 | 0,69 | 0,91 40,6 | 0,48 7,110 | 18 61,3 -1,0 38 Pos 40 6,6 5,68 | 20 59,5 -1,5 111 Pos 35 |
| Chem anal % m/m: Saturates Aromatics Resins Asphaltenes Ageing index | 9,1 36,2 54,0 9,6 9,4 | 6,6 29,7 45,7 13,9 5,4 | 7,8 35,7 34,8 15,1 4,7 | 7,2 16,8 34,7 23,2 2,2 | 7,1 20,3 42,8 18,6 3,4 | 5,5 17,4 34,5 23,3 2,2 | 6,9 22,2 39,4 19,5 3,1 | 7,2 17,2 36,9 23,2 2,3 | 5,6 23,2 46,5 17,1 4,1 | 6,8 20,1 37,3 22,0 2,6 | 7,8 21,1 38,5 19,4 3,1 |

Table 3 - Properties of original and recovered Binders from C - top and bottom Layers

inherent changes based on the chemical composition of the aged binder recovered from the deeper layers *, show that C changed the most and A the least, when differences in chemical composition from the original are expressed as percentages of the original chemical composition. However, the asphaltene values of A, both for the original as well as for the aged bitumens, are higher than B or C. The ductility of A also decreased substantially. The numerical differences between the physical and chemical properties of refinery to aged binders are shown in Figure 1. Increased asphaltene values and decreased ductilities were the outstanding features of this study.

A good correlation between the Ageing Indices (A.I.s) of the premix residual binders and the ductilities of the aged residual core binders were established. Values of below 2.8 for premix binders resulted in substantial loss of ductility while premix binders with values in excess of 3.5 retained their cohesive properties.

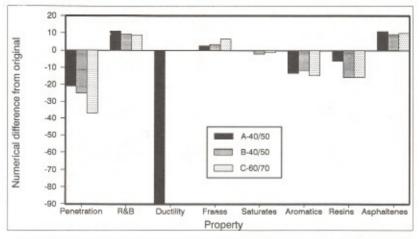


Figure 1 - Comparison of inherent Ageing of refinery to aged Binders

An evaluation of the residual bitumen from two distressed hot-mix applications

Both these "problem" pavements, D and E, were constructed in the Highveld region of the Transvaal. Both were of continuously graded design. The hot-mixes were produced by different plants and according to the manufacturers, the penetration grade bitumens used for the asphalt conformed to the local specifications. The specified depth of the compacted layer of asphalt was 25 mm. The mean thickness of the D-asphalt was slightly below this value, but the depth of the E-surfacing varied excessively. A 60/70 pen grade bitumen from refinery A and granite aggregate were used for the D-mix. The aggregate and binder types used for the E-surfacing have not been established conclusively.

Only chemical compositions could be determined on the small sample sizes recovered at the discrete levels.

Extensive investigations into the probable causes of the distress, indicated that the binders recovered from these distressed sections showed striking similarities with respect to some of the properties which were tested.

Premix surfacing D exhibited severe distress in the form of ravelling and disintegration of the pavement, particularly at stressed areas such as bends. Visual examinations by independent bodies, concluded that the distress had originated in the surfacing and that marginally inadequate design could have contributed to the early distress. The other contributing factor related to properties of the binder.

Blocks and cores of asphalt were taken randomly from these surfacings. These samples were tested extensively, both with respect to engineering properties of the compacted surfacing and properties of the residual binders. The properties of the asphalt and the residual binder recovered from them, are shown in Table 4.

| Sample | 1 | 2 | 3 | 4 | 5 |
|------------------------|------|------|---------|------|---|
| Mix properties | | | | | |
| Binder content % | 6,1 | | 5,6 | | 6,1 |
| Voids % | 7,5 | | 7,6 | | 7,5 |
| Tensile strength | 286 | | 286 | | 250 |
| (kN/m²) | | | | | 100000000000000000000000000000000000000 |
| Residual binder | | | | | |
| Pen (0,1 mm) @ 25 °C | 23 | 26 | 20 | 23 | 22 |
| Ductility (cm) @ 25 °C | 140 | 27 | 27 | 140 | 68 |
| Soft. Pt. °C | 57,0 | 60,8 | 60,8 | 58,3 | 59,1 |
| Spot test | Pos | Pos | Pos | Pos | Pos |
| Chem anal. % m/m: | | | 2000000 | | |
| Saturates | 9,1 | 8,6 | 7,7 | 7,1 | 8,6 |
| Aromatics | 29,4 | 29,6 | 29,4 | 32,1 | 32,4 |
| Resins | 38,5 | 35,8 | 34,6 | 39,9 | 40,6 |
| Asphaltenes | 20,0 | 22,6 | 25,4 | 21,3 | 22,4 |
| Ageing index # | 3,4 | 2,9 | 2,5 | 3,4 | 3,3 |
| Tar contamination | | | X | X | X |

^{# (}Aromatics + resins)/asphaltenes

Table 4 - Results on recovered Binders from distressed and "sound" Areas of D Discussion of results from pavement D.

The mean asphalt depth was about 2 mm lower than specified, although this varied in some instances to as much as 7 mm. The gradation of the aggregate recovered from the samples complied with the specifications. The binder film thickness based on the mean binder content of 5,8 per cent (specified binder content of 6,0 per cent) was a minimum of 6 micron which is acceptable for this particular design.

The voids content was higher than would normally be expected from a continuously-graded asphalt. The residual binders showed significantly reduced penetration, ductility and high asphaltene values when compared with average values for RTFOT-residues of similar bitumens, i.e. penetration 43, ductility 100cm and asphaltenes 18 - 21 per cent. The xylene-heptane values were in excess of 35/65. Laboratory tests have shown that for 40/50 and 60/70 penetration grade bitumens, which have been subjected to the rolling thin film oven test, this value is a maximum of 30/70 and 25/75 respectively. Furthermore, the presence of tar compounds which have percolated through the wearing course is an indication of inadequate compaction and when substantial amounts are present they may cause spurious results, e.g. lower viscosity and positive "spot test" due to presence of mineral fines in tar. The presence of oil and tar residues originating from both the surface and bottom of the cored material, distorted both physical and chemical properties of the recovered binders to a degree, especially with regard to the aromatic fractions. The additional boost in aromatic compounds afforded by the tar and oily residues, deceptively raised the A.I.s.

Low penetration and ductility values correlated well with both high asphaltene and n-heptane/xylene values and the degree of distress. These results indicate a very aged binder. Since this was a 'new' construction, it was concluded that the premature ageing was caused by exposure to prolonged high temperatures either during mixing or hot storage. Higher than expected viscosity of the aged binder could have contributed to inadequate compaction and high voids which in turn could have accelerated the ageing of the already prematurely aged binder.

Patches of the most severely affected areas were replaced and maintenance procedures were proposed as holding operations. The expected life time of the surfacing was estimated at eight years maximum depending on usage and maintenance.

Discussion of results from pavement E

Premix surfacing E was severely distressed within six months of service. Ravelling and crumbling of large areas of the surfacing were obvious. The surface had disintegrated almost completely in areas where storm-water overflowed onto the pavement.

The same procedure for testing and sampling as previously described was followed. The test results are shown in Table 5. They show a similar trend to the properties of binders recovered from the D-surfacing, but are obviously more aged. This is also in accordance with the differences in performance of D and E. Again tar compounds were detected in the recovered binders although thick layers of sand/tar prime were removed prior to testing. Chemical analyses of the discrete top to bottom layers of the sampled asphalt material showed that the tar compounds originated from the prime layer and that they had percolated through at least half of the layer thickness and in most cases, right through the layer thickness. The binder content and voids vary substantially and it seemed as if the compaction was not adequate. The role of high viscosity of a prematurely aged binder during compaction, and high voids in the acceleration of the ageing and oxidation processes is even more marked in this surfacing. The aged state of the binder seemed to have been one of the

| Sample | 1 | 2 | 3 | 5 | - 6 | 8 | 9 | 10 | 12 | 13 | 15 | 16 | 18 |
|------------------------|------|------|--------|---------|-------|---------|-------|------|------|------|------|------|------|
| Mix properties | | | | | | | | | * | | | | |
| Binder content % | 11,0 | 7,6 | 5,6 | 6,9 | 5,8 | 7,2 | 6,0 | 7,4 | 4,6 | 5,8 | 5,1 | 5,1 | 5,8 |
| Voids % | 10,6 | 4,1 | 9,5 | 10,5 | 7,9 | 8,1 | 7,7 | 7,4 | 9,8 | 6,6 | 3,1 | 11,1 | 10,2 |
| Residual binder | | | | | | | | | | | | | |
| Pen (0,1 mm) @ 25 °C | 38 | 37 | 27 | 25 | 25 | 23 | 25 | 21 | 18 | 21 | 21 | 18 | 34 |
| Ductility (cm) @ 25 °C | 140 | 88 | 91 | 140 | 67 | 129 | 55 | 19 | 26 | 17 | 27 | 21 | 140 |
| Soft. pt. °C | 1000 | | | 1000000 | | 1000000 | 2000 | | 200 | | 1000 | | |
| Spot test | Neg | Pos | Pos | Pos | Pos++ | Pos++ | Pos++ | Pos | Pos | Pos | Pos | Pos | Neg |
| Chem. anal. % m/m: | | | 22.000 | | | | | | | | | | |
| Saturates | 7.3 | 9,0 | 7,9 | 8,6 | 7,9 | 7,0 | 8,2 | 8,0 | 7,3 | 9,1 | 8,4 | 7,7 | 9,0 |
| Aromatics | 33,0 | 33,7 | 29,8 | 28,7 | 29,7 | 39,5 | 30,8 | 27,1 | 29,7 | 28,4 | 27,0 | 26,8 | 33,9 |
| Resins | 37,5 | 32,3 | 35,0 | 30,2 | 30,0 | 38,7 | 34,6 | 34,2 | 33,0 | 30,9 | 31,3 | 32,7 | 40,5 |
| Asphaltenes | 22,7 | 24,7 | 23,3 | 26,4 | 25,9 | 23,8 | 25,4 | 26,5 | 25,2 | 25,3 | 26,1 | 24,3 | 17,3 |
| Ageing index # | 3,1 | 2,7 | 2,8 | 2,2 | 2,3 | 3,3 | 2,6 | 2,3 | 2,5 | 2,3 | 2,2 | 2,4 | 4,3 |
| Tar contamination | X | X | X | X | X | X | X | X | | | | | |

Table 5 - Results on recovered Binders ex very distressed premix Surfacing E

primary causes for the distress, although the poor compaction could have accelerated the distress. At this stage, taking cognisance of the lack of quality control during the project, it is difficult to state emphatically that the poor quality of the binder in the manufactured hot-mix at the time of construction was the only reason for the unsuccessful compaction. Although some of the results were obscured by large proportions of residual tar compounds, a relation between low A.I., decreased ductility and the Oliensis spot test value, was clearly indicated.

AUSTRALIAN ROAD BOARD DURABILITY TEST AND CHEMICAL COMPOSITION OF BITUMENS

Results from the ARRB-durability test on 60/70 penetration grade bitumens which varied in their original (virgin) chemical composition are depicted in Figure 2. The samples have been identified by their penetration values. A direct relationship was found between A.I., low temperature ductility of the RTFOT-residues and days of exposure to the ARRB-test condition to reach the target viscosity, while the amount of asphaltenes are inversely related to ARRB durability. These results support the assumption that the ratio of soluble (n-hexane) aromatic compounds to the insoluble compounds (asphaltenes) is indicative of potential durability as measured by the ARRB-test and that durability is not necessarily directly related to the penetration value at 25°C.

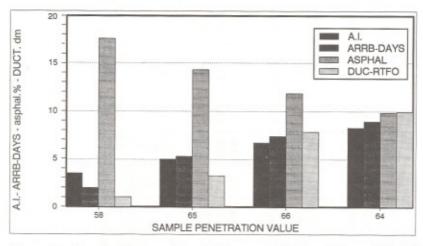


Figure 2 - Chemical Composition of Bitumen, ARRB-durability and Ductility

RAPID EVALUATION OF BITUMEN QUALITY

The durability of bitumen is determined by the amount and degree of polymerisation of the asphaltenes (alkane insolubles) and their ratio to the residual alkane soluble aromatic compounds. The Oliensis spot test was used to investigate the relationship between the minimum ratio of xylene required to solubilise the bitumens and the n-hexane insoluble asphaltenes (4) of bitumen samples from the 40/50, 60/70 and

80/100 penetration grades. The bitumen samples were dispersed in solvent-mixtures of increasing xylene:n-heptane ratios (5% increments of xylene), until a negative spot was obtained directly after the preparation of the suspension. The xylene value at this point minus 5 (the xylene value which produced a definite spot), was used as the end point of the test, and is referred to as the maximum xylene value (MXV). The proposed method was used to estimate the asphaltene values of unknown bitumens. The MXVs, estimated asphaltenes and gravimetrically determined n-hexane asphaltenes which were subsequently determined, are compared in Table 6. Viscosities of bitumens which vary in asphaltene content, were measured at 80 and 100 °C and are shown in Figure 3. The temperature/viscosity relationships are similar for bitumens of equal asphaltene content.

The MXV is directly related to asphaltene content of a bitumen. The method clearly differentiates between high (>15 %) and low (<10 %) n-hexane asphaltenes, even if very high ratios of maltene aromatic compounds should affect the MXV.

The asphaltene content correlates well with the temperature/viscosity properties of a bitumen. These properties can therefore be predicted from appropriate reference tables once the asphaltene content has been evaluated by means of the proposed test.

| Sample | % Xyle | ne in n-h | eptane | Viscosity* 100 °C | % Asphaltene | % Asphaltene | | |
|----------|--------|-----------|--------|----------------------|-----------------|-----------------|--|--|
| | 15 | 20 | 25 | Pa.s | estimated | determined | | |
| A 80/100 | +++ | ++ | + | 2,240 | 15 - 18 | 15,0 | | |
| B 80/100 | + | - | - | 1,723 | 9 - 11 | 8.5 | | |
| A 60/70 | +++ | ++ | + | 3,025 | 15 - 18 | 16,7 | | |
| A2 40/50 | +++ | ++ | + | 5.753 | 16 - 20 | 17,6 | | |

^{*} Absolute viscosity, measured by viscowaage viscometer, ball rod no 2.

Table 6 - Verification of estimated Asphaltene Values

CONCLUSIONS

Chemical analyses and low temperature ductility tests can be used to predict the potential durability of penetration grade bitumens. Higher A.I. ratios indicate a greater potential to absorb ageing. An upper limit for the A.I. has not been established.

Comparison of data obtained from distressed and successful new asphalts indicated a relationship between the ratio of soluble aromatic compounds to n-hexane insoluble asphaltenes (A.I.), ductility and the Oliensis spot test value. Residual bitumens recovered from unsatisfactory premix surfacings had A.I.s of less than 2,5, ductilities of less than 30 cm (25 °C) and they showed positive spots when dispersed in a mixture of 35:65 xylene-n-hexane solvent.

⁺ Spot intensity

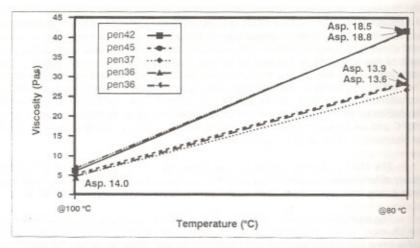


Figure 3 - Asphaltene Content and temperature/viscosity Properties

A relatively simple field test for asphaltene content estimation can be used for on-site quality control (Rapid Evaluation of Bitumen Quality). The asphaltene values are directly related to residual durability and temperature/viscosity performance of penetration grade bitumens.

KEYWORDS

Asphalts, Bitumen, Binder hardening, Durability, Quality control

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