ASPHALT BINDER HARDENING IN THE MICHIGAN TEST ROAD AFTER 18 YEARS OF SERVICE

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Analysis of six binders used in a Michigan test road has given an indication of the mechanism of compositional change and resultant hardening occurring during service. All the binders show the same directional changes in composition, namely a decrease in naphthene aromatics, which convert to polar aromatics and in turn to asphaltenes. This conversion is more pronounced in the top ⅛-in. (3.2-mm) layer than in the underlying ¼-in. (6.4-mm) layer. The mechanism proposed here clearly explains the increase in softening point, increase in hardness, and loss of ductility. Change in composition and physical properties also accounts for the slight but observable differences in the wear, weathering, and raveling qualities of the different sections. The Michigan test road and its overlays are still in service, although they show considerable reflection and joint cracking. Superficial judgment indicates that two of the test sections show more wear and weathering than the others. Although this is of technical interest, these differences are not large enough to permit quality judgment or selectivity between sources.

•IN 1954 the Michigan highway department constructed a 6-mile (9-km) test section on US-10, a four-lane highway between Pontiac and Flint. This was identified as M63-30, C8-R and consisted of 3 in. (76 mm) of hot mix (binder and surface) placed over existing portland cement concrete in six 2,400-ft-long (732-m) sections. The purpose of this test project was to correlate the comparative behavior of six typical asphalt binders available in Michigan by observing actual construction handling qualities and in-service response. Meticulous care was exercised in controlling aggregate gradation, binder content, temperatures, placing and compacting techniques, etc., so that only the source of the binder was a major variable. Details of planning, construction, and earlier observations are documented in three Michigan reports (2, 3, 5). Based on this work, the Michigan highway department approved all six sources of asphalt cement for use in bituminous construction on state trunk lines. Figure 1 shows scenes of this test road taken in March 1974, almost 20 years after the placement of the overlays.

After this test project had served its purpose, the original binders and binders extracted from each section were sampled and analyzed to

1. Determine the extent of change in chemical composition of the asphalt binders over long periods of service,
2. Relate those changes to the mechanism of binder hardening, and
3. Relate, if possible, the compositional changes with respect to wear and weathering qualities of the pavement.

Because the experiment was so well controlled, it was felt that such additional analyses offered an excellent opportunity for relating binder source, or its composition, with service observations. After binders extracted from cores taken in 1967 were examined, a final set of cores was taken in 1972, 18 years after the initial placement of the overlays. Although these test sections had been examined many times by the Michigan highway department and others interested in the project, a final rating was made by five engineers for the purpose of this report. This paper summarizes the

Figure 1. (a) Section 6 north and (b) section 1 south.

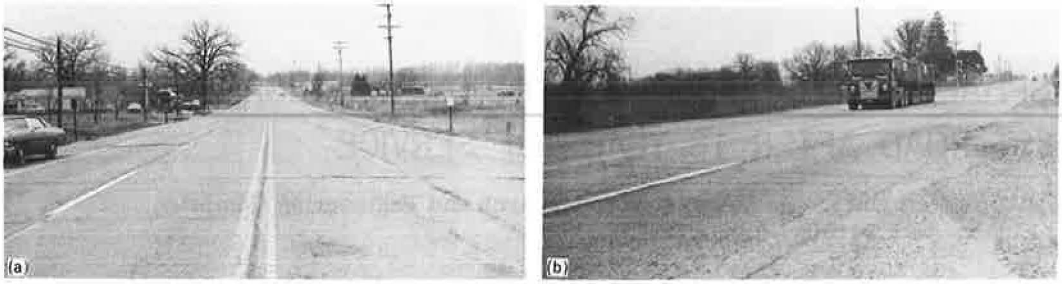


Table 1. Test data on asphalt cements (1954).

Property	Section 1	2	3	4	5	6
Softening point, F	120	125	119	123	120	124
Penetration at 77 F	63	60	67	60	61	65
Ductility at 60 F, cm	150+	150+	150+	150+	150+	77
Ductility at 77 F, cm	150+	150+	150+	150+	150+	150+
Kinematic viscosity at 140 F[a], stokes	2,457	4,336	2,361	3,159	3,869	4,418
Absolute viscosity at 140 F[a], poises	2,467	4,392	2,370	3,149	3,830	4,458
Saybolt furol viscosity at 275 F, sec	192	313	197	217	276	337
Thin film oven test at 325 F for 5 hours						
Percentage of loss	0.02	0.13	0.08	0.09	+0.09	0.02
Res. penetration at 77 F	36	38	40	42	39	45
Percentage of original penetration at 77 F	56	63	60	70	64	69
Res. ductility at 77 F, cm	150+	150+	150+	150+	150+	77

Note: 1 F = 1.8 C + 32; 1 stoke = 0.0001 m^2/s; 1 poise = 0.1 Pa·s.
[a]1965 data.

Table 2. Compositional analysis of original and recovered asphalts.

Sample	Fraction	Section 1	2	3	4	5	6
Original	Saturates	9.8	6.0	8.6	13.9	7.9	8.6
	Naphthene aromatics	32.5	28.8	32.6	31.3	42.0	38.7
	Polar aromatics	41.7	45.1	46.7	40.9	36.5	32.4
	Asphaltenes	16.0	19.2	12.0	12.8	13.3	19.7
Recovered, top ⅛-in.	Saturates	9.8	7.1	9.7	15.7	9.6	9.9
	Naphthene aromatics	25.9	20.7	25.9	22.7	28.4	24.2
	Polar aromatics	43.9	43.8	41.2	40.5	40.5	35.2
	Asphaltenes	19.3	27.7	19.4	20.3	20.7	28.8
Recovered, ¼-in. minus	Saturates	8.9	5.7	8.0	13.7	7.8	8.7
	Naphthene aromatics	34.6	28.9	35.5	30.4	32.7	35.0
	Polar aromatics	39.5	40.4	40.1	40.3	43.4	31.5
	Asphaltenes	16.9	22.8	15.6	15.0	15.9	24.7

Note: 1 in. = 25 mm.

compositional changes that occurred and relates these changes to the final ratings.

TESTING PROCEDURES

The original bulk retained asphalt binders are numbered from 1 to 6 as identified with the same number used for the six test sections in which they were used. Each binder was graded in the 60 to 70 penetration range to meet the Michigan specifications as well as ASTM D946. Complete physical properties and source identification are given elsewhere (2).

A summary of the physical properties, as measured on the original binders (2), is given in Table 1. Included are data on kinematic viscosity at 140 F (60 C) from the 1967 report (5) and an estimated value for absolute viscosity at 140 F. It is of interest to note that, relative to the current viscosity grading system, two of the binders would have been close to the AC-20 grade and four binders would have fallen in, or close to, the AC-40 grade.

Table 2 gives the results of compositional analyses that were performed on the original bulk samples in 1972 and the recovered samples. In accordance with Corbett's method (7), each binder was separated into four generic fractions: saturates, naphthene aromatics, polar aromatics, and asphaltenes. Although some aging may have occurred during the long-term storage of the bulk samples before this analysis, we believe that such changes were minimal. In any event, these differences are minor, especially since the hardening changes occurring in binders from the road cores were substantially larger.

Asphalt binders from pavement cores taken in 1972 were extracted with benzene (10) and recovered by using the Abson method (6). The cores were sawed into two layers, as shown in Figure 2. The surface mix in each section was 1¼ in. (32 mm), and a binder course comprised the remainder of the 3-in. (76-mm) overlay. All cores were taken in the passing lane in the wheel track, where vehicle drippage is minimal. Thus, each core provided a binder sample from the top ⅛-in. (3.2-mm) layer and another sample from the ¼-in. (6.4-mm) minus layer. All asphalt binders recovered from the core layers were then analyzed.

DISCUSSION OF RESULTS

Binder Inspections

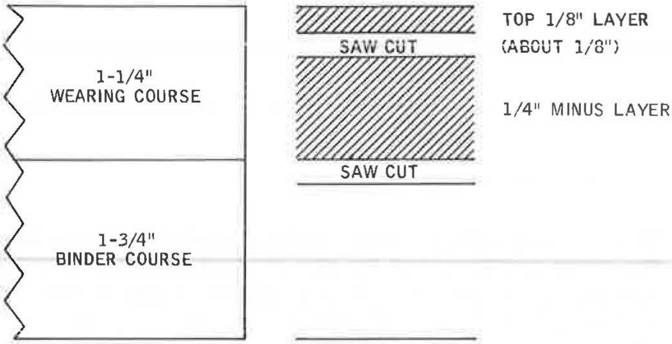
Compositional analyses, together with the physical properties of both the original (Table 2) and recovered binders (Table 3), led to the following observations.

In all cases, softening points and viscosities increased, and penetrations and ductilities decreased. These changes were greater in the binder from the ⅛-in. (3.2-mm) top layer than in the binder from the ¼-in. (6.4-mm) minus layer. This seems to indicate that hardening changes are more pronounced at or near the surface of a pavement because of greater exposure to air, sunlight, and other atmospheric effects. The binders from sections 2 and 6 in general had the highest consistencies and lowest ductilities, in both the top and minus layers.

The saturate content from recovered binders was virtually unchanged from that of the original binders. The saturate content in the top ⅛ in. (3.2 mm) showed some change, generally an increase, which is attributable to drippage. This drippage effect has been observed in other analyses of pavement cores (8). The amount of naphthene aromatics decreases in all cases in a manner similar to that reported when asphalt is air blown (4). There also seems to be greater change in composition in the ⅛-in. (3.2-mm) top layer than in the ¼-in. (6.4-mm) minus layer, and this appears consistent in all examples. Asphaltenes also consistently increase, especially in the top layer; the polar aromatics show no distinct pattern.

Figure 3 shows the average change in hardening for all sections. Basically, the

Figure 2. Method of preparing core for binder recovery.



1-1/4" = 3.18 cm
1-3/4" = 4.45 cm
1/8" = 0.32 cm
1/4" = 0.64 cm

Table 3. Physical properties of original and recovered samples.

		Section					
Sample	Property	1	2	3	4	5	6
Original	Softening point, deg F	126	124	124	125	125	126
	Penetration at 77 F	46	52	48	49	51	49
	Ductility at 60 F, cm	150+	150+	150+	67	16	27
	Viscosity at 140 F[a], poises	3,880	5,310	3,290	4,480	6,410	7,740
Recovered, top ⅛-in.	Softening point, deg F	136	150	140	147	147	165
	Penetration at 77 F	26	19	23	23	23	19
	Ductility at 77 F, cm	150+	7	150+	6	7	5
	Ductility at 60 F, cm	5	0.5	4.5	—	3	2.5
Recovered, ¼-in. minus	Softening point, deg F	129	136	133	135	138	147
	Penetration at 77 F	37	34	36	35	36	32
	Ductility at 77 F, cm	150+	150+	150+	150+	150+	40
	Ductility at 60 F, cm	8	8.5	8.5	6.5	6	4.5
	Absolute viscosity at 140 F, poises	7,320	17,041	7,752	9,705	11,787	34,414

Note: 1 in. = 25 mm; 1 F = 1.8 C + 32; 1 poise = 0.1 Pa·s.
[a]1965 data.

Figure 3. Mechanism of change for (a) top ⅛-in. layer and (b) ¼-in. minus layer.

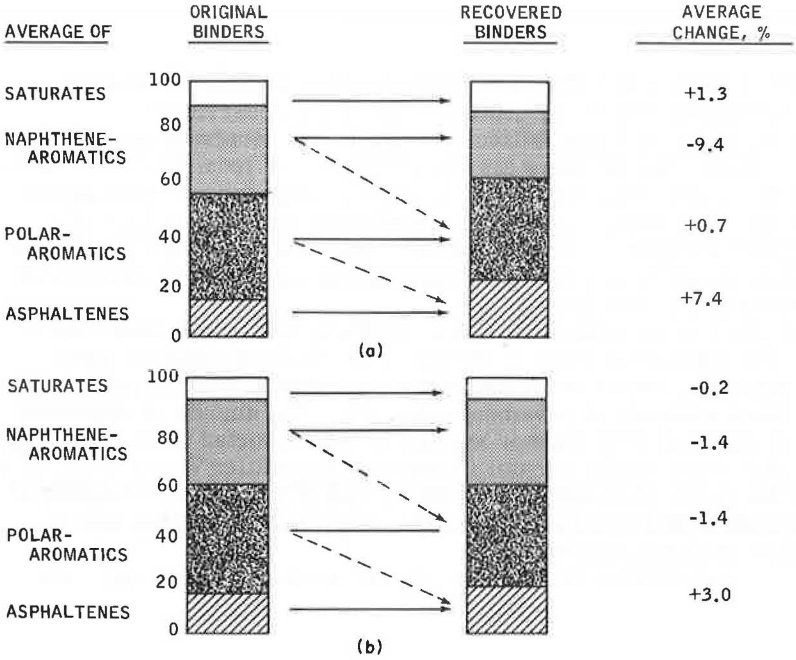


figure reveals a significant reduction of naphthene aromatics and an increase in asphaltenes. This figure confirms that the chemical change is consistently greater in the top layer than in the minus layer.

The compositional changes due to aging decrease in the liquid component (naphthene aromatics) and increase in the solid components (polar aromatics and asphaltenes), which is in line with the expected and observed physical data showing hardening. The average change in physical properties of the binders emphasizes the very substantial difference between the top and minus layers.

Property	Top Layer	Minus Layer
Softening point, F	26	15
Penetration at 77 F	-41	-28
Percentage of original penetration	35	56
Ductility at 77 F, cm	7	150+

This comparison and the detailed data in Table 3 tend to confirm that below the top ⅛ in. (3.2 mm) the binders are less affected, and thus the major bulk of the binder continues to be capable of performing its functional role for long periods. Furthermore, there is little distinction among binder sources in aging resistance in the ¼-in. (6.4-mm) minus layer.

It is known that volatilization of light components from the binder during pavement construction and aging is a factor that should be considered in evaluation of the hardening tendency. However, the volatility factor did not appear to be significant with these six binders. They all showed negligible losses by thin film oven testing (Table 1), and the slight differences reported in the original binder testing did not appear to correlate with the physical changes of the aged binder or with pavement performance.

Pavement Inspections

Over the past year or so, each of the test sections was inspected and rated by five engineers. Although pavement cracking was prevalent in all sections, this was not considered in the ratings because much of the cracking was obviously reflection cracks from the underlying portland cement concrete. Cracks from transverse expansion joints as well as joints made by the paving machine were very much in evidence. Other cracks, which probably result from cracking within the concrete slabs, were also in evidence but would be expected in pavements of this age. The ratings given in Table 4 are thus the average of the five appraisals in which judgment was based largely on wear and weathering of the surface, with some consideration of edge raveling. The method used by the engineers was to visually observe the surface, and to note the extent of aggregate exposure and loss of mortar. Because the differences in these qualities are difficult to judge, ratings were based on selecting the two best and the two poorest and leaving the other two as intermediate. Averaging all of these factors gave sections 1, 3, and 5 as best, 2 and 6 as poorest, and 4 as intermediate. There was complete unanimity in rating sections 2 and 6 as the poorest in wear and weathering qualities. Ratings on the other sections were not so decisive. Again, these do not represent great differences, as is shown by photographs in Figure 4 (taken in March 1974), but there were enough differences to permit superficial ratings.

Although the nature of this study did not permit complete identification of the chemical mechanisms involved in aging, the study does shed light on the natural overall hardening process. There is definite conversion of the naphthene aromatics fraction and a conversion of some of the polar aromatics to higher molecular weight asphaltene fractions, postulated by various condensation mechanisms. The apparent greater reactivity of naphthene aromatics seems to be in line with the laboratory photooxidation studies carried out by Thurston and Knowles (13), who showed that greater oxygen

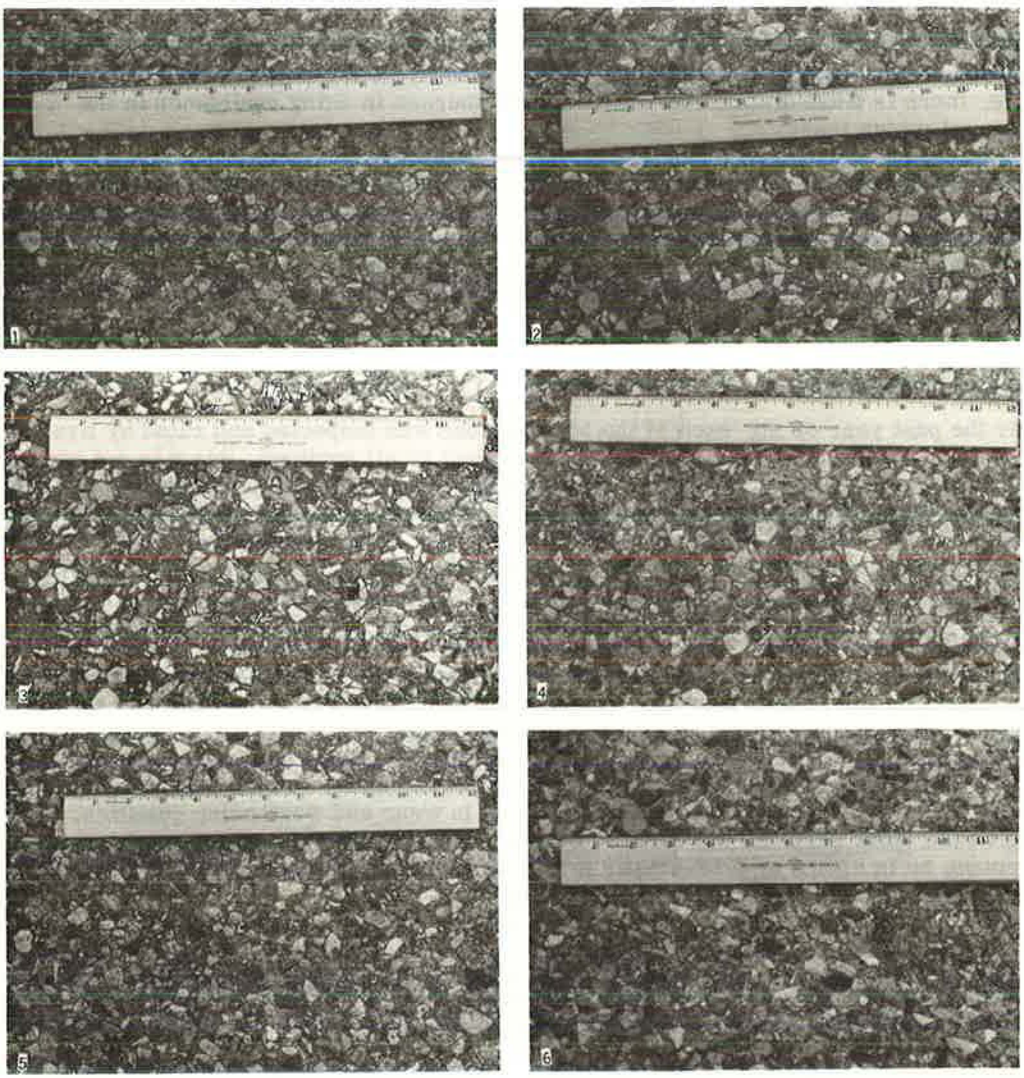
Table 4. Relation of visual inspection of pavement to binder changes.

Section	1	2	3	4	5	6
Average pavement rating[a]	1.4	3.0	1.4	1.8	1.4	3.0
Changes in top ⅛-in. layer						
Δ Softening point, F	+16	+25	+21	+24	+27	+41
Δ Penetration at 77 F	-37	-41	-44	-37	-38	-46
Percentage of original penetration at 77 F	41	32	34	38	38	20
Δ Ductility at 77 F, cm	0	-143	0	-144	-143	-145
Changes in ¼-in. minus layer						
Δ Softening point, F	+9	+11	+14	+12	+18	+23
Δ Penetration at 77 F	-26	-26	-31	-25	-25	-33
Percentage of original penetration at 77 F	59	57	54	58	59	49
Δ Ductility at 77 F, cm	0	0	0	0	0	-110

Note: 1 in. = 25 mm; 1 F = 1.8 C + 32.

[a] 1 = best and 3 = poorest.

Figure 4. Sections 1 through 6.



consumption occurs with an isolated naphthenic fraction of asphalt than with other fractions.

It is of interest to note that binders 2 and 6, which had the highest asphaltene contents both before and after aging, showed greater change in physical properties and somewhat more wear and weathering. It is believed that these higher consistency properties can be related to the amount of plasticizing components present in the binder. As shown in other fractionation work (4), the saturates and naphthene aromatics fractions are low-viscosity components, and it is believed that they, therefore, function as plasticizers for the high-viscosity components, i.e., the polar aromatics and asphaltenes. Binders 2 and 6 appear to show relatively less of the plasticizing components and thus are associated with binders of higher consistencies. It is noted, of course, that binders 2 and 6 had the highest viscosity among these penetration-graded cements. This is generally expected in view of their higher asphaltene contents. Binder 5 also had a relatively high original viscosity, not far from binders 2 and 6, and it showed fairly aggressive changes with aging in increasing its asphaltene content and its softening point. The favorable road evaluation of section 5 may be attributable to a higher proportion of the plasticizing components present in its original form. These being carried through during its service life resulted in the relatively better rating as compared to sections 2 and 6.

CONCLUSIONS

1. Although this test project on US-10 has shown considerable reflection and joint cracking for some time, the road is still serviceable for secondary traffic and does carry heavy-duty vehicles. Wear, weathering, and raveling are evident in all sections, although more pronounced in sections 2 and 6.
2. Increases in binder consistency and loss in ductility are prevalent in binders from all sections, but the same two sections noted above showed greatest changes in these qualities.
3. Changes in composition occurring in binder hardening are directionally the same in all cases; naphthene aromatics converted to polar aromatics and then in turn to asphaltenes. Again, sections 2 and 6 consistently show larger compositional changes.
4. The top ⅛-in. (3.2-mm) layer consistently exhibits greater changes in consistency and larger changes in composition than the ¼-in. (6.4-mm) minus layer.
5. This study alerts the paving technologist to how core depth can affect results. That is, to obtain a more meaningful characterization in core analysis may require a workup of the various layers involved rather than an overall composite of the entire core.
6. Based on the present level of hardness of these binders and the age of the road overlay, there is no gross distinction among binders involved and no practical need to distinguish one binder from another.

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