Asphalt Aging in Texas Roads and Test Sections

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In 1982–1983, test sections were laid at three Texas locations using five asphalt sources and two grades. Not all combinations were used at all locations and two roads failed early, but results are reported on 16 sections. These roads were cored in 1984, 1985, and 1987. Viscosities, penetrations, voids, gel permeation chromatography (GPC), and Fourier transform infrared (FT-IR) analyses were run. Voids and aging are strongly correlated whether the aging is measured by a viscosity aging index, growth in carbonyl peak, or GPC. Although GPC, carbonyl peak, and sulfoxide generally increase with age, sulfoxide and other aging indexes are poorly correlated. Growth in sulfoxide correlates weakly with sulfur content. In general, the number of voids reveals large ranges in asphalt performance. Asphalts showing nearly the same aging index at low numbers of voids might differ several-fold at high numbers of voids. When comparison was possible, higher viscosity grades resulted in greater aging. Results also were obtained for several south Texas highways, ranging in age from 5 to 19 years. Although original tank asphalt, hot-mix, and early cores were not available for study, recovered asphalt properties from highway-aged cores followed trends similar to those for the test sections with respect to viscosity, penetration, FT-IR, and GPC measurements.

In 1982–1983, test sections were constructed at three locations in Texas using asphalts from five refineries. AC–10 and AC–20 grades were used in a total of 20 sections. In 1982, seven sections were placed east of Dickens, on the westbound travel lane of US-82. Seven sections were located north of Dumas on the northbound travel lane of US-287. These sections were also constructed in 1982. In 1983, six sections were installed on the northbound lane of two-lane US-96, about 25 mi south of San Augustine. These sections are referred to as the "Lufkin sections."

The aggregate used at Dickens was mined near the site. The coarse and intermediate fractions were crushed siliceous gravel, and the fines were similar material. The Dumas aggregate was an absorptive crushed limestone. The Lufkin aggregate consisted of a mixture of limestone and iron ore gravel plus field sand.

The principal objective of the latter study was to relate physical and chemical laboratory properties to field pavement performance. Some of the properties that were measured on the original asphalts are presented in Table 1 (1). Following construction, the Dumas and Dickens sites had high void contents. Cores were taken at Dickens and Dumas after 1 week and after 1 and 2 years, and at Lufkin after 1 week and 1 year. The voids after each period are shown in Table 1. At Dumas, the Dorchester section failed after 1 year and had to

be replaced. The Exxon section was also in bad condition and had to be partially replaced. At Dickens, all the test sections began to show raveling and were fog sealed by 1985.

In 1986, a new study was begun with the objective of correlating certain chemical properties to physical properties and road performance. In connection with this objective, the test sections were recored in 1987. By this time, the Exxon and Diamond Shamrock AC–10 at Dumas had been completely replaced, and the remaining sections at Dumas had been seal coated. Only the Lufkin sections still had their original surfaces and these were all in good condition. One Lufkin section listed as Dorchester AC-20 was omitted from the study, because the asphalt source was actually believed to be Texaco. This procedure left 16 sections to be cored, as presented in Tables 2 and 3.

The study was expanded by including some old roads that were still in good condition. District 21, in the southern tip of Texas, was chosen because this area has a generally hot, dry climate significantly different from the other locations. This district, in cooperation with the Texas State Department of Highways and Public Transportation (SDHPT) in Austin and Texas A&M University, has established a data base on their roads containing pertinent information about the construction, use, and condition of these roads. Using this data base, only six roads could be found that were uncracked and at least 5 years old. A road found with slight transverse cracking was also included. These roads, cored in June 1988, are presented in Table 4.

State Highway 186 in Willacy County was sampled at two locations. At Milepost (MP) 25.8, the pavement was laid in the fall of 1982. The road showed a small amount of raveling but was otherwise in good condition. At MP 34, the pavement was laid in August 1980.

US-77 was also sampled in two locations. At MP 16 in Willacy County, the road was in good condition although it was laid in August 1982 and was a high-traffic area. In Cameron County, US-77 was sampled at MP 27, near Harlingen. This location had the highest traffic level. It was paved in August 1982 and was seal coated in 1987, possibly to solve a raveling problem.

The only other high-traffic road sampled was US-281 in Hidalgo County. This site, at MP 37.5 near McAllen, was laid in January 1979 and seal coated in May 1985. Raveling was obviously a problem with this stretch of road, because the verge of the road was covered with aggregate from the original asphalt, not from the seal coat.

Two Texas farm-to-market (FM) roads were included. In Cameron County, FM-2925 was sampled at MP 12. The asphalt there was laid in April 1983. This pavement was cracked

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TABLE 1 VISCOSITIES OF VIRGIN ASPHALTS AND CORE VOIDS

	Viscosity (Poise)		% Voids		
	140°F	275°F	1 week	1 year	2 years
Dickens					
D.S. AC-10	1220	4.51	16.0	14.3	9.1
D.S. AC-20	2175	7.15	13.4	10.0	12.7
McM AC-20	2523	4.64	15.6	9.9	14.0
EXX AC-20	2576	3.55	14.8	9.9	13.6
Dorch AC-20	2151	4.53	11.7	14.1	13.0
Cos AC-10	1264	2.55	14.4	14.5	10.6
Cos AC-20	1515	2.87	15.0	10.5	12.3
Dumas					
D.S. AC-10	958	4.65	20.4	7.2	2.1
D.S. AC-20	2155	6.39	13.9	11.6	9.2
McM AC-10	961	3.63	12.2	13.7	10.5
EXX AC-10	1388	3.06	16.5	15.4	
Dorch AC-10	1030	3.21	12.6	12.2	8.6
Cos AC-10	1038	2.48	20.4	13.8	7.3
Cos AC-20	2354	3.17	17.1	10.5	10.2
Lufkin					
D.S. AC-20	1728	5.05	8.6	2.6	
McM AC-10	932	3.63	4.8	6.2	
EXX AC-20	1811	3.19	6.6	3.0	
Dorch AC-10	1040	2.88	3.2	4.2	
Dorch AC-20	1913	3.96	7.4	2.2	
Cos AC-20	1858	2.83	6.5	3.2	

Diamond Shamrock (D.S.)   McMillan (McM)   Exxon (EXX)
Dorchester (Dorch)   Cosden (Cos)

TABLE 2 PHYSICAL PROPERTIES OF TEST SECTION 1987 CORES AND EXTRACTED ASPHALT

	Percent Voids	Viscosity		Penetration at 77°F (0.1 cm)	Percent Asphalt
		140°F (Kilopoise)	275°F (Poise)		
Dickens					
McM AC-20	8.0	159	23.1	16	6.2
Dorch AC-20	13.0	222	22.4	10	4.6
EXX AC-20	9.0	900	70.7	12	5.1
D.S. AC-20	12.0	260	34.2	18.5	---
D.S. AC-10	9.0	48.9	17.9	25	5.3
Cos AC-20	11.0	376	26.4	10.5	4.0
Cos AC-10	12.0	342	18.2	11.5	5.6
Dumas					
McM AC-10	5.5	13.3	9.2	38	5.5
D.S. AC-20	8.1	32.5	18.9	27.5	5.9
Cos AC-10	9.5	23.4	26.7	18	5.9
Cos Ac-20	8.5	55.6	11.7	15.5	5.9
Lufkin					
McM AC-10	2.4	4.8	6.5	39	6.6
Cos AC-20	1.8	5.4	5.7	22	6.9
D.S. AC-20	2.1	9.1	11.2	30	6.5
EXX AC-20	2.5	3.9	5.8	40.5	7.7
Dorch AC-10	2.1	2.4	4.6	38.5	5.6

McMillan (McM)   Dorchester (Dorch)   Exxon (EXX)
Diamond Shamrock (D.S.)   Cosden (Cos)

TABLE 3   PARAMETERS USED IN AGING CORRELATIONS

	Carbonyl Peak Height	Sulfoxide Peak Height	Percent LMS	Aging Index 140°F	Aging Index 275°F
Dickens					
McM AC-20	34	23	33.6	63	5.0
Dorch AC-20	29	18	25.4	103	4.9
EXX AC-20	37	20.5	38.8	350	19.9
D.S. AC-20	35.5	13.5	53.9	119	4.8
D.S. AC-10	29.5	16.25	46.0	40	4.0
Cos AC-20	32	18	26.8	247	9.2
Cos AC-10	31	22	30.1	271	7.1
Dumas					
McM AC-10	15.5	30	24.4	13.8	2.5
D.S. AC-20	25	22.25	45.9	15.0	3.0
Cos AC-10	17	29	19.6	22.5	10.7
Cos AC-20	20.5	25	23.2	20.5	3.7
Lufkin					
McM AC-10	10	13	33.7	10	1.8
Cos AC-20	8.5	32.25	18.5	8.5	2.0
D.S. AC-20	22	10.25	45.0	22	2.2
EXX AC-20	8	25.75	23.5	8	1.8
Dorch AC-10	5	19	23.5	5	1.6

McMillan (McM)   Dorchester (Dorch)   Exxon (EXX)
Diamond Shamrock (D.S.)   Cosden (Cos)

TABLE 4   DATA FOR SOUTH TEXAS ROADS

Highway	Carbonyl Peak Height	Sulfoxide Peak Height	% LMS	Viscosity 140°F (kpoise)	Viscosity 275°F (poise)	Penetration (0.1 cm)	Age (yrs)
FM 2925	23.5	26.75	26.4	67.8	15.4	14	5
US 77/27[a]	10.75	33	25	27.5	12.3	23.5	5+1[b]
US 77/16[a]	18	28.5	24	43.3	12.5	14.5	6
SH 186/25[a]	30.75	36	30.5	130	21.0	14	6
US 281	22	28.75	26.4	22.0	8.5	22	6+3[b]
SH 186/36[a]	30.75	23	29.7	282	23.5	10	8
FM 1017	36.75	13.5	33.8	248	21.1	13	19

[a] Milepost
[b] Years Under Sealcoat

transversely about every 8 ft. The road was made up of a thin pavement over the base, and the high level of boat traffic to the Gulf of Mexico may have contributed to the cracking. The other FM road was officially listed as having a seal coat only. FM-1017 in Hidalgo County was cored at MP 7.5. This road was easily the oldest in the study, having been laid in May 1969 over what appeared to be an old gravel road. The pavement was rutted by the oil-field trucks traveling it, but it was in good condition considering its age and construction. In fact, only about 0.6 mi of the 1969 road still existed, the rest having been replaced. All of the pavements in the south Texas test sites were constructed with aggregate resembling

river gravel, which may have contributed to the raveling in many roads. These locations and those of the original test sections are shown in Figure 1.

PROCEDURES

All of the cores were extracted by a modified ASTM D2172–81, Method A, except at Lufkin, where all but the Cosden AC–20 were extracted by Method B. The procedure was changed after it was realized that Method B can change the asphalt. In both instances, ethanol was added to improve removal from the aggregate. In the Method B procedure, a 95 percent trichloroethylene, 5 percent ethanol mixture was used. For Method A, several extractions with trichloroethylene were followed by a 90 percent trichloroethylene, 10 percent ethanol mixture. The small amount of ethanol considerably enhanced removal of the remaining amounts of asphalt.

Solvent was removed either by the Abson or by the Rotovap procedures. Gel permeation chromatography (GPC) analysis of the recovered material indicated that many samples still contained solvent, and additional solvent removal was required to avoid erroneously soft asphalt. (The times specified in the standard procedures were frequently inadequate for complete solvent removal, especially from viscous core material.)

Voids and the percentage of asphalt were calculated (see Table 2). Penetrations at 77°F using ASTM D5 and viscosities at 140°F and 275°F using ASTM D2173 were run on the recovered material. These data are presented in Tables 2 and 4.

GPC chromatograms were obtained using an IBM 9533 Liquid Chromatograph controlled by an IBM 9000 computer. Two Polymer Laboratories chromatographic gel columns were used in series, a 500-Å pore size followed by a 50-Å pore size. The detector was a Waters R401 differential refractometer. Purified tetrahydrofuran (THF) was used as the solvent. Asphalt

samples were made to exactly 7 percent by weight in THF and sonicated for about 3 hr. The sonicated material was then filtered through a 0.45-μm filter to remove fines. The filtered material was placed in a 1.8-mL septum-capped vial and placed in an automatic sampler. A 100-μL sampling loop was used.

Infrared spectra of the recovered material were obtained on a Nicolet 60 SX B Fourier transform infrared (FT-IR) spectrometer. All the data were obtained by a KBr pellet procedure (2), which, though time consuming, gives a strong, reproducible signal. In a precise manner, 0.9750 g of KBr and 0.025 g of asphalt (frozen to enhance handling) were ground together in a mortar until the mixture was homogeneous. Then 0.300 g was taken to prepare a pellet. The mixture was placed in a pellet die and 34,000 lb of force was applied for 30 sec. A blank of pure KBr was prepared at the same time by the same procedure. Before sample preparation, the KBr was heated to about 700°F for 8 hr to remove moisture.

In order to use either GPC or IR spectra to correlate properties, some simplified characteristic was desired because the spectra are complex. For GPC, the percentage of large molecular size (LMS), as suggested by Jennings (3), was used. Arbitrarily, the entire spectrum was taken as 20 to 35 min and the LMS interval as 20 to 25 min during elution. The LMS percentage was calculated from the areas under the curve during these intervals.

Two areas of the IR spectrum relate specifically to oxidation. One is the carbonyl peak, occurring at about a wave number of 1,700, and the other is a sulfoxide peak at a wave number of 1,030. Because these peaks both occur in areas where absorption of other entities is present, the choice of peak height is somewhat arbitrary. For the sulfoxide peak, the method recommended by Peterson (4) is used as shown in Figure 2. A tangent line is drawn below the peak of interest touching the low points on either side. A similar procedure was used with the carbonyl peak. The parameter was the height of each peak above this tangent line.

RESULTS AND DISCUSSION

Properties of the 1987 test section cores and the extracted asphalts are presented in Table 2. Sulfoxide and carbonyl peak
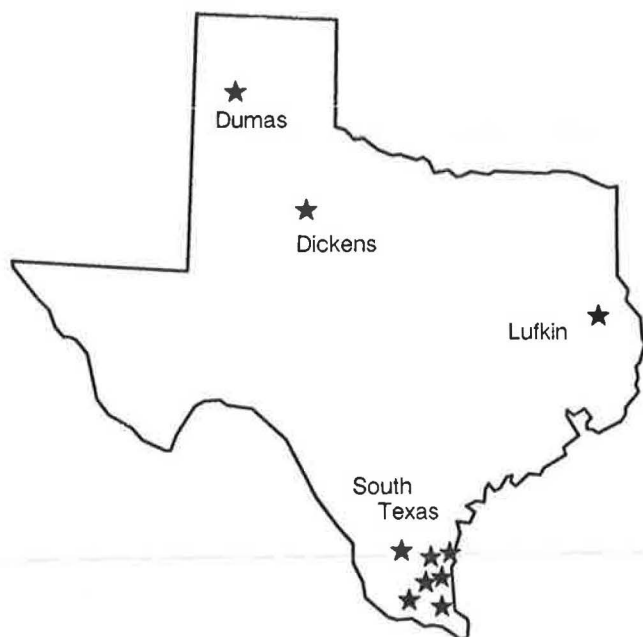

FIGURE 1   Location of test sections near Dickens, Dumas, and Lufkin and of highways cored in south Texas District 21.
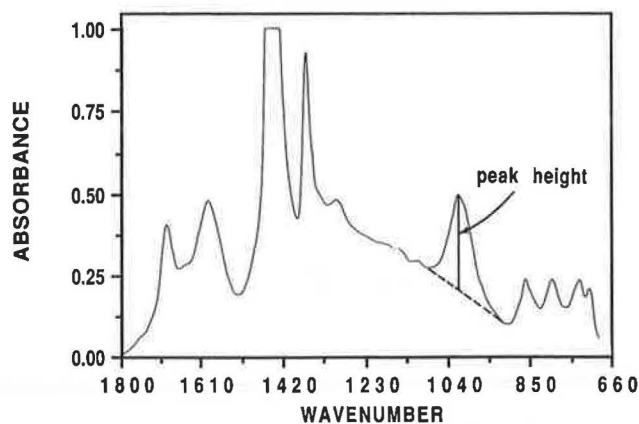

FIGURE 2   Example FT-IR spectrum showing the method of determining the sulfoxide band peak height near 1,000 cm^{-1}. The carbonyl peak height near 1,700 cm^{-1} is determined similarly.

heights from the IR analyses and LMS percentages from the GPC experiments are presented in Table 3, along with viscosity aging indexes at both 140°F and 275°F. The aging index is the ratio of the viscosity of the 1987 core extracts divided by the viscosity of the virgin asphalt from Table 1. Properties of the asphalt extracted from the south Texas cores are presented in Table 4.

Correlation of Voids and Aging

One of the surprising elements of the data in Tables 1 and 2 is the inexplicably high void level in the test sections at Dumas and Dickens, which has led to rapid aging and the early demise of three sections. For decades, high void levels have been known to shorten road life, yet they continue to be a problem, even in carefully monitored test sections.

An interesting case in point is a study of Pennsylvania test sections conducted during the 1960s and 1970s. This work, summarized by Gotolski et al. (5) and Roberts and Gotolski (6), included a variety of asphalts and aggregates. Performance was difficult to correlate with air voids because of the variability in voids from point to point, the steep variation across the road, and the effect of aggregate and gradation. Even so, it was found that: "Air voids are one, if not the greatest, factor affecting the rate of hardening of an asphalt pavement. The influence of the variable appears to be so pronounced that it completely overshadows the performance of asphalt type, aggregate type, traffic density and microclimate differences." The great effect of voids does seem incontrovertible, but the data indicate that asphalt quality becomes more critical at higher voids. One of the more interesting observations and conclusions from the Pennsylvania study, confirmed by the experience of the Texas test sections nearly 20 years later, is the following: "The pavements studied received better than average design and field control; yet these pavements were constructed with void contents as high as 13 percent. This underscores the need for more restrictive specifications and closer field control."

In Figure 3, carbonyl peak height is plotted versus percentage of voids. These are voids from 1987 cores that give better results than averaging the void data. This is probably because of variation in voids within the roadbed, so that earlier cores may not represent the cores being studied. Oxidation, as reflected in the growth of the carbonyl peak, increases rapidly with voids. The Dickens sections, which are represented by the seven highest carbonyl peaks, showed a greater response than the Dumas sections. In general, those sections constructed with the lower viscosity grade were also oxidizing less rapidly. In the three direct comparisons with the same supplier and site, Cosden at Dickens, Diamond Shamrock at Dickens, and Cosden at Dumas, this was the case. Though the Exxon at Lufkin appeared to be oxidizing no more than the other asphalts, the Exxon at Dickens was the most oxidized of all. The data are not sufficient, however, to conclude that any asphalt is more susceptible to oxidation.

In Figure 4, the aging index at 275°F is plotted versus the percentage of voids. The results are similar to those in the previous graph. The Exxon had a high aging index and a high carbonyl content. Both of the Cosdens appeared to be abnormally aged at Dickens. However, the Dumas-Cosden datum
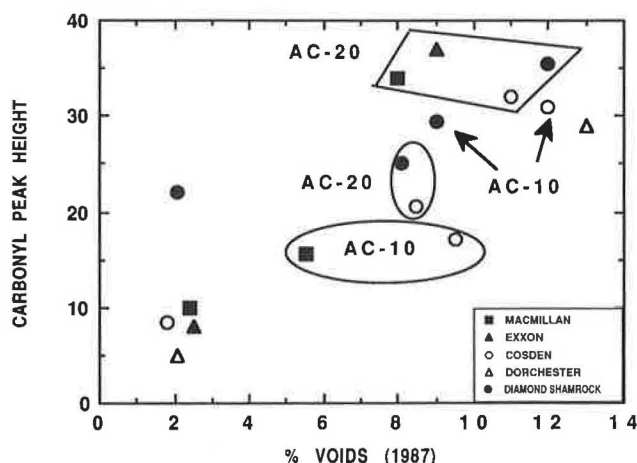

FIGURE 3 Carbonyl aging versus voids for asphalt from 1987 cores of the test sections.
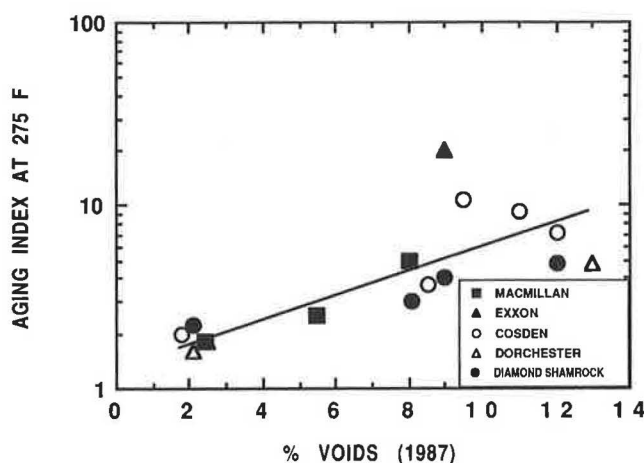

FIGURE 4 The 275°F aging index versus voids for asphalt from 1989 cores of the test sections.

at 8.5 percent voids was inconsistent with the 140°F data and with earlier cores and was probably in error. Figure 5 is a similar plot with 140°F aging indexes. Again the Exxon value was quite high, with high values for the Cosdens and perhaps for the McMillan. At Dumas, both Cosdens were near the curve. As mentioned earlier, the inherent problem in the study of voids versus aging is that roadbed variability makes it impossible to obtain an accurate voids history of the particular core being studied. So 1987 voids were used rather than average voids, no doubt accounting for much of the scatter.

Correlation of Carbonyl Content and Physical Properties

Because both the viscosity aging index and carbonyl peak height correlate with voids, they should cross-correlate. Figures 6 and 7 indicate that they do. Somewhat surprisingly, however, viscosity seems to correlate as well as the aging
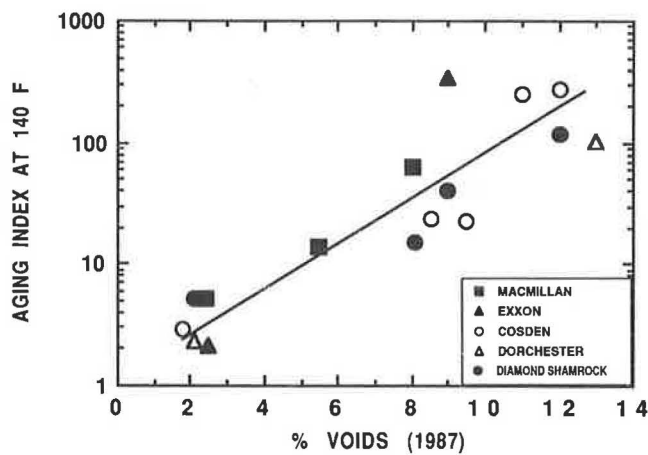
FIGURE 5   The 140°F aging index versus voids for asphalt from 1987 cores of the test sections.
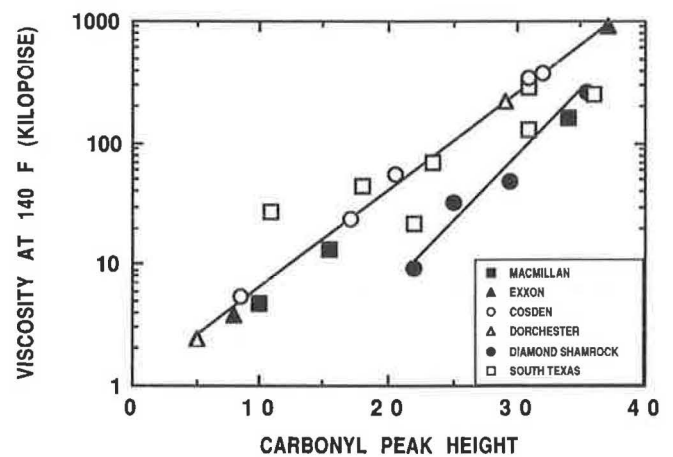

FIGURE 7   Viscosity at 140°F versus carbonyl peak height from 1987 cores of the test sections and from the south Texas highway cores.
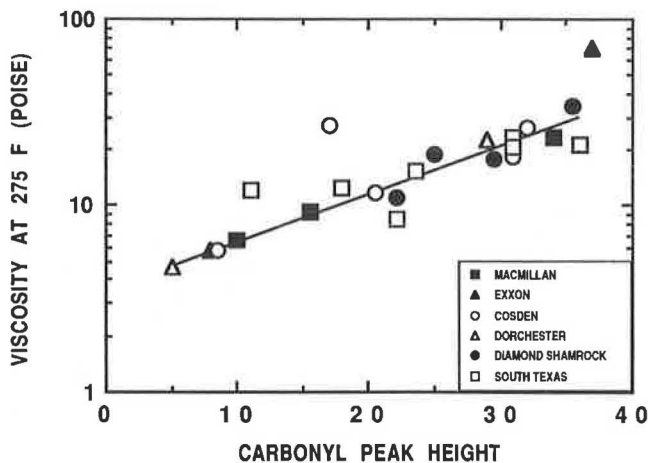

FIGURE 6   Viscosity at 275°F versus carbonyl peak height from 1987 cores of the test sections and from the south Texas highway cores.
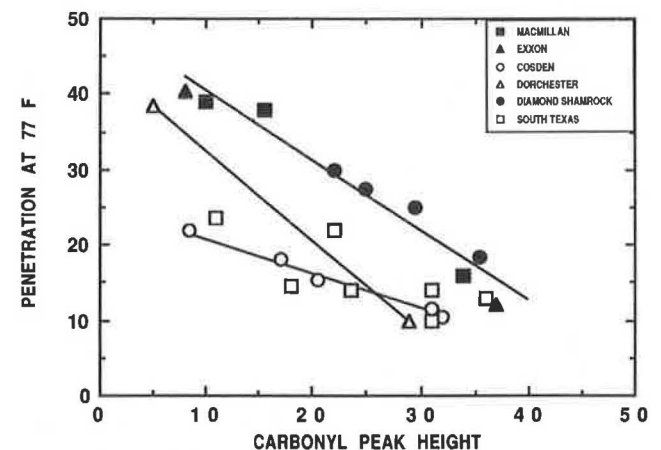

FIGURE 8   Penetration at 77°F versus carbonyl peak height from 1987 cores of the test sections and from the south Texas highway cores.

index, and this correlation allows the inclusion of the south Texas data, for which original viscosities are not available. At 275°F (see Figure 6), the Exxon Dickens value was very high. The high Cosden value is the suspect point previously discussed. Otherwise, most south Texas data fit very well.

At 140°F (see Figure 7), the Diamond Shamrock data had completely separated from the others. The Dickens Exxon was on the curve, but the plot of south Texas data had a different slope than the others. These data imply that the south Texas data in Figure 6 would also plot better with a lower slope.

In Figure 8, penetrations at 77°F are plotted versus carbonyl peak height. Individual asphalts are further separated, but Exxon and McMillan have almost merged with Diamond Shamrock, whereas Cosden has approximately joined the south Texas plot with an entirely different slope. The 19-year-old south Texas FM-1017 had only a slightly higher penetration than the 5-year-old Dickens Exxon. Several of the Dickens roads were approaching dangerously low penetrations, as was
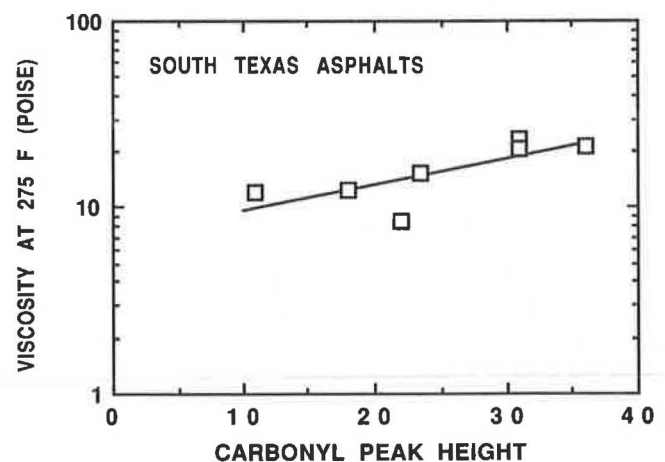
FIGURE 9   Viscosity at 275°F versus carbonyl peak height for asphalts from the south Texas highway cores.

Highway 186 at MP 36. This low penetration on Highway 186 was consistent with its high viscosity at both 140°F and 275°F.

Except for this road and Highway 281 at MP 37.5, which had abnormally high penetration and low viscosities, the south Texas roads show remarkable conformity. In Figures 9 and 10, viscosities of the south Texas roads are plotted versus carbonyl peak height at 275°F and 140°F. The 140°F correlation is particularly good, except for the two data referred to previously. All of these data tend to indicate that carbonyl peak height is a good measure of road aging for any particular asphalt with respect to both viscosity and penetration and can probably be used as an effective parameter in laboratory aging tests. However, carbonyl peak height cannot be related to road age in years, because the percentage of voids and probably other factors exert too much influence on aging. One of these other factors is almost certainly asphalt compatibility (7), which could account for much of the divergence of individual asphalts noted.
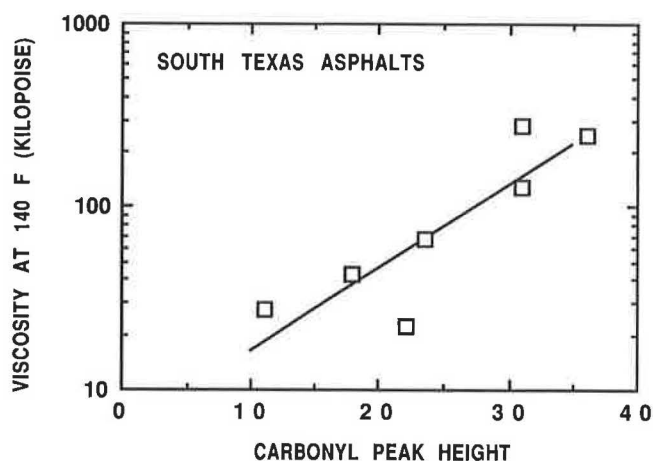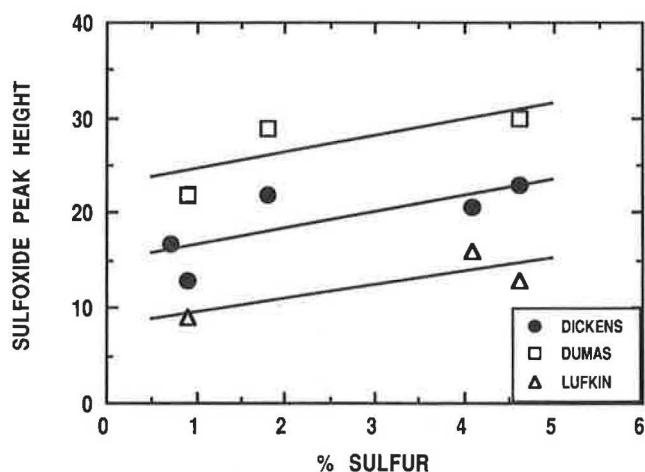
Effect of Sulfoxides

Surprisingly, the correlations so far have been made without reference to the height of the sulfoxide peak, which for some of the aged asphalts was higher than the carbonyl peak. Sulfoxide formation should be complicated by the wide variations encountered in sulfur content. Figure 11 shows sulfoxide peak height versus sulfur content for those asphalts for which data on sulfur content were available. Although a distinctly upward bias with increasing sulfur content was present overall, location had a much stronger effect. The Dickens sites had consistently higher carbonyl peaks and viscosities and generally lower penetrations, yet the Dumas asphalt had consistently higher sulfoxide peaks. These differences are even stronger when the asphalts from the same supplier are compared.

This reverse correlation between sulfoxide and carbonyl peak heights is shown in Figure 12. The scattered data at the bottom, represented by the lower curve, are from Lufkin, where both carbonyl and sulfoxide tended to be lower because of the small extent of aging. All of the other data fell reasonably well on the upper curve except for Highway 186 at MP 25, which showed a high value for both peaks. The asphalt extracted from this road fit the other correlation well, further giving evidence that sulfoxide formation has little effect on physical properties except as it affects carbonyl formation.

Apparently, there is competition between sulfoxide and carbonyl formation; because the former contributes little to hardening, it is desirable. The question remains why sulfoxide formed preferentially at Dumas. A possible answer is the aggregate, which at Dumas is an absorptive limestone and at Dickens is sandstone. Sulfoxide formation is reported to be base catalyzed (8).

Correlation with GPC

Figure 13 shows GPC results for Cosden AC–20 at all three locations. The progressive aging with time and the higher aging at Dickens and Dumas are clearly shown. Figure 14 shows a plot of the percentage of LMS versus carbonyl content. The correlation is not good, because the chromatograph
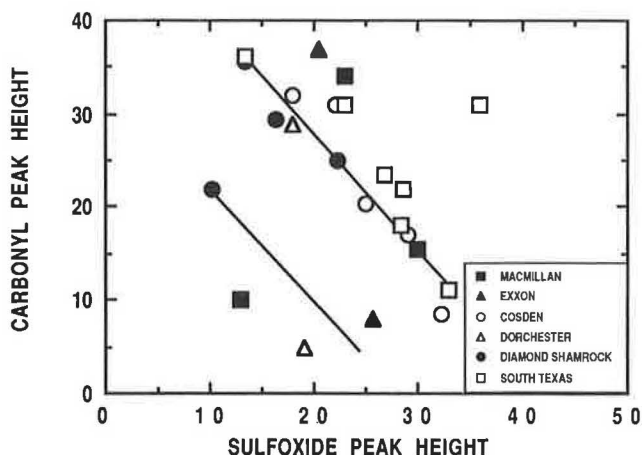

FIGURE 10   Viscosity at 140°F versus carbonyl peak height for asphalts from the south Texas highway cores.

FIGURE 11   Sulfoxide peak height versus sulfur content for asphalt from 1987 cores of the test sections.

FIGURE 12   Carbonyl FT-IR peak height versus sulfoxide peak height from 1987 cores of the test sections and from the south Texas highway cores.
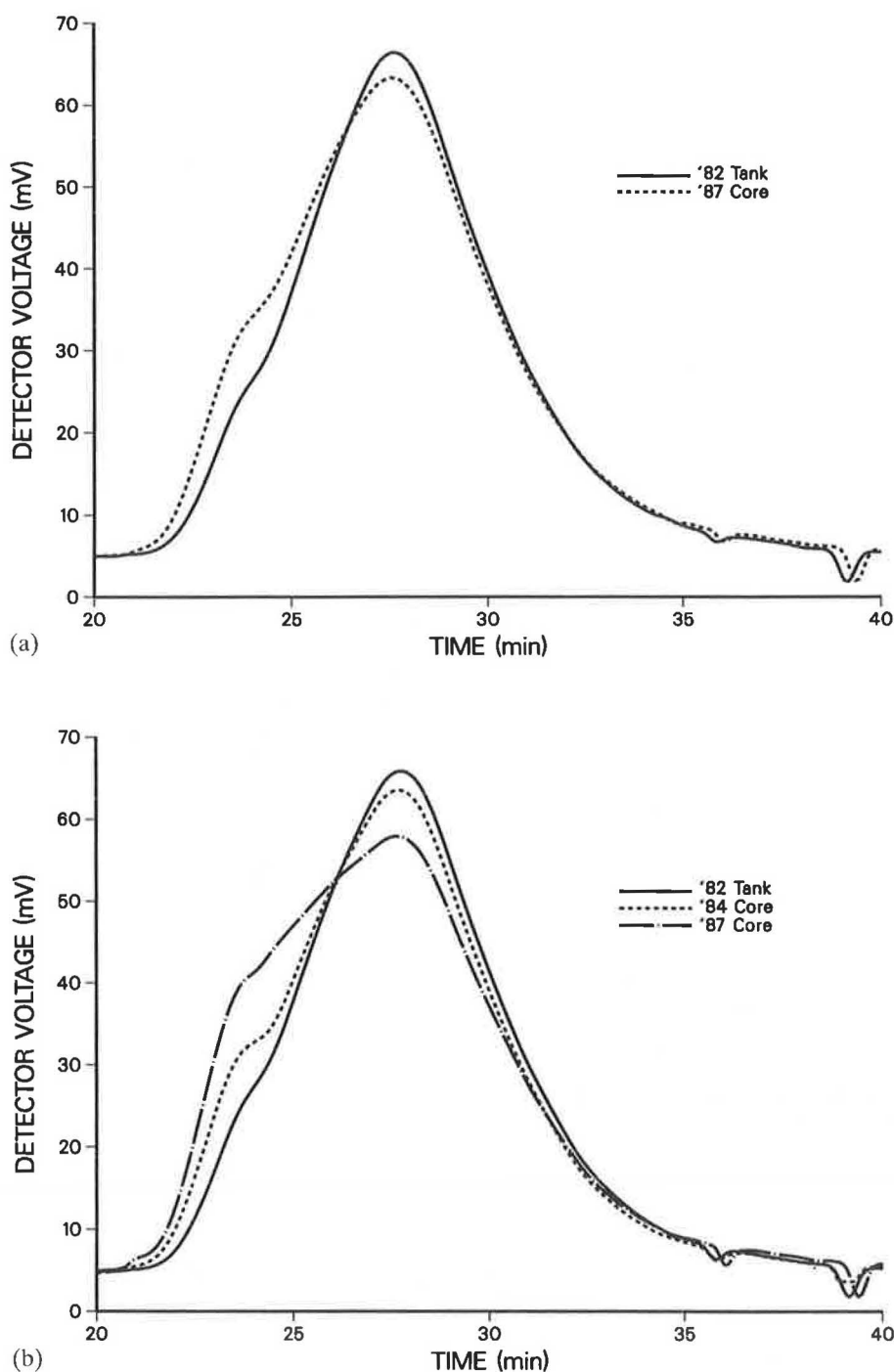
(a)

(b)

FIGURE 13 GPC chromatograms of the Cosden AC–20 asphalt as sampled before hot-mixing (tank) and as extracted and recovered from a, Lufkin, b, Dumas, and c, Dickens. (*continued on next page*)

of each asphalt source tends to have its distinctive shape, only crudely represented in LMS. The unique shape of Diamond Shamrock chromatographs is reflected in this graph, but the scatter is disappointing, as it is for several others.

The largest and most consistent sets of data are for Cosden and south Texas. Figure 15 is a plot of the percentage of LMS versus carbonyl peak height for south Texas; except for Highway 77 at MP 16, the correlation is excellent. Figure 16 shows

the GPC chromatograms for south Texas. The chromatogram for this asphalt is anomalous, having the lowest shoulder but not the lowest LMS value. In Figures 17 and 18, viscosity is plotted versus LMS percentage for the south Texas asphalts. Most of the points off the line are the same ones that did not correlate in the plot of carbonyl versus viscosity. In Figure 18, the highest and lowest points are for the same asphalts that are off the line in Figure 10. The third point represents
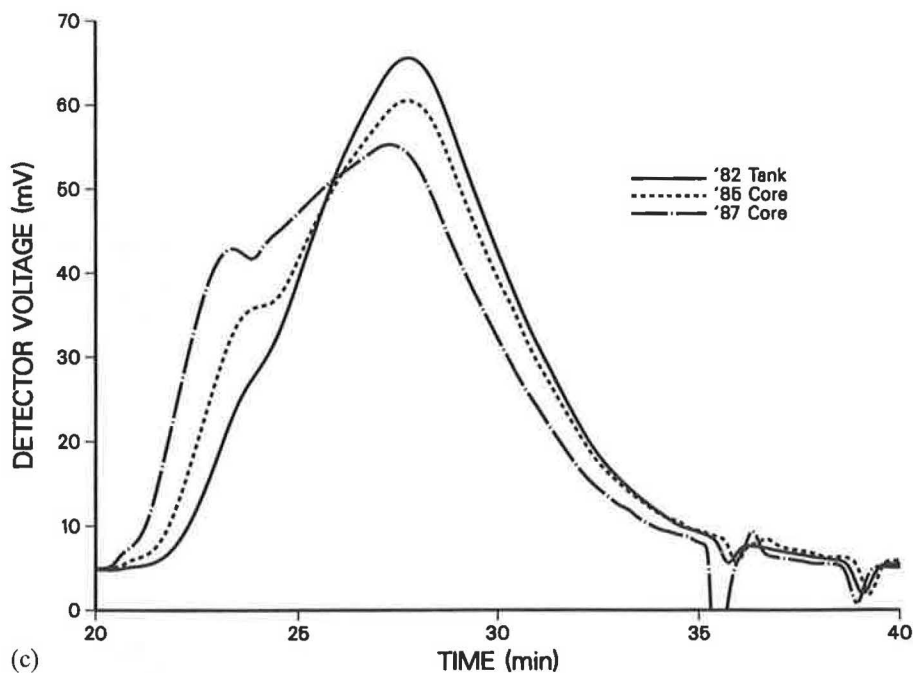
FIGURE 13 (*continued*)
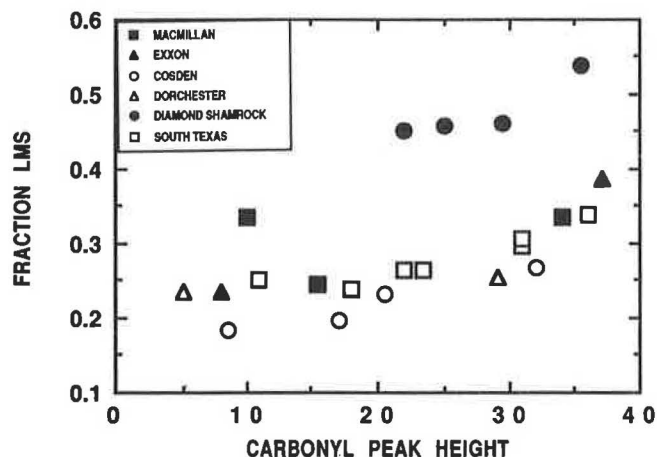
FIGURE 14 GPC chromatogram of LMS fractions versus carbonyl peak height from 1987 cores of the test sections and from the south Texas highway cores.
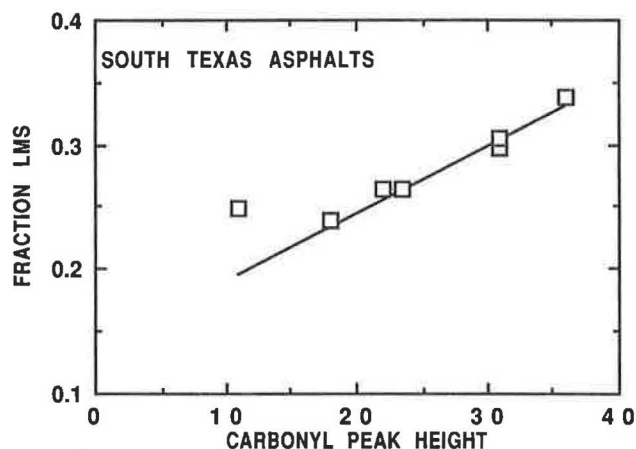

FIGURE 15 GPC chromatogram of LMS fractions versus carbonyl peak height for asphalts from the south Texas highway cores.

the asphalt that deviated from the line in Figure 15. The remaining four points form an almost perfectly straight line.

CONCLUSIONS

The extremely detrimental effect of high voids has been demonstrated again. This effect has been known for decades but seems to resist implementation. The progressive hardening of road asphalt with age will be impossible to predict apart from

knowledge of the percentage of voids and perhaps knowledge of the aggregate used. Sulfoxide formation may be desirable. In this event, high-sulfur asphalts and aggregates that promote the oxidation of sulfur would be preferred. Lower viscosity grades should be used where possible.

Carbonyl formation is an excellent measure of oxidative aging and correlates with changes in physical properties. If other properties, such as compatibility, were included, the correlations would probably improve. The use of GPC to measure asphalt aging is also very useful, but GPC results are
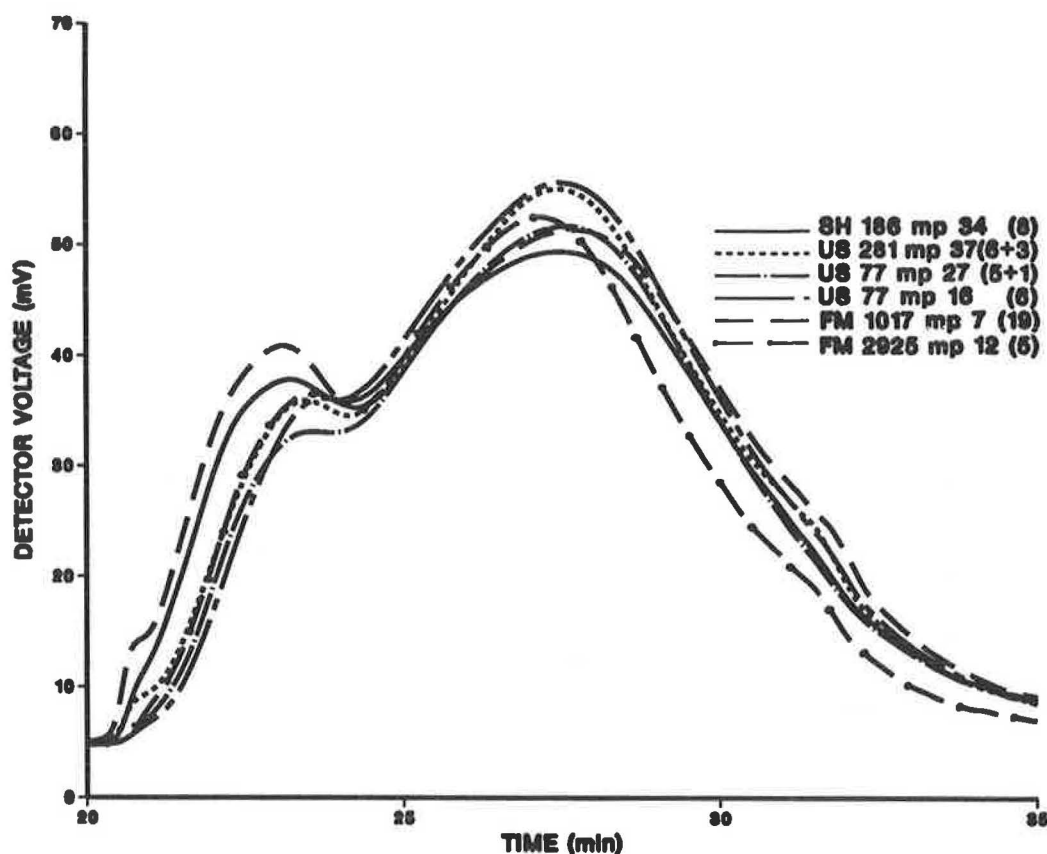
FIGURE 16   GPC chromatograms for asphalts from the south Texas highway cores.
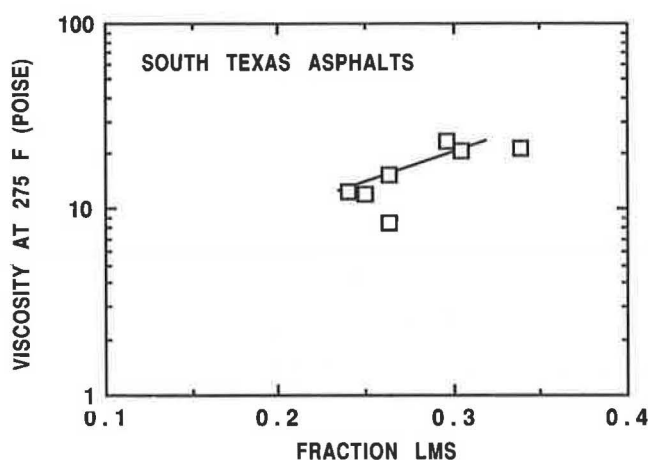

FIGURE 17   Viscosity at 275°F versus GPC LMS fractions for asphalts from the south Texas highway cores.

FIGURE 18   Viscosity at 140°F versus GPC LMS fractions for asphalts from the south Texas highway cores.

more difficult to correlate, because each asphalt yields a chromatogram with a distinctive shape that is dependent on the procedures used.

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