AN INVESTIGATION OF THE HARDENING OF ASPHALT RECOVERED FROM PAVEMENTS OF VARIOUS AGES

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ABSTRACT

The purpose of this study was to determine how the hardening of asphalt in actual pavement cores varied with depth below the pavement surface. Each core, 1-13 years old, was sliced in one-fourth inch layers parallel to the pavement surface, and the asphalt from each layer was recovered by the Abson Technique. The absolute viscosity was determined and related to depth, age of pavement, and original viscosity.

The results show that there is an increase of approximately 50 per cent in the viscosity of asphalt recovered from the top one-fourth inch over asphalt recovered from the next lower slice. There is a very thin film at the pavement surface which has a higher viscosity than the average viscosity in the top one-fourth inch layer.

Increases in viscosity with age are more apparent in the top one-half inch than at lower depths; there is only a small change in viscosity at lower depths, except for an initial increase occurring before or during placing.

In the upper one-half inch, asphalts with lower viscosities were found to increase in viscosity more rapidly than asphalts with high original viscosities.

INTRODUCTION

In the study of asphalt durability there have been many studies of the causes and effect of hardening. However, few studies have dealt with the aging of thin asphalt layers parallel to the surface of the pavement.

Pauls and Halstead (1) found that after a 19 year period there was a considerable variation in the hardening of asphalt within the same core. Asphalt near the surface hardened to a greater extent and lost a larger percentage of ductility than did the material in the center or bottom of the pavement. Both top and center samples showed more hardening than did the bottom. Very little change was indicated in the penetration of asphalt in the bottom quarter inch of the 19 year old sample as compared to a sample extracted immediately after mixing. Only one sample was reported, making definite conclusions impossible.

Simpson, Griffin, and Miles (2) found that, in general, the asphalt in the top one-fourth inch of pavement has a higher viscosity than the rest of the pavement, including both the surface and base. They used the microviscometer to study 32 and 35 month old cores taken from the Zaca-Wigmore Experimental Road in California. At depths lower than one-half inch, within the surface course, there was less change than in

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the top half inch. Higher viscosities were sometimes encountered in the top of the base course indicating that appreciable hardening occurred between the laying of base and surface courses. Also, they found a general agreement between air void content of the compacted mix and viscosity: cores with higher air void content had higher viscosities at equal depths.

Numerous studies have been made concerning asphalt aging. Neppe (3) and the Highway Research Board (4) have published bibliographies which together list about 300 publications. Only a few of the more important can be listed here.

Hubbard and Reeve (5) made what was probably the first study of aging in 1913 by exposing thin films of asphalt for one year. They observed a 75 per cent decrease in penetration in this period. Traxler and Schweyer (6) in 1936 established the first conclusive evidence that viscosity increased with time, temperature held constant.

Brannon (7) studied actual pavements in 1937 and found that decrease in penetration varied from 22 per cent at 4 months to 47 per cent at 30 months. As much as 24 per cent was found to occur before or during placement. Identical asphalts decreased in penetration by different amounts when placed in different areas.

White (8) found that asphalt content is the greatest single factor determining the amount of drop in penetration of asphalt with service. Asphalt in cutback asphalt wearing surfaces hardens in about six years to the point of incipient raveling. Pauls and Welborn (9) concluded that an increase in hardening as indicated by decrease in penetration is accompanied by changes in other properties, such as an increase in softening point and a decrease in ductility.

Brown, Sparks, and Larsen (10) developed an equation to relate early changes in ductility, softening point, or penetration to ultimate pavement performance:

$$T/\Delta Y = a + bT$$

where T is time since paving (yr), Y is softening point, ductility, or penetration, and a and b are empirically determined constants. When Y is plotted as a function of T the hyperbolic form appears with asymptotic values reached at about nine to ten years.

Parr and Serafin (11) found that penetration of asphalt extracted from cores decreased up to 17 months and then remained relatively constant or increased slightly at ages of 29, 40, and 52 months. Penetration was found to be greater under the center of the traffic lane than under the wheel paths. Simpson, Griffin, and Miles (12) substantiated this finding by noting that most hardening occurred in the first 16 to 20 months and then decreased considerably.

Skidmore and Abson (13) studied hardening during mixing. They pointed out that even under good control during manufacture, a 10 to 20 point decrease in penetration may be expected. Lang and Thomas (14) reported an average of 9.1 per cent loss in penetration of 50-60 penetration asphalts immediately after mixing.

Bissett (15) recovered asphalt from pavements and tested them for ductility, penetration, and softening point. He found a 28 per cent decrease in penetration during mixing and placing. He concluded that early loss of penetration and ductility, primarily ductility, causes cracking of the pavement surface.

The purpose of this research was to obtain a precise measure of asphalt viscosity taken from various layers in existing pavement surfaces and by studying pavement cores of various ages to measure the magnitude of asphalt hardening that occurs within these layers.

PROCEDURE

The asphalt samples used in this project were obtained from existing pavements in Georgia through the cooperation of the Georgia State Highway Department. In accordance with the objectives of the project, asphalt samples were obtained by extraction and recovery from cores taken from existing pavements. These cores are well representative of ages from 4 months to 12 years. A summary of projects and asphalt data appears in Table I. All cores were obtained from locations near Atlanta, Georgia, with the exception of numbers one and two, which came from southern Georgia. To the authors' knowledge no special conditions were encountered during placing or service life which would effect the results.

Two six-inch diameter cores were obtained from each project. These cores were transported to the laboratory, placed in water, and

Table I. Sample Description

Sample Number	Project	Location	Date Placed	Age (mo)	Asphalt Grade	Specific Gravity	Original Penetration	Original Viscosity* (megapoises)
1	F-007-2(N) ct 1	Thomasville-Quitman Rd Brooks Co., Ga.	Mar. 1965	4	AC-6	1.028	67	2.60*
2	PR-1421-B(1)	Excelsior Rd Tift Co., Ga.	Jan. 1965	6	AC-6	1.026	66	2.69*
3	RA 15-8(5)	Decatur-Tucker Rd DeKalb Co., Ga.	July 1964	9	AC-6	1.032	64	2.82*
4	I-20-1(4) ct 1	I-20 West Fulton Co., Ga.	Sept. 1964	10	AC-6	1.029	71	2.36
5		Ga. Tech Electrical Engr. Parking Lot	Sept. 1964	10	AC-6	1.038	69	2.43
6	I-20-1(4) ct 1	I-20 West Fulton Co., Ga.	Sept. 1964	10	AC-6	1.046	66	2.72
7	F-074-2(4)	W. Fayetteville Rd College Park, Ga.	Aug. 1964	11	AC-6	1.029	79	1.94
8	US-1394(2)	N. Druid Hills Rd Atlanta, Ga.	July 1963	24	AC-6	1.041	63	2.90*
9	SAP 719A(9)	College Ave. Atlanta, Ga.	June 1961	47	AC-8	1.040	95	1.43*
10	DS-0696(2)	Lawrenceville-Duluth DeKalb Co., Ga.	Aug. 1959	70	AC-8	1.026	94	1.46*
11	F-074-2(4)	S.R. 85 North of Fayetteville, Ga.	June 1956	109	AC-8	1.029	96	1.41*
12	SAP 719A(5)	East Lake Dr Atlanta, Ga.	Oct.-Nov. 1954	126	AC-8	1.032	93	1.48*
13	SAP 1083 c(1)	Atlanta-Buford Rd DeKalb Co., Ga.	Feb. 1954	135	AC-8	1.032	94	1.46*
14	SAP 1214 B(3)	Stone Mt.-Tucker Rd DeKalb Co., Ga.	Oct. 1952	151	AC-8	1.042	90	1.57*

*Original viscosity obtained from empirical equation: Abs. Visc. = $3591.3(\text{Pen})^{-1.719}$
See Reference 16

covered to prevent further aging by air or light. Balls of steel wool were placed in the water to lessen the possibility of oxidation by dissolved oxygen in the water.

In order, each core was removed from water and the surface course sliced into one-quarter inch slices with a diamond blade saw. Care was taken not to include the binder or underlying layers. The top layer was placed in the oven while the other slices were returned to the water for later use.

After drying for 20 minutes at 250 F., the sample was broken into small pieces and returned to the oven for ten minutes. This softened the mixture and permitted placement in the extraction bowl. An analytical reagent grade benzene was used to extract the asphalt. A total of 240 ml. of benzene was used in three steps on the approximately 275 gram sample. The asphalt-benzene solution was then centrifuged at 770 times gravity for 30 minutes to remove dust and other suspended matter.

The Abson Technique, ASTM D-1856-63, was used with slight modification to recover the asphalt. This modification was the use of a 275 gram sample since it was not possible to obtain a 1200 gram sample from the one-quarter inch slice. This necessitated extreme care in the distillation to prevent oxidation or overheating.

A study was made to determine the influence of sample size and of operator on viscosity. The per cent difference between the viscosities determined from a 1200 gram sample and a 275 gram sample was only about six per cent. The difference between operators was about eight per cent. These were considered to be within satisfactory limits and not to adversely affect results.

In accordance with standard procedure, all extraction distillations were completed in less than eight hours. The redovered asphalt samples were placed in sealed tin boxes until they were tested.

Viscosity testing was performed with the sliding plate microviscometer developed by Shell Development Company. Absolute viscosities were determined in accordance with procedures proposed by Griffin, et al. (17) and are reported at a shear rate of 0.05 reciprocal seconds at 77 degrees F.

DISCUSSION OF RESULTS

The results of the 12 asphalt viscosity determinations are shown in Table II. The one-fourth inch layers are referred to as layers A, B, C, D, and E in succession beginning at the pavement surface proceeding downward. Since the diamond blade used in cutting the slices was one-eighth inch thick, the depths of each slice are not multiples of one-fourth inch. The actual depths are as shown in the tabulation on the next page.

It will be noted that samples 1 through 8 are 60-70 penetration grade and samples 9-14 are 85-100 penetration grade asphalt cement. Due to a general change from AC-8 to AC-6 asphalt by the Georgia Highway

Layer	Actual Depth
A	0-1/4"
B	3/8-5/8"
C	3/4-1"
D	1-1/8-1-3/8"
E	1-1/2-1-3/4"

Department in 1963, it was not possible to obtain recent samples of AC-8 asphalt. For this reason the viscosity data for the AC-6 and AC-8 samples will be analyzed separately as well as a unit. Average viscosity results are shown for all 14 samples and for the AC-6 and AC-8 samples respectively.

Viscosity versus Depth

Figure 1 shows the average viscosity of all 14 samples at each layer. In general, it is apparent that the greatest change in viscosity occurs in the top half-inch. The average viscosity in layer A is 23.4 megapoises and the average in layer B is 15.3 megapoises. This shows the average viscosity of layer A is about 50 per cent greater than layer B. The differences between layers B and C, C and D, and D and E, respectively are 11 per cent, 7 per cent and 1 per cent.

To better interpret these results a computer program was used to make an analysis of variance on the relative viscosities. Relative viscosity is defined as the ratio of the viscosity of the hardened sample divided by the original viscosity. It is a measure of the hardening of

Table II. Absolute Viscosity Results*

Sample Number	1	2	3	4	5	6	7	8	Ave. AC-6	9	10	11	12	13	14	Ave. AC-8	Ave. of 5**	Overall Ave.
Age (mo)	4	6	9	10	10	10	11	24		47	70	109	126	135	151			
Layer A	22.8	24.4	6.4	22.8	17.5	22.4	19.8	21.4		20.4	20.3	31.8	22.1	11.4	56.0			
	23.2	24.4	6.6	24.4	18.4	23.1	21.5	20.9		21.4	24.1	29.5	22.8	11.2	58.5			
									20.1							27.7	22.6	23.4
Layer B	14.7	13.6	23.0	13.3	13.2	13.9	14.2	12.0		7.7	17.5	11.9	14.1	12.6	30.0			
	14.9	13.2	23.9	13.1	11.7	13.4	15.1	13.1		7.4	18.6	12.6	15.2	12.3	30.6			
									14.8							15.9	13.3	15.3
Layer C	14.9	14.4	25.3	13.3	10.6	13.7	12.7	10.7		5.7	15.1	9.2	14.4	10.9	-			
	15.7	14.6	25.6	14.3	11.1	13.9	13.1	9.9		5.9	16.1	9.7	13.9	11.3	-			
									14.6							11.2	12.8	13.3
Layer D	14.8	14.4	23.6	12.5	-	12.5	12.2	11.7		3.4	13.3	7.3	-	9.6	-			
	15.3	14.5	24.7	12.5	-	12.7	14.0	10.9		3.3	13.7	7.1	-	9.8	-			
									14.7							8.4	11.8	12.4
Layer E	12.5	12.8	20.6	-	-	13.3	-	-		-	-	6.6	-	6.9	-			
	12.6	13.5	21.8	-	-	13.5	-	-		-	-	6.1	-	7.2	-			
									15.1							6.8	10.6	12.3
Ave.	16.1	15.9	20.2	15.8	13.8	15.4	15.3	13.8		9.5	11.7	13.2	17.1	10.3	43.8			

*All values megapoises

**Average of samples 1, 2, 6, 11, and 13.

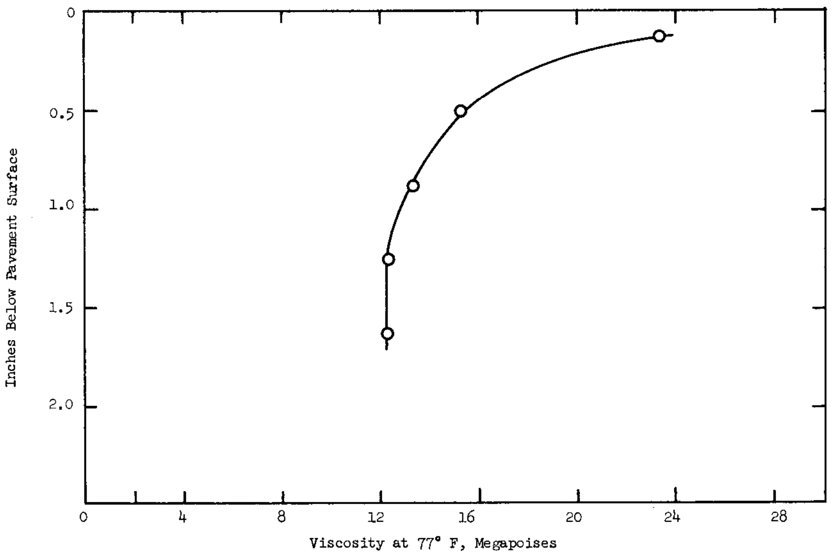

Fig. 1. Average Viscosity of Samples 1-14 versus Depth.

the asphalt. A high value of relative viscosity indicates much hardening and a low value indicates little hardening and, thus, probable superior pavement durability in service. Relative viscosity was used rather than absolute viscosity to remove the effect of initial viscosity so that only change in viscosity would be tested. Table III shows the original viscosity of each layer and the relative viscosity of each extracted sample.

The analysis indicated a significant effect at the 0.1 per cent level due to age. It also showed that there was a significant effect at the 0.1 per cent level due to depth.

To further interpret this data, Duncan's Multiple Range Test was used to indicate which layers were the sources of the difference with depth. It showed a significant difference between layers A and B at the 1.0 per cent level and between layers B and C at the 5.0 per cent level. No difference was found at the 5.0 per cent level among the three lower layers.

The previous paragraphs clearly indicate that the major increase in viscosity is in layer A. The question now arises as to how the viscosity varies within layer A itself. To answer this question three sample numbers, 8, 10 and 12, were selected for further study. Taking the duplicate cores of these samples, the top 1/16 inch was shaved off with the saw. A one-fourth inch layer (actual depth 1/16-5/16 inch and designated "A_s") was then sliced and the viscosity determined. The results are shown in Table IV.

Table III. Relative Viscosity Results

Sample Number	1	2	3	4	5	6	7	8	Ave. AC-6	9	10	11	12	13	14	Ave. AC-8	Ave. of 5*	Overall Ave.
Age(mo)	4	6	9	10	10	10	11	24		47	70	109	126	135	151			
Original Viscosity (Mega-poises)	2.60	2.69	2.82	2.36	2.43	2.72	1.94	2.90		1.43	1.46	1.41	1.48	1.46	1.57			
Layer A	8.8	9.1	2.3	9.7	7.2	9.0	10.2	7.4		14.3	15.8	22.6	14.9	7.7	35.7			
	8.9	9.1	2.3	10.3	7.6	8.5	11.1	7.2		15.1	16.5	20.9	15.4	7.8	37.3			
									8.0							18.7	11.2	12.6
Layer B	5.7	5.1	8.2	5.6	5.4	5.1	7.3	4.1		5.4	12.2	8.4	9.5	8.6	19.1			
	5.7	4.9	8.4	5.5	4.8	4.9	7.8	4.5		5.2	12.5	8.9	10.3	8.4	19.5			
									5.9							10.7	6.6	7.9
Layer C	5.7	5.4	9.0	5.6	4.4	5.0	6.6	3.7		4.1	10.6	6.5	9.7	7.5	-			
	6.0	5.4	9.0	6.1	4.5	5.1	6.8	3.4		4.2	10.9	6.9	9.4	7.7	-			
									5.7							7.7	6.1	6.5
Layer D	5.7	5.4	8.4	5.3	-	4.6	6.3	4.0		2.4	9.3	5.2	-	6.6	-			
	5.9	5.4	8.7	5.3	-	4.7	7.2	3.8		2.3	9.3	5.1	-	7.0	-			
									5.8							5.9	5.5	5.8
Layer E	4.8	4.8	7.3	-	-	4.9	-	-		-	-	4.7	-	4.9	-			
	4.9	5.0	7.7	-	-	5.0	-	-		-	-	4.7	-	4.9	-			
									5.5							4.8	4.9	5.3
Ave.	6.2	5.9	7.1	6.7	5.7	6.7	7.9	4.8		6.6	12.1	9.4	11.5	7.1	27.9			

*Average of samples 1, 2, 6, 11 and 13.

The average viscosity of these three A_s samples was 16.6 megapoises, whereas the average in layer A of the three original cores was 22.4 and in layer B was 15.1. This represents a 25.8 per cent decrease in viscosity from layer A upon the removal of the top one-sixteenth inch. However, it is only ten per cent greater than the original viscosity of layer B. A straight line interpolation between the original viscosities at the average depth of layer A, 0.125 inch, and the average depth of layer B, 0.50 inch, would give an expected viscosity of 21.0 megapoises at the average depth of A_s, 0.1875 inch. See Figure 2. Since the recorded viscosity is only 79 per cent of this value, it is apparent that most of the hardening occurs in a very thin layer (less than 0.1875 inch) immediately below the pavement surface.

Figures 3 and 4 show the average viscosity as a function of depth for the AC-8 and AC-6 samples, respectively. It should be noted that

Table IV. Viscosity in Top One-Half Inch

Layer	Average Depth (inch)	Sample Number 8	10	12	Average
A	0.125	21.4 20.9	23.0 24.1	22.1 22.8	22.4
A_s	0.188	14.2 13.5	14.3 15.9	17.0 16.5	16.6
B	0.500	12.0 13.1	17.5 18.6	14.1 15.2	15.1

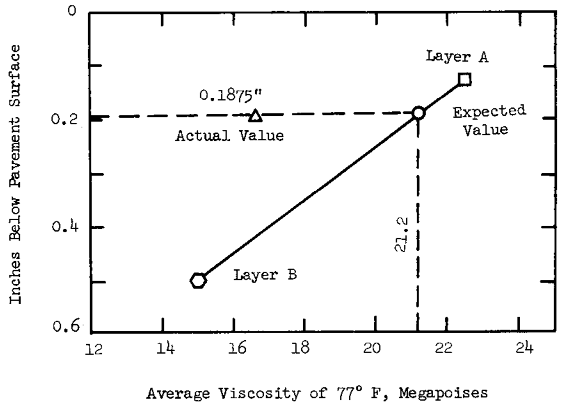

Fig. 2. Average Viscosity versus Depth in Top One-Half Inch.

the AC-8 samples are all 47 months or more old, and the AC-6 are all 24 months or less old.

For the AC-8 samples the curve appears very similar to Figure 1. The increase in layer A, 27.7 megapoises, over layer B, 15.9 megapoises, was about 75 per cent. Per cent differences of 42, 33, and 24 were found between layers B and C, C and D, and D and E, respectively. In the analysis of variance, a significant difference was found between

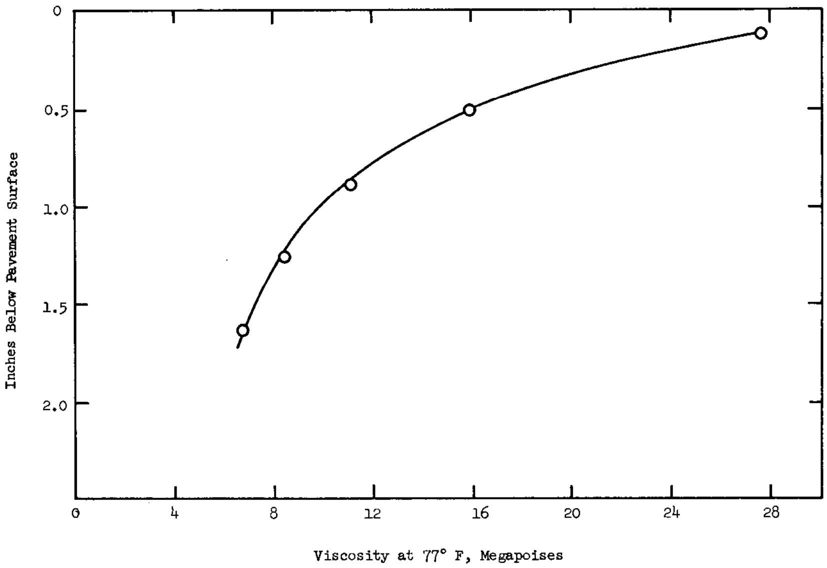

Fig. 3. Average Viscosity of AC-8 Samples versus Depth.

ages and also between depths at the 0.1 per cent level. Duncan's Multiple Range Test showed that each layer was significantly different from all other levels at the 1.0 per cent level.

The curve of the AC-6 samples shows that the viscosity in the top quarter inch is about 36 per cent greater than that in layer B. But the difference in viscosity of layers B through E is less than three per cent in all cases. In the analysis of variance, the relative viscosities at different depths and different ages were found to be significantly different at the 0.1 per cent level. Duncan's Multiple Range Test showed a significant difference between layers A and B at the 1.0 per cent level but no significant difference between any other two consecutive layers at the 5.0 per cent level.

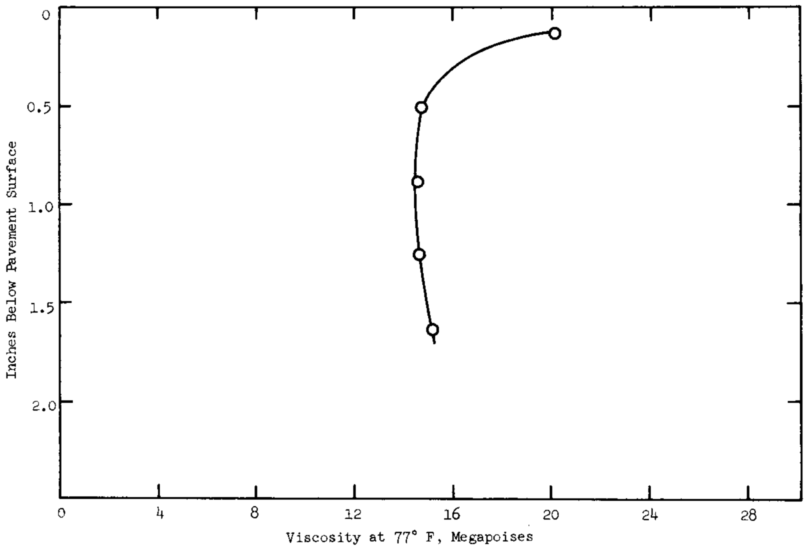

Fig. 4. Average Viscosity of AC-6 Samples versus Depth.

Considering Figures 1, 3, and 4 together it appears that as a unit there is a higher viscosity in the top quarter inch than at greater depths below the pavement surface. As depth becomes greater the per cent difference between adjacent layers is less. The older AC-8 samples show essentially the same results but the AC-6 samples by themselves indicate that the greatest change in viscosity occurs only in the top quarter inch and that in the first two years there is no appreciable change in viscosity with depth except in layer A. The viscosity increase in the lower layers is approximately uniform indicating that it probably occurred during mixing, transporting, or placing.

From Table II it can be seen that all samples did not have the full depth of five layers; consequently any individual value has more relative

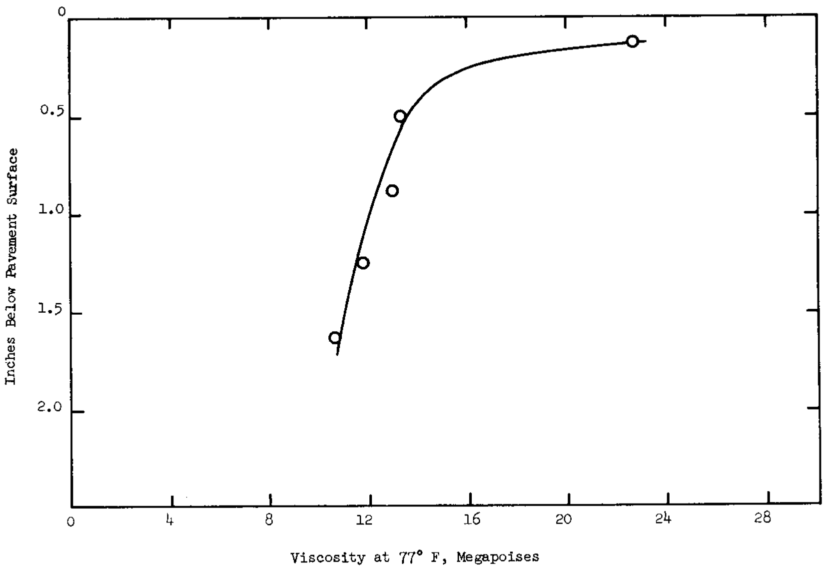

Fig. 5. Average Viscosity of Samples 1, 2, 6, 11 and 13 versus Depth.

weight on the average than a value at a higher layer of the same sample. For this reason samples 1, 2, 6, 11, and 13, each having five layers, were analyzed separately. Sample number 3 was not included because of the low result found in layer A. A graph of average viscosity versus depth is shown as Figure 5. It shows a 70 per cent increase in viscosity of layer A over layer B. The difference between other layers is from four to eleven per cent.

An analysis of variance was run on the results of the five selected samples. A significant difference was found between the relative viscosities at the 0.1 per cent level with both age and depth. Duncan's Multiple Range Test showed a significant difference between each pair of adjacent layers at the 1.0 per cent level.

The graphs of viscosity versus depth of each individual sample were plotted but are not included in this paper. Except for samples 3 and 13 an appreciable increase was seen in viscosity in layer A over layer B. Duplicate recoveries were performed on these samples, and the results were within 14 per cent for sample 3 and two per cent for sample 13 of the original tests. The most apparent reason for the viscosity of layer A to be less than layer B is the possibility of a fog seal or emulsion slurry seal being used to rejuvenate the pavement. This is quite possible for sample 13, as it is over 12 years old. However, there is no record of this having been done in either case.

Viscosity versus Age

Figure 6 shows a composite graph of the relative viscosity as a function of age for each layer. In general, it shows that relative viscosity increases with age, except for layer E. The rate of change is more rapid in the top layer and decreased with increasing depths. Layer D is of interest in that there is not appreciable change in viscosity with time except for an initial increase. The initial increase probably occurred before or during placing and the later oxidation did not penetrate to this depth in the pavement. (The original data points to which the lines in Figure 6 were fitted are shown in Figures A-1 through A-5, Appendix.)

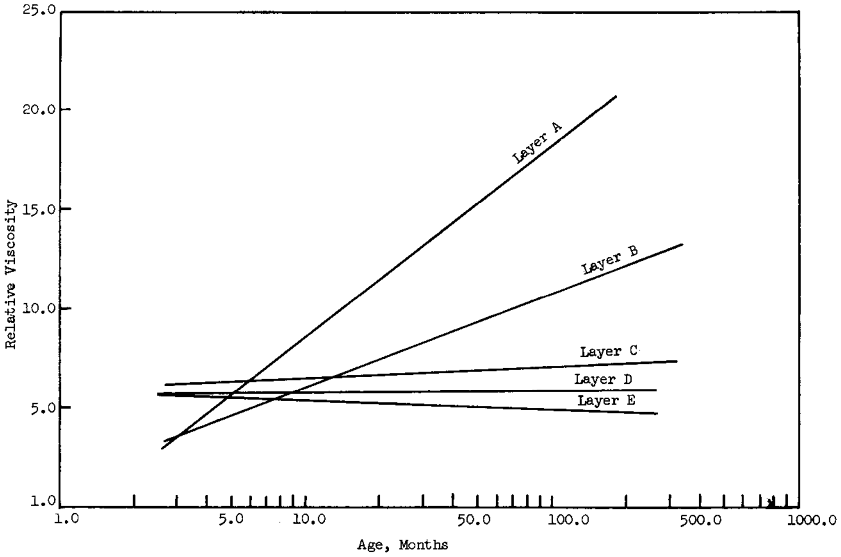

Fig. 6. Relationship between Relative Viscosity and Age.

Relative Viscosity versus Original Viscosity

The question logically arises as to what Figure 6 means with respect to original viscosity. It will be remembered that all samples 47 months or older are AC-8; those less than 47 months are AC-6. There is a difference between the original average viscosities of the two series. In this case, the difference is 1.08 megapoises. This suggests the possibility that the change in relative viscosity with age could be a function of original viscosity instead.

To evaluate this possibility a composite graph of relative viscosity versus original viscosity for each layer is shown in Figure 7. (The original data points for these lines may be seen in Figures A-6 through A-10, Appendix.) It shows that for layers A and B, and to some extent

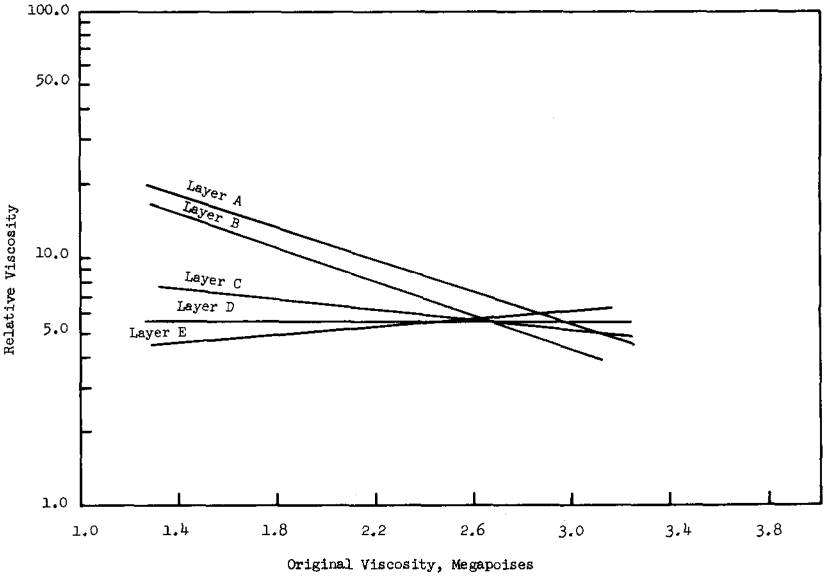

Fig. 7. Relationship between Relative Viscosity and Original Viscosity.

C, a high initial viscosity will result in less change than a low initial viscosity. Again, layer D is of special interest, showing that relative viscosity, essentially, is independent of original viscosity at this depth. Regardless of the grade and original viscosity, the relative change at this depth will be the same.

Returning to Figure 6, one would conclude from the previous paragraph that for layer D at least, the relationship is true and independent or original viscosity versus age, there was no significant difference at the 5.0 per cent level between layers C, D, and E. Thus the relative viscosity versus original viscosity relationships for layers C and E in Figure 7 may also be considered true.

For layers A and B no conclusive statement may be made as a result of Figures 6 and 7. Relative viscosity is influenced by both age and original viscosity, but the relative importance of each may not be determined. It can be stated, however, that viscosity does increase with age. But how this increase is influenced by original viscosity was not determined.

CONCLUSIONS

The results of this study indicate the following conclusions for the asphalt pavements studied:

1. There is about 50 per cent increase in the viscosity of asphalt extracted from the top quarter inch of a pavement over asphalt extracted from depths to one-half inch.

2. Within the top quarter-inch layer there is a greater viscosity immediately under the surface than lower in the layer.

3. Viscosity in the upper one-half inch increases with age while little change with age occurs at greater depths.

4. At a depth of about one and one-half inches below the pavement surface, there is little change in viscosity with age except for an initial increase during or before placing.

5. At a depth of about one and one-half inches below the pavement surface, the relative viscosity is independent of original viscosity.

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APPENDIX

Figures A-1 through A-10

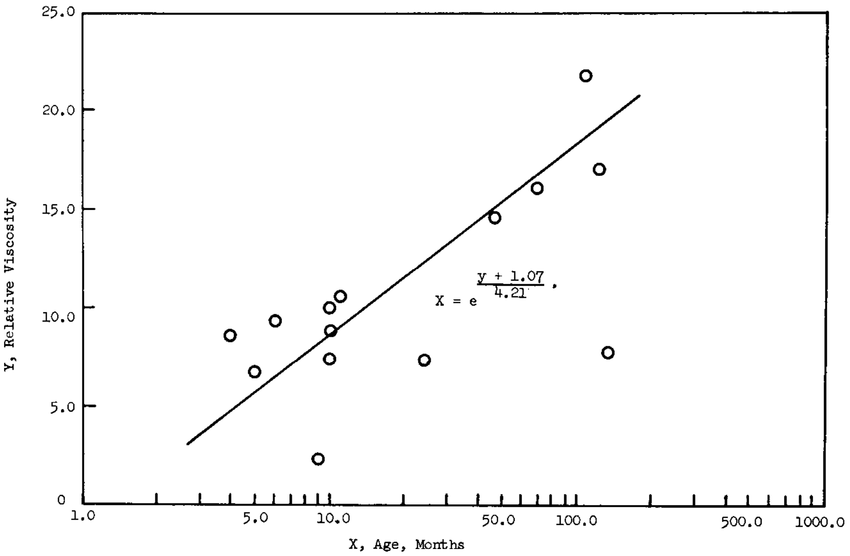

Fig. A-1. Relative Viscosity in Layer A versus Age.

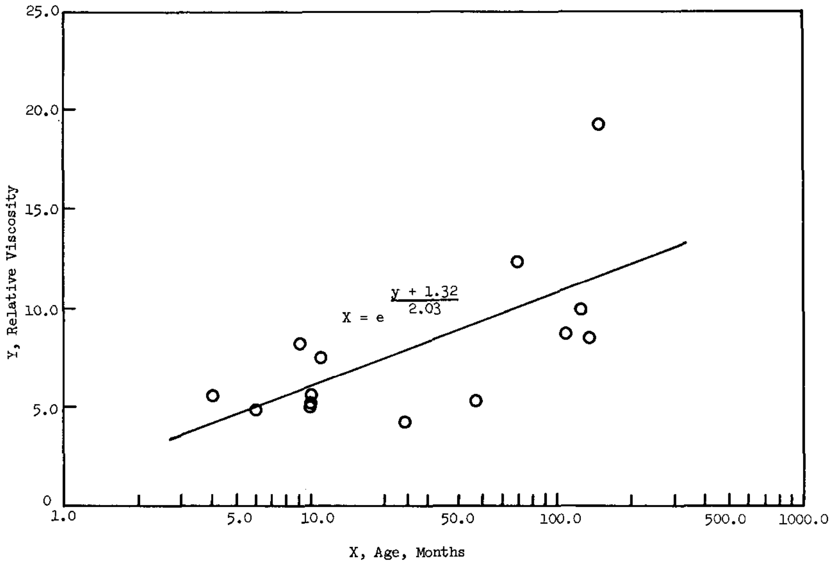

Fig. A-2. Relative Viscosity in Layer B versus Age.

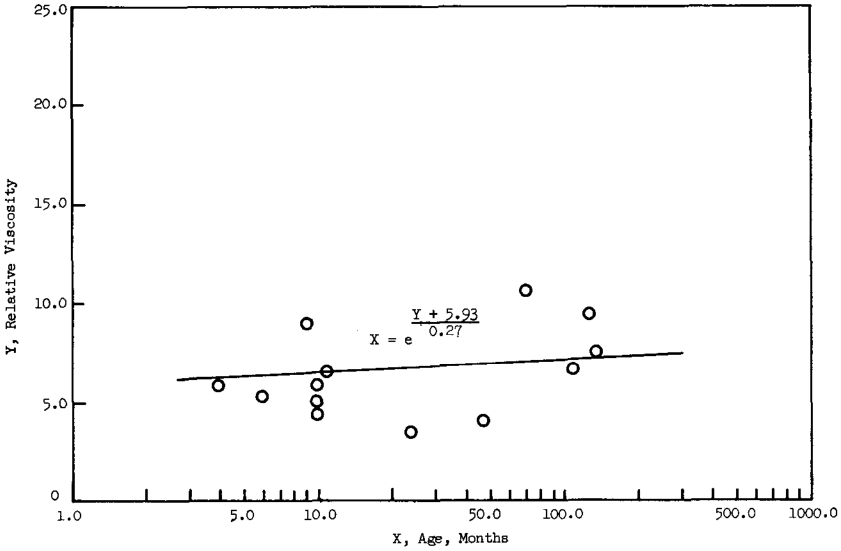

Fig. A-3. Relative Viscosity in Layer C versus Age.

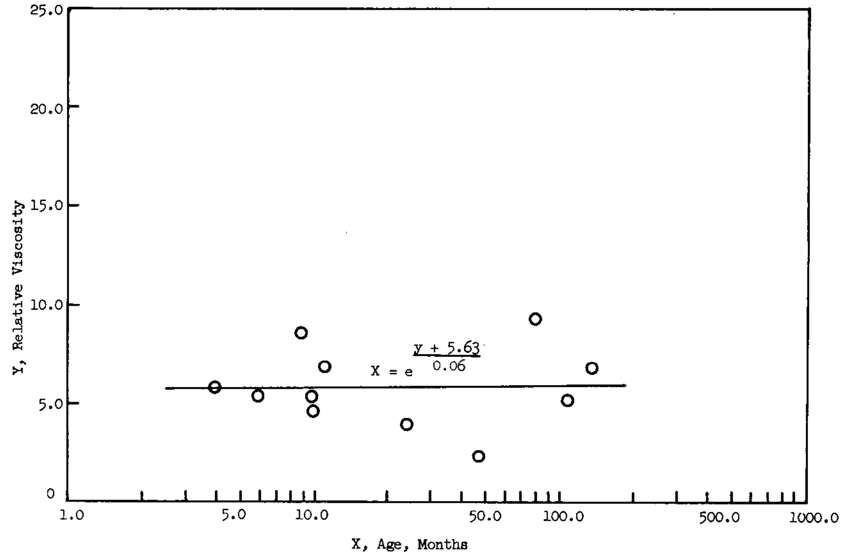

Fig. A-4. Relative Viscosity in Layer D versus Age.

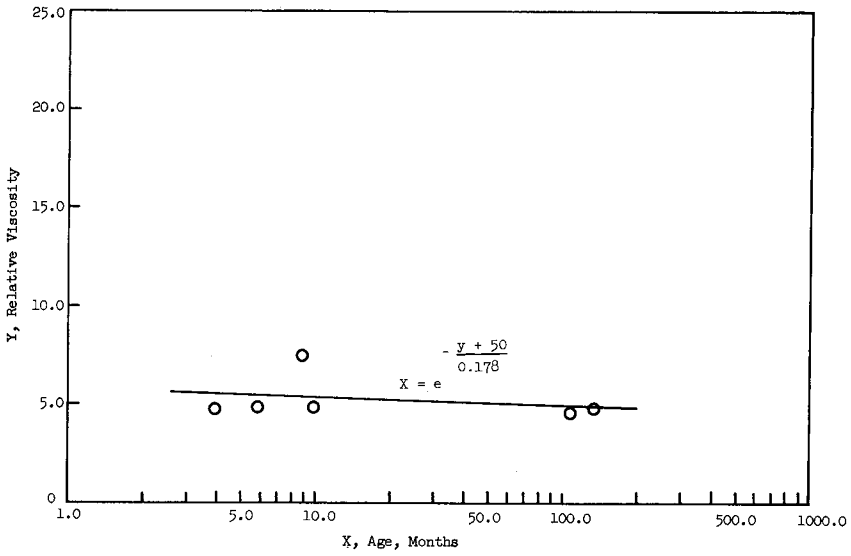

Fig. A-5. Relative Viscosity in Layer E versus Age.

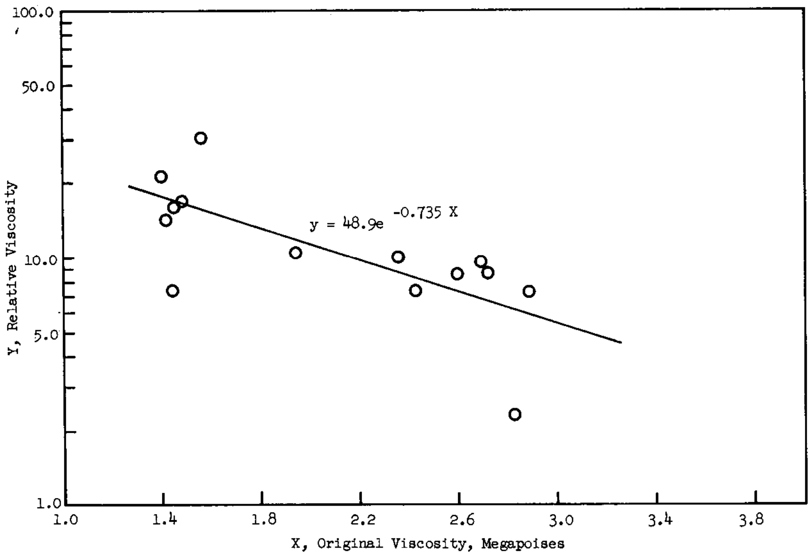

Fig. A-6. Original Viscosity versus Relative Viscosity for Layer A.

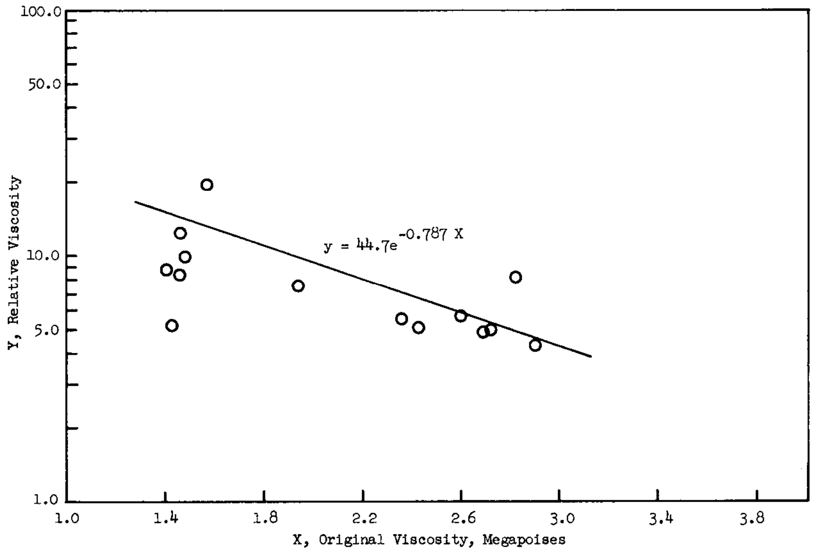

Fig. A-7. Original Viscosity versus Relative Viscosity for Layer B.

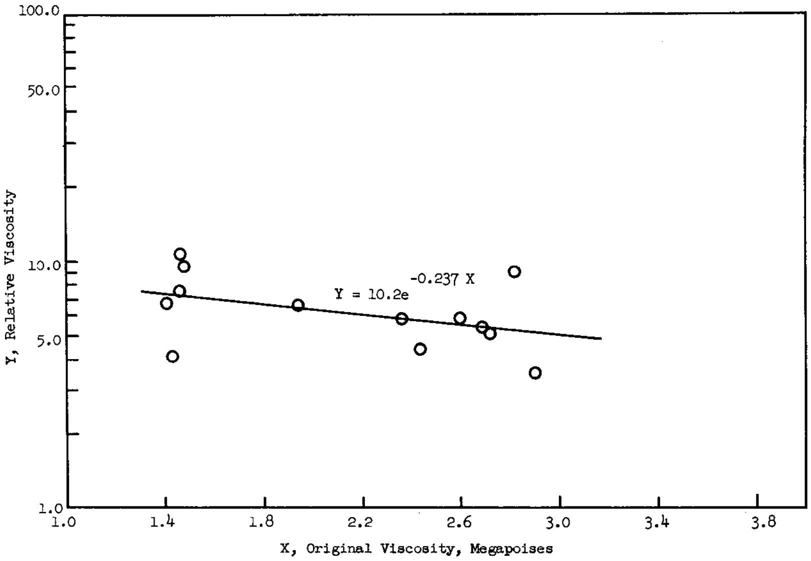

Fig. A-8. Original Viscosity versus Relative Viscosity for Layer C.

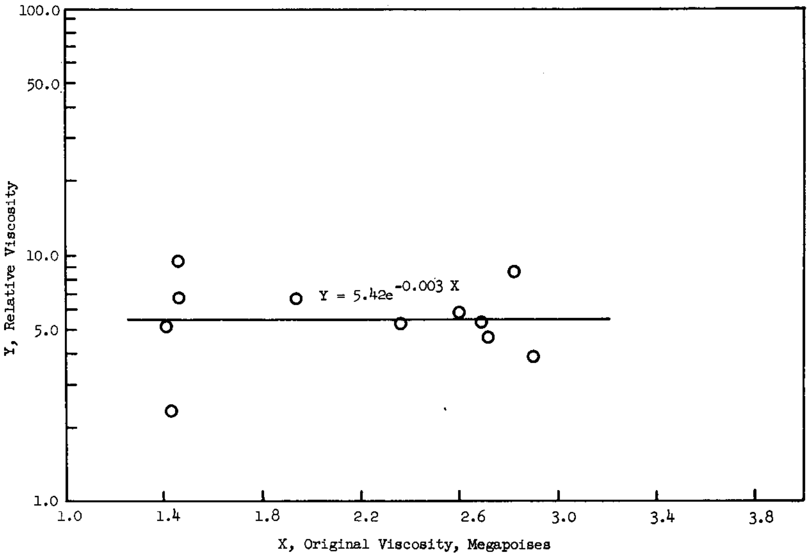

Fig. A-9. Original Viscosity versus Relative Viscosity for Layer D.

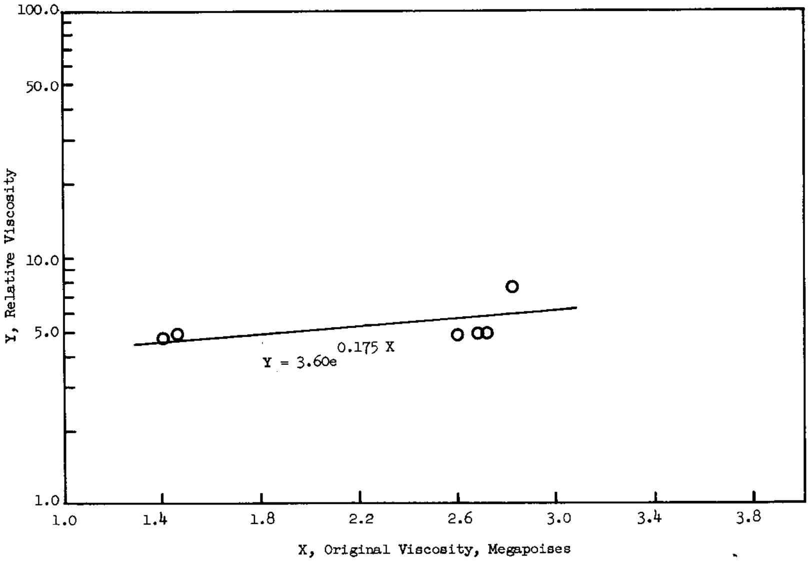

Fig. A-10. Original Viscosity versus Relative Viscosity for Layer E.