Effects of Mineral Additives on the Durability of Coating-Grade Roofing Asphalts

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[List continued on cover page III]

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Effects of Mineral Additives on the Durability of Coating-Grade Roofing Asphalts

Sidney H. Greenfeld

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Sidney H. Greenfeld*

The effects of 14 mineral additives on the durability of coatings made from three asphalts were evaluated in accelerated durability machines. It was found that while the durability of the coating is largely a function of the asphalt used, it increases, generally, with coating thickness and mineral additive concentration. Additives with flat, platelike particles finer than 75 microns in diameter (U. S. Standard Sieve No. 200) were most effective in producing coatings of increased durability. Complete dispersion of the additives in the base asphalts is necessary to produce consistent results.

1. Introduction

In the late 1920's and early 1930's, the asphalt-roofing industry began to use mineral additives in the asphalt coatings of roll roofing and shingles in order to stabilize these coatings, i. e., prevent their flowing, at service temperatures. It was observed that some of these materials also appreciably increased the durability of the coatings.

Because little was known of how mineral matter affected the weathering of asphalts, a project was started at the National Bureau of Standards by the Asphalt Roofing Institute [1] to investigate the effects of adding finely divided mineral matter of various kinds to an asphalt. The results indicated that many types of minerals up to concentrations of 35 percent materially improved the durability of the asphalt [1, 2].[2] However, the study left many questions still unanswered.

The investigation was resumed and extended in 1947 to cover concentrations of mineral matter in excess of 35 percent in several different asphalts. In addition, the effects of coating thickness, softening point of the base asphalt, particle-size distribution, particle shape, and some natural variations of the minerals and methods of mixing the minerals and asphalts are covered by this work.

2. Materials

2.1. Mineral Additives

O. G. Strieter [2], working with but one asphalt, found that although many mineral additives enhanced durability, there were large differences in the magnitude of the increase. Based partly on his findings and largely on the results of service performance, six additives were selected for evaluation in three asphalts in this investigation. The characteristics of these additives are reported in detail in table 1. Figure 1 contains photomicrographs of these materials at convenient magnifications to illustrate their particle shapes. Table 2 lists the properties of other materials used in only one phase of this study. Some of the properties of three of the minerals in table 1 have been repeated in table 2 in order to facilitate the comparison of these minerals with the others in table 2.

2.2. Asphalts

The asphalts investigated were obtained from three widely different crudes and are typical of asphalts used in the manufacture of asphalt shingles. Each crude was commercially processed into a flux and five asphalt products of different softening points, as shown in table 3. The fluxes are the residual products of steam- or vacuum-refining processes that had removed the lower-boiling fractions of the crudes. The fluxes were oxidized with air at about 450° F in industrial blowing stills. As oxidation progressed, the charge became harder, and as it attained the characteristics of each of the desired products, that product was removed from the still. (Although rarely exposed to the same climate conditions, service performance of the three asphalts indicated that asphalt II should be the most durable and I the least. Asphalt III should be close to asphalt II.)

To facilitate the differentiation of the various coating terms, the following designations are used throughout this report:

Asphalt. A particular source of asphalt products, designated by the numerals I, II, and III.

Asphalt product. An asphalt blown to a desired softening point.

Asphalt blend. A mixture of two adjacent products in suitable proportions to have a desired softening point.

Coating. An asphalt blend with or without mineral matter having a softening point in the range of shingle coatings.

Asphalt coating. An asphalt blend without mineral matter having a softening point in the range of shingle coating asphalts, and exposed for evaluation, designated A, B, and C.

Additive coating. An asphalt blend with added mineral matter having a softening point in the range of shingle coatings.

Asphalt base. An asphalt blend that is mixed with mineral matter to produce an additive coating.

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[1] Now the Asphalt Roofing Industry Bureau.

[2] Figures in brackets indicate the literature references at the end of this report.

Table 1. *Characteristics of mineral additives*

Property	Blue-black slate	Florida clay[a]	Niagara dolomite	Low-carbon fly ash	Tennessee mica	Lake Erie silica
	Additive					
Specific gravity[b]	2.94	2.64	2.87	2.62	3.01	2.68
Surface area[c] ... m^2/g	1.0	27.2	2.0	2.0	2.7	2.5
Oil absorption[d] ... %	29.5	63.9	19.4	30.0	97.2	19.5
Water absorption[d] ... %	32.7	36.4	18.5	33.8	61.5	20.2
Loss on ignition at 1,000° F ... %	2.1	11.8	1.8	4.9	0.9	0.7
Loss on ignition at 1,800° F ... %	5.4	13.3	43.7	7.3	4.4	2.5
Moisture ... %	0.2	2.7	0.1	0.4	0.2	0.2
Solubility ... %	.00	0.04	.00	5.90	.46	.00
Free alkali[e] ... %	.0	.0	[f].0	[f]0.0	.0	.0
Chemical analysis:[g]						
SiO_2 ... %	56	47	6	40	50	98+
$Al_2O_3+Fe_2O_3$ ... %	32	38	1	48	35	
$CaO+MgO$ ... %			49	2.5	1	
K_2O+Na_2O ... %	4				10	
SO_3 ... %					0.5	
Carbon ... %	2			7.6		
Particle size (*mils* / *microns*) — Finer than U. S. Std. Sieve						
9.84 / 250 — No. 60 ... %	99.8	100.0	99.9	99.6	100.0	99.9
6.97 / 177 — No. 80 ... %	99.3	99.9	99.9	98.9	96.0	98.2
5.86 / 149 — No. 100 ... %	97.9	99.8	99.6	97.5	91.0	80.8
4.92 / 125 — No. 120 ... %	96.2	99.8	99.3	96.7	85.6	53.7
3.47 / 88 — No. 170 ... %	91.3	99.6	96.6	94.2	65.9	15.3
2.91 / 74 — No. 200 ... %	86.9	99.3	93.4	92.3	56.8	8.1
2.44 / 62 — No. 230 ... %	83.6	99.1	89.9	90.6	50.3	5.4
1.73 / 44 — No. 325 ... %	76.7	98.9	81.0	84.8	37.8	3.4
1.57 / 40 — Sed[h]	68	97	73	73	65	9
0.79 / 20 — Sed[h]	54	12	39	60	22	6
.39 / 10 — Sed[h]	8	2	26	43	4	4
.16 / 4 — Sed[h]	2	1	13	15	2	1
.8 / 2 — Sed[h]	1		7	2		
Mixture with asphalt:[i]						
Ease of mixing	Good	Poor	Fair	Fair to good	Good	Good
Softening-point increase ... °F	15	28	13	20	40	5

[a] Plasticity index, 34. Plastic limit, 34. ASTM Method D424–39.
[b] Isopropyl alcohol displacement.
[c] Low-temperature nitrogen adsorption—B. E. T. Method.
[d] ASTM D281–31, using a mineral oil and water instead of the specified oil.
[e] W. Lerch and R. H. Bogue, Ind. Eng. Chem. **2**, 296–300 (1930).
[f] Turns phenolphthalein pink in aqueous solution.
[g] Supplier's analyses.
[h] Sedimentation in isopropyl alcohol. It should be noted that the results obtained by this method do not necessarily agree with those of the sieve analysis.
[i] 40 percent of additive in product 4 of asphalt III (softening point, 224° F). See table 3.

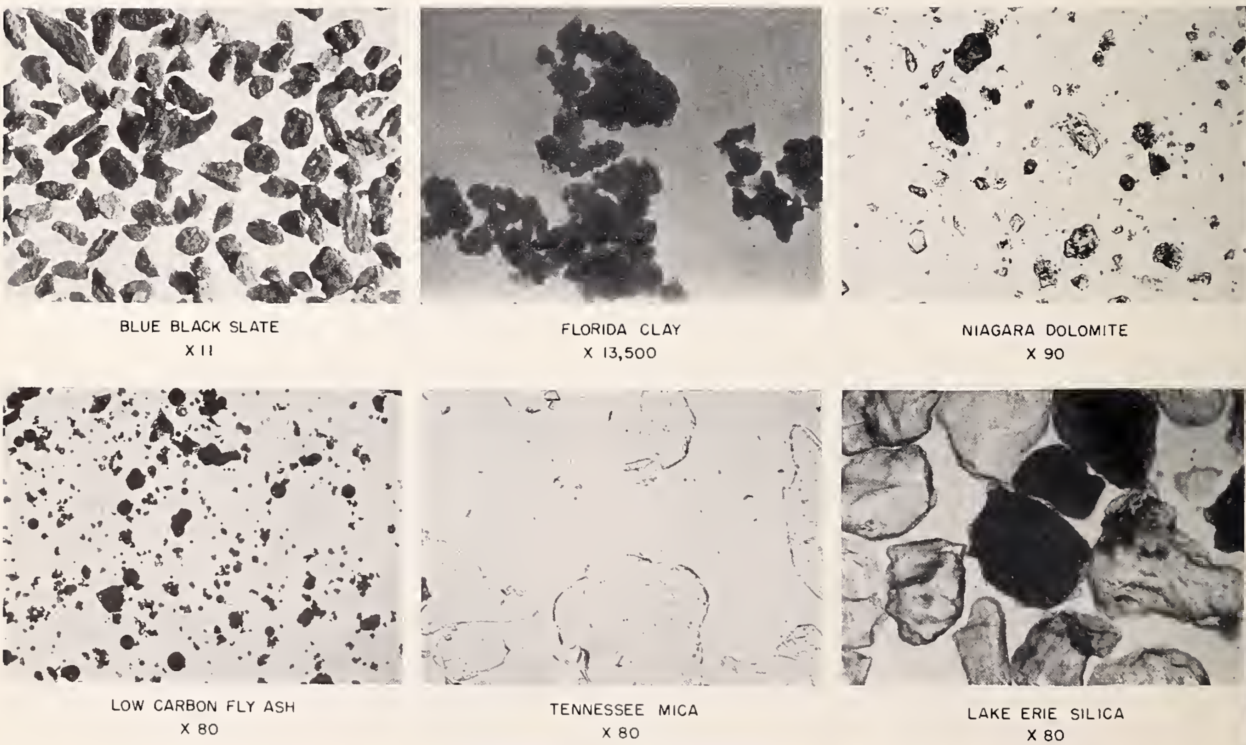

Figure 1. *Mineral additives.*

Table 2. *Properties of clays, fly ashes, silicas, and oyster shell*

Property [a]	Florida [b] clay	Kyanite	Bradbury clay	Low carbon [b] fly ash	High carbon fly ash	Chicago fly ash	Lake Erie [b] silica	Bank silica	Ottawa silica	Special silica	Oyster shell
Moisture ... %	2.7	0.0	1.6	0.4	0.4	0.4	0.2	0.1	0.0	0.6	0.4
Loss on ignition at 1,000° F ... %	11.8	.1	4.4	4.9	22.8	2.1	.7	.2	.1	.7	.9
Loss on ignition at 1,800° F ... %	13.3	.1	5.7	7.3	25.9	4.2	2.5	.4	.2	1.4	43.5
Water solubility ... %	0.0	.5	1.0	5.9	4.9	5.6	0.0	.2	.1	3.0	0.5
Free alkali ... %	.0	.0	0.0	[c] 0.0	[c] 0.1	[c] 0.6	.0	.0	.0	0.0	[c] .0
Particle sizes finer than U. S. Standard Sieve:											
No. 60 ... %	100.0	99.9	100.0	99.6	98.7	99.9	99.9	100.0	100.0	98.2	97.7
No. 80 ... %	99.9	99.0	99.7	98.9	97.4	99.7	98.2	97.8	99.0	98.1	93.6
No. 100 ... %	99.8	95.8	99.1	97.5	95.5	99.4	80.8	85.8	96.8	98.0	88.3
No. 120 ... %	99.8	91.9	98.8	96.7	93.9	99.1	53.7	72.2	90.2	97.8	84.4
No. 170 ... %	99.6	80.3	97.9	94.2	89.2	98.2	15.3	42.7	65.0	94.3	75.5
No. 200 ... %	99.3	72.4	97.3	92.3	85.4	97.4	8.1	34.3	51.7	91.2	69.5
No. 230 ... %	99.1	65.8	96.8	90.6	82.5	96.6	5.4	30.8	43.0	87.9	65.2
No. 325 ... %	98.9	58.9	95.0	84.8	72.9	94.0	3.4	19.9	27.8	83.1	57.5
Mixture with asphalt: [d]											
Ease of mixing	Poor	Good	Fair	Good	Good	Good	Good	Good	Good	Good	Good
Softening point increase ... °F	28	5	4	20	17	22	5	7	7	20	15

[a] Refer to table 1 for test methods. [b] Reproduced from table 1 for comparison purposes. [c] Turns phenolphthalein pink in aqueous solution. [d] 40 percent of mineral matter in product 4 of asphalt III (softening point, 224° F). See table 3.

Table 3. *Asphalt products*

Product	Asphalt I	Asphalt II	Asphalt III
Product 1:			
Softening point [a] ... °F	189	190	190
Penetration [b]	28	27	22
Specific gravity [c]	1.013	0.995	1.015
Product 2:			
Softening point [a] ... °F	201	204	192
Penetration [b]	23	23	22
Product 3:			
Softening point [a] ... °F	213	217	207
Penetration [b]	21	20	20
Product 4:			
Softening point [a] ... °F	218	227	226
Penetration [b]	19	18	17
Product 5:			
Softening point [a] ... °F	230	239	232
Penetration [b]	14	15	16
Specific gravity [c]	1.017	1.003	1.021
Flux:			
Furol viscosity [d] at 210° F	84	286	595
Flash point (COC) [e] ... °F	445	580	620

[a] Softening point, ring, and ball, ASTM Method D36-26.
[b] Penetration at 77° F, 100 g, 5 sec (0.1-mm units), ASTM Method D5-52.
[c] Specific gravity at 77° F was determined only on products 1 and 5.
[d] Furol viscosity, ASTM Method D88.
[e] Flash point, Cleveland Open Cup, ASTM Method D92.

3. Procedure

3.1. Preparation of Specimens

Preliminary to preparing the additive coatings, the softening-point increases produced by the minerals at their test concentrations were determined. These increases were used to select the asphalt base required for the coating. Suitable proportions of the asphalt products straddling the softening point of the asphalt base were blended to form the base. The blend was mixed with the desired percentage of mineral matter to produce an additive coating with a softening point in the range 217° to 227° F. In some instances the softening point of 227° F was exceeded even though only the softest product was used, because that particular concentration of that additive resulted in a very large softening-point increase.

The selected blend of products was melted, the mineral matter added, and the mixture stirred continually by hand at about 420° to 430° F until the surface became free from foam and bubbles. When viscosity measurements were desired, the temperature was increased to 450° F and the viscosity measured with a Brookfield viscometer.[3] While the temperature was dropping slowly to 190° F, viscosity measurements were made at intervals of about 10 deg F after thorough stirring of the mix.

The mix was reheated to its working range for the preparation of exposure panels [4] by the hydraulic-press method [3]. The molten coating was poured on a sheet of aluminum, 3 by 6 by 0.064 in., which was fastened to a 6- by 8-in. sheet of kraft paper with masking tape (the tape covered ¼ in. on three sides and ½ in. on one of the narrow sides of the aluminum sheet). A sheet of dextrin-coated paper was placed immediately over the surface of the molten coating and the assembly put between the heated platens (220° F) of a hydraulic press. The press was closed with sufficient thrust to cause the coating to flow and assume the thickness defined by spacers at two sides of the platens. After about 30 sec, the assembly was removed from the press, and the dextrin paper was soaked from the panel surface. The masking tape was stripped from the panel, and the thickness of the coating was recorded. All acceptable coatings had a deviation of less than 1 mil from the required thickness.

Just prior to making the first panel, two specimens for the softening-point determination and

[3] A Furol viscometer is customarily used in asphalt work when no mineral matter is present.
[4] "Panels" and "specimens" are synonymous.

one shatter specimen [5] were poured. After half the panels had been prepared, another shatter specimen and a water-adsorption specimen (see footnote 5) were poured. A third shatter and two additional softening-point specimens were poured after all the panels had been made. On occasions, ash determinations were made to confirm uniform mineral distribution in the molten stabilized coating.

3.2. Exposure of Specimens

Six accelerated-durability machines were used in this investigation. Duplicate specimens were exposed in two different machines in order to obtain an average value of durability that would be considered more representative. The specimens were supported on aluminum panel supports, two to a support, one above the other. The specimens and supports, respectively, were inverted on alternate days.

The accelerated-durability machines were operated 22 hr a day, 7 days a week, during the entire course of exposures. As recommended [4], the enclosed arcs were operated at 120 to 145 v and 15 to 17 amp. The exposure cycle, identified as 51–9C, consisted of 51 min of radiation, followed by 9 min of radiation and cold (40° $\pm$2 deg F), demineralized water spray, delivered at 25 $\pm$5 psig. This cycle was selected for its effectiveness, based on a comparison of numerous combinations of light, water spray, and refrigeration [5]. It provides the frequency and quantity of cold water needed for removal of the water-soluble weathering products, and also induces frequent thermal shocks, which make a low-temperature refrigeration period unnecessary [6]. It also has the advantage of yielding consistent results, regardless of the part of the cycle in which the inspection is made, providing the panels are dry when inspected.

A thermostat was embedded in an asphalt-coated panel and rotated with the test specimens. This thermostat controlled forced-air circulation to maintain the panel in which it was mounted at 140° to 145° F for about half of each cycle. The panel temperature rose from about 50° to 140° F, within about 30 min, the exact time being dependent on the number of panels in each machine and the ambient conditions.

3.3. Inspection of Specimens

The exposure panels were examined weekly, both visually and with a high-voltage probe [7]; spark patterns were taken whenever there was any break in the coating [8]. The patterns were examined through a transparent grid of 60 squares covering the central 2- by 5-in. portion of the coating (⅜ in. around the edge of the coating was not included). When breaks in the coating appeared in 30 or more squares, the specimen was considered failed and removed from the machine.

[5] The methods are described in the appendix, page 13.

4. Results

All durability results reported on coatings are the averages of two or more exposures made in two different accelerated-durability machines. In all cases, the replicate individual specimens failed within 15 percent of their reported average. Of course, because of the large number of variables involved in the materials, preparation, and exposure of each coating, occasional replicate specimens deviated widely from each other. In these few instances additional sets of four specimens were exposed to obtain additional results from which more reliable conclusions could be drawn.

4.1. Effect of Softening Point of Asphalt Products on Durability

Four specimens of each of the asphalt products [6] in table 3 were exposed to determine the relationship between softening point and durability. The results are presented in figure 2. For all three asphalts the durability increased as the softening point of the products decreased. This characteristic is used to good advantage when mineral matter is added to asphalts in commercial production, because it permits the use of softer, more durable asphalt products without the danger of having the coating flow at service temperatures.

[6] About a year had passed between the time the additive coatings were exposed and that at which these specimens were made. The change in softening point of each product during storage may be obtained from figure 2 and table 3.

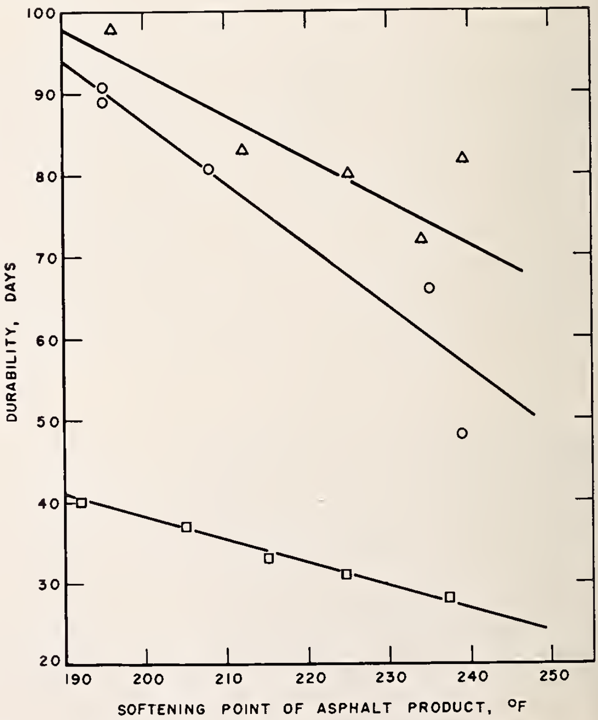

FIGURE 2. *Durability of asphalt products.*

25-mil coating thickness. 51–9C cycle. □, products of asphalt I; △, products of asphalt II; ○, products of asphalt III.

4.2. Effect of Softening Point of Asphalt Base on Durability of Coatings Containing Mineral Additives

Additive coatings having a common asphalt base (blend of asphalt II; softening point, 225° F) with 35, 50, and 60 percent of mineral matter were made and compared with additive coatings made from progressively softer blends of the same asphalt with the same percentages of mineral matter (variable base). The variable-base coatings were all compounded to have softening points in the range of 217° to 227° F. As seen in figure 3, with both blue-black slate and dolomite, the durability of the coatings increased with mineral-matter concentration, with the "common-base" coatings appearing slightly, but not significantly, more durable than the "variable-base" coatings at concentrations below 50 percent and slightly less durable above 50 percent. Thus, in the case of this particular asphalt, the increased durability of the softer products was not manifested significantly, and the softening point of the asphalt base was not of controlling importance.

The data in figure 3 are plotted on a ratio basis, i. e., the durability of the coating divided by the durability of the corresponding asphalt coating. In this way it is possible to see readily the magnitude of the change in durability produced by the additive, and the effects of the additives on the various asphalts are reduced to a comparable basis.

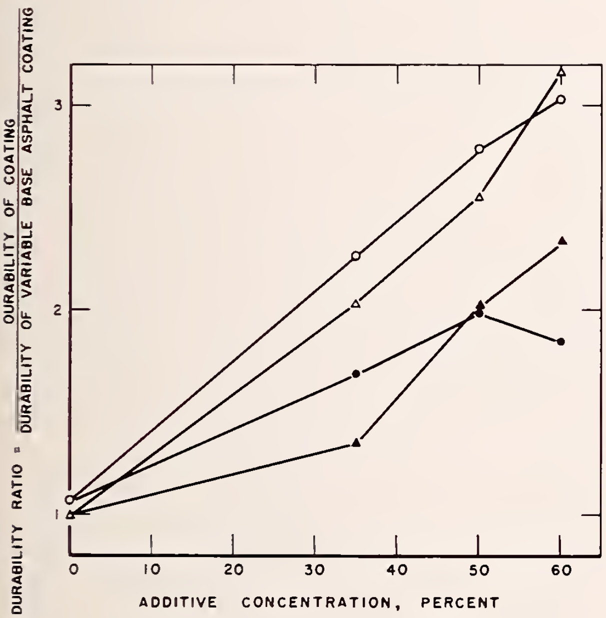

FIGURE 3. *Accelerated-durability data.*

25-mil coating thickness. 51–9C cycle. Durability ratio of 1=75 days.

Concentration	Softening points of coatings with blue-black slate		Softening points of coatings with dolomite	
	Common base, ○	Variable base, △	Common base, ●	Variable base, ▲
%	°F	°F	°F	°F
0	225	228	225	228
35	231	222	230	225
50	246	217	236	219
60	270	[a] 237	248	--------
	Base softening points			
35	225	211	225	217
50	225	202	225	206
60	225	190	225	197

[a] See page 3.

4.3. Effect of Degree of Mixing of Additives With Asphalt on Durability of Coatings

The mineral additives investigated had wide ranges of particle sizes, as shown in table 1. The effect of the thoroughness of mixing on the durability and uniformity of durability was investigated. Three mineral additives were used: (1) blue-black slate, representing a material that mixes readily and easily with asphalt, (2) Niagara dolomite, representing a material that is incorporated with asphalt with moderate difficulty, and (3) Florida clay, representing a material extremely difficult to mix with asphalt. These materials were mixed in concentrations of 35, 50, and 60 percent with asphalt III by three different procedures:

Prolonged hand mixing (P)—The melt was stirred continually until all bubbling and foaming ceased, about 1 hr.

Continuous mechanical mixing (M)—The mixture was stirred for 30 min with a high-speed laboratory stirrer after the mineral matter had been added to the molten asphalt.

Brief hand mixing (B)—Panels were made as soon as the mineral matter was folded into the asphalt. Only enough stirring was used to keep the mineral matter suspended.

Figure 4 covers the results of the accelerated durability tests on these coatings. The data in figure 4 are also plotted on a ratio basis. It can be seen that with blue-black slate, a mineral that mixes readily with molten asphalt, the type of

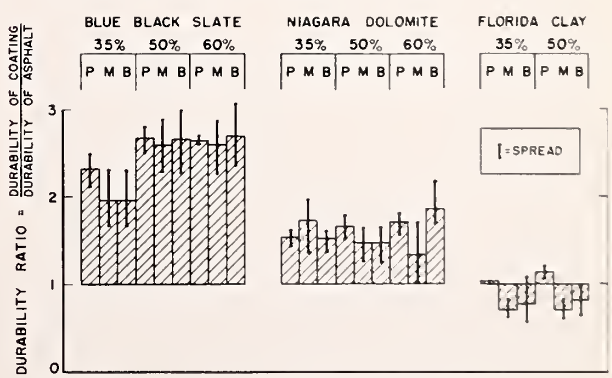

FIGURE 4. *Effect of mixing on durability.*

25-mil coating thickness. 51–9C cycle. Durability ratio of 1=75 days (Asphalt III). P, prolonged hand mixing; M, 30-min mechanical mixing; B, brief hand mixing.

mixing had little effect on the average durability at the two higher concentrations. With only 35 percent of slate, however, prolonged hand mixing produced more durable coatings than either the 30-min mechanical mixing or the brief hand-mixing procedure.

The most durable coating at each concentration of dolomite resulted from a different mixing procedure: mechanical mixing at 35 percent, prolonged hand mixing at 50 percent, and brief hand mixing at 60 percent. Of the coatings containing clay, only those in which the clay was incorporated by means of prolonged hand mixing proved to be slightly more durable than the corresponding asphalt coating without mineral matter; the other mixing procedures resulted in coatings of significantly less durability. Thus, it may be concluded that with clay, the mineral matter that was difficult to blend homogeneously with the asphalt base, the degree of mixing was important. Even with the blue-black slate and dolomite, thorough mixing was essential to produce uniform durability in duplicate specimens. As seen by the solid vertical lines in the center of each bar in figure 4, regardless of the mineral additive used, the differences in the durability among the replicate specimens were always least when prolonged hand mixing was employed.

4.4. Effect of Six Selected Commercially Available Additives on Durability of Coatings

A series of exposures was made to determine the effects of six commercially available additives on the durability of asphalts from three crudes. The investigation included the effects at concentrations of 35, 50, and 60 percent when exposed in films 13, 25, and 43 mils thick. Because of the difficulty of handling coatings containing 50 and 60 percent of Tennessee mica and 60 percent of Florida clay, specimens were not made from these compositions, but exposures were made of all three concentrations of the No. 50 blue-black slate, Niagara dolomite, low-carbon fly ash, and Lake Erie silica in the three asphalts.

Where possible, each coating was made to a softening point in the range 217° to 227° F, but in the cases of 60 percent of blue-black slate and fly ash and 35 percent of mica, a softening point of 227° F was exceeded, even though the softest asphalt products were used.

For control purposes, coatings containing no additives were made of the three asphalts. The characteristics of the asphalt coatings that were exposed are shown in table 4. The durabilities of these coatings are reported in figure 5 and are used as the reference coatings in the durability ratios in figures 6 to 10.

In the range of film thicknesses investigated, 13 to 43 mils, the durability of each of the three asphalt coatings increased almost linearly with thickness. Coatings of A were less durable than corresponding coatings of B and C.

The characteristics of all of the coatings, both with and without additives, are presented in table 5. The softening points and penetrations of the asphalt bases, as estimated from the proportions of the two products blended, and the volume composition and specific gravity, as calculated from the specific gravities of the components, are presented for information. The temperature of preparation and viscosity data are reported as background information pertinent to the preparation of the specimens. The water-absorption and shatter determinations are related to two factors

TABLE 4. *Characteristics of asphalt coatings used as controls*

Test	Asphalt 1 Coating A	Asphalt 11 Coating B	Asphalt III Coating C
Softening point [a] ----------°F--	223	224	227
Penetration [b] at 32° F----------	10	11	11
Penetration [b] at 77° F----------	17	17	14
Penetration [b] at 115° F---------	30	26	21
Penetration index [b]-------------	4.7	4.7	4.5
Suseeptibility [b]----------------	1.16	0.87	0.73
Loss on heating [c] ----------%--	0.22	.03	.10
Penetration after heating [b]------	17	17	14
Speeifie gravity at 77° F---------	1.015	0.999	1.018
Viseosity [d] at 400° F--------cp--	280	420	375
Viseosity [d] at 450° F--------ep--	100	140	130
Viseosity [d] at 500° F--------ep--	25	53	28
Water absorption [e] at 28 days------------------g/ft²--	0.67	0.43	0.34
Shatter resistance [f] (average) ---------------in.--	6.3	8.3	2.7

[a] ASTM Method D36–26.
[b] ASTM Method D5–52. Units are 0.1 mm. Penetration index from nomograph in referenee [9], and

$$\text{susceptibility} = \frac{\text{penetration } 115°\text{F} - \text{penetration } 32°\text{ F}}{\text{penetration } 77°\text{ F}}.$$

[c] ASTM Method D6–39T.
[d] Brookfield viscometer.
[e] Specimens, 3-in. diameter by 3/16 in. thick, submerged 1/4 in. in distilled water at 75° F. See appendix for method.
[f] A 1/2-lb. weight was dropped on a 3-in.-diamter by 3/16-in.-thick specimen in a mixed iee and water bath. See appendix for method.

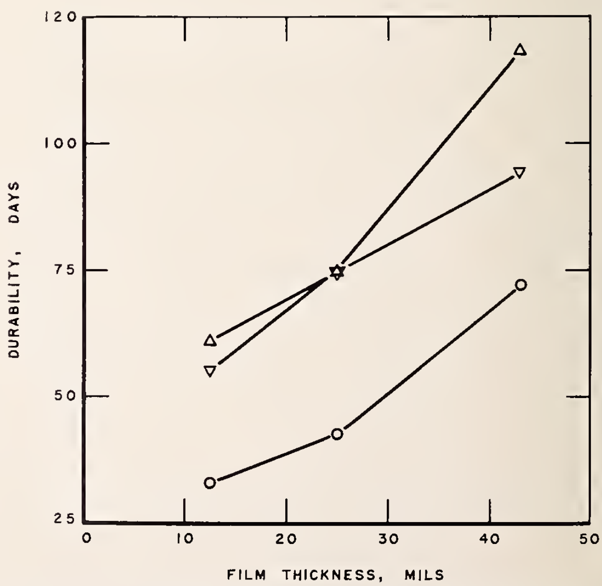

FIGURE 5. *Accelerated durability data af asphalt caatings.*
51–9C cycle. ○, Coating A; ▽, eoating B; △, coating C.

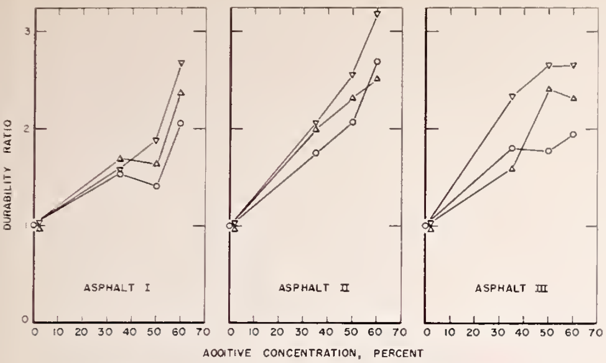

Figure 6. *Coatings containing blue-black slate.*

51–9C cycle

Asphalt	Coating thickness		
	○, 13 mils	▽, 25 mils	△, 43 mils
	Durability ratio of 1=		
	Days	*Days*	*Days*
I	33	43	72
II	55	75	94
III	61	75	118

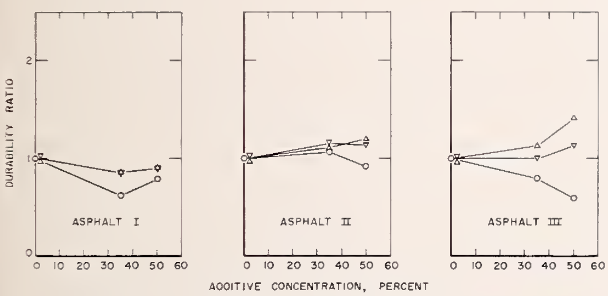

Figure 7. *Coatings containing Florida clay.*

See figure 6 legend.

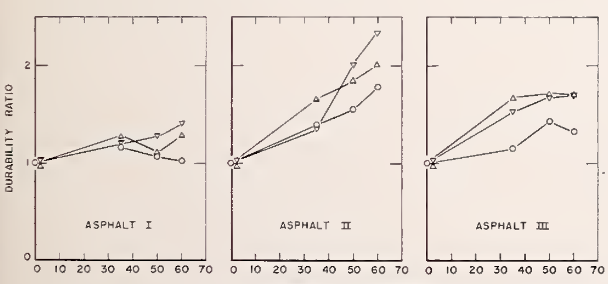

Figure 8. *Coatings containing Niagara dolomite.*

See figure 6 legend.

of natural degradation that are not usually considered in accelerated durability tests. However, because these characteristics may be significant under certain types of exposure conditions, they have been included.

The durability of each of the coatings containing additives is presented in table 6 and figures

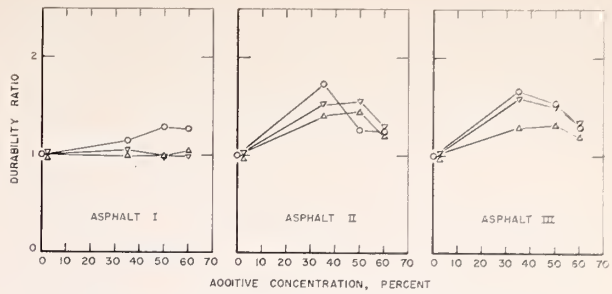

Figure 9. *Coatings containing low-carbon fly ash.*

All coatings containing fly ash were examined only visually.

See figure 6 legend.

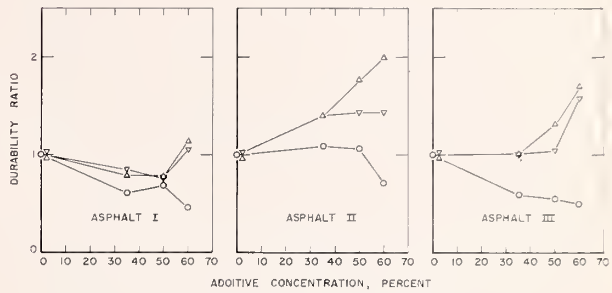

Figure 10. *Coatings containing Lake Erie silica.*

See figure 6 legend.

6 to 10, according to the mineral used. The data in the figures, again, are on a durability-ratio basis in order to simplify their interpretation.

At all film thicknesses, the Tennessee mica was most effective in increasing the durability of asphalts from all three sources.

Many of the coatings containing mica were removed before failure, as shown in table 6, but long after all of the other coatings had failed. Although 35 percent of mica increased the durability of all three asphalts at least 500 percent, it could not be used at the higher concentrations selected for these tests, because it increased the viscosity of even the softest asphalt products to the extent that they became unworkable.

It is evident from the data that different mineral additives may affect durability to different degrees. However, one generality applies. Durability increases with coating thickness both with and without mineral additives. In some instances this conclusion may be evident only when the ratio data are translated into observed durability in days. For example, in figure 6, the durability ratio for the 25-mil-thick coating of 50 percent blue-black slate in asphalt I was about 1.9, whereas for the 43-mil-thick coating it was only 1.6, but the durabilities of these coatings were 81 and 116 days, respectively.

Each of the asphalt-additive systems must be considered separately in order to interpret the data accurately. Blue-black slate, which is widely used in commercial roofing, improved the durability of all three asphalts in all three film thicknesses. However, in asphalt I, there was

TABLE 5. *Characteristics of coatings*

Property	Additive (%) in asphalt I				Additive (%) in asphalt II				Additive (%) in asphalt III			
	0	35	50	60	0	35	50	60	0	35	50	60
Blue-black slate												
Volume percentage[a]	0	15.7	25.7	34.1	0	15.5	25.4	33.8	0	15.7	25.7	34.2
Specific gravity of mixture[a]	1.015	1.32	1.51	1.68	0.999	1.30	1.49	1.66	1.018	1.32	1.51	1.68
Softening point °F	223	222	226	240	224	222	217	237	227	218	221	256
Softening point of base[b] °F	223	210	202	189	224	211	202	190	227	205	191	190
Penetration of base at 77°F[b]	17	21	23	28	17	22	23	27	14	20	22	22
Temperature of preparation °F	365–428	410–428	410–428	446–482	410–464	437–446	446–464	467–473	401–437	410–437	448–473	464–490
Viscosity at 400°F cp	280	540	1,210	11,100	420	640	1,480	13,500	375	580	1,450	14,500
Viscosity at 450°F cp	100	180	580	3,900	140	290	600	4,150	130	224	535	6,200
Viscosity at 500°F cp	25	58	260	2,100	53	130	275	1,850	28	87	240	2,350
Water absorption at 28 days g/ft²	0.67	0.77	0.92	0.92	0.43	0.70	0.91	0.99	0.34	0.53	0.66	0.70
Shatter resistance[c] in.	6.3	11.7	18.3	21.0	8.3	12.0	18.1	21	2.7	9.2	15.5	21
Florida clay												
Volume percentage[a]	0	17.3	27.8	36.6	0	16.9	27.5	36.3	0	17.3	27.8	36.7
Specific gravity of mixture[a]	1.015	1.30	1.47	1.61	0.999	1.28	1.45	1.59	1.018	1.30	1.47	1.61
Softening point °F	223	[d]219	226	------	224	221	220	------	227	221	227	------
Softening point of base[b] °F	223	199	189	------	224	206	191	------	227	205	190	------
Penetration of base at 77°F[b]	17	24	28	------	17	23	27	------	14	20	22	------
Temperature of preparation °F	365–428	410–428	437	------	410–464	428–455	446–482	------	401–437	437–464	446–482	------
Viscosity at 400°F cp	280	590	3,000	------	420	830	3,600	------	375	990	5,000	------
Viscosity at 450°F cp	100	225	1,400	------	140	340	1,550	------	130	335	2,100	------
Viscosity at 500°F cp	25	94	570	------	53	135	700	------	28	117	850	------
Water absorption at 28 days g/ft²	0.67	1.32	1.96	2.04	0.43	1.42	2.18	------	0.34	1.88	1.99	------
Shatter resistance[c] in.	6.3	8.7	9.0	12.0	8.3	8.3	15.7	------	2.7	9.3	6.3	------
Niagara dolomite												
Volume percentage[a]	0	16.0	26.1	34.7	0	15.8	25.8	34.3	0	16.0	26.1	34.7
Specific gravity of mixture[a]	1.015	1.31	1.50	1.66	0.999	1.29	1.48	1.64	1.018	1.31	1.50	1.66
Softening point °F	223	220	224	226	224	225	219	-----	227	221	222	224
Softening point of base[b] °F	223	209	205	198	224	217	206	197	227	204	202	194
Penetration of base at 77°F[b]	17	22	22	25	17	20	23	25	14	20	21	22
Temperature of preparation °F	365–428	428–437	419–455	455–464	410–464	428–464	392–410	401–419	401–437	437–464	446–482	436–455
Viscosity at 400°F cp	280	500	780	1,380	420	690	880	1,380	375	590	900	1,280
Viscosity at 450°F cp	100	175	295	485	140	240	360	550	130	222	275	450
Viscosity at 500°F cp	25	90	125	225	53	100	180	260	28	80	140	230
Water absorption at 28 days g/ft²	0.67	0.88	1.07	1.30	0.43	0.86	1.15	1.41	0.34	0.91	1.15	1.26
Shatter resistance[c] in.	6.3	6.3	5.8	14.0	8.3	15.7	15.5	18.3	2.7	7.3	8.3	12.0
Low-carbon fly ash												
Volume percentage[a]	0	17.2	27.9	36.8	0	17.0	27.6	36.4	0	17.3	28.0	36.8
Specific gravity of mixture[a]	1.015	1.29	1.46	1.60	0.999	1.27	1.44	1.58	1.018	1.29	1.46	1.60
Softening point °F	223	223	219	256	224	219	225	253	227	224	224	266
Softening point of base[b] °F	223	205	189	189	224	205	197	190	227	205	191	190
Penetration of base at 77°F[b]	17	22	28	28	17	23	25	27	14	20	22	22
Temperature of preparation °F	365–428	410–473	437–455	464–482	410–464	435–464	437–455	428–437	410–437	419–446	457–475	509–527
Viscosity at 400°F cp	280	480	1,500	18,500	420	560	------	------	375	820	2,200	33,000
Viscosity at 450°F cp	100	205	590	7,750	140	200	------	------	130	320	860	14,500
Viscosity at 500°F cp	25	96	255	4,300	53	120	------	------	28	94	365	5,700
Water absorption at 28 days g/ft²	0.67	0.95	1.12	1.14	0.43	0.88	1.08	1.23	0.34	0.70	0.93	1.08
Shatter resistance[c] in.	6.3	7.8	------	11.5	8.3	7.3	12.0	21.0	2.7	5.3	10.7	12.7
Tennessee mica												
Volume percentage[a]	0	15.4	25.2	33.6	0	15.2	24.9	33.2	0	15.4	25.2	33.6
Specific gravity of mixture[a]	1.015	1.33	1.52	1.69	0.999	1.31	1.50	1.67	1.018	1.33	1.52	1.69
Softening point °F	223	270	------	------	224	270	------	------	227	270	------	------
Softening point of base[b] °F	223	189	------	------	224	190	------	------	227	190	------	------
Penetration of base at 77°F[b]	17	28	------	------	17	27	------	------	14	22	------	------
Temperature of preparation °F	365–428	428–446	------	------	410–464	482–491	------	------	401–437	491–518	------	------
Viscosity at 400°F cp	280	----	------	------	420	----	------	------	375	----	------	------
Viscosity at 450°F cp	100	----	------	------	140	----	------	------	130	----	------	------
Viscosity at 500°F cp	25	----	------	------	53	----	------	------	28	----	------	------
Water absorption at 28 days g/ft²	0.67	2.05	------	------	0.43	0.83	------	------	0.34	0.52	------	------
Shatter resistance[c] in.	6.3	21	------	------	8.3	21	------	------	2.7	21	------	------
Lake Erie silica												
Volume percentage[a]	0	16.9	27.5	36.2	0	16.7	27.2	35.8	0	16.9	27.5	36.2
Specific gravity of mixture[a]	1.015	1.30	1.47	1.62	0.999	1.28	1.45	1.60	1.018	1.30	1.47	1.62
Softening point °F	223	----	226	[d]217	224	[d]217	[d]217	222	227	226	227	222
Softening point of base[b] °F	223	213	208	196	224	207	200	197	227	206	205	191
Penetration of base at 77°F[b]	17	21	22	25	17	23	24	25	14	20	20	22
Temperature of preparation °F	365–428	410–419	410–437	419–446	410–464	383–401	392–410	383–419	401–437	401–410	419–446	446–464
Viscosity at 400°F cp	280	600	790	------	420	480	860	------	375	600	1,140	------
Viscosity at 450°F cp	100	200	250	------	140	220	300	------	130	215	485	------
Viscosity at 500°F cp	25	50	77	------	53	92	190	------	28	65	200	------
Water absorption at 28 days g/ft²	0.67	0.64	0.73	0.73	0.43	0.56	0.62	0.65	0.34	0.63	0.59	0.59
Shatter resistance[c] in.	6.3	7.3	14.7	16.7	8.3	11.7	20.0	17.7	2.7	6.3	7.3	8.3

[a] Calculated. [b] Estimated. Penetration units=0.1 mm. [c] 3- by 3/16-in. disk. Average of 3 specimens. [d] Rate slow—probably 3 to 4 deg F higher.

Table 6. *Durability ratio* [a] *of coatings containing 35 percent of mica*

Coating thickness	Asphalt		
	I	II	III
Mils			
13	5.7	6.7	6.6
25	13.3	[b]>13.3	[b]>13.3
43	[b]>13.9	[b]>10.6	[b]>8.5

[a] See caption of figure 6 for durability-ratio figures.
[b] Removed at 1,000 days (before failure).

little difference in durability between 35 and 50 percent blue-black slate and in asphalt III, almost no difference in durability between 50 and 60 percent; in the 13-mil coating all three concentrations were of about the same durability. The greatest relative improvement was observed in the 25-mil coating.

Florida clay behaved as an extender in asphalt I, leaving the durability almost unchanged in all concentrations. In asphalt II, similar results were observed, although some slight increase in durability could be seen. Although improvement was shown in asphalt III in the 43-mil film, there was almost a corresponding decrease in durability at 13 mils.

Niagara dolomite, in all three concentrations, improved the durability of asphalt I slightly, and that of asphalts II and III appreciably more, especially in the 25- and 43-mil coatings.

Because fly ash conducts electricity, coatings made with it could not be inspected with the aid of the high-voltage probe; they were inspected visually. Because the majority of the other coatings failed electrically before they would have failed by visual inspection, the durabilities of the fly-ash coatings were probably less than reported. It is not possible to estimate the magnitude of this effect; therefore, these data must be considered without attempting to compare them with those of the other minerals. In asphalt I, the fly ash apparently had little effect on the durability. In asphalts II and III, the fly ash seemed to improve the durability at concentrations of 35 and 50 percent, but less so at 60 percent. Fly ash was the only additive in which the 60 percent coatings were consistently less durable than the coatings with 35 and 50 percent of additives.

The Lake Erie silica decreased the durability of asphalt I at the lower concentrations, but had almost no effect at 60 percent (except in the 13-mil coating). In both asphalts II and III it decreased the durability in the 13-mil films and increased it in the 43-mil films. It improved the durability of asphalt II about the same at all three concentrations in the films 25 mils thick, but did not change the durability of asphalt III at concentrations lower than 60 percent.

Thus all six additives increased or did not appreciably alter the durability of asphalts II and III at all three concentrations in films 25 and 43 mils thick. Blue-black slate and mica also improved the durability of asphalt I appreciably in all film thicknesses and concentrations. In those instances in which the 13-mil additive coatings behaved irregularly this effect may be attributed to the presence of additive particles approximating in diameter the thickness of the coating.

4.5. Effect of the Particle-Size Distribution of the Mineral Additives on the Durability of Coatings

Because the particle-size distribution is one of the attributes of the additives that may affect performance and one that may be modified, its effects on durability were evaluated in all three asphalts. Three minerals, blue-black slate, Niagara dolomite, and Lake Erie silica, were sieved into 8 fractions and reconstituted into 5 different synthetic size distributions, designated as follows and reported in detail in table 7:

+200 mesh—Material retained on U. S. Standard Sieve No. 200.
−200 mesh—Material passing a U. S. Standard Sieve No. 200.
−325 mesh—Material passing a U. S. Standard Sieve No. 325.
−140 +325 mesh—Material passing a U. S. Standard Sieve No. 140 but retained on a No. 325.
"Equal" size—Approximately equal weights retained on U. S. Standard Sieves Nos. 120, 170, 230, and 325.

Table 7. *Particle-size distributions*

U. S. Standard Sieve No.	Particle size	Size distribution [a]					
		Regular	+200	−200	−325	−140; +325	Equal size
		Blue-black slate finer than Standard Sieve					
	Mils						
60	9.8	99.8	99.1	----	----	----	100.0
80	7.0	99.3	94.7	----	----	100.0	98.7
100	5.9	97.9	85.0	100.0	----	94.7	95.1
120	4.9	96.2	74.1	99.9	----	92.4	90.2
170	3.5	91.3	35.2	99.9	----	72.9	66.6
200	2.9	86.9	5.7	99.3	----	40.2	54.5
230	2.4	83.1	1.0	95.4	----	13.5	42.8
325	1.7	76.7	0.7	85.5	100.0	4.1	21.7
		Niagara dolomite finer than Standard Sieve					
60	9.8	99.9	99.6	----	----	----	----
80	7.0	99.9	98.6	----	----	----	100.0
100	5.9	99.6	95.8	----	----	----	97.9
120	4.9	99.3	91.1	100.0	----	100.0	94.5
170	3.5	96.6	61.4	99.9	----	93.0	79.8
200	2.9	93.4	20.5	99.9	----	74.1	70.0
230	2.4	89.9	5.9	97.9	----	41.2	50.0
325	1.7	81.0	0.7	87.0	100.0	9.5	19.1
		Lake Erie silica finer than Standard Sieve					
60	9.8	99.9	100.0	----	----	----	
80	7.0	98.2	99.3	100.0	----	----	100.0
100	5.9	80.8	92.7	99.9	----	----	97.9
120	4.9	53.7	76.9	99.9	----	100.0	93.1
170	3.5	15.3	26.5	99.7	----	43.7	71.2
200	2.9	8.1	7.7	98.2	----	18.8	56.5
230	2.4	5.4	1.8	85.7	----	7.3	46.7
325	1.7	3.4	1.1	52.5	100.0	1.2	28.2

[a] A more complete description of these distributions is in the text. Because of the uncertainties involved in separating irregular particles by means of sieves with square openings, the distributions deviate somewhat from their designations.

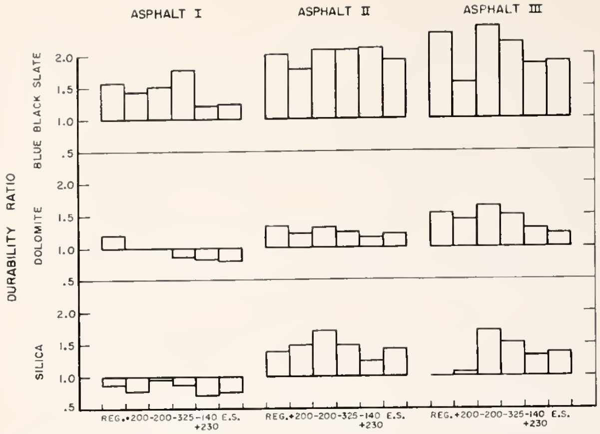

FIGURE 11. *Effect of particle-size distribution on durability.*

35 percent mineral matter. 25-mil coating thickness. 51–9C cycle.

Asphalt	Durability ratio of 1=
	Days
I	43
II	75
III	75

Exposures were made of coatings (all 25 mils thick) containing 35 percent of additives with each of the above distributions. Average durability figures are shown graphically in figure 11. The data are presented as a ratio of the durability of the additive coating divided by that of the corresponding asphalt coating. The results obtained with the regular commercial-size distributions are also presented for comparison.

In asphalt I, only the coating with −325-mesh blue-black slate proved to be superior to that with the regular particle-size distribution, and in asphalt II and III, many of the distributions were approximately equal. In general, the coatings containing −200-mesh materials were somewhat more effective in increasing the durability of the asphalts and those with the +200-mesh additives less effective.

Additional exposures were made from asphalt II and 50 percent of the +200- and −200-mesh portions of each of these three minerals in film thicknesses of 13 and 25 mils. The results are shown in figure 12. Film thickness was of extreme importance when an experimental grading such as the +200-mesh materials was used. In every instance the 13-mil films containing 50 percent of +200-mesh material did not weather as well as the 13-mil films without additives, but in the 25-mil films, the durability was considerably improved by this coarse material as well. However, when the −200-mesh material was used, there was an improvement of about the same magnitude in both the 13-mil and the 25-mil films.

To generalize for coatings 25 mils thick containing 35 percent of additive, the −200-mesh fraction increased the durability most and the

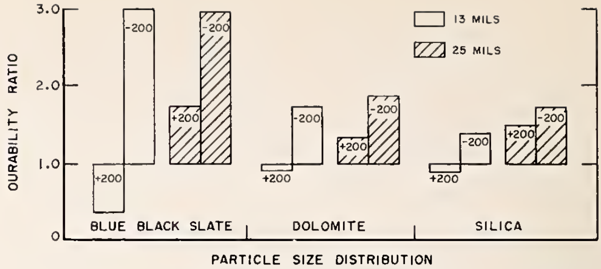

FIGURE 12. *Effect of particle-size distribution on durability.*

50 percent of mineral matter. 51–9C cycle.

Film thickness	Durability ratio of 1=
Mils	*Days*
13	55
25	75

+200-mesh material was least effective, whereas the regular, −325, equal size, and uniform size (−140 +230) follow the −200 in descending order, with little difference between consecutive distributions. When higher concentrations of mineral additives are used, the maximum particle size should be considerably less than the coating thickness in order to obtain greater durability.

4.6. Effect of Particle Shape of Additives on Durability of Coatings

Because particle shape was one property of the minerals studied that seemed to be related to the efficacy of their stabilizing action, some quantitative method of expressing particle shape was sought. A relationship between a bulk density and the true density of the minerals seemed worth considering for this purpose. Franz Pöpel [10], in 1929, proposed a relationship of this type, which he called "fineness factor," and defined as

$$f=\frac{\text{density}-\text{compacted weight,}}{\text{compacted weight}}$$

where the compacted weight is an empirical bulk density determined under any set of conditions that would yield reproducible results.

The bulk density used was obtained by fluffing the mineral matter by letting it flow slowly through a column of 1-in. inside diameter, 21 in. long. Baffles set at an angle of 45° were placed 12 and 16 in. from the top of the column and the column was constricted to a diameter of ¾ in. at the bottom. The column discharged into a 100-ml, weighed and calibrated cylinder from a point ¼ in. above the cylinder. In practice the mineral matter was poured slowly into the column until it just overflowed the receiver. The excess was struck off level with the top of the receiver and the receiver and contents weighed. The weight of the mineral matter divided by its volume is termed the "fluffed bulk density" or simply, the "fluff density." A correlation of durability with fineness factor [7] is presented in figure 13. The individual points are tabulated in table 8.

The only points that deviated considerably from the curves were those for oyster shell and mica, both being materials characterized by flat, platelike particles. Because the deviations are in the direction of greater durability than indicated by the lines, the lines may be taken as conservative indications of durability.

Despite the fair correlations indicated in figure 13, it must be emphasized that these data were obtained for specific materials under very specific test conditions and that the results may vary under other conditions. The fact that a different line is plotted for each asphalt emphasizes the importance of the asphalt itself in respect to durability.

[7] The durabilities of the coatings with mica, although not shown in figure 13, are considerably greater than would be expected from linear extrapolation of the curves.

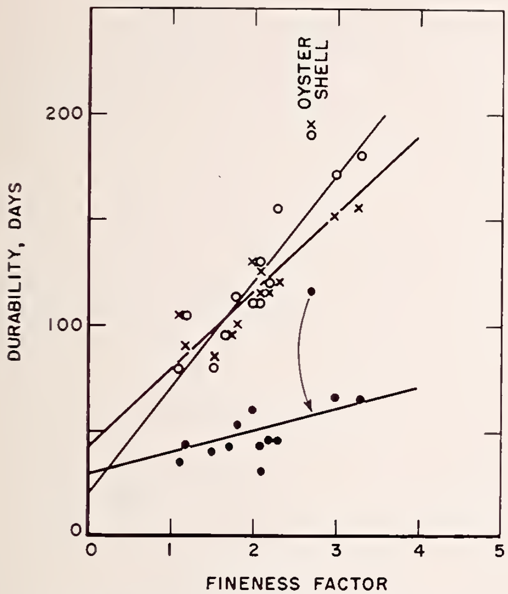

FIGURE 13. *Accelerated durability data.*

35 percent of mineral matter. 25-mil coating thickness. 51-9C cycle. ●, Coatings with asphalt I; ×, coatings with asphalt II; ○, coatings with asphalt III.

TABLE 8. *Effect of fineness factor on the durability of additive coatings (25 mils thick)*

Additive	Particle shape (visual)	Density	Fluff density	Fineness factor	Durability, 35 % of mineral in asphalts—		
					I	II	III
		g/cm^3	g/cm^3		*Days*	*Days*	*Days*
Mica	FP[a]	3.01	0.29	9.7	572	>1,000	>1,000
−200 blue-black slate.	FP	2.94	.68	3.3	65	157	182
Blue-black slate	FP	2.94	.73	3.0	68	152	174
Oyster shell	AP	2.68	.72	2.7	115	198	193
Special silica	BR	2.58	.78	2.3	45	123	155
Low-carbon fly ash.	H	2.62	.62	2.2	46	116	119
−200 dolomite	BS	2.87	.94	2.1	43	99	127
−325 dolomite	BS	2.87	.93	2.1	37	93	111
+200 blue-black slate.	FP	2.94	.99	2.0	62	133	116
Niagara dolomite	BS	2.87	1.03	1.8	52	101	115
Kyanite	I	3.60	1.34	1.7	43	98	96
Florida clay	I	2.64	1.04	1.5	38	86	76
+200 dolomite	BS	2.87	1.29	1.2	43	90	107
Lake Erie silica	BR	2.68	1.31	1.1	37	105	76

[a] FP=Flat plates; BR=blocky, rounded corners; H=heterogeneous; AP=acicular plates; BS=blocky, sharp corners; I=indefinite (requires higher magnification than on light microscope).

4.7. Effect of Clay, Fly Ash, and Silica From Different Sources on Durability of Coatings

All coatings tested in this series were 25 mils thick and contained 35 percent of mineral matter. The materials investigated (described in table 2) are representative of the wide variety of each type available. Although the three clays tested varied from a disordered kaolinite (Florida clay) to a mullite (Kyanite), there was little difference in their effects on the durability of any of the asphalts (fig. 14). Because of the flocculated nature of the Florida and Bradbury clays, the reported size distributions are probably not accurate. These clays were not dispersed readily in the asphalts. However, even with Kyanite, which was mixed easily with the asphalts, there was no appreciable improvement in durability. In general, therefore, all three clays produced a small increase in the durability of asphalts II and III and a small decrease in asphalt I.

As previously mentioned, fly ash contains some components that are conductors of electricity;

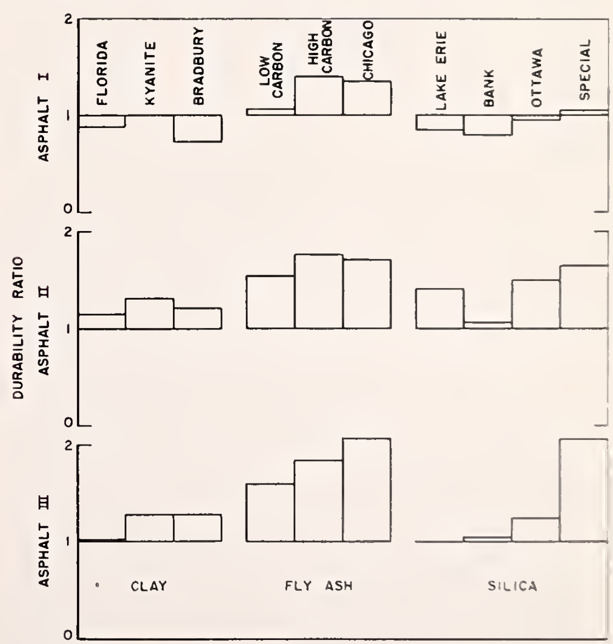

FIGURE 14. *Effects of selected materials on durability.*

Specimens containing fly ash were inspected only visually. See figure 9 legend.

therefore, the high-voltage probe could not be used to determine failures in coatings containing fly ash. All end-point determinations on coatings containing fly ash were made by visual inspection. Because the durability based on visual inspection is generally greater than that based on inspection with the high-voltage probe, the durability ratios reported in figure 14 must be considered only as approximations and the following discussion evaluated accordingly.

Because fly ash is a byproduct of the combustion of coal, its composition, both physical and chemical, varies with the source of the coal, the way it is burned, and the efficiency of the combustion. Therefore, its effect on the durability of asphalt roofing varies considerably. The three samples of fly ash tested varied from one produced under very efficient combustion conditions (2% carbon) to one with almost 23 percent of carbon. All three additives were very finely divided and had appreciable amounts of water-soluble material present; two had small amounts of free alkali. The low-carbon fly ash (actually 4.9% carbon), with no free alkali, apparently did not increase the durability of asphalt I, but seemed to increase that of II and III. However, the high-cabon fly ash apparently increased the durability of all of the asphalts. The Chicago fly ash also increased the durability of all three asphalts, slightly for asphalt I and appreciably for asphalt III.

Two of the samples of silica were in natural form, and the other two (Ottawa and Special) had been processed through grinding and sieving operations. Because the special silica had been used to polish plate glass, the corners and edges of the particles were rounded. In addition, small particles of glass were present. With the exception of this additive, which contained 3 percent of material soluble in water, the silicas were rather coarse and inert.

None of the silicas affected the durability of asphalt I; both of the processed silicas and Lake Erie silica increased the durability of asphalt II; and only the special silica appreciably increased the durability of asphalt III. The efficacy of the special silica is probably due to its fine degree of subdivision, being 82 percent less than 44 microns (325-mesh sieve).

5. Summary and Conclusions

A number of variables pertinent to the durability of 3 asphalts in combination with 14 mineral additives were investigated under accelerated exposure conditions. The results follow:

As the softening point of a blown asphalt coating is increased, its durability is decreased.

In additive coatings, the greater durability of the asphalt bases of the variable-base coatings compared with those of the constant-base coatings was not significant.

Concentrations of up to 60 percent of selected additives resulted in substantially increased durability in all three asphalts.

Thorough mixing of the mineral additives with the asphalts was of primary importance for uniform results.

The durability of all coatings increased with coating thickness in the range of 13 to 43 mils.

Although the additives produced effects of varying magnitude in the three asphalts, in general, mica, oyster shell, and blue-black slate were most effective in increasing durability; dolomite and fly ash of less influence; and silica and clay, least. In other words, minerals with platelike particle shapes were most effective in increasing durability.

Blue-black slate, dolomite, and silica were more effective at higher concentrations in thicker coatings, but fly ash was less effective. Satisfactory specimens with 50 or 60 percent of mica or with 60 percent of clay could not be produced with the facilities available.

The particle-size distributions of the mineral additives had significant effect on the durability of coatings.

Clays of three different types all produced nearly the same small effect on the durability of all three asphalts. Three samples of fly ash all produced some increase in durability. Only special silica, of the four silicas evaluated, produced a significant increase in durability (especially in asphalt III).

In general, the asphalt was the most important constituent in a stabilized coating in determining durability, but the durability was improved appreciably by the judicious incorporation of selected mineral additives.

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7. Appendix

7.1. Volume Composition

Several calculated quantities are reported in table 5 along with the measured data. The volume composition was calculated from the weight composition and the specific gravities of the components as follows:

$$\frac{100W_S}{W_A+W_S}=\%\ S\text{ (by weight)};\quad \frac{W_S}{d_S}=V_S;$$

$$\frac{W_A}{d_A}=V_A;\quad \frac{100V_S}{V_A+V_S}=\%\ S\text{ (by volume)},$$

where S=stabilizer, W=weight, V=volume, d=specific gravity, and the subscripts A=asphalt, S=stabilizer, and C=coating.

7.2. Specific Gravity

The specific gravity of each stabilized asphalt coating was calculated from the composition of the coating and the specific gravity of the individual components, as follows:

$$d_C=\frac{W_A+W_S}{V_A+V_S}=\frac{W_A+W_S}{\frac{W_A}{d_A}+\frac{W_S}{d_S}}.$$

In order to check these calculations, a number of specific-gravity measurements were actually made on some of the stabilized coatings. The calculated and observed specific gravities for 13 coatings are presented in table 9.

TABLE 9. *Specific gravities*

Asphalt	Composition	Specific gravity	
		Calculated	Observed
I	50% blue-black slate	1.51	1.50
	35% clay	1.30	1.30
	50% clay	1.47	1.48
	60% dolomite	1.66	1.66
	35% silica	1.30	1.29
	50% silica	1.47	1.47
II	50% blue-black slate	1.49	1.50
	50% fly ash	1.44	1.39
	35% silica	1.28	1.24
	50% silica	1.45	1.43
III	50% clay	1.47	1.47
	50% dolomite	1.50	1.49
	60% silica	1.62	1.62

7.3. Shatter Test on Coatings

Preparation of Specimens.

Several disks of asphalt 3 in. in diameter and 3/16 in. thick are cast, using a suitable glycerine-coated brass mold.

Test Method.

The test apparatus provides a means for dropping a constant weight from a variable and measured height on the cast disk and for recording the height of drop required to split the disk in one or more places, each split extending from the center to the edge of the specimen.

The apparatus consists of a 21-in. vertical brass tube, 1 in. in internal diameter; a solenoid sliding within the tube and adjustable to any height up to 21 in.; an electric connection to a standard 110-v line (either a-c or d-c) with the solenoid in series with a 60-w lamp and a switch for shorting the solenoid; a falling steel weight, 15/16 in. in diameter, and weighing ½ lb; and a stationary steel contact rod, 15/16 in. in diameter, weighing ½ lb, and having a hemispherical end contacting the asphalt.

In operation the specimen is brought to a temperature of about 40° F by submersion in a bath of ice and water for a period of not less than 1 hr. It is then placed under the vertical brass tube, being submerged in water at 40° F during the test, and the contact rod placed in the tube and in contact with the center of the specimen. The circuit is closed and the ½-lb weight raised to a height of 1 in. by means of the solenoid holding it. The solenoid is then shorted, the weight allowed to drop, and the specimen quickly examined for fracture. If it has not split, the weight is raised to 1½ in. and the drop again made. This procedure is repeated with ½-in. increments in height until the specimen fails. With subsequent specimens the weight should be released from a height 1 in. below the failing height of the first test. At least three determinations are made on each asphalt.

Results.

Failure is recorded when the specimen splits, in one or more places, from the center to the edge. Fractures that do not extend to the edge of the disk are ignored. The average height of drop required to break the specimen is recorded as its shatter resistance.

7.4. Water Absorption of Asphalt—Disk Method

Application.

This method is applicable to all asphalts having softening points (ring and ball) of 170° F or over.

Apparatus and Materials Required.

1. Brass mold 3/16-in.-thick, 3-in.-diameter hole.
2. Brass plate, 4 by 4 in.
3. Glycerine.
4. Hotplate.
5. 100 g of asphalt to be tested.
6. Pyrex-glass tray, 1½ in. deep, any convenient length and width.
7. Distilled water.

Preparation of Specimen.

Apply glycerine to the surfaces of the clean brass plate and mold, which will come in contact with the asphalt. Assemble the mold and place it on the brass plate. Heat the sample of asphalt to be tested until fluid and free from air bubbles. If the sample contains mineral matter, it should be stirred slowly with a piece of stiff wire to keep the matter properly suspended without incorporating air bubbles.

Pour a sufficient amount of the sample to fill the mold. The pouring must be done in a manner such that air bubbles are not occluded. The surface may be flamed lightly to remove a few that might form. Not more than 1/16 in. of the sample should show above the top of the mold. After the specimen has cooled thoroughly, remove it from the mold and wash it to remove the attached glycerine. Allow the specimen to dry and mark identification on both sides.

Procedure.

Weigh the specimen to 0.001 g and record the weight. Place the specimen in the glass tray and fill it with sufficient distilled water to submerge the specimen at least 1/4 in. Place the glass tray and specimen in a dark cabinet at room temperature.

Make periodic weighings in the following manner to determine the amount of water absorbed:

Remove the specimen from the water at the end of each specified period. Do not wipe, but blot both sides and edges carefully until each surface is as uniformly dry as possible. Weigh the specimen and record the weight. Return the specimen to the distilled water tray. Renew with fresh water at each weighing.

Compute the water absorbed, and convert the result to grams of water absorbed per square foot of asphalt surface exposed, including the edges of the disk.

This program was jointly designed and sponsored by the National Bureau of Standards and the Asphalt Roofing Industry Bureau. The author thanks the members of the Floor, Roof, and Wall Coverings Section for their advice and cooperation, and William H. Appleton, John Marchi, and Robert F. Jackson for their help in obtaining the original data. George Clarvoe, Johns-Manville Corp., supplied the water-absorption and shatter-resistance procedures.

WASHINGTON, March 28, 1956.

U. S. GOVERNMENT PRINTING OFFICE: 1956

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