

Rejuvenator Diffusion in Binder Film for Hot-Mix Recycled Asphalt Pavement

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In hot-mix recycling of bituminous mixes a rejuvenator is commonly used to restore the aged asphalt cement to a condition that resembles that of virgin asphalt cement. The type and amount of rejuvenator to be used are generally determined from a characterization of the recovered weathered binder. However, the extent to which the salvaged bitumen will be softened by the recycling agent during the hot-mix operation, and hence the characteristics of the rejuvenated binder and the resulting performance of the recycled pavement, has not been widely reported. An investigation was undertaken to determine the extent to which certain types of rejuvenators diffuse into the hardened asphalt film coating the aggregate and affect its properties during a specified period of time. A partial extraction technique that had the effect of dividing the asphalt film into microlayers was used. The binder recovered from each microlayer was characterized by means of consistency tests. This technique was used to evaluate the consistency distribution of the binder film around the aggregate in (a) the extracted mix containing recycled asphalt pavement (RAP) only; (b) the extracted mix containing RAP and a rejuvenator; and (c) the extracted mix containing RAP, virgin aggregate, and a rejuvenator.

Recycling operations have grown rapidly in recent years. This growth has resulted from increased awareness of the potential for cost savings and material conservation. More important, the effort put forth by equipment manufacturers has also increased. In recent years there have been rapid advances in pulverizers, millers, and hot-mix plants that facilitate recycling operations. With the increase in recycling operations has come an increased awareness that the recycled material must be properly characterized in order to ensure a quality pavement. If the recycled pavements show excessive deterioration, the cost and energy savings realized during construction may be lost through excessive maintenance. Initial indications are that a quality pavement is being constructed. However, these pavements have not been in service long enough to permit a definite judgment of their long-term performance.

The aged binder present in a recycled asphalt pavement (RAP) has physical properties that make it undesirable for reuse without modification. Materials have been developed to restore these old binders to a condition suitable for reuse. This concept is not new and has been the subject of a number of extensive studies during the last several years (1-3).

The common procedure for analyzing a recycled mixture involves (a) extraction of the asphalt cement, measurement of its properties, and selection of a proper amount and type of rejuvenator to restore the classification properties to a pre-selected level and (b) examination of the gradation of the

salvaged aggregate fraction to determine the amount and gradation of new aggregate that may be required (4-6).

The comparison of laboratory performance predictions for recycled materials and new construction requires that the binder content and consistency be the same. These two values represent the parameters that are influenced by the recycling operation. The consistency of the recycled asphalt cement should be similar to that of the virgin asphalt samples that represent new construction (7, 8).

It has been generally recognized that the effectiveness of a recycling agent is related to its uniform dispersion throughout the pavement binder. This is an important issue for recycling because changes in properties with time have been attributed to inadequate mixing of the reclaimed bitumen and rejuvenating agent during processing (9).

An investigation was undertaken to determine the extent to which certain types of rejuvenators diffuse into the hardened asphalt film coating the aggregate and affect its properties during a specified period of time. A partial extraction technique that had the effect of dividing the asphalt film into microlayers was used. The binder recovered from each microlayer was characterized by means of consistency tests.

This technique was used to evaluate the consistency distribution of the binder film around the aggregate in (a) the extracted mix containing RAP only; (b) the extracted mix containing RAP and a rejuvenator; and (c) the extracted mix containing RAP, virgin aggregate, and a rejuvenator.

CONCEPTUAL DISCUSSION OF DIFFUSION PROCESS

Common practices call for totally extracting and recovering the weathered asphalt and thoroughly mixing it with various percentages of the rejuvenator in order to determine the amount of rejuvenator required. The Asphalt Institute (10) has recommended an easy way to determine an initial value for this amount, if the viscosities at 140°F for the weathered asphalt and the rejuvenator and the target classification (consistency) of the resulting binder are known, by using a group of curves. Hence, it was assumed that (a) the rejuvenator is thoroughly mixed with the weathered asphalt and (b) the weathered asphalt film around the aggregate has a uniform consistency throughout its layer. Unfortunately this may not be the case in a typical hot-mix recycling project.

Carpenter and Wolosick (9) outlined the way in which a rejuvenator diffuses into the weathered asphalt film of a cold-mix recycled bituminous material (given that no virgin aggregate is used) as follows:

1. The rejuvenator forms a very low-viscosity layer that surrounds the aggregate that is coated with aged asphalt cement (Time Step 0).

2. The rejuvenator begins to penetrate into the asphalt cement layer, thereby decreasing the amount of raw rejuvenator that coats the particle and softening the old asphalt cement (Time Step 1).

3. No raw rejuvenator remains and penetration continues; the viscosity of the inner layer is lowered and gradually the viscosity of the outer layer is increased (Time Step 2).

4. Equilibrium is approached over the majority of the film of asphalt except right at the asphalt-aggregate interface, which may remain at a higher viscosity level (Time Steps 3 and 4).

The study also indicated that the time span between these four phases may be critical and hence the structural parameters may or may not be sufficiently developed to provide resistance to wheel loads during the initial portion of the life of the recycled pavement.

Careful selection and testing of recycling agents must be conducted to shorten this time span and cause structural parameters to develop more rapidly.

SAMPLING PLAN AND MATERIALS

Salvaged Asphalt Pavement Samples

A stockpile of representative salvaged bituminous pavement was obtained for laboratory evaluation. The material used was milled from US-52 (south of Indianapolis, Indiana) and randomly selected under the supervision of the Indiana Department of Highways for the purpose of this study. Sampling of the stockpile was also done at random to obtain statistically representative bituminous materials for this study.

Virgin Aggregate Samples

Crushed limestone and local sand were selected to represent the coarse and fine aggregate material for virgin aggregate samples. The selection was based on materials that are generally used in Indiana to produce hot-mix bituminous pavements.

Recycling Agents

Three types of recycling agents were selected for use in combination with the age-hardened salvaged bituminous binder. The selection was based on previous use of these agents in

other recycling techniques and the knowledge of their physical and chemical properties (3, 11). The following recycling agents were used: an AC-2.5 obtained from Amoco Oil Company, an AE-150 (Indiana-designated, high-float, medium-setting type of asphalt emulsion), and Mobilsol-30 (Type-101 oil, ASTM designation in an emulsified form) supplied by McConaughay, Inc.

TEST RESULTS AND ANALYSIS

Salvaged Material

Samples of the RAP were randomly chosen, reduced in size, and characterized. Asphalt extraction and recovery were conducted using ASTM D 2172-67 Method A and the Abson method (ASTM D 1856), respectively. The salvaged binder was characterized by penetration, softening point, and viscosity tests. The amount of asphalt present was determined, and the salvaged aggregate obtained from extraction was characterized by sieve analysis.

Tables 1 and 2 give the characteristics of the extracted hard asphalt and the gradation of salvaged aggregate, respectively. The values given represent an average of 10 samples. The Indiana specifications for No. 12 surface are also included in Table 2 for purposes of comparison and for use in future determination of the feasibility of using the salvaged aggregate in a high-quality hot surface mix. It should be noted that the amount of hardening that occurred in the old binder was relatively low compared with that in previous recycling projects. In addition, the sieve analysis of the salvaged aggregate indicated a gradation that is within the specification for No. 12 surface (Indiana DOT specifications). However, a lower percentage of material passing the $\frac{3}{8}$ -in. sieve was noted.

TABLE 1 CHARACTERISTICS OF EXTRACTED HARD ASPHALT

Test	Value
Penetration at 77°F, 100 g, 5 sec	28
Viscosity at 140°F (poises)	20 888
Kinematic viscosity at 275°F (cSt)	726
Softening point (°F)	137
Asphalt content (percentage of total weight)	6

TABLE 2 GRADATION OF SALVAGED AGGREGATE

	Sieve Size							
	$\frac{3}{8}$ in.	No. 4	No. 8	No. 16	No. 30	No. 50	No. 100	No. 200
Percentage passing	93	74	62	44	28	15	7.5	5
Indiana specification for No. 12 surface (%)	96-100	70-80	36-66	19-50	10-38	5-26	2-17	0-8

Recycling Agents (rejuvenators)

Three types of recycling agents were used to restore the old binder to the AC-20 classification range. Selection of the three types was based on their previous usage in recycling techniques other than hot-mix recycling. In addition, the AC-20 classification range was a target because AC-20 is widely used in producing high-quality hot-mix paving mixtures in Indiana.

The three types used were AC-2.5 ASTM designation; AE-150 Indiana designation; and Mobilsol-30, which is a commercial type. Table 3 gives the characteristics of Mobilsol-30.

TABLE 3 CHARACTERISTICS OF MOBISOL-30

Characteristic	Measurement
Asphaltenes (%)	0
Polar compounds (%)	8
Aromatics (%)	79
Saturates (%)	13
Solids in emulsified form (%)	66.7
Flash point (°F)	505
Kinematic viscosity at 140°F (cSt)	164

NOTE: Constituents were obtained using clay-gel analysis (ASTM D 2007-75). All characteristics except solids in emulsified form are those of residue.

Determination of the Amount of Rejuvenator

Asphalt Institute curves (10) were used to determine an initial value for the percentage of rejuvenator (AC-2.5 and AE-150) to be added to the old binder to restore the properties to the AC-20 range of classification. The curves suggest the percentage of rejuvenator on the basis of its viscosity at 140°F, the old binder viscosity at 140°F, and the required viscosity for the new rejuvenated binder at 140°F. The initial value for the percentage of Mobilsol-30 was chosen on the basis of previous recycling projects (3, 11).

A series of extraction and recovery tests was conducted to justify these initial values. Table 4 gives the characteristics of the salvaged asphalt, the rejuvenators, and the three rejuvenated binders.

TABLE 4 CHARACTERISTICS OF SALVAGED ASPHALT, REJUVENATORS, AND REJUVENATED BINDERS

Binder	Penetration	Viscosity at 140°F (poises)
Old asphalt	28	20 888
AC-2.5	200	292
AE-150 residue	200	270
40% old asphalt plus 60% AC-2.5	62	2 112
45% old asphalt plus 55% AE-150 residue	68	1 994
85% old asphalt, 15% Mobilsol-30 residue	69	1 974
AC-20 specification	60+	1 600–2 400

NOTE: Characteristics of Mobilsol-30 are given in Table 3.

Concept of Stage Extraction

A stage extraction technique was used to determine the extent to which the salvaged bitumen would be softened by the recycling agent during the laboratory-simulated hot-mixing operation. The method used (explained later in detail) divides the asphalt binder film coating the aggregate into four successively extracted microlayers. Each layer is then characterized separately to determine how much it is affected by the rejuvenator (in other words, to what extent does the rejuvenator diffuse into the old asphalt binder film and affect its properties). The same technique was used to investigate the consistency distribution of the binder film around the aggregate in (a) the extracted mix containing RAP only; (b) the extracted mix containing RAP and a rejuvenator; and (c) the extracted mix containing RAP, virgin aggregate, and a rejuvenator.

Method

The RAP sample was heated in an oven at 240°F for 30 min. The rejuvenators (AC-2.5, AE-150, and the Mobilsol-30) were heated in an oven at 180°F. The RAP, virgin aggregate, and one of the rejuvenators were mechanically hot mixed for 2 min to ensure proper mixing. The loose samples were stored in an oven for 15 hr at 140°F and directly extracted at different stages using Method A (ASTM D 2172). To fully extract the binder from a 1200-g sample of RAP, 1400 mL of trichloroethylene (TCE) solvent were used. Seven samples were used to obtain the proper amount of recovered asphalt from each microlayer for characterization. The solvent was applied to the mix in increments of 200, 200, 300, and 700 mL, respectively, to have the extracted asphalt film in four components. A 5-min soaking period was required between the successive increments. Asphalt binders were then recovered separately from each of the four fractions by using the Abson method (ASTM D 1856) and characterized by means of penetration and viscosity tests. For those mixes that called for the addition of virgin aggregate, the aggregate was heated at 240°F for 30 min before it was mechanically mixed with the RAP and the rejuvenator.

Results of Fractional or Stage Extraction Process

RAP Only

The RAP, without the addition of either virgin aggregate or recycling agent, was stage extracted for purposes of comparison. Stage extraction gave rise to some interesting results. Table 5 gives the penetration and viscosity (140°F) values of the reclaimed stage-extracted old binder. The original asphalt used was AC-20, and it can be observed that the outer microlayer of the asphalt was severely hardened by direct exposure to weathering actions. However, the second microlayer was less hardened and the third one was almost unchanged (compared with original AC-20 characteristics). On the other hand, the last microlayer at the binder-aggregate interface was slightly hardened, probably because of the tendency of limestone (commonly used in Indiana) to absorb light fractions of the binder. Figure 1 is a schematic diagram of these four

TABLE 5 TEST RESULTS ON RECLAIMED STAGE-EXTRACTED RAP

TCE Increment (mL)	Binder (% by weight)	Penetration	Viscosity at 140°F (poises)
200	55.5	24	24 000
200	26.5	33	15 000
300	11.2	65	2 500
700	6.8	57	3 300

NOTE: The results are averages of three replications, each conducted on seven samples of 1200 g; percentage of asphalt cement is 6 percent by weight of mix; and original asphalt was AC-20.

microlayers and the penetration and viscosity distribution along the old asphalt film.

Rejuvenator Effect, No Virgin Aggregate

It was decided in this portion of the study not to add any virgin aggregate in order to clarify the effect of the rejuvenator on the older binder during the laboratory-simulated hot-mix operation. Table 6 gives the penetration and viscosity (140°F) values of reclaimed stage-extracted treated binder. The results suggest that the three rejuvenators used (AC-2.5, AE-150, and Mobilsol-30) restored the two outer layers of the old binder to the AC-20 range of specification to almost the same extent and that the other two inner layers were almost unaffected. However, these two layers were not significantly hardened as

previously indicated by the results of stage extracting the RAP only. Figures 2-4 are schematic diagrams of the four layers and the penetration and viscosity distributions along the treated asphalt film.

Effect of Rejuvenator in Combination with Virgin Aggregate

Because a hot-mix recycling operation generally requires the use of virgin aggregate, it was imperative to include the effect of rejuvenators on old binder in the presence of virgin aggregate. The amount and gradation of aggregate added were selected to keep the treated binder content at 6 percent by weight of mix (same as binder content in RAP) and the total aggregate fraction gradation within the No. 12 surface range of specification, which is commonly used in Indiana for producing hot-mix bituminous pavement. These two requirements were met by using 60, 55, and 15 percent of virgin aggregate by total aggregate weight for the mixes treated with AC-2.5, AE-150, and Mobilsol-30, respectively. The gradation used was the specification midpoint of No. 12 surface given in Table 2. The heated rejuvenator (AC-2.5, AE-150, or Mobilsol-30) was added during the mixing of the heated virgin aggregate-RAP combination except for the Mobilsol-30 that was mixed with the RAP directly before the addition of hot virgin aggregates. Table 7 gives the penetration and viscosity (140°F) values of the reclaimed stage-extracted binder. The results suggest that both rejuvenators (AC-2.5 and Mobilsol-30) were attracted to the old asphalt binder, softened it, and then covered the virgin aggregate. However, this was not the case for the AE-150; its

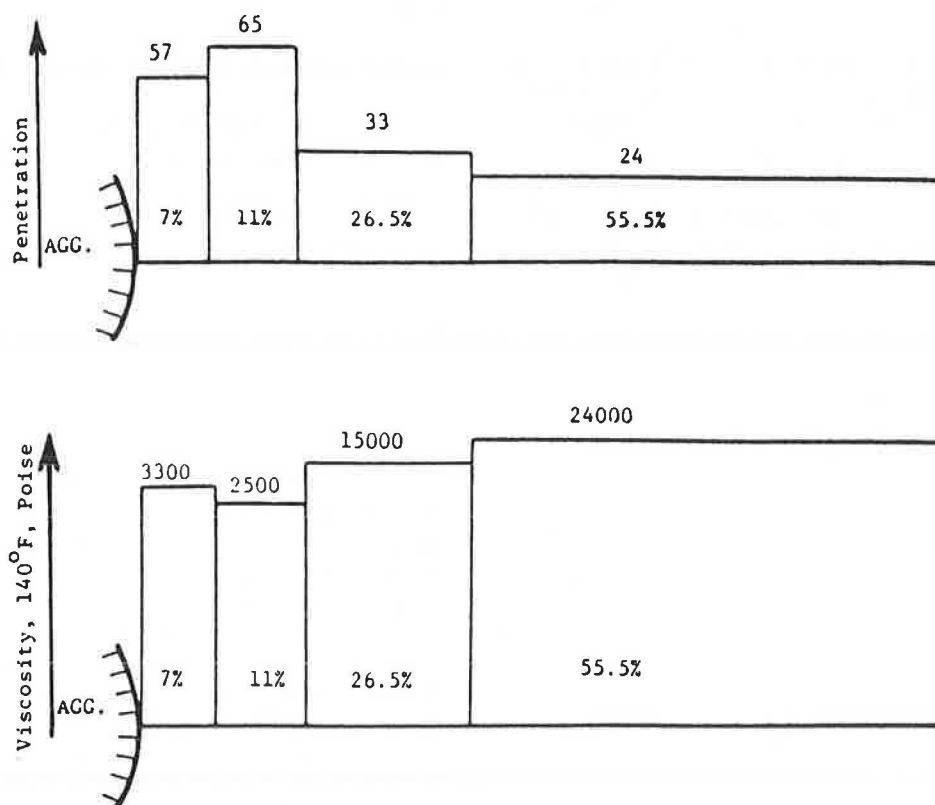


FIGURE 1 Consistency distribution throughout the binder film, RAP only.

TABLE 6 TEST RESULTS ON RECLAIMED, STAGE-EXTRACTED, TREATED BINDER—NO VIRGIN AGGREGATE

Binder	TCE Increment (mL)	Binder (% by weight)	Penetration	Viscosity at 140°F (poises)
60% AC-2.5, 40% old asphalt	200	67.5	67	1674
	200	21.5	68	1880
	300	7	59	2394
	700	4	50	3000
55% AE-150 residue, 45% old asphalt	200	69	75	1683
	200	16.5	70	2010
	300	8.5	62	2290
	700	6	49	3020
15% Mobilsol-30 residue, 85% old asphalt	200	71	75	1864
	200	18	69	1980
	300	6	63	2040
	700	4	48	3152

NOTE: It was not possible to keep the percentage of treated binder at 6 percent (original percentage in RAP) because no virgin aggregate was added. Treated binder contents by weight of mix were 13.75, 12.5, and 7 percent for the AC-2.5, AE-150, and Mobilsol-30, respectively.

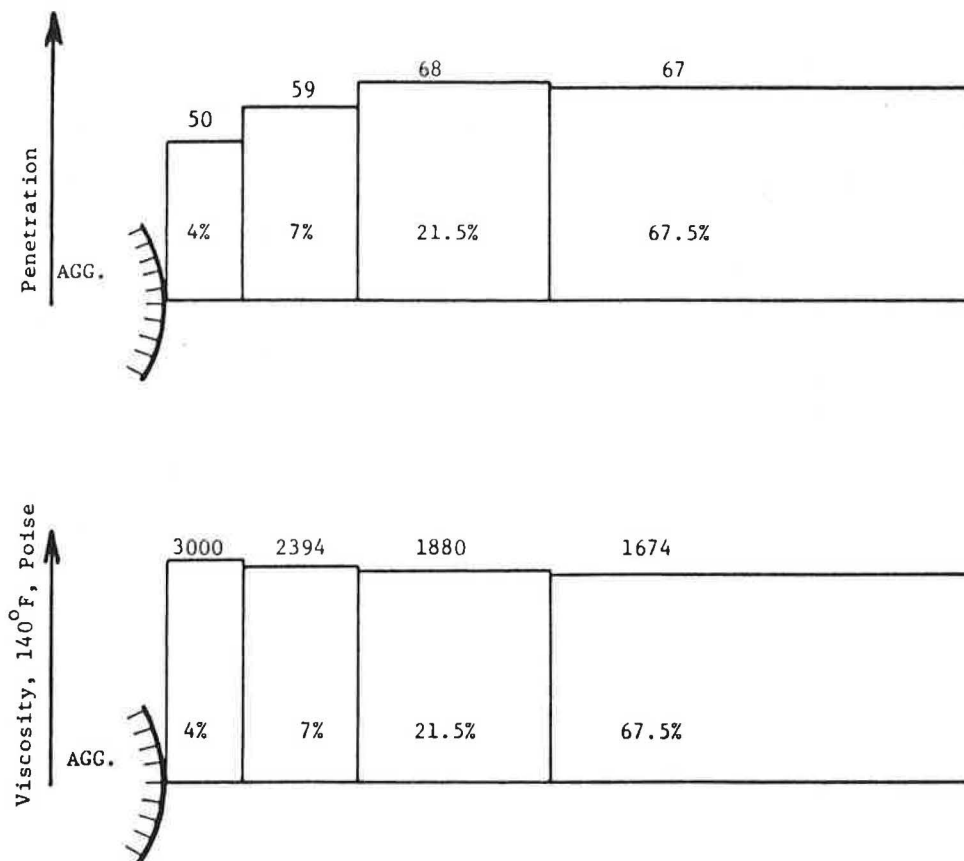


FIGURE 2 Consistency distribution throughout the binder film (RAP plus AC-2.5), no virgin aggregate.

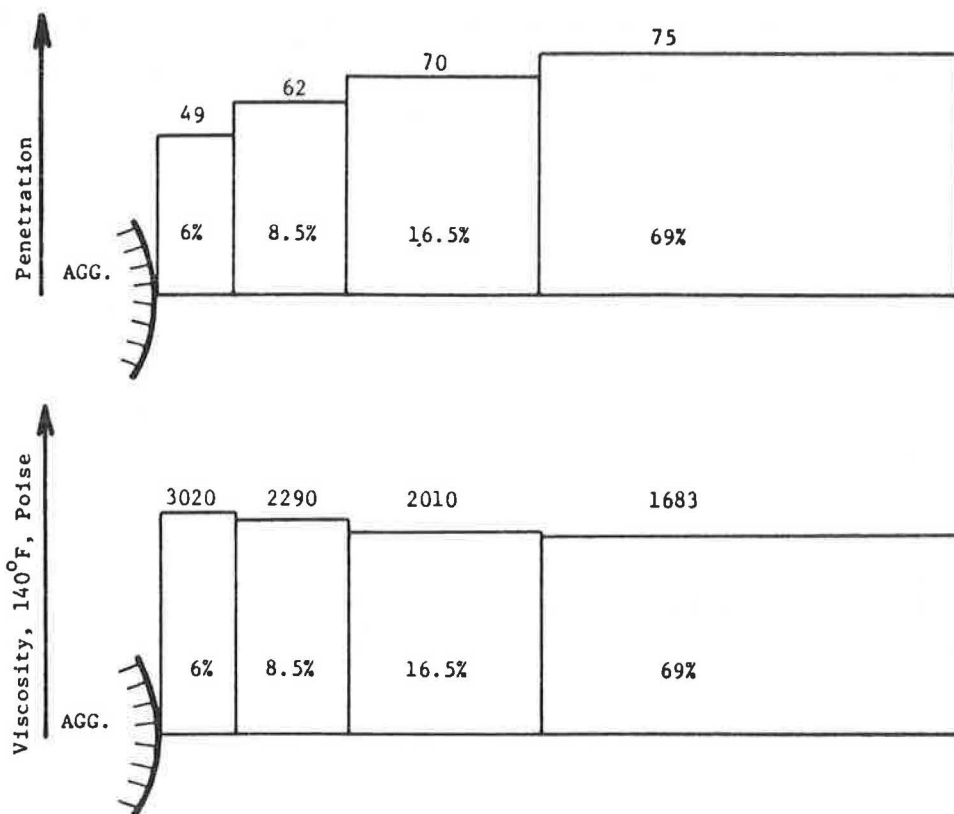


FIGURE 3 Consistency distribution throughout the binder film (RAP plus AE-150), no virgin aggregate.

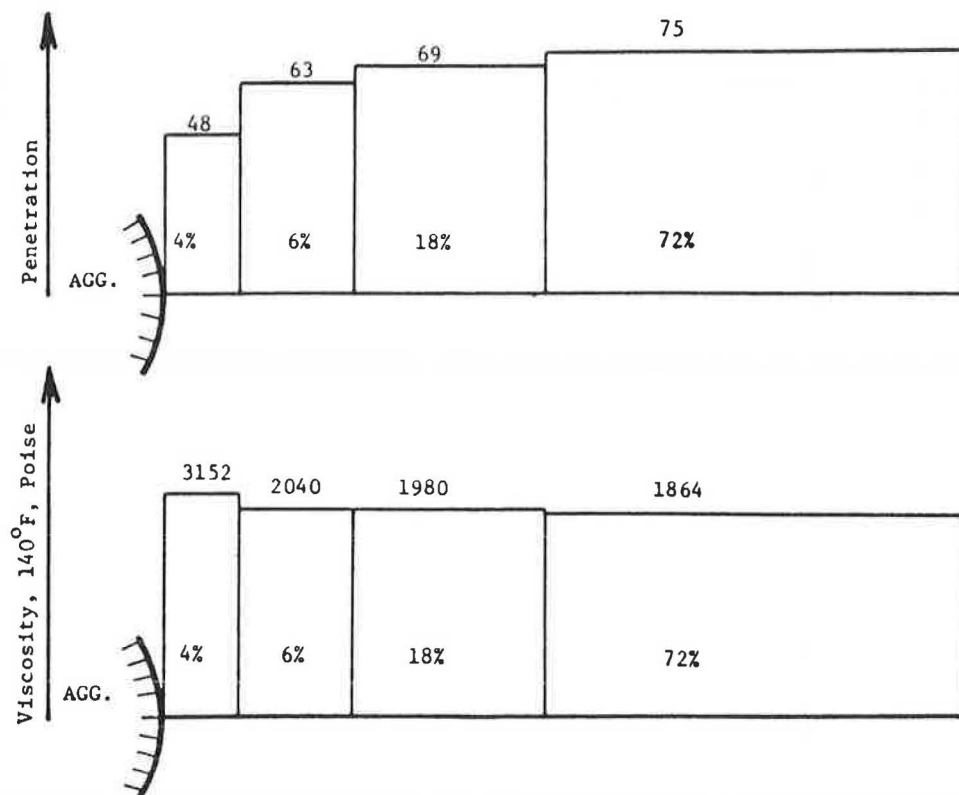


FIGURE 4 Consistency distribution throughout the binder film (RAP plus Mobilsol-30), no virgin aggregate.

TABLE 7 TEST RESULTS ON RECLAIMED, STAGE-EXTRACTED, TREATED BINDER—VIRGIN AGGREGATE USED

Binder	TCE Increment (mL)	Binder (% by weight)	Penetration	Viscosity at 140°F (poises)
60% AC-2.5, 40% old asphalt	200	72	60	2100
	200	19	51	2892
	200	19	51	2892
	300	5.5	52	2470
	700	3.5	130	809
55% AE-150 residue, 45% old asphalt	200	71	70	1972
	200	19	67	1734
	300	6	60	2424
	700	4	50	3616
15% Mobilsol-30 residue, 85% old asphalt	200	74	73	2049
	200	17.5	80	1664
	300	5.5	90	1260
	700	3.5	100	1240

NOTE: 6 percent binder was used in all mixes.

results indicated a consistency gradient that was almost identical to the one for no virgin aggregate.

In general, the consistency of the four microlayers of the treated binder (representing the whole film of asphalt coating the aggregate) characterized by penetration and viscosity (at 140°F) results was similar to that of AC-20, which indicates that the rejuvenators (AC-2.5, AE-150, and Mobilsol-30) diffused well through the hard asphalt film and restored its properties to the AC-20 specification range. Figures 5–7 are schematic diagrams of the four layers and the penetration and viscosity distributions along the treated asphalt film.

Development of Microlayers and Theoretical Implications

It has been observed that the penetration and viscosity (at 140°F) values for the four microlayers of asphalt film extracted and reclaimed from all samples used in this study are logarithmically additive. In other words, if $\text{Log}_{10}A$, $\text{Log}_{10}B$, $\text{Log}_{10}C$, and $\text{Log}_{10}D$ represent the logarithmic values for the penetration or the viscosity (140°F) of the four microlayers and $\text{Log}_{10}T$ represents that value for the whole asphalt film, it was observed that

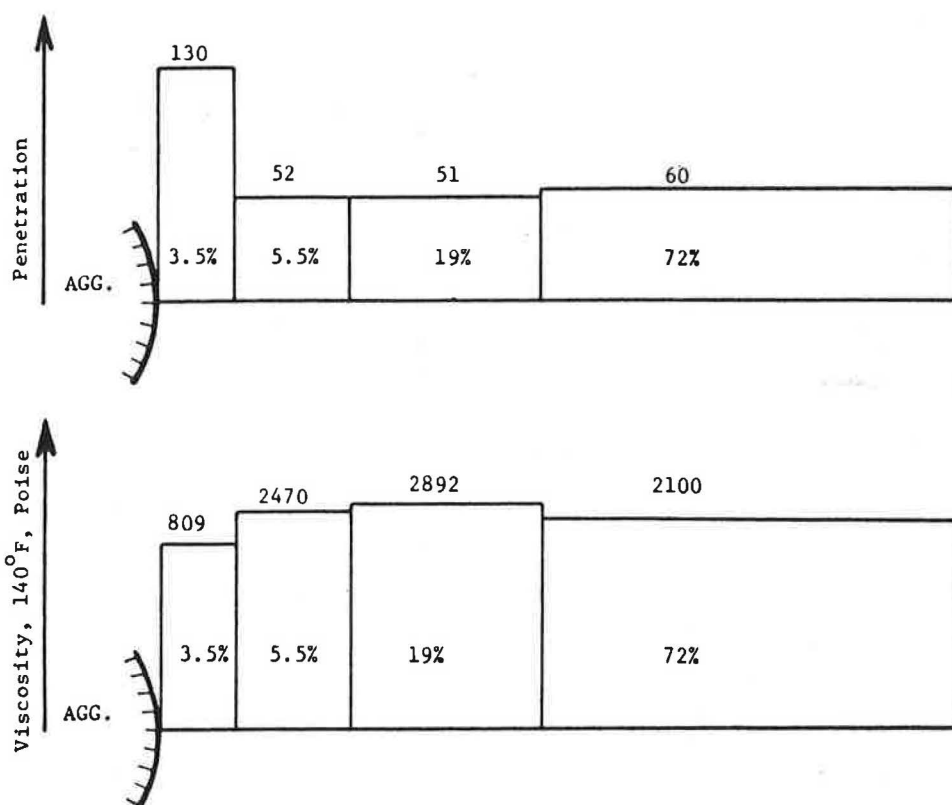


FIGURE 5 Consistency distribution throughout the binder film (RAP plus AC-2.5), with virgin aggregate.

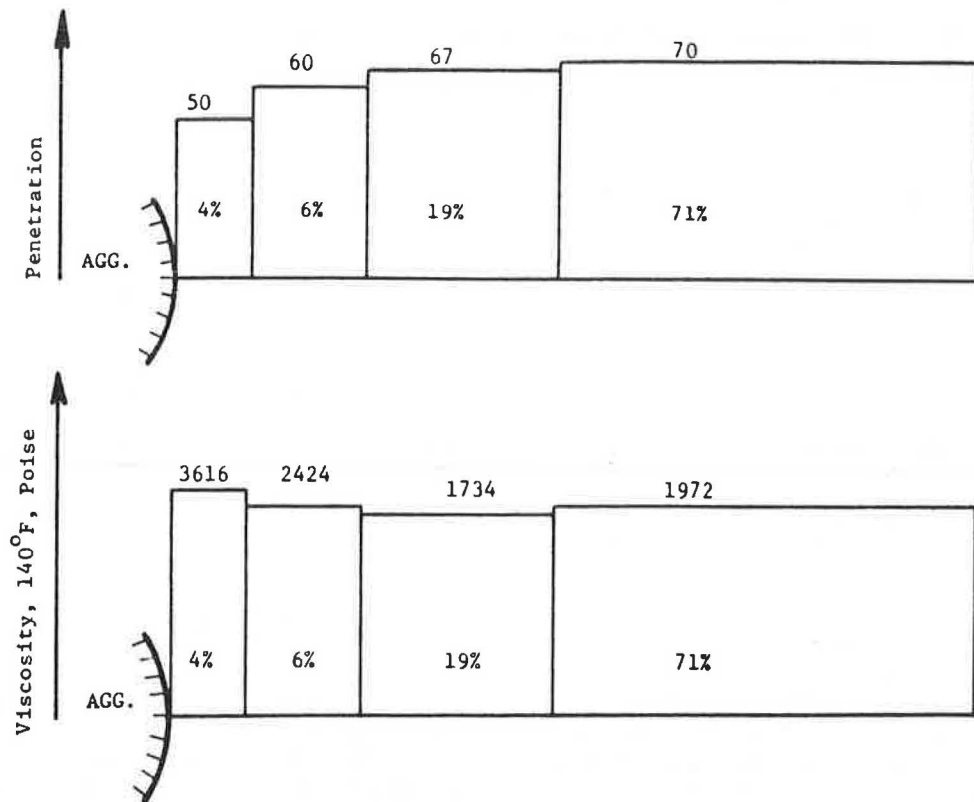


FIGURE 6 Consistency distribution throughout the binder film (RAP plus AE-150), with virgin aggregate.

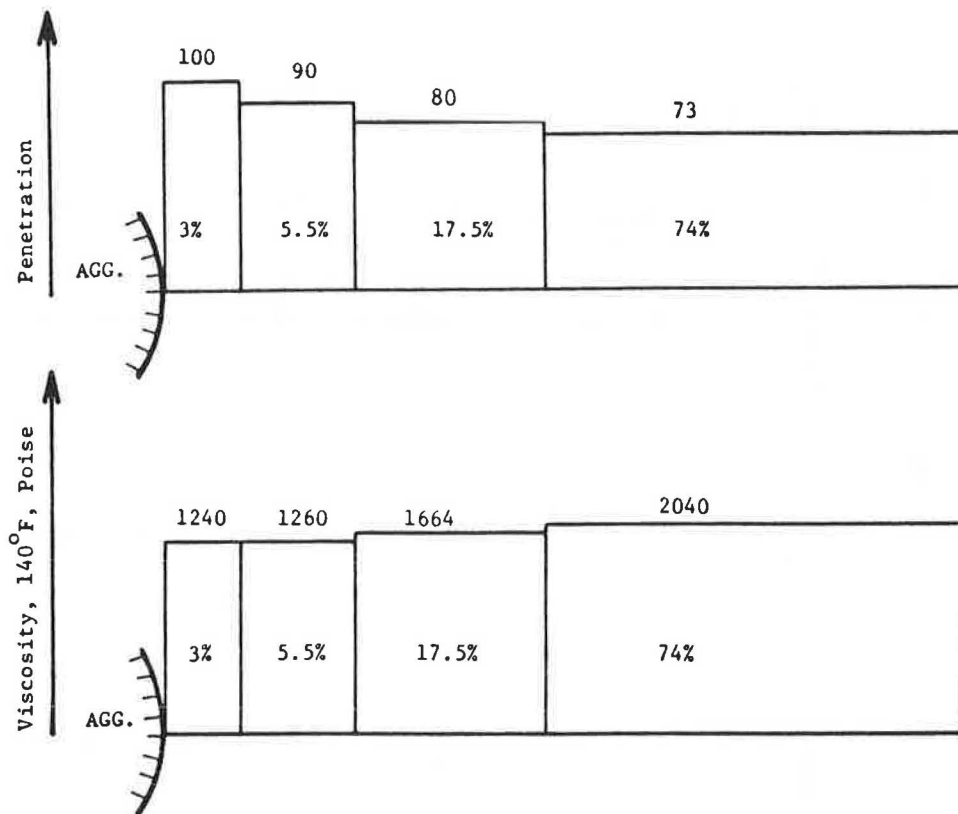


FIGURE 7 Consistency distribution throughout the binder film (RAP plus Mobilsol-30), with virgin aggregate.

$$\log_{10} T = P_1 \log_{10} A + P_2 \log_{10} B + P_3 \log_{10} C + P_4 \log_{10} D$$

where P_1 , P_2 , P_3 , and P_4 are the percentages by weight of the four microlayers. Taking the RAP rejuvenated by Mobilsol-30 as an example, values for P_1 , P_2 , P_3 , and P_4 are 0.72, 0.18, 0.06, and 0.04 (Table 6) and viscosity values A, B, C, and D are 1864, 1980, 2040, and 3152 poises (Table 6). If these values are substituted in the equation, T is 1935, which is close to its test value of 1974 (Table 4).

Because the proof of this relationship would entail a research effort that is beyond the magnitude of this study, it was necessary to include it only as an observation. However, this relationship can be used to develop the results for the four microlayers obtained in this study into 10 microlayers or more. Figures 8 and 9 show the relationship between the percentage of binder extracted and the penetration or the viscosity (at 140°F) of the extracted old binder (RAP) and the RAP treated with AC-2.5, AE-150, and Mobilsol-30 when virgin aggregate is used. It would be possible to predict the penetration or viscosity value of the last 5 percent microlayer (at the binder-aggregate interface) by obtaining the value of viscosity or penetration at 95 percent binder extracted (A) and at 100 percent binder extracted (T) and substituting these values in the previous expression. Taking the untreated RAP as an example, the A penetration value is 27 at 95 percent binder extracted

(Figure 8), and the T penetration value at 100 percent binder extracted is 28 (Figure 8). P_1 and P_2 are 0.95 and 0.05. Substituting these values in the equation yields a B penetration value of 56, which is close to the test value of 57 given in Table 5.

SUMMARY OF RESULTS

The salvaged material used in this study was obtained from US-52 in Indiana. The recycling agents applied to the salvaged material were AC-2.5, AE-150, and a commercial type (Mobilsol-30). Stage extraction was conducted using Method A (ASTM D-2172). Analysis and evaluation of the test data revealed some new aspects of hot-mix recycled bituminous pavement. However, it is imperative to indicate that the significant results obtained may be limited to the materials used and the test conditions applied in this study.

The main findings can be summarized as follows:

1. Stage extraction of hard asphalt film for the RAP indicated a nonuniform consistency distribution. The outer microlayer of the binder film was severely hardened by direct exposure to weathering actions. However, the second microlayer was less hardened and the third layer appeared to retain its original consistency. The slight hardening of the inner microlayer (at the asphalt-aggregate interface) may be due to the

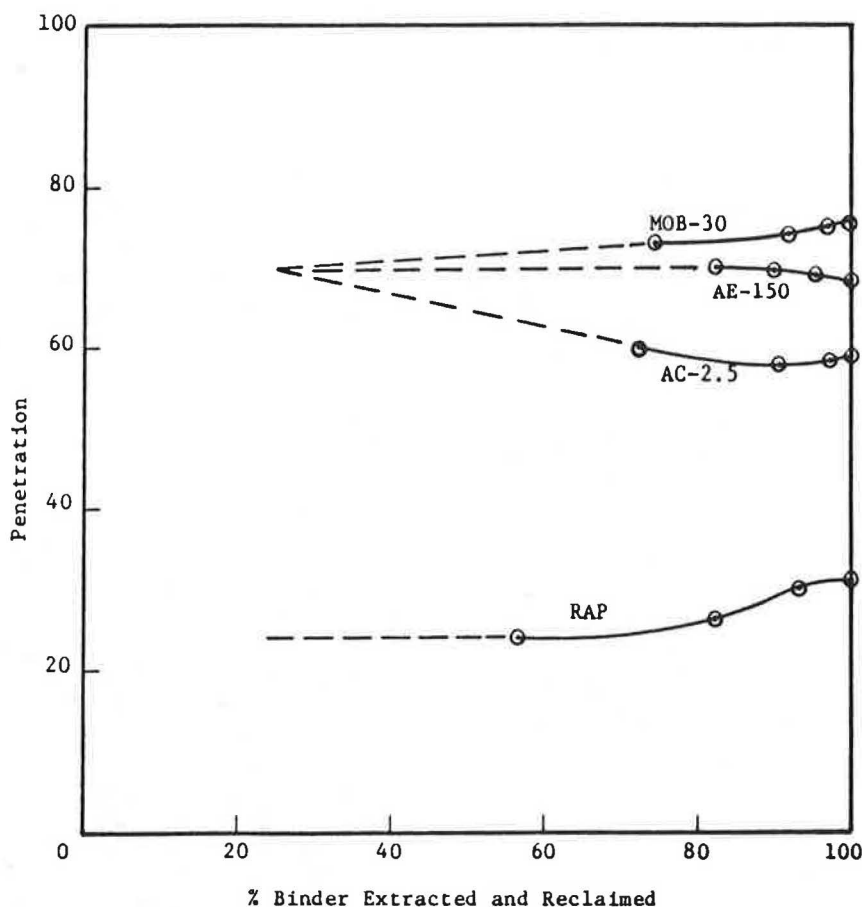


FIGURE 8 Relationship between percentage of extracted and reclaimed binder and penetration (with virgin aggregate).

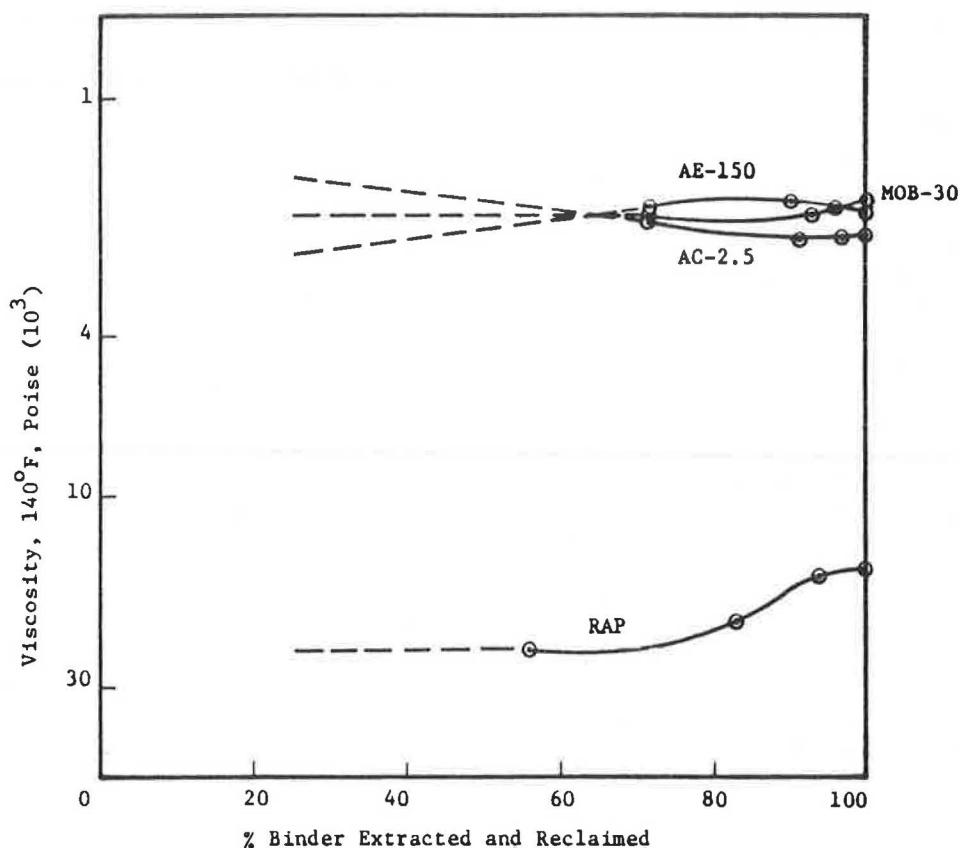


FIGURE 9 Relationship between percentage of extracted and reclaimed binder and viscosity (with virgin aggregate).

tendency of limestone (commonly used in Indiana) to absorb light fractions.

2. Stage extraction of the binder rejuvenated by AC-2.5, AE-150, or Mobilsol-30 without the addition of virgin aggregate indicated that the rejuvenators are most effective at softening the hardened binder on the outer two microlayers of the asphalt film.

3. Stage extraction of rejuvenated binders in the presence of virgin aggregate indicated variable trends in the consistency distribution of the asphalt film on the aggregate. The attraction of the low-viscosity rapidly softened binder to the virgin aggregate may have been the cause of these inconsistent trends.

4. In general, all three rejuvenators were successful in restoring the old hardened asphalt film to the AC-20 specification range.

5. The three recycling agents used displayed good efficiency in diffusing through the hard asphalt film as indicated by stage extraction test results after 15 hr.

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