Evaluating Recycled Asphalt Binders by the Thin-Film Oven Test

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Evaluation of long-term performance and identification of nonhomogeneity, incompatibility, and hardening rate of hot-mix–recycled bituminous pavement are described. The effects of artificial laboratory aging on a virgin binder (AC–20) and three other—extracted and recovered—rejuvenated binders were determined. Laboratory aging was achieved by means of the thin-film oven test (TFOT). Analysis and evaluation of the test data revealed some important aspects of hot-mix recycling. The TFOT was identified as a potential added criterion in identifying recycling agents that tend to cause a high rate of hardening, nonhomogeneity, and noncompatibility problems in recycled binders meeting the standard specifications of a virgin binder. This identification can be obtained by visually inspecting the residues after the TFOT and classifying their consistency using penetration and viscosity tests.

During the last 15 years, recycling of asphalt pavement has become an increasingly attractive rehabilitation alternative. The decreasing supply of locally available quality aggregate in some areas, growing concern over waste disposal, geometric difficulties of successive pavement maintenances, and unstable cost of asphalt cement and fuel have made recycling an environmentally and economically attractive alternative.

Pavement recycling is a technique in which hardened, deteriorated old pavement can be processed and reused. The fundamental concept lies in softening the old binder fraction by the addition of softening agents so that the original properties of that old binder are restored (*1*).

Millions of tons of hot-mix–recycled pavements have been used in the present highway systems. Recycled-mixture designs have been prepared using the test methods and criteria historically used for conventional asphalt concrete pavements. Initial results indicate that these methods and criteria are generally acceptable. However, there is still a need for assurance that long-term aging, with a potentially higher rate of hardening, and the effects of weathering, homogeneity, and compatibility on the mechanical and structural properties of the pavement are not problems (*2*).

Data collected from previous recycling projects indicated that the rejuvenated binders in a recycled mix may have a higher rate of hardening and be more susceptible to temperature than the virgin asphalt cement used in a conventional mix (*3*). Problems caused by incompatibility were also reported when field-aged asphalts were blended with recycling agents (*4*). The type of rejuvenating agent used and the nature of the aged asphalt could have a role in these observations.

The effectiveness of a recycling agent is a function of its uniform dispersion throughout the pavement binder. This issue is important in the process of recycling, because changes in properties with time have been attributed to the fact that old binder and rejuvenating agents may not have been thoroughly mixed (*5*). Some research efforts have been conducted to establish the ability of mixing operations to produce a homogeneous mixture (*4–6*). The specifications for recycling agents proposed by the Pacific Coast User-Producer Conference (June 1980) appear to be the best currently available to select the proper type of recycling agent for a specific project. However, these specifications alone cannot adequately identify incompatibility problems. Additional tests and criteria need to be developed to identify incompatibility (*2,7,8*).

If blends of aged asphalt and recycling agents are evaluated to ensure that they meet current ASTM or AASHTO specifications for virgin asphalt cements, viscosity and penetration measurements on samples of these blends after a thin-film oven test (TFOT) can help identify potential incompatibility (*2,7,8*).

To establish the time-temperature effect that three types of recycling agents have on aged asphalt binder, the TFOT was used to identify the rate of hardening in a weathered asphalt after it was treated with the following agents: AC–2.5 (ASTM designation), AE–150 (Indiana designation), and Mobilsol-30 (a commercial type). Samples of virgin asphalt (AC–20) were used for comparison purposes. The various samples were subjected to the oven exposure for three specific periods, after which the residues were classified by means of penetration and viscosity tests.

EXPERIMENTAL DESIGN

The experimental part of this study was statistically designed in advance to investigate the effects of binder type and time of oven exposure.

1. Binder Type (*B*). This factor consisted of four levels, each representing a specific type of bituminous binder. The first type was AC–20 (typically used in Indiana for producing hot-mix bituminous pavement). The second, third, and fourth levels were the recycled asphalt pavement (RAP) restored to an AC–20 specification range using the recycling agents AC–2.5, AE–150, and Mobilsol-30, respectively.
2. Time of Oven Exposure (*T*). The four levels of this factor were the periods of time over which a specific type of binder was subjected to oven exposure in the TFOT (ASTM D1754).

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The time spans were chosen to be 0 (no oven exposure), 2, 5 (the standard time), and 10 hr.

These two factors were selected for evaluation of the performance of three specific rejuvenated binders. These binders were compared with a virgin binder (AC–20) under the same conditions of time and temperature. Rejuvenated binders with higher hardening rates and greater sensitivity to long-term weathering actions (simulated artificially by the TFOT) than a virgin binder are believed to create cracking and compatibility problems when used in the field. If so, a recycled pavement would experience a more rapid deterioration rate than a conventional virgin mix.

The following mathematical model was used to introduce the data:

$$PV = M + B_i + T_j + BT_{ij} + E_{ijk} \quad (1)$$

where

- PV = penetration or viscosity response for any sample,
- M = overall mean,
- B_i = binder-type effect (fixed and qualitative),
- T_j = time of oven exposure during the TFOT (fixed and quantitative),
- BT_{ij} = interaction effect, and
- E_{ijk} = experimental error (random).

Indices i, j, k run over the number of samples.

A completely randomized design with two factors each having four levels (i.e., 16 treatment combinations) was applied (*9*). Three replications were prepared for each treatment combination, so that statistical significance could be detected if it existed, making the total number of samples 48

SAMPLING PLAN AND MATERIALS

Salvaged Materials

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. Samples were selected at random from the laboratory-created stockpile to obtain statistically representative bituminous materials.

Virgin Materials

Crushed limestone and local sand were selected to represent the coarse and fine aggregate material for virgin aggregate samples. An AC–20 from AMOCO Oil Company was chosen to produce conventional mixes. The selection was based on materials in the state of Indiana that are generally used to produce hot-mix bituminous pavements.

Three types of recycling agents were selected for use with the age-hardened salvaged bituminous binder. The agents were selected because they had been previously used in other recycling techniques and because their physical and chemical properties were known (*5,10*). The following recycling agents were used: an AC–2.5 obtained from AMOCO Oil Company; an AE–150; and Mobilsol-30, supplied by McConnaughay, Inc.

TEST RESULTS AND ANALYSIS

Salvaged Materials

Samples of RAP were randomly chosen, reduced in size, and characterized. Asphalt extraction and recovery were conducted using ASTM D2172-67, Method A, and the Abson method (ASTM D1856); respectively. The salvaged binder was characterized by means of penetration, softening point, and viscosity tests. Amount of asphalt present was determined, and the salvaged aggregate obtained from extraction was characterized by sieve analysis.

Tables 1 and 2 present the characteristics of the extracted hard asphalt and the gradation of salvaged aggregate, respectively. The values were an average of 10 samples. The Indiana specifications for No. 12 surface were also included in Table 2 for comparison purposes and for future determination of the feasibility of using salvaged aggregate in a high-quality, hot-surface mix. The amount of hardening that occurred in the old binder was not significant when compared to previous recycling projects. In addition, the sieve analysis of the salvaged aggregate indicated a gradation within the specification for No. 12 surface (Indiana Department of Transportation Specifications), except for a small margin at No. ⅜ sieve.

Recycling Agents (Rejuvenators)

Three types of recycling agents were used to restore the old binder to the AC–20 classification range. Selection of the agents was based on their previous use in recycling techniques other than hot-mix recycling. The AC–20 classification range was selected 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 (a commercial type). Table 3 presents the penetration and viscosity values of AC–20, AC–2.5, and AE–150 residue; Table 4 presents the characteristics of Mobilsol-30.

TABLE 1 CHARACTERISTICS OF EXTRACTED HARD ASPHALT

Test	Value*
Penetration, 77°F, 100 gm, 5 sec.	28
Viscosity, 140°F, Poises	20,888
Kinematic visc., 275°F, c. st	726
Softening Point, °F	137
Asphalt Content (Total wt.)	6%

*Average of 10 samples

TABLE 2 GRADATION OF SALVAGED AGGREGATE

Sieve size	3/8	#4	#8	#16	#30	#50	#100	#200
% Passing*	93	78	62	44	28	15	7.5	5
IND. spec. for #12 surface	96-100	70-80	36-66	19-50	10-38	5-26	2-17	0-8

*Average of 10 samples

TABLE 3 CHARACTERISTICS OF AC-20, AC-2.5, AND AE-150

Asphalt	Penetration	Viscosity, 140°F, Poise
AC-20	65	1890
AC-2.5	200	292
AE-150 Residue	200	270

TABLE 4 CHARACTERISTICS OF MOBILSOL-30

Percent Asphaltenes*	0
Percent Polar Compounds*	8
Percent Aromatics*	79
Percent Saturates*	13
Percent solids in Emulsified Form	66.7
Flash Point*	505
Kinematic Viscosity* at 140°F, c.st.	164

*Properties of Residue

Determination of the Amount of Rejuvenator

Asphalt Institute curves (*11*) 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 an AC-20 range of classification. The curves suggest the rejuvenator percentage on the basis of its viscosity at 140°F, the old binder's 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 (*5,10*).

A series of extraction and recovery tests were conducted to justify these initial values. Table 5 presents the characteristics of salvaged asphalt and the three rejuvenated binders.

Preparation of Samples for TFOTs

RAP samples and the virgin aggregate were heated in an oven at 240°F for 30 min. The rejuvenators were heated in an oven at 180°F when they were used. RAP, virgin aggregate, and one of the rejuvenators were mechanically hot-mixed for 2 min. The amount and gradation of virgin aggregate were selected in such a way that the resulting binder content was 6 percent of the total weight of mix (the original binder content in the RAP) and the resulting aggregate gradation was within the Indiana specifications for No. 12 surface (typically used for producing hot-mix bituminous surface mix). The loose samples were stored in an oven for 15 hr at 140°F for curing and were directly extracted using Method A of ASTM D2172. Asphalt binders were then recovered separately using the Abson Method (ASTM D1856).

Actual field conditions were simulated by adding virgin aggregate to the RAP followed by the rejuvenator; however, Mobilsol-30 was added before the virgin aggregate. In other words, the salvaged binder was treated before the extraction and recovery process was conducted.

Results and Analysis of the TFOT

Penetration and viscosity values at 140°F were obtained on recovered, rejuvenated asphalt samples (0 hr on TFOT) and on residues after 2, 5 (the standard time), and 10 hr in the thin-film oven. Identical conditions were applied to the AC-

TABLE 5 CHARACTERISTICS OF SALVAGED ASPHALT AND REJUVENATED BINDERS

Binder	Penetration	Viscosity, 140°F*
Old Asphalt	28	20,888
40% Old Asphalt +60% AC-2.5	62	2112
45% Old Asphalt +55% AE-150 Residue	68	1994
85% Old Asphalt 15% Mobilsol-30 Residue	69	1974
AC-20 spec.	60+	1600-2400

*Average of 10 samples

Note: Mobilsol-30 characteristics are given in Table 4.

20, and its penetration and viscosity values at 140°F were obtained for comparison purposes.

Tables 6 and 7 present average penetration and viscosity values (at 140°F) of the three replications at each treatment combination (binder type and time of oven exposure). Significant differences were obtained when conducting a two-way analysis of variance (ANOVA) on the data presented in Tables 6 and 7. Increasing the time of oven exposure resulted in a significant drop in penetration and a significant increase in viscosity for all the samples (which was expected). However, these changes varied significantly, depending on the binder type. The RAP rejuvenated by the AE–150 experienced the highest hardening rate, followed by the virgin AC–20, the RAP rejuvenated by AC–2.5, and the RAP rejuvenated by the Mobilsol-30. In addition, after the TFOT on RAP samples rejuvenated by AE–150, an easily removed, brittle skin was formed on the top of the sample in the pan. This was true for all the samples of RAP modified by AE–150, even those exposed for only 2 hr in the oven.

In general, these data indicate that using AE–150 as a recycling agent for hot-mix–recycled bituminous pavements may result in incompatibility, nonhomogeneity, and a high rate of hardening problems. Test results for the AC–2.5 and the Mobilsol-30 encourage their use as recycling agents. The RAP rejuvenated by AC–2.5 or Mobilsol-30 had a hardening rate slightly slower than that of the virgin AC–20.

TABLE 6 PENETRATION VALUES OF BINDER AFTER DIFFERENT TIMES OF OVEN EXPOSURE

	Time of Oven Exposure During TFOT			
Binder Type	Zero	2 hours	5 hours	10 hours
AC-20	65	43	33	25
RAP+AC-2.5	64	48	38	29
RAP+AE-150	62	34	26	18
RAP+Msol-30	64	50	43	33

Note: Values included are average of 3 replications.

TABLE 7 VISCOSITY VALUES (AT 140°F) OF BINDERS AFTER DIFFERENT TIMES OF OVEN EXPOSURE

	Time of Oven Exposure During TFOT			
Binder Type	Zero	2 hours	5 hours	10 hours
AC-20	1890	3920	8780	25,870
RAP+AC-2.5	1980	3410	7890	15,080
RAP+AE-150	2150	9770	18,740	62,340
RAP+Mobilsol-30	2220	4680	7490	14,880

Note: Values included are averages of 3 replications.

Relationship Between the Time of Oven Exposure and Consistency of Binder

Regression analyses were conducted to establish statistical relationships between the time of oven exposure during the TFOT (0, 2.5, and 10 hr) and the consistency of binder (AC–20, RAP + AC–2.5, RAP + AE–150, or RAP + Mobilsol-30) represented by the penetration and the viscosity at 140°F. Tables 8 and 9 present the regression equations for penetration and viscosity, respectively. The symbol x was used to represent the time spent in the TFOT. The regression parameter multiplied by x can be used as an indicator for the tendency of the rejuvenated binder to have a high hardening rate and hence create short-term aging and possible incompatibility and nonhomogeneity problems.

Figures 1 and 2 show graphical representations of the statistical relationships for penetration and viscosity at 140°F versus the time of oven exposure.

SUMMARY OF RESULTS

The salvaged material was obtained from US-52, Indiana. The recycling agents applied to the salvaged material were AC–2.5, AE–150, and a commercial type (Mobilsol-30). The virgin AC–20 used for comparison purposes was obtained from

TABLE 8 REGRESSION EQUATIONS FOR THE RELATIONSHIP BETWEEN PENETRATION OF BINDER AND TIME OF OVEN EXPOSURE DURING TFOT

Binder Type	Equation	R^2
AC-20	$\text{Penetration} = \frac{100}{\sqrt{2.45+1.35x}}$	0.999
RAP+AC-2.5	$\text{Penetration} = \frac{100}{\sqrt{2.45+0.95x}}$	0.999
RAP+AE-150	$\text{Penetration} = \frac{100}{\sqrt{2.45+2.45x}}$	0.993
RAP+Mobilsol-30	$\text{Penetration} = \frac{100}{\sqrt{2.45+0.75x}}$	0.993

NOTES: "x" is the time of oven exposure during the TFOT. R^2 is the coefficient of determination.

TABLE 9 REGRESSION EQUATIONS FOR THE RELATIONSHIP BETWEEN VISCOSITY (AT 140°F) AND TIME OF OVEN EXPOSURE DURING TFOT

Binder Type	Equation	R^2
AC-20	$\text{Viscosity} = (45.4+10x)^2$	0.999
RAP+AC-2.5	$\text{Viscosity} = (45.4+9x)^2$	0.982
RAP+AE-150	$\text{Viscosity} = (45.4+22x)$	0.975
RAP+Mobilsol-30	$\text{Viscosity} = (45.4+8x)^2$	0.977

NOTES: "x" is the time of oven exposure during the TFOT. R^2 is the coefficient of determination.

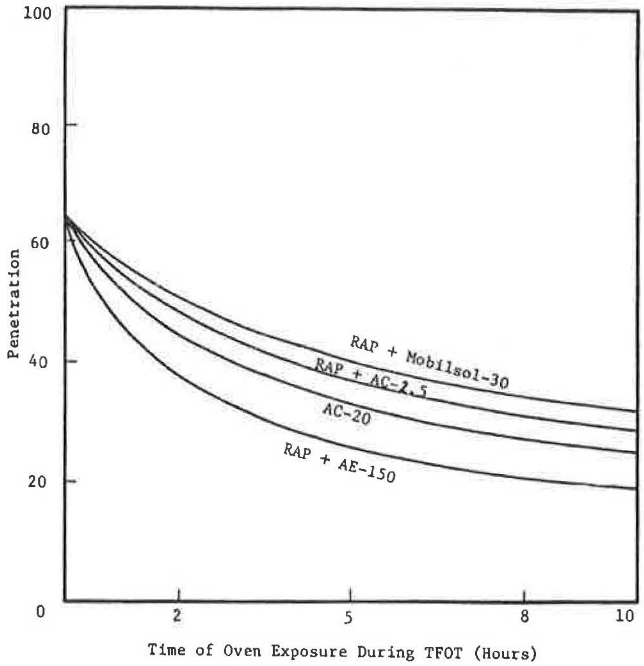

FIGURE 1 Relationship between penetration and time of oven exposure during the TFOT.

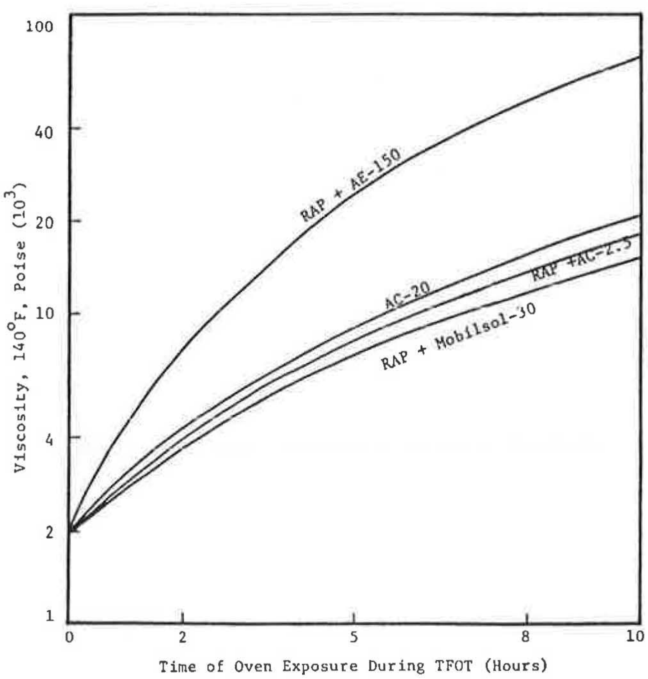

FIGURE 2 Relationship between viscosity and time of oven exposure during the TFOT.

AMOCO Oil Company. A completely randomized design was used for the design of the experiment and the analysis of data resulting from the TFOT (ASTM D1754). The analysis and evaluation of the test data revealed a number of important aspects of hot-mix–recycled bituminous pavement. However, the significant results obtained may be limited to the materials used and test conditions applied.

The main conclusions can be summarized as follows:

1. Rejuvenated binders having the same consistency as a virgin binder will probably have different long-term performances and hardening rates.
2. Having a rejuvenated binder meet the standard specifications for a virgin binder is not enough to ensure the success of a hot-mix–recycled pavement. Additional criteria and test conditions have to be developed.
3. The TFOT is suggested as a good tool to identify the rate of hardening, possible nonhomogeneity, and noncompatibility that may be expected from a rejuvenated binder in the hot-mix–recycled pavement.
4. Salvaged asphalt in the RAP may experience a high rate of hardening and create nonhomogeneity and noncompatibility problems in the hot-mix–recycled asphalt pavement if AE–150 is used as a recycling agent. However, AC–2.5 and Mobilsol-30 may not create these effects as recycling agents, and their use indicated a slightly slower hardening rate than that of the virgin AC–20.
5. When AE–150 was used for treating weathered asphalt, a brittle skin tended to form on all the TFOT residues; the skin was easily separated from the rest of the sample.
6. Careful selection and testing of a recycling agent (rejuvenator) is essential to ensure good-quality hot-mix–recycled asphalt pavement with an acceptable performance.

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